Appendix C

SOPs

OPEN PATH MEASUREMENT OF AMMONIA AND HYDROGEN SULFIDE WITH THE CEREX UVSENTRY ULTRAVIOLET DIFFERENTIAL OPTICAL ABSORPTION SPECTROMETER

Standard Operating Procedure (SOP) C1

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1. Scope and Applicability

1.1. This method involves open-path spectral measurements of NH_3 and H_2S made with a UV-DOAS instrument (UV Sentry, Cerex Environmental Services, Atlanta, GA).
1.2. The purpose of the method is to measure NH_3 and H_2S upwind and downwind of area sources at livestock production facilities.
1.3. One UV Sentry can measure multiple gases.
1.4. The path length is 50 to 150 m long.
1.5. The minimum detection limit for $NH_3$is 500 ppb-m in lagoon air matrix.
1.6. The minimum detection limit for H_2S is 2000 ppb-m in lagoon air matrix.
1.7. The concentration range when used at manure storage facilities is 1 to 800 ppb.

2. Summary of Method

The Open Path UV-DOAS is a bi-static, broadband, spectral absorption instrument designed to measure the concentration of various constituents in the open air along a path that is at least 50 m long. The instrument is built around a CCD spectrometer. The spectrometer and UV light source (emitter) are mounted on tripods or other stable platforms. Data processing is computerized. Ammonia is characterized by multiple sharp peaks with relatively small absorbance between the peaks, which is known as "fine feature structure", while H_2S is characterized by a broad absorbance band (peak absorbance at 196 nm with secondary peaks at 192 and 200 nm) that shows no fine feature structure, and therefore is known as a "broad absorber" (Fig. 1).

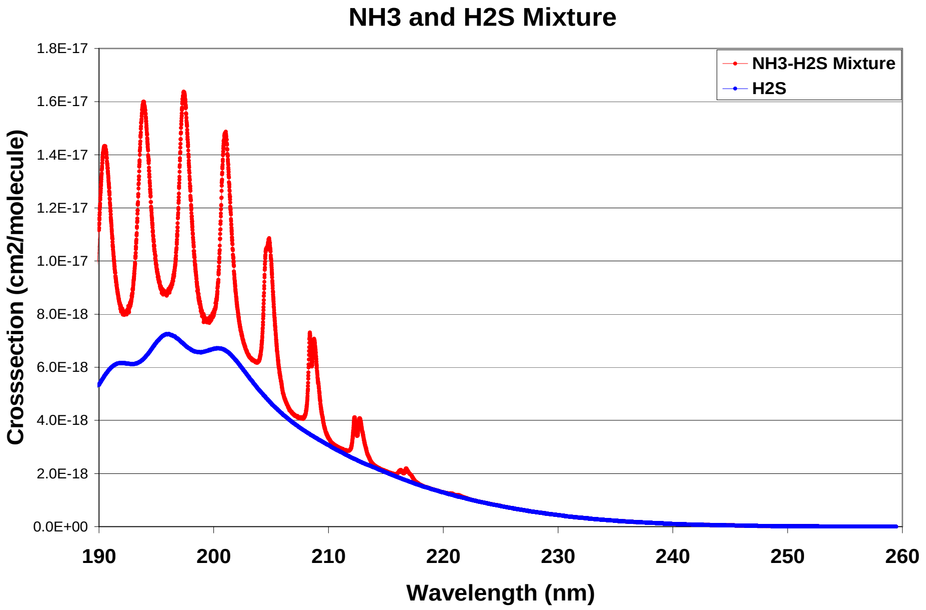


Figure 1. Absorbance characteristics: broad band and fine feature structures.

3. Definitions

3.1.	AU	Absorbance units
3.2.	CLS	Classic least squares
3.3.	CCD	Charge-coupled device
3.4.	EA	Explained Absorbance
3.5.	FA	Filtered Absorbance
3.6.	FOS	Field operations staff
3.7.	HD	Computer hard drive
3.8.	lollipop	A sealed cell that contains a stable calibration gas
3.9.	MDL	Minimum detection limit
3.10.	DOAS	Differential optical absorption spectrometer
3.11.	ppb	parts per billion (volume basis)
3.12.	PC	Personal computer (laptop)
3.13.	PIC	Path-integrated concentration
3.14.	PLS	Partial least squares
3.15.	PM	Program Manager
3.16.	QAPP	Quality Assurance Project Plan
3.17.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.18.	PAML	Purdue Applied Meteorology Laboratory
3.19.	RSD	Relative standard deviation
3.20.	RMS	Square root of a mean squared error
3.21.	USB	Universal serial bus
3.22.	UV	Ultraviolet
3.23.	UVS	UV Sentry

4. Health and Safety

4.1. The transmitter contains a UV-irradiating deuterium lamp. Within 10 ft of the casing, do not look directly at the light source unless wearing UV-filtering eyewear. It can cause permanent damage to eyesight and even blindness.

4.2. Use proper care with the electrical components of the sensor.

5. Interferences

5.1. General interferences that obscure light beam
  5.1.1. Dirty windows (loss of sensitivity and possible interruption)
  5.1.2. Dirty or deteriorated optical bench components (loss of sensitivity and possible interruption).

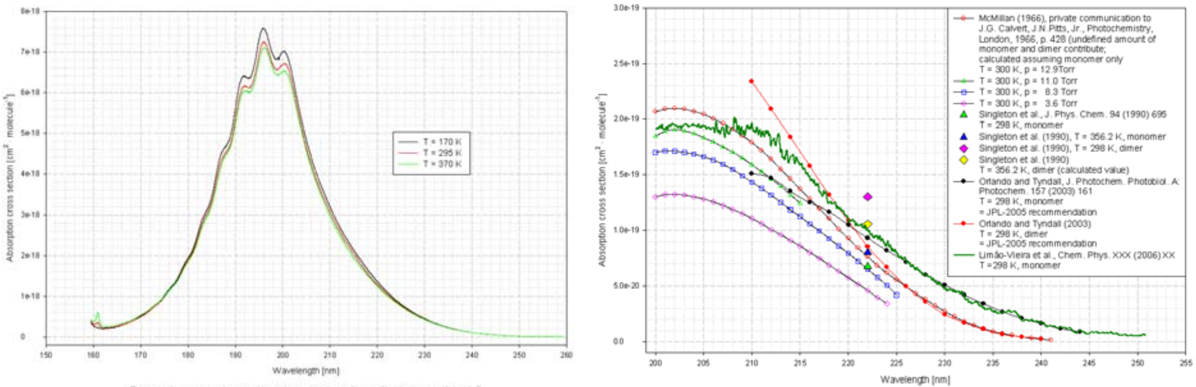
5.2. Degradation of backgrounds
  5.2.1. The characteristic output of a UV light source degrades over time so beware of older backgrounds (Section 8.2.4.2) because inaccuracies may be introduced to the analytical process when a recent sample is compared to an older sample.
  5.2.2. Program the UVS Control to automatically update backgrounds so that if an acquisition is less contaminated than the current background, it will begin using the latest acquisition as background.

5.3. Weather interferences that obscure light beam
  5.3.1. Heavy fog (temporary loss of sensitivity and possible interruption)
  5.3.2. Heavy rain (temporary loss of sensitivity and possible interruption)

5.4. Independent analysis of NH_3 and H_2S is not possible. Because both gases are analyzed together, it must be calibrated using mixtures of the two gases. Otherwise, each gas is an interferent for the other gas (Figure 1), causing inaccuracies.

5.5. Acetic acid is an interferent (Figure 2; broad band absorber with peak absorption at 202 nm) for H_2S analysis but anticipated to be at concentrations below the MDL of the instrument. The acetic acid absorption characteristic are stored in the UVS software and the acetic acid concentration are determined in combination with H_2S using a Partial Least Squares (PLS) analysis method.

5.6. Oxygen is an interferent for NH_3 and H_2S; however, the instrument's software is programmed to account for oxygen at atmospheric levels.



Temperature dependence of the absorption spectrum of hydrogen sulfide H_2S, Wu and Chen, J. Quant. Spectrosc. Radiat. Transfer 60 (1998) 17

Absorption cross sections of acetic acid $CH_3C(O)OH$ and its dimer

Figure 2. Absorbance spectra of Hydrogen Sulfide and Acetic Acid

Note that the maximum absorbance cross-section for H_2S is $8x10^{-18}$ cm^2 / molecule and for acetic acid is $2x10^{-19}$ cm^2 / molecule. (reproduced from Hannelore Keller-Rudek, Geert K. Moortgat, *MPI-Mainz-UV-VIS Spectral Atlas of Gaseous Molecules*,www.atmosphere.mpg.de/spectral-atlas-mainz)

6. Personnel Qualifications

6.1. Personnel must be trained to use the handheld multimeter, which takes 10 min.

6.2. Field staff members must read and understand the instrument instruction manual and this SOP, and have undergone specific training for the UVS.

6.3. Personnel must be trained in the use of the TEI Model 146 diluter (SOP C3).

7. Equipment and Supplies

- 7.1. UV-DOAS (UV Sentry, CEREX Environmental Services, Atlanta, GA)
- 7.2. XStream® RS-232/485 RF Modem
- 7.3. TEI Model 146 diluter
- 7.4. Environics Series 4040 Gas diluter
- 7.5. Laptop computer
- 7.6. DB9 modem cable
- 7.7. Nitrogen (N_2) gas cylinders with pressure regulators (SOP G2)
- 7.8. H_2S gas cylinders with pressure regulators (SOP G2)
  - 7.8.1. One disposable aluminum cylinder with regulator and about 50 ppm H_2S
  - 7.8.2. One aluminum cylinder with regulator and about 500 ppm H_2S
- 7.9. NH_3 gas cylinder with pressure regulator (SOP G2)
  - 7.9.1. One disposable aluminum cylinder with regulator and about 50 ppm NH_3
  - 7.9.2. One aluminum cylinder with regulator and about 500 ppm NH_3
- 7.10. “Lollipop” with benzene (C6H6) gas at a concentration greater than 100 ppm
- 7.11. 6 in x 4 in flow-through calibration cell to contain the H_2S gas
- 7.12. Tripods (2) to mount the 6” flow-through cells
- 7.13. Various lengths of tubing to connect the tanks to the cells
- 7.14. Adjustable wrench
- 7.15. Electrical tape

8. Procedures

- 8.1. Instrument acceptance
  - 8.1.1. Single-point checks of the NH_3 and H_2S calibrations are made using separate external chambers filled with known quantities of gas.
  - 8.1.2. Conduct single-point check with interference calibration (Section 8.4.4).
- 8.2. Instrument installation
  - 8.2.1. Field setup
    - 8.2.1.1. Set up tripods at the desired distance, and adjust legs until they are level according to bubble level at the head mount.
    - 8.2.1.2. Attach UVS by sliding the mounting bracket into the tripod head.
    - 8.2.1.3. Roughly align UVS using sighting scopes with crosshairs focused on opposing unit’s silver hood.
  - 8.2.2. Hardware setup
    - 8.2.2.1. Connect power and USB cables to the UV Sentry Receiver.
    - 8.2.2.2. Connect USB to the monitoring computer.
    - 8.2.2.3. Start the UVS Software.
    - 8.2.2.4. If the status window at bottom shows “Not Connected”
      - 8.2.2.4.1. Go to: Setup → Hardware
      - 8.2.2.4.2. Click “Manually Select”
      - 8.2.2.4.3. Under *USB Serial Number*, select: USBxxxxxx
      - 8.2.2.4.4. Click: *OK*, then *OK* again to return to the main program screen.
    - 8.2.2.5. Check alignment by clicking *Align*.
    - 8.2.2.6. Physically align UVS until maximum signal strength is reached..

- 8.2.2.7. Finally, adjust integration time so that peak value of the curve is between 2500 and 3500 counts, which is indicated by Done button turning green. Click Done to return to the main program screen.
- 8.2.3. Sampling interval
  - 8.2.3.1. Adjust the sample interval of the UVS to obtain 30-s, 1-min, or 5-min average according to the QAPP specifications.
  - 8.2.3.2. Go to: Setup → Analysis.
  - 8.2.3.3. Choose a sampling interval.
  - 8.2.3.4. Make sure "Dynamically adjust integration period" is selected.
  - 8.2.3.5. Set *Boxcar Smoothing* to 0.
  - 8.2.3.6. Select "Automatically update background".
- 8.2.4. Backgrounds
  - 8.2.4.1. The ideal background is one free of potential contaminants, if the winds change so that you are upwind of an emission source, a new background may be desirable. To acquire a new background:
    - 8.2.4.1.1. Go to: Acquire → Single Acquisition
    - 8.2.4.1.2. At end of sample period, select: Acquire→Background→Last Acquisition
  - 8.2.4.2. In many situations, a background is available that is known to be clean. To use a stored background:
    - 8.2.4.2.1. Select: Acquire → Background → From File…
    - 8.2.4.2.2. A menu will appear that will enable you to select a UVS xxxxx YYYY-MM-DD.csv file that you know to be a valid background.
- 8.2.5. Site names and file logging
  - 8.2.5.1. By default, the system logs samples to a directory corresponding to the site name entered at the left of the main program screen.
  - 8.2.5.2. Site Name is predefined (SOP D1) (Do not use dashes, '-',in file name.)
  - 8.2.5.3. Insert the correct distance between tripods in the *Path length* field.
  - 8.2.5.4. Double clicking on the *File Count* in the status bar resets count to 0.
- 8.2.6. Monitoring
  - 8.2.6.1. The system has two monitoring settings: *Single* and *Continuous*.
    - 8.2.6.1.1. In *Single Acquisition* mode, one data collection is performed and the system stops monitoring. Select *Acquire → Single Acquisition*.
    - 8.2.6.1.2. In *Continuous Acquisition* mode, the system will acquire data until user manually stops monitoring. Select Acquire → Continuous Acquisitions or click *Start Monitoring*. Click *Stop Monitoring* after enough samples have been obtained.

8.3. Communications installation

- 8.3.1. Connect serial cable from XStream Modem to laptop computer COM port.
- 8.3.2. Using PC Windows Hyperterminal program on PC, verify configuration of Modem (8 data bits, 1 stop bit, no parity, 19200 baud) (SOP U5).
- 8.3.3. Connect serial cable from XStream Modem serial output connector to UV-DOAS using 1/8"slotted screwdriver (Fig. 3).
- 8.3.4. Connect antenna coaxial cable to RF modem.
- 8.3.5. Verify destination address of Modem (SOP U5).
- 8.3.6. Provide power to UV-DOAS.

- 8.3.7. Provide power to radio modem by connecting power cable to power connector and switching on power switch (Fig. 3).
- 8.3.8. Verify power to radio modem by checking red light (Fig. 3).

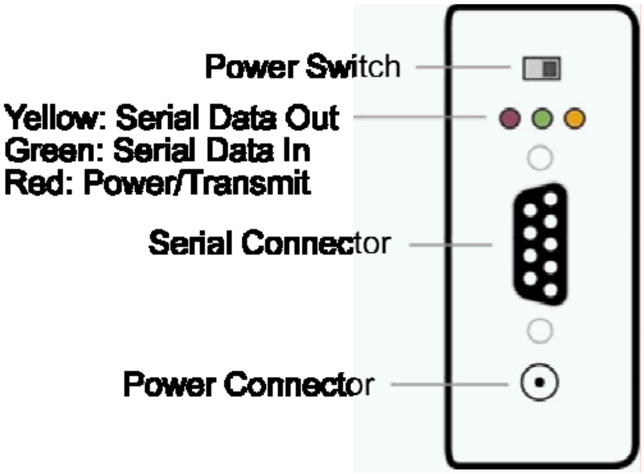


Figure 3. Diagram of radio modem.

- 8.3.9. Test communication link to PC in trailer.

8.4. Instrument calibration

- 8.4.1. External calibration chambers
  - 8.4.1.1. Utilize two 6″ x 10 cm flow-through calibration cells.
  - 8.4.1.2. The 6″ cell has a ¼″ Swagelok tubing connection
- 8.4.2. Calibration gas cylinder with 50 ppm NH_3 in N_2
  - 8.4.2.1. Verify concentration with FTIR (SOP G12).
  - 8.4.2.2. Calibration gas flowing through 10-cm long chamber is at 1% of inlet concentration per meter: 5 ppm-m.
- 8.4.3. Calibration gas cylinder with 50 ppm H_2S in N_2
  - 8.4.3.1. Verify concentration with FTIR (SOP G12) or fluorescence (SOP G5)
  - 8.4.3.2. Calibration gas flowing through 10-cm long chamber is at 10% of inlet concentration per meter: 5 ppm-m.
- 8.4.4. Single Point with Interference Calibration Procedure
  - 8.4.4.1. Single point calibrations will be performed in the field but next to the instrumentation trailer.
  - 8.4.4.2. Remove instruments from their respective support structures and mount on portable tripods next to trailer.
  - 8.4.4.3. Set path length between transmitter and receiver at 3 m.
  - 8.4.4.4. Use calibration gases defined in 8.4.2 and 8.4.3.
  - 8.4.4.5. Mount calibration cells on portable tripods next to trailer.
  - 8.4.4.6. Record cylinder reference numbers and chemical concentrations on calibration sheet for calibration cylinders (see verifications in SOP G2).
  - 8.4.4.7. Place the two calibration cells on tripods in beam path as close as possible to the receiver.
  - 8.4.4.8. Align the system to achieve best integration time: 20 – 25 ms for 3000+ counts. Use the UV-DOAS to monitor the alignment and ensure a sustained high signal strength.

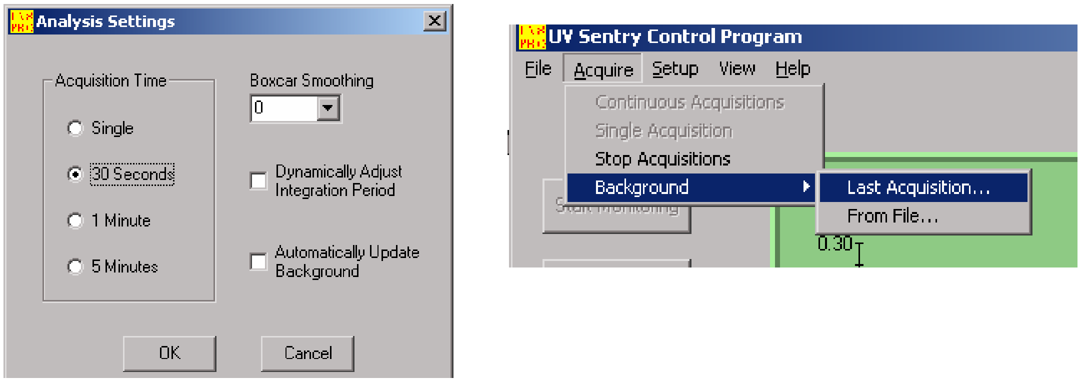
8.4.4.9. Make background scan

8.4.4.9.1. Purge first calibration cell with N_2 and cover inlet and outlet ports when finished. This should take anywhere from 1 – 5 min, depending on the flow rate.

8.4.4.9.2. Purge second calibration cell with N_2 at a low flow rate.

8.4.4.9.3. Begin taking a background scan while N_2 is flowing through the cell.

8.4.4.9.4. Set to 30-s averaging and uncheck auto-update integration time and auto-background update. Do not use boxcar smoothing (Fig. 4).



Analysis Settings

Acquisition Time
○ Single
● 30 Seconds
○ 1 Minute
○ 5 Minutes

Boxcar Smoothing: 0

☐ Dynamically Adjust Integration Period

☐ Automatically Update Background

Figure 4. Configuring UVS Analysis Settings and setting a background with the Background → Last Acquisition feature.

8.4.4.9.5. Set this acquisition as the background for UV-DOAS (Fig. 4).

8.4.4.10. NH_3 Calibration Point

8.4.4.10.1. Continue N_2 flow through second cell. Start *Continuous Acquisitions*. [Do not stop *Continuous Acquisitions* until end of test].

8.4.4.10.2. Acquire four 30-s data sets, then shut off the N_2.

8.4.4.10.3. Connect the NH_3 to the cell inlet port and begin flowing gas at 0.2 L/min.

8.4.4.10.4. Acquire fifteen 30-s data sets.

8.4.4.10.5. Calculate mean NH_3 concentration from recorded data in MS Excel.

8.4.4.10.6. Record mean NH_3 concentration on MS Excel Single Point Calibration Data Sheet (Appendix A).

8.4.4.10.7. Purge cell with N_2 for 2 min and cover ports with tape to retain N_2.

8.4.4.11. H_2S Calibration Point

8.4.4.11.1. Remove tape covering the ports to the first calibration cell.

8.4.4.11.2. Connect H_2S tank to the Inlet port of the calibration cell.

8.4.4.11.3. Begin flowing H_2S gas at 0.2 L/min.

8.4.4.11.4. Acquire fifteen 30-s data sets, then shut off the H_2S.

8.4.4.11.5. Calculate mean H_2S concentration from recorded data in MS Excel.

8.4.4.11.6. Record H_2S concentration on MS Excel Single Point Calibration Data Sheet (Appendix A).

8.4.4.11.7. Purge cell with N_2 for 2 min and cover ports with tape to retain N_2.

8.4.4.12. NH_3 and H_2S Combined Calibration Point

8.4.4.12.1. Connect NH_3 to the first cell and begin flowing gas.

8.4.4.12.2. Connect H_2S to the second cell and begin flowing gas.
8.4.4.12.3. Acquire fifteen 30-s data sets with this combined gas configuration.
8.4.4.12.4. Shut off NH_3 gas first. Purge the first cell with N_2.
8.4.4.12.5. Acquire fifteen 30-s data sets with only H_2S again.
8.4.4.12.6. Calculate mean NH_3 and H_2S concentrations from recorded data in MS Excel.
8.4.4.12.7. Record NH_3 concentrations on Calibration Data Sheet (Appendix A).
8.4.4.13. Evaluate measurement accuracy by calculating percent error:

$$\%\ \text{Error} = \frac{[(\text{Mean of 15 readings}) - (\text{Gas cylinder concentration})]}{(\text{Gas cylinder concentration})} \times 100\%$$

Use MS Excel. (Example results found in Appendix B)

8.4.4.13.1. Calculate percent error for NH_3 alone, and for NH_3 with H_2S present.
  8.4.4.13.1.1. If accuracy <10%, the system is working properly.
  8.4.4.13.1.2. If accuracy >10%:
    8.4.4.13.1.2.1. Verify placement of flow-through cells in beam path.
    8.4.4.13.1.2.2. Verify proper operation of N_2 gas cylinder.
    8.4.4.13.1.2.3. Verify proper operation of NH_3 and H_2S gas cylinders.
    8.4.4.13.1.2.4. Repeat test.
    8.4.4.13.1.2.5. If the system fails twice, pull it from the field.
8.4.4.13.2. Calculate percent error for H_2S alone, and for H_2S with NH_3 present.
  8.4.4.13.2.1. If accuracy <10%, the system is working properly.
  8.4.4.13.2.2. If accuracy >10%:
    8.4.4.13.2.2.1. Verify placement of flow-through cells in beam path.
    8.4.4.13.2.2.2. Verify proper operation of N_2 gas cylinder.
    8.4.4.13.2.2.3. Verify proper operation of NH_3 and H_2S gas cylinders.
    8.4.4.13.2.2.4. Repeat test.
    8.4.4.13.2.2.5. If the system fails twice, pull it from the field.
8.4.4.14. Reinstall instruments on their respective support structures.

8.4.5. Multipoint Calibration with Interference Procedure

Because NH3 and H2S concentrations are expected to span several orders of magnitude, verify linear system response with a 4-point calibration that spans the expected range.

8.4.5.1. Multipoint calibrations will be performed at PAML.
8.4.5.2. Calibration of NH_3 with 500 ppm calibration gas
  8.4.5.2.1. Verify concentration with FTIR (SOP G12).
  8.4.5.2.2. Dilute gas to 5 and 50 ppm using N_2 using a diluter (SOP C3).
  8.4.5.2.3. Calibration gas flows through 10-cm long chamber, resulting in an effective path-integrated calibration concentration for the sensor of 10% of that for a 1-m pathlength: Calibration gas inlet concentrations of 5 ppm and 50 ppm correspond to effective path integrated concentrations of 500 ppb-m and 5 ppm-m, respectively.
  8.4.5.2.4. Document results on MS Excel Multipoint Calibration Data Sheet (Appendix C).

- 8.4.5.3. Calibration of H_2S with 500 ppm calibration gas
  - 8.4.5.3.1. Verify concentration with FTIR (SOP G12).
  - 8.4.5.3.2. Dilute gas to 5 and 50 ppm with N_2 using a diluter (SOP C3).
  - 8.4.5.3.3. Effective path integrated concentrations are 500 ppb-m and 5 ppm-m (Section 8.4.5.2.3).
  - 8.4.5.3.4. Document results on MS Excel Multipoint Calibration Data Sheet (Appendix C).
- 8.4.5.4. Conduct calibration at zero concentration (pure N_2) and at each concentration in isolation and in combination. Follow procedures outlined in Section 8.4.4.9.
  - 8.4.5.4.1. Document results on MS Excel Multipoint Calibration Data Sheet (Appendix C).
- 8.4.5.5. *Linearity*: Reference spectra are included in the data analysis matrix that span the expected minimum and maximum concentrations. In order to ensure that non-linearity effects do not come into play, the field data must be within the highest and lowest gas concentrations within the analytical matrix. Because the PLS algorithm uses multiple concentrations for each gas, there are no issues associated with non-linearity of absorbance spectra. There is no need for adjustments for non-linearity due to Beer's Law.

8.5. Instrument operation

- 8.5.1. Setup
  - 8.5.1.1. Connect serial cable from XStream Modem to PC COM port.
  - 8.5.1.2. Using PC Windows HyperTerminal program, verify configuration of Modem (8 data bits, 1 stop bit, No parity, 19200 baud) (SOP U5.
  - 8.5.1.3. Connect serial cable from XStream Modem to UVS COM port.
  - 8.5.1.4. Test communication link to PC in trailer.
  - 8.5.1.5. Locate appropriate input serial channel on PC in trailer (SOP U4) and evaluate output for reasonable values.
  - 8.5.1.6. Generate a background spectrum according to Section 8.2.4.
  - 8.5.1.7. Depending on wind direction and overall monitoring situation, obtain a single beam spectra for the path length of interest when there is no contribution of the target gases from a source upwind.
  - 8.5.1.8. Check maximum peak emission for counts between 2500 and 3000.
  - 8.5.1.9. Set measurement scans for every ~0.5 s averaged to 5 min for each of two instruments.

8.6. Troubleshooting

- 8.6.1. Communications
  - 8.6.1.1. Verify communications link between radio modems (Yellow and Green lights on modems (Fig. 2).
  - 8.6.1.2. If no communications, verify firm connections for power, signal, and antenna for serial communications. Correct as needed.
  - 8.6.1.3. Verify power to modems by red light (see Fig. 2).
  - 8.6.1.4. Correct as needed.

8.6.2. UV Sentry

8.6.2.1. Integration time of 3500 counts at maximum signal strength should be less than 500 ms, in accordance with Table 1. If not:

8.6.2.1.1. Check system alignment. Correct as necessary

8.6.2.1.2. If alignment is good, either the source needs to be replaced or the optics may have become damaged. In either case, contact PM at PAML: the system should be returned to PAML for instrument check and calibration.

Table 1. Troubleshooting based on integration time.

Integration Time, ms	Deuterium Source (100-m path)
Less than 150	Factory Specification
150 – 500	Data is reliable
500 – 700	Source is degrading
Greater than 700	Replace source

8.7. Data acquisition, calculations & data reduction requirements

8.7.1. Store 5-min averaged spectra as individual 48K (a0) files in Flash memory

8.7.2. Analyze spectra in real time for NH_3 and stored in sequential record file containing scan #, background scan #, NH_3, background NH_3.

8.7.3. Utilize a partial least squares (PLS) data analysis routine to quantify gases that absorb UV light. The method (incorporated into the UVS software) minimizes effects of baseline shifts, cross interference of target gases, and interference of other gases. Key elements of the method are as follows:

8.7.3.1. A data analysis matrix is created that includes all of the target analytes. The matrix includes reference spectra containing gases of interest, as well as interfering species.

8.7.3.2. Absorption spectra are generated by ratioing the field data to a clean air background reference.

8.7.3.3. The absorbance spectra are then smoothed using a Savinski-Golay smoothing filter resulting in the filtered absorbance (FA) spectra. The filter eliminates high frequency noise in the absorbance spectra while maintaining target analytes' sharp spectral features.One peak of the NH_3 absorption feature in the FA spectra is aligned in frequency (pixel shifting for the CCD spectrometer detector) to the reference spectra. Maximum allowable pixel shift is ±1 pixel.

8.7.3.4. The ammonia concentration is determined from the relationship of the FA NH_3 absorption peak and the corresponding peak of the absorption reference spectra using a classical least squares (CLS) technique.

8.7.3.5. The entire ammonia absorption spectra corresponding to the CLS-determined concentration is subtracted from the FA spectra.

8.7.3.6. The remaining portion of the FA spectra is analyzed for H_2S and acetic acid using a PLS analysis method resulting in the determination of the concentration of all compounds found in the PLS calibration matrix.

8.7.3.7. The correlation coefficient between the original FA spectra and the combined explained H_2S, NH_3, and acetic acid (or other analyte) absorption spectra (EA spectra) is calculated

8.7.3.8. The root mean squared (RMS) residual from the regression, representing the unexplained variability in the measured absorption spectra is calculated based on the determined concentrations of NH_3, H_2S (and acetic acid, etc.) according to:

$$\text{RMS residual}=\sqrt{\frac{\sum_{waveband}(FA-EA)^2}{\sum_{waveband}\left(FA-\overline{FA}\right)^2}} \qquad \text{(Equation 1)}$$

8.8. Computer hardware & software

8.8.1. The instrument is controlled by an embedded computer, which is linked to the LAN.

8.8.2. Record and store data using a second PC (termed ‘bLS computer’).

9. Data and Records Management

9.1. 5-min integration spectra are stored as individual 48k (a1) files.

9.2. Format of file is: (background PIC, SignalStrength, IntegrationTime, PIC NH3, NH3 measurement error, PIC H2S, H2S measurement error, PixelShift, CorrelationCoeff., RMSresidual).

9.3. Store spectra for each instrument in separate directories (SOP D1).

9.4. Send spectra for each instrument to Purdue through ftp (SOP D1).

9.5. Receipt of raw and post-processed data at PAML will be logged on the transmittal sheet and transferred to the database (SOP D1).

9.6. WINDTRAX/ bLS Emissions measurement method

9.6.1. Measured NH3 PIC is passed from UVS to WINDTRAX program for on-site flux estimation using BLS method (SOP O1) on ‘bLS’ PC (SOP D1).

10. Quality Control and Quality Assurance

10.1. Precision and accuracy

10.1.1. While TO-16 (USEPA, 1999a) gives no definitive test method for determining precision and accuracy of open-path sensors, it states that a flow-through cell is appropriate for measuring precision and accuracy if the entire light beam passes through an external cell in the beam path.

10.1.2. The UV-DOAS system’s precision and accuracy are determined by using an external flow-through cell in its beam path (Appendix B).

10.1.2.1. Determine accuracy according to Section 8.4.4.

10.1.2.2. Determine precision according to Sections 8.4.4.1 through 8.4.4.10.

10.1.2.2.1. Calculate RSD the computed analyte concentration from 15 measurement spectra.

10.1.2.2.2. Precision = RSD of 15 measurements.

10.1.2.2.2.1. If RSD <10% of the difference between the cylinder and average measured values, the system is working properly.

10.1.2.2.2.2. If RSD >10% of the difference between the cylinder and average measured values.

10.1.2.2.2.2.1. Verify the external flow through cells are correctly placed in the beam path.

10.1.2.2.2.2.2. Verify the N_2 gas cylinder is operating correctly.

10.1.2.2.2.2.3. Verify the NH_3 and H_2S gas cylinders are operating correctly.

10.1.2.2.2.2.4. Perform test again.

10.1.2.2.2.2.5. If the system fails the test a twice, replace it.

10.2. Dark Current Correction Check

10.2.1. Charged diode array spectrometers build up a charge over time that is independent of the actual light being detected. This charge buildup is defined as “dark current”, and must be subtracted from each single-beam spectrum before data analysis can occur. Although the system can be setup in a manner where the dark current is measured and then manually subtracted out of each individual spectrum, the UVS software performs this task automatically.

10.2.2. Examine the first 15 pixels of a single beam spectrum to determine whether the UVS software is performing this function properly.

10.2.3. Record results on QA sheet (Appendix D).

10.2.4. The average of these pixels should fall within ± 5 counts. Dark current is shown in the bracketed rows of Fig. 4.

10.3. Spectral Feature Match Check

10.3.1. Check is guided and results documented by the “Q-Soft System Calibration Software”

10.3.2. Use absorption peaks of benzene.

10.3.3. Insert a ‘lollipop’ containing benzene and SO_2 into beam path.

10.3.4. Collect a data spectrum and compare the locations of the measured benzene absorption peaks with the location in the standard benzene peak found in the set of QA reference spectra that are matched to the spectrometer. The QA reference spectra output files are denoted by the letters QA.

10.3.5. Record results on QA sheet (Appendix D).

10.3.6. Difference in peak location should be <2 pixels. If >2 pixels, contact manufacturer for remediation or repair.

10.3.7. Correlation coefficient should be ≥ 0.90. If <0.90, contact manufacturer for remediation or repair.

10.4. Single point calibration check (procedures found in section 8.4.4)

10.4.1. Check is guided and results documented by the “Q-Soft System Calibration Software”

10.4.2. Set path length between transmitter and receiver at 3 m.

10.4.3. Fill first external cell with a known concentration of NH_3.

10.4.4. Fill second external cell with a known concentration of H_2S.

10.4.5. Set signal strength to 3000 counts.

10.4.6. Collect a background scan.

10.4.7. Insert calibration cell in path.

10.4.8. Verify that signal strength is between 2700 and 3300 counts.

10.4.9. Collect five consecutive data sets and observe reported concentrations.

10.4.10. Record results on MS Excel Single Point Calibration Data Sheet (Appendix A).

10.5. Multipoint Calibration Check

10.5.1. Procedures for this are found in Section 8.4.5.

10.5.2. Record results on MS Excel Multipoint Calibration Data Sheet (Appendix C).

10.6. Baseline Stability Test

10.6.1. Set the following system parameters:

10.6.1.1. Integration Time: < 500 ms

10.6.1.2. Sample Time: 30-s average

10.6.1.3. Number of Samples: 12

10.6.2. Collect background spectrum.

10.6.3. Collect designated number of samples.

10.6.4. Convert samples to absorbance spectra.

10.6.5. Open the UVS absorbance viewing software.

10.6.6. Record results on QA sheet (Appendix D).

10.6.7. The total drift at 270 nm should be less than ± 0.002 AU.

10.7. Single Beam Ratio Test.

The single beam ratio test checks the adequacy of the UV Source. A weak UV source that is weak will produce data with low signal-to-noise ratios.

10.7.1. Align instrument at the shortest path length of the study.

10.7.2. Adjust integration time until maximum signal strength is 3500 counts.

10.7.3. For a deuterium source, the maximum peak energy should be near 250 nm. Compare the signal value at 250 nm to the signal value at 210 nm. Record the two signal values and ratio the value at 210 nm to the value at 250 nm.

10.7.4. Record results on QA sheet (Appendix D).

10.7.4.1. The signal value at 210 nm should be greater than 20% of the signal at 250 nm.

10.7.4.1.1. If the signal value at 210 nm is equal to or less than 20% of the signal at 250 nm, contact PM at PAML for remediation.

10.8. Spectral alignment

10.8.1. Alignment of the measured spectrum to the reference spectrum is determined using one peak of the NH_3 absorption feature (Figure 1). This alignment consists of a pixel shift in the spectrometer array. Maximum allowable pixel shift is 1 pixel.

10.8.1.1. If the pixel shift > 1 and NH_3 PIC < MDL, set NH_3 PIC to ½ MDL and the H_2S PIC is indeterminate by automated processing.

10.8.1.1.1. Post-process FA for H_2S using PLS assuming NH_3 is zero. If still no H_2S quantified, set to ½ MDL.

10.8.1.2. If the pixel shift > 1 and NH_3 PIC > MDL, compare pixel shift against the spectral feature alignment check (paragraph 10.3)

10.8.1.2.1. If the spectral feature alignment check < 2, set H_2S PIC to ½ MDL.

10.9. Correlation coefficient

10.9.1. The correlation coefficients of the measured spectra compared to the spectral quantification of H_2S, NH_3 and acetic acid absorption spectra is calculated. Values must be greater than 0.75.

10.9.1.1. If $r^2 < 0.75$ and NH_3 PIC < MDL, set NH_3 and H_2S PIC to ½ MDL.

10.9.1.2. If $r^2 < 0.75$ and the NH_3 PIC > MDL, set H_2S PIC to ½ MDL.

10.10. Analysis residual

10.10.1. The residual of the spectral quantification, or unexplained variability in the absorption spectra, is calculated based on the determined concentrations of NH_3, H_2S and acetic acid. Values must be less than 0.25.

10.10.1.1. If residual > 0.25 and the NH_3 PIC < MDL, set the NH_3 and H_2S PIC to ½ MDL.

10.10.1.2. If residual > 0.25 and the NH_3 PIC > MDL, set H_2S PIC to ½ MDL.

10.11. If the instrument records no measured PIC, set PIC to ½ MDL.

11. References

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11.3. SOP D1. 2006. Management of Open Path and Weather and Lagoon Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.

11.4. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab

11.5. SOP G5. 2006. Measurement of Hydrogen Sulfide (H2S) with the Thermo Electron Corporation Model 450C Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab.

11.6. SOP G8. 2006. Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.

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11.11. SOP U5. 2006. Installation of Open-source Measurements. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.

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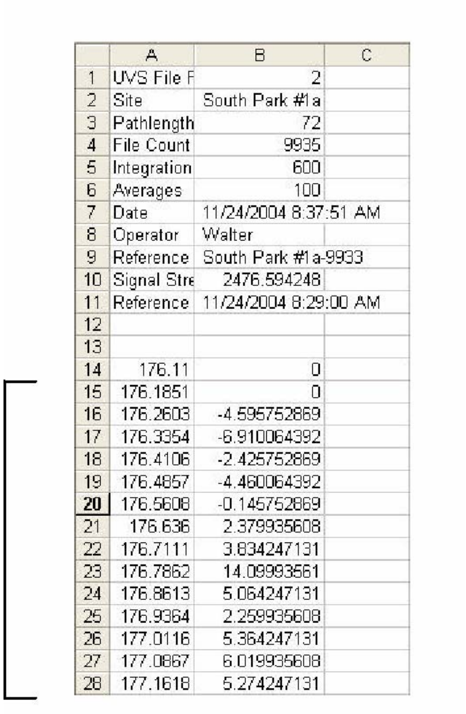
11.13. USEPA. 1999b. General Guidelines for On-Site Meteorological Data Collection for N.C. Air Quality Analysis (NCDAQ). USEPA, Wash., D.C.
11.14. USEPA. 1995. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV- Meteorological Measurements. Document No. EPA/600/R-94/038d, USEPA, Washington, D.C.
11.15. USEPA. 2006. Interim Standard Operating Procedure for Open Path Measurement of Air Pollutants with the CEREX UVSentry Ultraviolet Differential Optical Absorption Spectrometer. ERA-ORD-NRMRL ORS MOP 6844, Rev. 1B.
11.16. UV Sentry Software Manual. 2004. Software Guide and Operations Manual, Draft v. 1.1, CEREX Environmental Services Inc., Atlanta, GA.
11.17. Xstream Model Manual. 2005. XStream-PKG-R RS-232/485 RF Modem. Product Manual v4.2B, Maxstream, Lindon, UT.

12. Contacts

12.1. UV-DOAS Manufacturer: CEREX Environmental Services Inc., 739 Trabert Avenue, N.W., Building 2, Suite D, Atlanta, GA 30318 USA
  12.1.1. Phone: 404-352-0210
  12.1.2. Fax: 801-858-7844
  12.1.3. e-mail: don@cerexenv.com
12.2. Modem Manufacturer: Maxstream, Inc., 355 South 520 West, Suite 180, Lindon, UT 84042
  12.2.1. Phone: 801-765-9885
  12.2.2. Fax: 801-765-9895
  12.2.3. e-mail: rf-xperts@maxstream.net



	A	B	C
1	UVS File F	2	
2	Site	South Park #1a	
3	Pathlength	72	
4	File Count	9935	
5	Integration	600	
6	Averages	100	
7	Date	11/24/2004 8:37:51 AM	
8	Operator	Walter	
9	Reference	South Park #1a-9933	
10	Signal Stre	2476.594248	
11	Reference	11/24/2004 8:29:00 AM	
12			
13			
14	176.11	0	
15	176.1851	0	
16	176.2603	-4.595752869	
17	176.3354	-6.910064392	
18	176.4106	-2.425752869	
19	176.4857	-4.460064392	
20	176.5608	-0.145752869	
21	176.636	2.379935608	
22	176.7111	3.834247131	
23	176.7862	14.09993561	
24	176.8613	5.064247131	
25	176.9364	2.259935608	
26	177.0116	5.364247131	
27	177.0867	6.019935608	
28	177.1618	5.274247131	

Figure 4. Dark current rows.

Appendix A:Single point UV-DOAS Calibration Sheet

UVDOAS Serial Number ___________________ Date (MMDDYY) _____________________ Time (HH:MM) _________________

NH_3 cylinder reference number ________________ H_2S cylinder reference number ________________

PAML SOP C1

Certified Gases			**Measured Analyte**				
Analyte: Gases present	**Conc.**	**Conc.**	**Mean**	**SD**[1]	**Accuracy**[2]	**RSD**	**Operator**
		ppm-m	**ppm-m**	**ppm-m**	%	%	
NH_3							
H_2S							
NH_3: NH_3+H_2S ppm-m							
H_2S: NH_3+H_2S							

[1] Standard deviation

[2] Accuracy = 100% * (Measured concentration – Certified concentration) / (Certified concentration)

Appendix B
Assessments of Detection Limits, Precision, and Accuracy

1) Detection Limits

Laboratory (negligible sources of NH_3 or H_2S):

The procedure for determining the detection limits for ammonia (NH_3) and hydrogen sulfide (H_2S) with a 2 m physical pathlength was:

- Generate 16 sequential spectra using a UVS.
- Create 15 absorption spectra using the back to back spectra.
- Quantify the target analytes using the standard PLS matrix for NH_3 and H_2S,
- Calculate the standard deviation of the 15 quantifications.
- Calculate the MDL by multiplying the standard deviation by three.

Results from CEREX, Inc laboratory test, Spring 2006:
The MDL for NH_3 was 138 ppb-m.
The MDL for H_2S was 317 ppb-m.

Field (lagoon environment with acetic acid, NH_3, H_2S):

The procedure for determining the detection limit for hydrogen sulfide (H_2S) with a 100 m physical pathlength with negligible H_2S present was:

- Generate 10 sequential spectra using a UVS.
- Create 10 absorption spectra using the back to back spectra.
- Quantify the NH_3 using CLS technique.
- Subtract quantified NH_3 from absorption spectra and quantify H_2S using the standard PLS matrix.
- Calculate the standard deviation of the 10 quantifications.
- Calculate the MDL by multiplying the standard deviation by three.

Results from three replicates under field conditions on 10-9-2006 indicated the MDL for H_2S was 1903 ppb-m.

2) Precision and Accuracy:

The laboratory precision and accuracy of the UVS for H_2S and NH_3 was determined using:

- 50 ppm H2S calibration gas cylinder with 15-cm flow through cell
- 311 ppm NH3 calibration gas cylinder with 1-cm flow through cell

The procedure for determining the precision and accuracy of a target analyte in the field was as follows:

- Place the external cell in the 2-m beam path of the open-path UV system
- Flow N2 through the cell until three volume changes occurred
- Collect a data spectrum.
- Flow the target analyte through the cell for a total of three volume changes
- Collect fifteen data spectra

- Purge the system with N2 and obtain another data spectrum

Example results from CEREX, Inc laboratory test, Spring 2006:
Determination of Accuracy:

Gas	**Reference** ppm-m	**n**	**Mean** ppm-m	**Accuracy** %
NH_3	31	15	31.77	1.7
H_2S	75	15	77.60	2.6

Determination of Precision

Gas	**n**	**Mean** **ppm-m**	**St. Deviation** **ppm-m**	**Precision** **%**
NH_3	15	31.77	1.34	4.23
H_2S	15	77.60	2.64	3.39

Appendix C: Multipoint UV-DOAS Calibration Sheet

UVDOAS Serial Number __________________ Date (MMDDYY) ____________________ Time (HH:MM) ________________

NH_3 cylinder reference number _______________ H_2S cylinder reference number _______________

Certified Gases							Measured Analyte				
Nominal NH_3 Inlet Conc.	**Dilution**	**Actual NH_3 Inlet Conc.**	**Nominal H_2S Inlet Conc.**	**Dilution**	**Actual NH_3 Inlet Conc.**	**Analyte: Gases present**	**Mean**	**SD[1]** PAML SOP CI	**Accuracy[2]**	**RSD**	**Operator**
ppm			**ppm**				**ppm-m**	**ppm-m**	%	%	
500			**0**			**NH_3:NH_3**					
50			**0**			**NH_3:NH_3**					
5			**0**			**NH_3:NH_3**					
0						**NH_3:NH_3**					
0						**H_2S:H_2S**					
0						**H_2S:H_2S**					
0		**0**				**H_2S:H_2S**					
0		**500**				**$_2S$:H_2S**					
500		**50**	**500**			**NH_3: NH_3+H_2S**					
50		**5**	**500**			**NH_3: NH_3+H_2S**					
5		**0**	**500**		**H**	**NH_3: NH_3+H_2S**					
500			**50**			**NH_3: NH_3+H_2S**					
50			**50**			**NH_3: NH_3+H_2S**					
5			**50**			**NH_3: NH_3+H_2S**					
500			**5**			**NH_3: NH_3+H_2S**					
50			**5**			**NH_3: NH_3+H_2S**					
5			**5**			**NH_3: NH_3+H_2S**					

[1] Standard deviation [2] Accuracy = 100% * (Measured concentration – Certified concentration) / (Certified concentration)

Appendix D:UV-DOAS QA Sheet

UVDOAS Serial Number ___________________

Date	Time	Dark current correction check	+/- 5 counts?	Spectral feature match	<2 pixels?	R[1]	>0.90?	Baseline stability drift at 270 nm	<+/- 0.002AU?	Beam energy at 250 nm	Beam energy at 210 nm	210 nm energy /250 nm energy	<20%?	Operator
mm/dd/yy	hh:mm	# counts	Y/N	pixels	Y/N			AU						

1: R is correlation coefficient

If <u>No</u> is answer any of the above questions, Remediation is needed.

SOP C2. STANDARD OPERATING PROCEDURE FOR MEASUREMENT OF AMMONIA WITH THE BOREAL LASER GASFINDER TUNABLE DIODE LASER ABSORPTION SPECTROMETER (TDLAS)

SOP C2. STANDARD OPERATING PROCEDURE FOR MEASUREMENT OF AMMONIA WITH THE BOREAL LASER GASFINDER TUNABLE DIODE LASER ABSORPTION SPECTROMETER (TDLAS)

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Reviewed by

Bill W. Bogan and Albert J. Heber

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Rev. 1.0 Effective Date: February 11, 2008

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
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West Lafayette, IN 47907

Table of Contents

1. Scope and Applicability

1.1. The absorption of a laser tuned to 1512 nm will be used to measure ammonia gas concentrations upwind and downwind of open manure storage areas and lagoons.
1.2. The open-path concentration data will be obtained with a Tunable Diode Laser Absorption Spectrometer (TDLAS).
1.3. This procedure applies only to the Boreal Laser GasFinder2.0 TDLAS, which can measure only one gas at a time.
1.4. The stated detection limit for ammonia using this TDLAS is 5.3 ppm-m at 100 m (the test length used in this SOP).
1.5. The reported range of NH3 concentrations in the atmosphere around agricultural open-source manure storages is 1 to 800 ppb.
1.6. The path length of this TDLAS is 10 to 1000 m long.

2. Summary of Method

The open-path TDLAS is a monostatic narrowband spectral absorption instrument designed to measure the concentration of various constituents (here NH_3) in the open air along a path of at most 1 km. The TDLAS consists of an integrated transmitter/receiver unit and a remote, passive retro-reflector array. The transceiver houses the laser diode source, the transmitter and receiver electronics, the detector module, and microcomputer subsystems. The laser light emitted from the transceiver unit propagates through the atmosphere to the retro-reflector and returns to the instrument, where it is focused onto a photodiode detector. A portion of the laser beam is passed through an onboard reference cell to provide a continuous calibration update. These two optical signals are converted into electrical waveforms, which the microcontroller processes to determine the actual concentration of gas along the optical path.

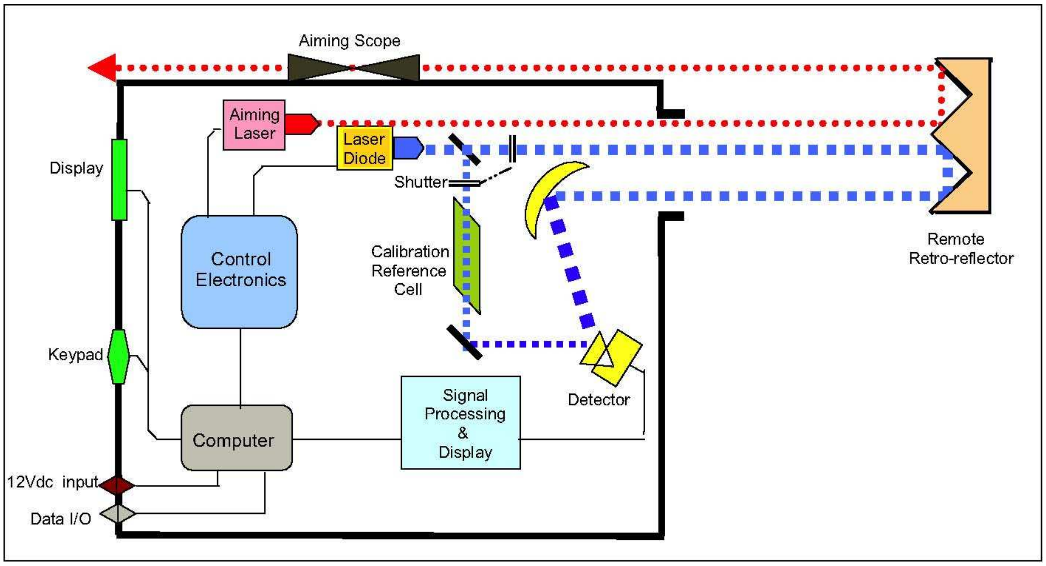

Figure 1. Schematic representation of the GasFinder2.0 TDLAS.

The computed gas concentration is then displayed on the back panel of the instrument. The TDLAS is controlled by a laptop computer linked by hard-wire or radio modem. The TDLAS is mounted on a scanner, which is controlled by the same computer and by the same communications method, and which provides lateral scanning of at least 90° and vertical scanning of at least 15°. The scanner is stepper motor controlled, with a resolution of 0.02 °. The concentration data from the TDLAS data, along with the associated scanner position data, is processed by a computer.

3. Definitions

3.1.	FOS	Field operations staff
3.2.	HD	Computer hard drive
3.3.	TDLAS	Tunable Diode Laser Absorption Spectrometer
3.4.	MDL	Minimum detection limit
3.5.	PIC	Path integrated concentration
3.6.	ppm	Parts per million by volume
3.7.	QAPP	Quality Assurance Project Plan
3.8.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.9.	PAML	Purdue Applied Meteorology Laboratory
3.10.	PC	Personal computer
3.11.	Retro-reflector	A reflector composed of a number of reflecting corner cubes resulting in reflections that always return in the same direction as the incoming light.
3.12.	SMP	Site Monitoring Plan

4. Health and Safety

4.1. The Boreal Laser GasFinder 2.0 Instrument contains both visible and invisible (infrared) laser sources. Both sources conform to Class 3a as per ANSI Z136.1-1993, and do not require the use of protective eyewear, protective equipment or special safety measures. There is no optical ignition hazard presented by lasers of this type.
4.2. Misuse or mishandling of electrical equipment may cause electrocution, severe burns, injury, or death.

5. Cautions

5.1. The sensors have electrical components, and proper care should be used as with all electrical equipment.
5.2. The TDLAS outer window is a thin plastic membrane that can tear if mishandled.
5.3. Optical components in the equipment may be damaged if the equipment is dropped.
5.4. The scanner is made of precision mechanical components that may be damaged if dropped.
5.5. Failure to tightly secure the scanner and TDLAS to its tripod can result in damage to the scanner and/or the TDLAS, and possible injury or death.

6. Interferences

- 6.1. General interferences
  - 6.1.1. Dirty windows (loss of sensitivity and possible interruptions)
  - 6.1.2. Dirty retro-reflectors (loss of sensitivity and possible interruptions)
- 6.2. Weather interferences that obscure light beam
  - 6.2.1. Heavy fog (temporary loss of sensitivity and possible interruptions)
  - 6.2.2. Heavy entrained dust (temporary loss of sensitivity and possible interruptions)
  - 6.2.3. Heavy precipitation (temporary loss of sensitivity and possible interruptions)
- 6.3. Gases that interfere with NH_3 concentration measurements include CO_2 and H_2O (ETV report, Reference 12.4):

7. Personnel Qualifications

- 7.1. Personnel must be trained in use of the handheld multimeter. Training takes 10 min.
- 7.2. Field staff members must read and understand the sensor instruction manual and this SOP, and undergo specific training on this instrument, before operating the TDLAS equipment.

8. Equipment and Supplies

- 8.1. (2) Boreal Laser GasFinder 2.0 Tunable Diode Laser Absorption Spectrometers with scanner heads
- 8.2. Distilled water
- 8.3. Anti-static spray solution
- 8.4. Tissues
- 8.5. NH3 calibration gas cylinder and pressure regulator (SOP G2)
  - 8.5.1. One disposable aluminum cylinder with regulator and about 500 ppm
  - 8.5.2. One aluminum cylinder with regulator and about 1000 ppm
- 8.6. CEM Zero nitrogen (N_2) or CEM zero air gas cylinder with regulator (SOP G2)
- 8.7. Environics Series 4040 Gas diluter (SOP G11)
- 8.8. TEI Model 146 diluter (SOP C3)
- 8.9. Lumitek International Model Q-32-R IR Sensor Card
- 8.10. Calibration chamber
- 8.11. XStream® RS-232/485 RF Modems
- 8.12. Laptop computer
- 8.13. D300 test cable
- 8.14. D300 interface cable (Model D300AC-CAB-25BO).
- 8.15. 24-VDC power supply (Model D300AC-APS-30V).
- 8.16. 12-VDC power supply
- 8.17. DB9 modem cables
- 8.18. (2) Short-haul radio telephones
- 8.19. Tripod

9. Procedures

9.1. TDLAS acceptance

9.1.1. Connect to a 12-VDC, 3-A power supply and switch on (Figure 2).

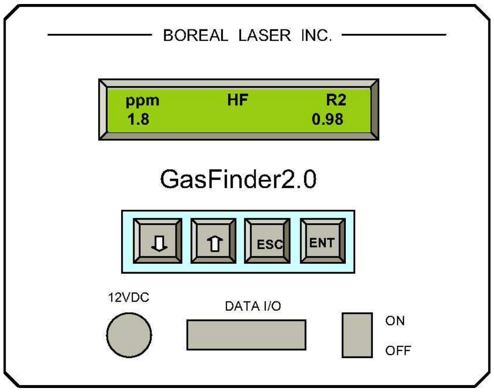

Figure 2. Back panel of TDLAS.

9.1.2. Warm-up cycle lasts approximately 3 min. The display will read:

```
GasFinder2.0   Warm_up
 Ver:0.37   HF    180
```

After the warm-up cycle, the following display may appear:

```
Low Light
No Reading
```

This indicates that the TDLAS is off-target and requires alignment with the reflector tape.

9.1.3. Press the ENT key twice on the keypad, then the ⇩ key once, and the Light Value display will appear:

```
Light Value      dist   m
     64           10
```

This indicates a pre-set default distance of 10 m, and a minimum returning light value of 64 (a light value of < 100 indicates a no-light situation).

9.1.4. Align TDLAS with the visible red aiming laser to point at piece of reflective tape at least 3 m away. Verify the Light Value ranges from 2000-12000. Light level can be decreased by tilting the reflector or rotating the calibration chamber.

9.1.4.1. Verify the distance in the GasViewMP program reads 1.0 meter.

9.1.4.1.1. If the distance is a value other than 1.0 meter, change the value to 1.0 meter in the Aim menu and click OK.

9.1.5. Press the ESC key twice on the TDLAS to return to the normal operating mode. After the computer checks the calibration, the display will read something like:

```
ppm      HF        R2
4                0.99
```

9.1.5.1. This indicates a measured path-averaged hydrogen fluoride (HF) concentration of 4 ppm with an R^2 of 0.99 (99%). Note that the actual value for ppm will be displayed in the GasViewMP program if the path length (distance) is set correctly.

9.1.6. The TDLAS instrument is shipped already calibrated.

9.1.7. The TDLAS will undergo an internal calibration check on power-up (See Section 9.3.2).

9.1.8. Check calibration with single-point calibration according to Section 9.3.4.

9.2. Scanner acceptance

9.2.1. Mount scanner on provided tripod.

9.2.2. Connect scanner at D300 base connector (Figure 3) to computer serial COM port and 24-VDC, 2.3-A power supply using the D300 interface cable.

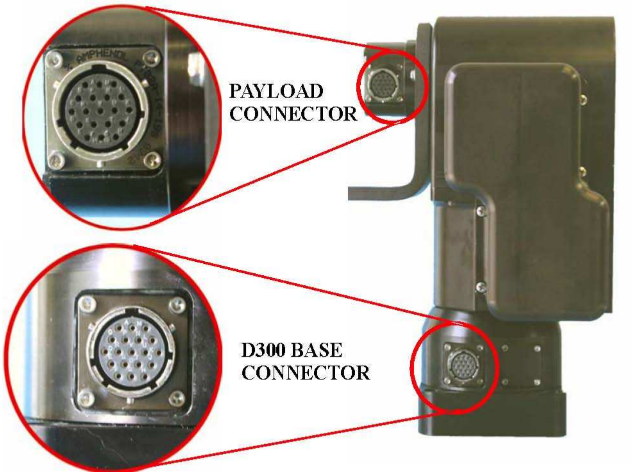

Figure 3: Scanner cable connections.

9.2.3. Using HyperTerminal, set computer port to 9600 baud, 1 start bit, 8 data bits, 1 stop bit, and no parity or handshaking.

9.2.4. Power up the scanner. If power is working, the unit may go through a power-up calibration as soon as power is supplied (not all scanners are programmed to do so).

9.2.5. At power up, the scanner defaults to providing splash text on the computer screen that will identify the unit configuration. The scanner should go through a reset cycle (pan and tilt axes will cycle through their full range of motion). This reset is completed when an asterisk ('*') appears.

9.2.5.1. If the unit does not reset properly, recheck the power source and cables.

9.2.5.2. If the unit went through its reset procedure, but no text or garbled text appears on the screen, then:

9.2.5.2.1. Check that the host RS-232 host port settings are correct.

9.2.5.2.2. Check that the RS-232 cable is correct for the host computer.

9.2.6. Test scanner motion under TDLAS load.

9.2.6.1. Attach TDLAS to the scanner as a side mount using provided bracket and bolts.

9.2.6.1.1. Test the pan axis: Move the pan axis through its range using the following sequence of commands in HyperTerminal (HT):

Enter in HT	System response in HT	Scanner physical response
dr_	*	
Pp1000_	*	Moves approx. 6° to right
pp-1000_	*	Moves approx..6° to left
pp_	"current pan position is -1000"	

Note: Underline indicates a "space"

9.2.6.1.1.1. A load that is too heavy or moved too quickly will cause unit to lose synchrony, resulting in an audible "rrrr" sound from the pan-tilt unit motors. If this occurs, contact Boreal Laser for remediation.

9.2.6.1.2. Test the tilt axis. Move the tilt axis through its range of motion to test whether the pan-tilt can handle the load using the following sequence of commands in HyperTerminal (HT):

Enter in HT	System response in HT	Scanner physical response
dr_	*	
tp1000_	*	Moves approx. 12° down
tp-1000_	*	Moves approx. 12° up
pp_	"current tilt position is -1000"	

9.2.6.1.2.1. A load that is too heavy or moved too quickly will cause the unit to lose synchrony, which will be accompanied by an audible "rrrr" sound from the scanner. If this occurs, contact Boreal Laser for remediation.

9.3. TDLAS calibration

9.3.1. The factory calibration data are stored in the instrument's software as a standard reference waveform.

9.3.2. At system startup

9.3.2.1. When powered up, the GasFinder automatically centers the laser frequency using the internal reference cell and precise control over the internal laser diode temperature and drive current. The system will display "Calibrating" while it is calibrating, and the associated GasFinder data status code (Appendix A) will display 2001.

9.3.2.1.1. Upon completion, the GasFinder will be in a standard operating mode displaying gas data and R2 values on the front panel (Figure 2). If the GasFinder automatic calibration fails, the display will read “CAL_ERR” and the status code within the GasFinder serial data string will display a value other than 1. If the system fails this calibration, contact Boreal Laser Inc. for further instructions.

9.3.2.1.2. Verify that the units setting is ‘ppm-m’. If set on ‘ppm’, change to ‘ppm-m’.

9.3.3. During operation

9.3.3.1. The internal reference cell scan is compared with the stored waveform at specified intervals with significant deviations generating a QA status code to alert the user to a potential calibration problem.

9.3.3.2. The drift in the absolute absorption of the reference cell is assessed periodically and recorded in the instrument. The sample, reference and calibration waveforms are used to verify that the internal calibration system is functioning correctly.

9.3.4. A single-point calibration check is made using an external chamber filled with a known quantity of gas and measured.

9.3.4.1. Record the gas concentration (50 ppm) of the calibration cylinder (verified as below) on the calibration sheet.

9.3.4.1.1. Concentration of the calibration gas should be directly checked by FTIR (SOP G12), or checked against that of a cylinder which has been checked by FTIR.

9.3.4.1.2. Calibration gas can be mixed with zero air or zero N_2 using an Environics Model 4040 diluter (SOP G11) or a TEI Model 146 diluter (SOP C3) to achieve the desired concentration, if other than 50 ppm. The calibration concentration, either directly from the cylinder or as indicated by the diluter, is the ‘Expected concentration’.

9.3.4.2. Insert external calibration chamber in laser path.

9.3.4.2.1. 50-cm chamber made of Teflon tube with Lexan® windows at Brewster’s angle.

9.3.4.3. Attach zero air/zero N_2 cylinder to a rotameter, open gas cylinder to a pressure of 20 psi, and adjust the flow control 0.5 L/min (flow verified by rotameter). Once verified, remove tubing connection to rotameter and attach to calibration chamber.

9.3.4.4. Align GasFinder through the calibration chamber according to Sections 9.1.4-9.1.5.

9.3.4.5. Flow zero air into chamber for 5 min.

9.3.4.6. Collect background scans over 1 min while flowing zero air/zero N_2 through the chamber.

9.3.4.7. Attach tubing from zero air/zero N_2 cylinder to the TEI 146I diluter’s zero air port, and the calibration cylinder to port A (or other programmed Port).

9.3.4.8. Open both gas cylinders (calibration gas and zero air) to a pressure of 20 psi

9.3.4.9. Connect output port to calibration gas to chamber with tubing.

9.3.4.9.1. Effective concentration of the calibration gas flow through 50-cm chamber is 50% of inlet concentration per meter. For example, 50 ppm concentration calibration gas yields 25 ppm-m path.

9.3.4.9.1.1. Dilute calibration gas with zero air/zero N_2 as necessary with diluter.

9.3.4.9.1.2. Verify flow rate through diluter.

9.3.4.9.2. Allow three exchanges of the chamber volume prior to measurement.

9.3.4.9.3. Rotate calibration cell until the linear correlation r^2 is maximized and concentrations are stabilized. This should occur when the reflected light off the incoming light window is near a minimum.

9.3.4.10. Collect 5 min of consecutive NH_3 measurements.

9.3.4.10.1. In Microsoft Excel, determine the accuracy [(expected-measured)/expected] based on the valid measurements (r2>0.84), and determine the standard deviation of the five (5) 1-min mean measured concentrations. Calculate the relative standard deviation (RSD) based on the measured mean concentration of gas supplied to the external chamber. Results are stored on form similar to Appendix B.

9.3.4.10.2. The ETV Report for TDLAS (Ref. 12.4) documented an MDL of TDLAS for NH_3 is 5.3 ppm-m with dwell time of 1 minute. Accuracy was 1.3% to 9.8% for a 220-m pathlength.

9.3.4.10.3. If the calculated accuracy (RSD) is greater than 10%, then further evaluation of the instrument is indicated.

9.3.4.10.3.1. Repeat calibration verification with 5 min of measurements using NH_3 calibration gas diluted to 200 ppm and recalculate RSD.

9.3.4.10.3.2. If the accuracy (RSD) of this test is less than 10%, the diluter may be introducing noise in the response. Repeat calibration verification with 5 min of measurements using NH_3 calibration gas undiluted (at the full concentration of the calibration gas cylinder) and recalculate the RSD.

9.3.4.10.4. If the accuracy (RSD) continues to be greater than 10% regardless of concentration of supplied calibration gas to the chamber, decommission the instrument. The ETV Report documented an RSD precision of 3.14%.

9.3.4.10.5. If the calculated precision (RSD) is greater than 5% then further evaluation of the instrument is indicated.

9.3.4.10.5.1. Repeat calibration verification with 5 min of measurements using NH_3 calibration gas diluted to 200 ppm and recalculate RSD.

9.3.4.10.5.2. If the RSD is greater than 5%, the diluter may be introducing noise in the response. Repeat calibration verification with 5 min of measurements using NH_3 calibration gas undiluted (at the full concentration of the calibration gas cylinder) and recalculate RSD.

9.3.4.10.5.3. If the RSD continues to be greater than 5% regardless of concentration of supplied calibration gas to the chamber, decommission the instrument is and send to the manufacturer for repair.

9.3.5. Multipoint calibration check. Because the anticipated gas concentrations are expected to span several orders of magnitude, a linear system response should be verified. Thus, perform a three-point calibration spanning the anticipated concentration range.

- 9.3.5.1. Align TDLAS to have laser reflect off reflector tape at an approximate 3-m distance.
- 9.3.5.2. Insert 50-cm external calibration chamber into the laser path.
  - 9.3.5.2.1. Use calibration gas concentration of about 1000 ppm (exact value determined following Section 9.3.4.1).
  - 9.3.5.2.2. Dilutions of the 1000-ppm calibration gas can be made using an Environics Model 4040 diluter (SOP G11) or a TEI Model 146 diluter (SOP C3) to achieve the desired concentration.
  - 9.3.5.2.3. Make all dilutions with CEM zero air or CEM zero N_2.
  - 9.3.5.2.4. Calibrations should be made at inlet concentrations of 1000 ppm (undiluted), 500 ppm, and 200 ppm.
  - 9.3.5.2.5. Corresponding integrated path concentrations are 500 ppm-m, 250 ppm-m, and 100 ppm-m. Adjust these values as necessary based on the verified composition (Section 9.3.4.1) of the calibration gas cylinder.
- 9.3.5.3. For each calibration gas concentration:
  - 9.3.5.3.1. Insert external chamber into laser path.
  - 9.3.5.3.2. Verify signal strength is within 2000 – 12000 range.
  - 9.3.5.3.3. Open zero air/zero N_2 flow into chamber at 0.5 L/min for 5 min and collect background scans as in Sections 9.3.4.3 – 9.3.4.6..
  - 9.3.5.3.4. Collect measurements for 1 min.
  - 9.3.5.3.5. Introduce calibration gas (or dilution thereof) into the cell.
  - 9.3.5.3.6. Adjust orientation and position of the calibration cell until r^2 is maximized and concentrations are stabilized.
  - 9.3.5.3.7. Collect measurements for 5 min according to Section 9.3.4.9.
- 9.3.5.4. In Microsoft Excel, determine the standard deviation of the discrepancy between actual and measured concentrations at each point of the multipoint calibration. Calculate the percent relative accuracy at each concentration as the absolute value of the difference between the monitor response and the reference gas concentration, divided by the reference gas concentration. Calculate the average accuracy by averaging the percent accuracy at all of the individual calibration concentrations.
  - 9.3.5.4.1. The ETV Report documented an accuracy of 1.3 to 9.8% for a 220-m pathlength.
  - 9.3.5.4.2. If the calculated accuracy (RSD) is greater than 10%, decommission the instrument and send to the manufacturer for repair.
- 9.3.5.5. Calculate instrument linearity in Microsoft Excel using the regression function, making sure to only use measurements with the same integration time and pathlength.
  - 9.3.5.5.1. The ETV Report documented a linearity of 0.99.
  - 9.3.5.5.2. If the linearity is less than 0.95, decommission the instrument and send to the manufacturer for repair.

9.4. Scanner calibration

- 9.4.1. The scanner is controlled by step motors, and their proper function dictates proper movement.
- 9.4.2. The scanner may be configured to reset both the pan and tilt axes automatically upon

power up or when the reset command is issued. If necessary, transmit a manual rest using the GasViewMP program.

9.4.2.1. The reset calibration allows the pan-tilt unit to determine axis coordinates, hence the pan-tilt axis should be reset every time the scanner is powered up (default).

9.5. TDLAS and scanner installation

9.5.1. Power and communication with the TDLAS and scanner can either be directly through a laptop computer (with separate power supplies for each component), or through a combined power supply/RF modems.

9.5.2. Serial communications configuration: 8 data bits, 1 stop bit, No parity, No handshaking, 9600 baud

9.5.3. If using separate power supplies and direct serial connection to laptop:

9.5.3.1. Connect the TDLAS serial/power cable from the TDLAS DATA I/O connector and power connector. Connect the D300 test cable from the TDLAS/scanner D300 base connector to the laptop COM port and USB COM port, the TDLAS power supply, and the scanner power supply.

9.5.3.2. Connect the two power supplies to line power (110VAC).

9.5.3.3. In the field, connect the power/communications unit to the scanner's D300 base connector (Fig. 3) and the payload connector (Fig. 3).

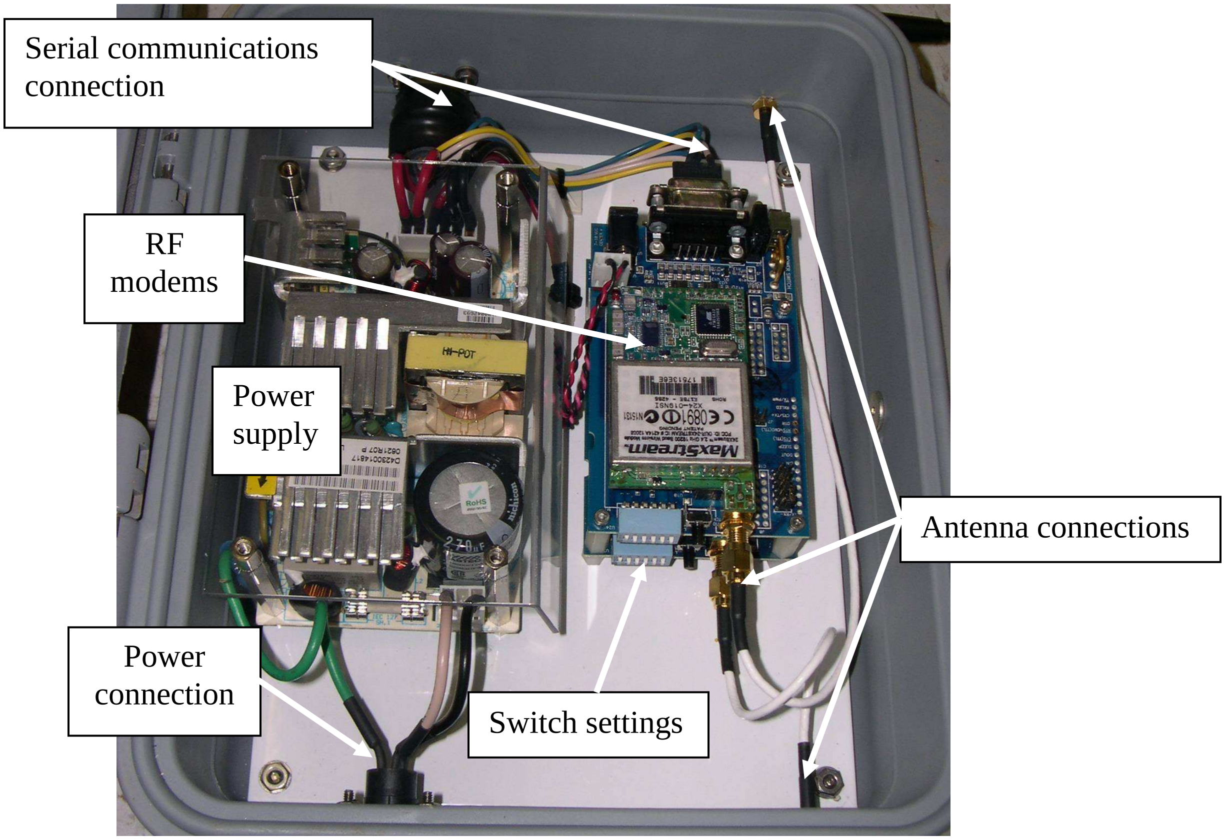

Figure 4. Combined power supply/RF modem unit. Top modem is for the TDLAS, and bottom modem is for the scanner.

9.5.4. If using combined power supply/RF modems:

9.5.4.1. Install laptop in trailer and connect the serial cable from the TDLAS RF modem (Fig. 4) in the instrumentation trailer to first laptop computer COM port and connect the serial cable from the scanner RF modem in the instrumentation trailer to the second laptop COM port (a USB port).

9.5.4.2. Verify that the switch settings on the two RF modems in the TDLAS/scanner power/communications unit (Fig. 4) are identical to the paired modems (one for the scanner and one for the TDLAS) attached to the laptop computer in the trailer.

9.5.4.3. Attach antennae from the trailer roof to the two RF modems in the trailer using coaxial cables.

9.5.4.4. Attach wall power transformers to RF modems in trailer and connect wall power transformers to 110 VAC power.

9.5.4.5. Verify power to radio modems by checking red lights on the modems (Fig. 5).

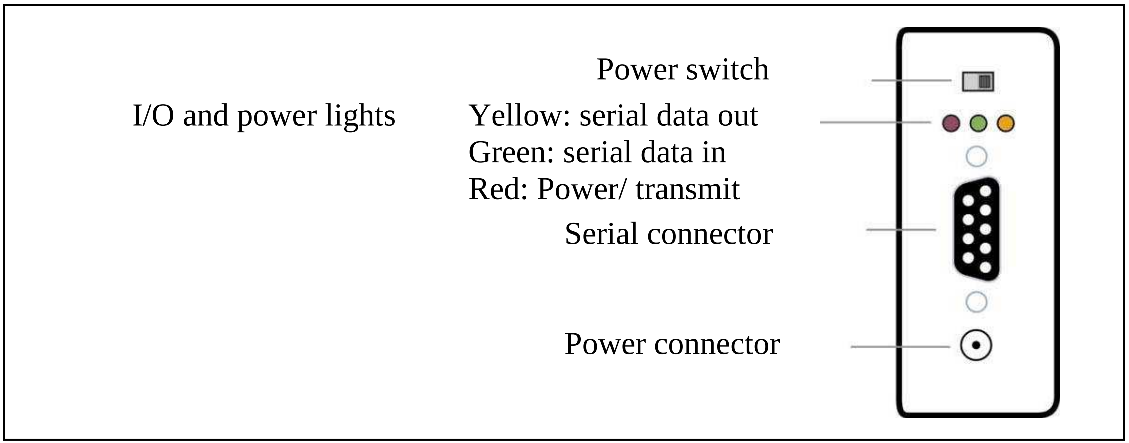

Figure 5. Trailer RF Modem connections.

9.5.4.6. In the field, connect the cable from the power/communications unit (Fig. 6) to the scanner's D300 base connector (Fig. 3), and the TDLAS serial/power cable from the TDLAS DATA I/O connector and power connector.

9.5.4.6.1. Use PC Windows HyperTerminal program on the laptop computer to verify configuration of modem in accordance with SOP U6.

9.5.4.7. Attach both antennae to the power/communications unit.

9.5.4.8. Connect power cord from power/communications unit to 120-VAC line power.

9.5.4.9. Verify power to radio modem by checking red lights on the modems in the power/communications unit (Fig. 6).

9.5.4.10. Test communication link to PC in trailer.

9.5.5. Switch on the TDLAS instrument (the scanner has no start switch but starts on provision of power). The TDLAS requires a 3-min warmup period.

9.5.5.1. Switch power on at TDLAS. Warm up cycle for TDLAS takes about 3 min.

9.5.5.2. Establish communications according to Section 9.5.

9.5.5.3. Locate appropriate input serial channel on computer in trailer (SOP U4) and evaluate output for reasonable values. If TDLAS does not indicate reasonable values, refer to Section 9.8.

9.5.6. Scanner operation
- 9.5.6.1. Establish communications according to Section 9.5.
- 9.5.6.2. Warm up consists of scanner going through a reset cycle where pan and tilt axes will cycle through their full range of motion. If scanner does not reset properly, refer to Section 9.10.

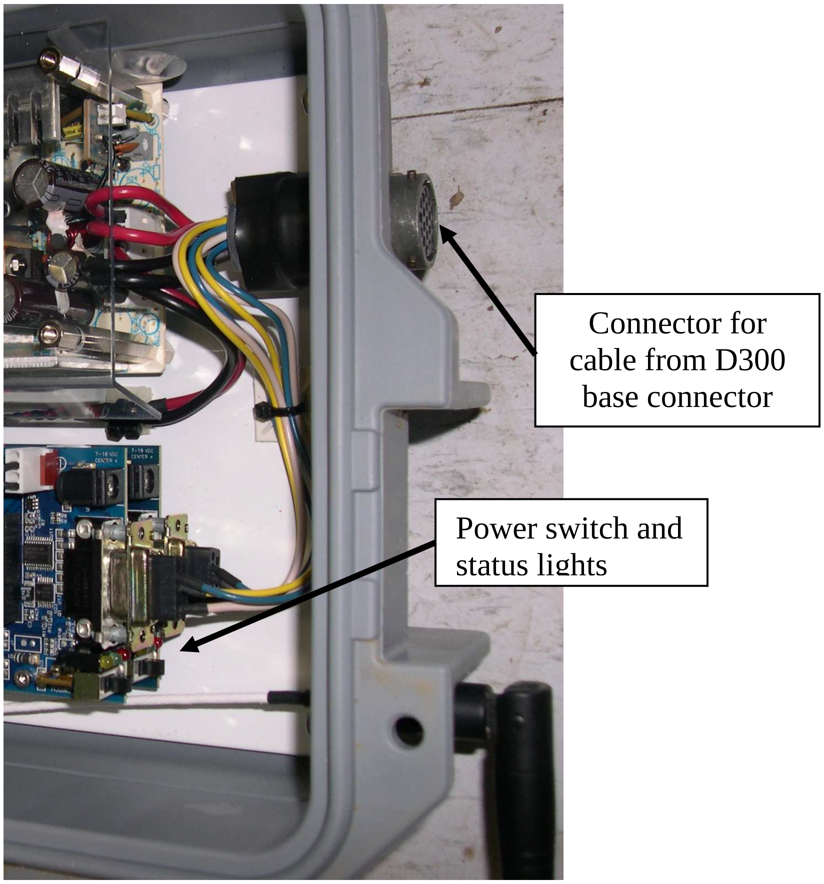

Figure 6. Connections on the combined power supply/RF modem unit.

9.5.7. Start GasViewMP software program on the laptop computer in the trailer.
- 9.5.7.1. Set number of retro-reflectors to be scanned by the scanner under ‘Options‘, ‘Pan-Tilt Unit’ dropdown menu. Setting is in upper left-hand corner.
  - 9.5.7.1.1. Set scanner parameters as:
    - 9.5.7.1.1.1. Pan and Tilt Hyst to 100
    - 9.5.7.1.1.2. Minimum Acceptable to 3000, Maximum Acceptable to 11000, and Optimum Level to 8000
      - 9.5.7.1.1.2.1. Exceptions will occur at sites with longer or shorter than typical path lengths.
    - 9.5.7.1.1.3. Obstruction Level to 100
    - 9.5.7.1.1.4. Size to 2
    - 9.5.7.1.1.5. Maximum Cross Excursion to 0 and Spiral Excursion to 5

9.5.7.1.1.6. Timeout to 900 sec

9.5.7.2. Click on ‘optimize enable’ under ‘Options‘ dropdown menu.

9.5.8. Sequentially align the retro-reflectors configured according to SOP U5 with the laser head. At 250-m path length, the diameter of the beam is about 375 mm, so the beam area is larger than the area of the retro-reflector (260 mm x 229 mm).

9.5.8.1. Procedure generally requires two people: one at the computer and one at the TDLAS/scanner.

9.5.8.2. Enter “light meter” mode on back of TDLAS as per Section 9.1.3. The red aiming laser will be visible.

9.5.8.3. Position retro-reflectors according to SOP U5.

9.5.8.4. Use alignment routine accessed from ‘AIM’ radio button to center the TDLAS aiming scope crosshairs on a retro-reflector target. The software will have an adjustment for movements of 1-500 steps/move.

9.5.8.5. With the crosshair centered on the retroreflector, you should be able to see a large reflection of the visible laser back through the GasFinder aiming scope. Using the light value displayed on the GasFinder front panel, maximize the GasFinder return light signal using approximately 1 step/move. There is a 1 measurement time delay (roughly 1 sec) associated with a position move and the resulting change of light level.

9.5.8.6. Fine-tune the adjustment so that the displayed light value is near maximum level (16368) or as high as possible.

9.5.8.6.1. If the light value is below 2000, the system is in a low-light situation, and may fail to make a correct measurement. When the light value drops below a factory set value of 500, the system will give a status message on the display indicating “low light -no reading” and an error code will be stored in the instrument (termed ‘checksum’ in Appendix A). To increase the light value, move the crosshairs closer to the center of the retroreflector array or increase the number of retroreflectors.

9.5.8.6.2. If the light value is above the optimal range, the receiver electronics may saturate and spurious readings may be displayed (e.g., unrealistically high concentration values and low confidence factors). Program will optimize light level if optimization has been enabled.

9.5.8.7. Set Dwell Time for 15 sec.

9.5.8.8. Indicate path length next to ‘Distance’ setting. Distance between scanner and retro-reflector is documented in appropriate SMP.

9.5.8.9. Record settings with ‘APPLY’ radio button.

9.5.8.9.1. Record the coordinates of the retro-reflector on paper.

9.5.8.10. Repeat Sections 9.5.8.5 through 9.5.8.8 for each retro-reflector to be scanned by the scanner.

9.5.8.11. Manually perform one full cycle between targets by stepping down through path numbers and record GasFinder light values. If light values are not greater than 2000, return to Sections 9.5.8.4 through 9.5.8.6 for alignment.

9.5.8.12. Verify that each path is aiming at its own designated location.

9.5.8.13. Once complete, click radio button ‘OK’

9.5.8.14. Under ‘Options’, ‘LogFile’ dropdown menu, check “By Clock” and place checkmarks next to 00:00, 06:00, 12:00, and 18:00. Check “Append Timestamp”.

9.5.8.15. Under ‘File’, ‘OpenLogFile’ dropdown menu, browse to find the appropriate directory for storing the new data. If necessary, create this new directory. Name the output file, e.g., TDL1_OH1A.

9.5.8.16. Exit “light meter” mode on back of TDLAS by pressing ESC key twice. The screen on the TDLAS should appear similar to Fig. 2.

9.5.8.17. Initiate scanning by clicking radio button ‘RUN’

9.5.8.17.1. Be sure that the Alarm output on the main GasViewMP screen says 1 when valid light levels are present. If not, then be sure that the instrument is out of alignment mode

9.6. Power up the pan-tilt unit and test its operation according to Section 9.2.6.

9.7. Instrument operation

9.7.1. Two configurations of the TDLAS and scanner are used. Both configurations will scan ten retro-reflectors per cycle (SOP U5).

9.7.1.1. TDLAS and scanner communicating with the control and data acquisition computer in the trailer (SOP U4) by hard-wired D300 interface cable.

9.7.1.2. TDLAS and scanner communicate with the control and data acquisition computer in the instrumentation trailer (SOP U4) by RF modems.

9.7.2. Ensure that the power cables for the TDLAS and scanner on the D300 interface cable are connected to the 12VDC power supply (TDLAS) and 24VDC power supply (scanner). Ensure that the TDLAS Data I/O and 12V power cables are connected to the scanner payload cable (Fig. 3).

9.7.3. Ensure that the scanner and TDLAS serial data connectors on the D300 interface cable are connected to their respective RF radio modems or directly to the COM ports of the control and data acquisition PC.

9.7.4. TDLAS operation

9.7.4.1. Switch power on at TDLAS. Warm up cycle for TDLAS takes about 3 min.

9.7.4.2. Establish communications according to Section 9.5.

9.7.4.3. Locate appropriate input serial channel on computer in trailer (SOP U4) and evaluate output for reasonable values. If TDLAS does not indicate reasonable values, refer to Section 9.8.

9.7.5. Scanner operation

9.7.5.1. Establish communications according to Section 9.5.

9.7.5.2. Warm up consists of scanner going through a reset cycle where pan and tilt axes will cycle through their full range of motion. If scanner does not reset properly, refer to Section 9.10.

9.7.5.3. From the GasViewMP software program running on the laptop computer in the trailer, perform the following:

9.7.5.3.1. Perform one full cycle between targets and record GasFinder light values. If light values are not in the range of 2000-12000, return to Section 9.5 for alignment.

9.7.5.3.2. Initiate scanning.

9.7.5.3.3. Verify that each path is aiming at its own designated location.

9.8. Communications troubleshooting

- 9.8.1. Verify communications link between radio modems (Yellow and Green lights on modems (Fig. 5).
  - 9.8.1.1. If no communications:
    - 9.8.1.1.1. Verify firm connections for power, signal, and antenna for serial communications.
    - 9.8.1.1.2. Verify power to modems.
    - 9.8.1.1.3. Verify Destination Address of radio modems in accordance SOP W6.
    - 9.8.1.1.4. Check that the correct baud rate is selected (See Serial Communications section).

9.9. TDLAS troubleshooting

- 9.9.1. If the system is not operating:
  - 9.9.1.1. Check power supply integrity. Verify that the unit is plugged in, and that the fuse and power cable are intact.
- 9.9.2. If the status message: “Low Light -No Reading” appears:
  - 9.9.2.1. Check that open path is clear, and that no obstruction (vehicle, personnel, etc) is interfering with the signal.
  - 9.9.2.2. Check for dirty windows, and clean as necessary (Section 11.1).
  - 9.9.2.3. Check alignment (Section 11.2)
  - 9.9.2.4. If trying to operate the unit in heavy fog or heavy rain, it may be necessary to wait for improved weather conditions.
  - 9.9.2.5. Verify optical alignment (see Operating Instructions)
- 9.9.3. If a problem develops with the internal thermoelectric cooler or the associated electronics, the system will protect the laser diode by shutting the laser off. Contact Boreal Laser to resolve this problem.
- 9.9.4. If the signal being analyzed by the onboard electronics falls below a certain set value, and the returning light level is within the acceptable bounds, this may indicate a problem with the internal receiver electronics. Contact Boreal Laser to resolve this problem.
- 9.9.5. If the voltage of the portable battery or power supply falls below 11.5V, a ‘B’ will appear in the top right corner of the display. If the voltage falls below 10.0V, the instrument will shut down and enter a “low power” mode after saving all the system variables. In this mode, internal logging of the data will be suspended. After a voltage greater than 11.5V is applied, the instrument will resume normal operations.
- 9.9.6. If a constant measurement is recorded for more than 1 min
  - 9.9.6.1. Check for clicking sound of shutter changing position of laser path between internal reference cell and outside retro-reflector.
    - 9.9.6.1.1. If there is no clicking sound, contact manufacturer for possible repair.
- 9.9.7. If no data is being transmitted to the storage computer:
  - 9.9.7.1. Check that the correct handshaking option is selected. (See Section 9.5.2).
  - 9.9.7.2. Check that the TDLAS is on line in the GasView MP program (TDLAS serial number indicated at the top of the graph), and that the data communications cable(s) is (are) connected.

9.10. Scanner troubleshooting

9.10.1. If scanner does not show an introduction page (In HyperTerminal) and go through a reset cycle (pan and tilt axes will cycle through their full range of motion):

9.10.1.1. Recheck power source and cabling, unplug power and replug power to scanner.

9.10.1.2. If problems continue, contact Boreal Laser.

9.10.2. If the scanner went through its reset procedure, but no text or garbled text appears in HyperTerminal:

9.10.2.1. Check that the host RS-232 host port communications settings are correct.

9.10.2.2. Check that the RS-232 cable is correct for the host computer.

9.10.2.3. If problems continue, contact Boreal Laser.

9.10.3. If there is an audible “rrrr” sound from the pan-tilt unit motors during movement, contact Boreal Laser.

9.10.4. If scanner is operating properly but no TDLAS measurements are indicated on either the TDLAS itself or the GasViewMP program, realign retro-reflector.

9.10.4.1. A record of the coordinates of the retro-reflector is beneficial as it provides a good starting point for re-alignment.

9.11. Data acquisition, calculations & data reduction requirements

9.11.1. The data are transmitted as a comma-delimited ASCII string. The format of the string is as follows:

$GFDTA, scanline#,Conc, R^2,dist,light,date_time,ser_num,status_code,* checksum

such as: $GFDTA,3,8.9,18,125.0,6292,2007/12/10 08:05:42, NH3OP-1029,1*52

9.11.1.1. $GFDTA indicates a data value

9.11.1.2. scanline: ranging from 1 to 10, integer

9.11.1.3. Conc: concentration in ppm-m (parts per million meter), real number

9.11.1.4. R2 : value between 0 and 99, Integer

9.11.1.5. dist: user set distance to reflector in meters, real number

9.11.1.6. light: returning light level, Integer

9.11.1.7. date_time: current date and time, YYYY/MM/DD hh:mm:ss

9.11.1.8. ser_num: identification number of the system, String

9.11.1.9. status_code: code giving system operation parameters, Integer. QA status codes are described in Appendix A of this SOP.

9.11.1.10. An asterisk (*) signifies end of string. Each string is terminated by a carriage return and a line feed.

9.11.1.11. checksum: check summing option to verify data integrity, String.

9.11.2. The [NH_3] PIC values are stored as individual files on laptop hard drive according to SOP D1.

9.12. Computer hardware & software

9.12.1. The instrument is controlled internally and communicates with a computer by two serial modems. The computer will record and store the data.

10. Data and Records Management

10.1. Store the measurements of each scan path as individual record strings as (a1) files.

10.1.1. Store measurements for each instrument in separate directories on the HD as specified in SOP D1.

10.1.2. Send data from each instrument to Purdue through ftp according to SOP D1.

10.1.3. Receipt of raw data at Purdue will be logged automatically, and the data will be transferred to a directory for daily quality assurance checks.

11. Quality Control and Quality Assurance

Currently there are no EPA guidance documents that specifically address Quality Assurance (QA) for TLDAS spectroscopy. However, because these systems operate in a similar manner as infrared air monitoring systems, we can borrow from the vast array of QA documentation that has been established for the air monitors.

11.1. Cleaning windows

11.1.1. The instrument relies on a clear optical path to obtain readings of gas concentration. Dust settling on the instrument’s window and/or the window of the retroreflector enclosure must be cleaned off whenever it degrades the instrument’s performance. The average value for the light level should be kept between 2,000 and 10,000.

11.1.2. Many dusts are very abrasive and care must be taken not to scratch the windows when cleaning them. Follow the recommended procedure for cleaning in the manual.

11.1.3. The need for cleaning will be indicated by a consistent loss of light, which may result in poor gas concentration readings. Cleaning intervals may vary from once per week to once per year.

11.2. Alignment

11.2.1. Difficulty in obtaining an adequate light level after cleaning the windows may indicate that the instrument has moved out of alignment. This may be caused by movement of the base due to vibration or other factors. Details of the alignment procedures can be found in the Operation Manual.

11.2.1.1. Alignment is automatically adjusted within the GasViewMP program optimization routine if optimization has been enabled.

11.2.1.1.1. Scanner moves in search pattern defined by setup parameters (Section 9.5.7.1.1).

11.2.1.2. Optimization routine begins when light level is above obstruction light level and below minimum acceptable light level. GasViewMP reports each position in the optimization routine (resulting from low light level) as a $GFDTA measurement (Section 9.11.1). After specified number of trial positions, the optimized position value is reported as:

```
$GFMPO, opt_channel, opt_light, opt_status, opt_pan, opt_tilt, opt_date_time ',
Checksum
```

A typical optimization sequence on path #1 is illustrated below:

```
$GFDTA,1,0.0,0,44.0,6736,2007/12/10 07:50:00, NH3OP-1029,30001*5F
$GFDTA,1,0.0,0,44.0,80,2007/12/10 07:50:03, NH3OP-1029,30010*50
$GFDTA,1,0.0,0,44.0,80,2007/12/10 07:50:05, NH3OP-1029,30010*56
$GFDTA,1,0.0,0,44.0,80,2007/12/10 07:50:06, NH3OP-1029,20010*54
$GFDTA,1,0.0,0,44.0,14067,2007/12/10 07:50:06, NH3OP-1029,20001*68
$GFMPO,1,12165,3,6533,-229,2007/12/10 07:50:08*5A
$GFDTA,1,0.0,0,44.0,12165,2007/12/10 07:50:08, NH3OP-1029,1*61
$GFDTA,1,0.0,0,44.0,9799,2007/12/10 07:50:09, NH3OP-1029,70001*58
$GFDTA,1,0.0,0,44.0,16368,2007/12/10 07:50:11, NH3OP-1029,60001*64
$GFDTA,1,0.0,0,44.0,6103,2007/12/10 07:50:13, NH3OP-1029,60001*58
$GFMPO,1,5227,3,6535,-229,2007/12/10 07:50:14*62
```

11.2.1.2.1. channel, opt indicates channel being optimized

11.2.1.2.2. opt_status values indicate:
- 0 – minimum light level not met (keep coordinates)
- 1 – new path coordinates accepted
- 2 – Low light detected and optimizing
- 3 – High light detected and optimizing
- 4 – Pan-tilt movement error/timeout during optimization(see status code n20 from $GFDTA line).

11.2.1.2.3. opt_pan and opt_tilt are the optimized pan and tilt coordinates

11.3. Internal linearity check

11.3.1. During operation, the instrument's internal reference cell is periodically compared with the stored waveform at specified intervals with significant deviations generating a QA status code to alert the user to a potential calibration problem.

11.3.2. The drift in the absolute absorption of the reference cell is assessed periodically and recorded in the instrument.

11.3.3. Internal check indicated as:

```
$GFDBG, internaltemp, centerlinedutycycle, refcellquality, refR2, refstatus, supplyvoltage, gain1, gain2, Date_Time, Checksum
```

For example:

```
$GFDBG, 12.6, 165, 10344, 97, 0, 1341, 1, 0,2007/12/10 08:06:02*64
```

11.3.3.1. "internaltemp" is the TDLAS internal temperature (^{o}C)

11.3.3.2. "centerlinedutycycle" is the output of an internal pulse-width modulator that generates a small DC offset current to make adjustments to the laser diode drive current in order to compensate for changes in laser wavelength due to temperature changes. The value is adjusted by the microcontroller by measuring the peak position of the LD wavelength and comparing it to a pre-set value.

11.3.3.3. "refcellquality" is a comparison of the current, live reference cell waveform with the factory-stored calibration waveform. A comparison of 100% would be shown as 10000.

11.3.3.4. “refR2” is the correlation coefficient of the linear regression of the reference cell to the stored waveform of the reference cell on installation

11.3.3.5. “refstatus” value “0” is the normal operation code. Additional codes are given in Appendix A.

11.3.3.6. “supplyvoltage” is the voltage into the GasFinder, in 1/100^{ths} of a volt

11.3.3.7. “gain1” and “gain2” are internal circuit gain values, and are used primarily during setup of the instrument at the factory. Values range from 0 to 3, with 0 being a maximum gain (lowest attenuation) and 3 being lowest gain (maximum attenuation). These values are not necessarily indicative of any particular problem or status of the instrument.

11.3.4. Comparison of repeated measured concentrations in reference cell to actual: Coefficient of Determination (r^2) should be greater than 0.96.

11.4. Each reported measurement has corresponding QA status codes (Appendix A) indicating instrument performance.

11.4.1. Comparison of structure of internal reference cell absorption spectrum to measured spectrum, as evaluated by Coefficient of Determination (r^2), provides a measure of accuracy of measurement.

11.4.2. Company-provided relationship of NH_3 measurement accuracy to r^2:

r^2	Accuracy		
>90	+/-	2	%
80	+/-	13	%
70	+/-	24	%
60	+/-	35	%
50	+/-	46	%
40	+/-	57	%
30	+/-	67	%
20	+/-	78	%
10	+/-	89	%
0	+/-	100	%

11.5. Determination of Root Mean Square noise

11.5.1. RMS noise is used to determine the inherent noise of the system. This is an important QA element, as it determines the MDL for the gases of interest.

11.5.1.1. The procedure to measure RMS noise is to collect successive single-beam spectra. The spectra should simulate the standard field setup, including a typical path length for the equipment (100 m), typical integration time for the data collection (1 sec), and typical sample time for each spectrum.

11.5.1.2. Measure concentrations for 15 sec.

11.5.1.3. In Microsoft Excel, determine the standard deviation of the measurements where the r^2 value is greater than 0.90. The MDL is two times the standard deviation.

11.5.1.3.1. The stated detection limit for ammonia is 0.79 ppm-m at 100 m.

11.5.1.3.2. The ETV Report documented a MDL of 5.32 ppm-m for ammonia.

11.6. The sample, reference and calibration waveforms are used to verify that the internal calibration system is functioning correctly.

11.7. If light levels range from 100-3000 or above 11000 on a particular retro-reflector path, the instrument conducts an automatic re-alignment routine to maximize signal strength on that individual path. New pan/tilt coordinates are passed to the GasViewMP program.

11.8. If light levels are between 2000 and 10,000, but r^2 is less than 0.10, no PIC value can be resolved; set PIC to a value one-half the MDL.

12. References

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12.3. Maxstream, Inc. 2005. XStream-PKG-R RS-232/485 RF Modem. Product Manual v4.2B. Maxstream, Inc., Lindon, UT.
12.4. Myers, J., T. Kelly, C. Lawrie, K. Riggs. 2000. Environmental Technology Verification Report: Boreal Laser Inc. GasFinder 2.0 Tunable Diode Laser Open-Path Monitor. ETV Advanced Monitoring Systems Center, Battelle Laboratories, Columbus, OH
12.5. USEPA. 1989. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV- Meteorological Measurements.
12.6. USEPA. 1999. Compendium Method TO-16: Long-Path Fourier Transform Infrared Monitoring Of Atmospheric Gases. Office of Research and Development, Washington, D.C. EPA/625/R-96/010b.
12.7. SOP D1. 2006. Management of Open path and Weather and Lagoon Characterization Data. Standard Operating Procedure D1, v. 0.0. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
12.8. SOP G2. 2006. Gas Cylinders Standard Operating Procedure G2, v. 0.0. Purdue Ag Air Quality Lab.
12.9. SOP G8. 2007. General calibration of Gas analyzers. Standard Operating Procedure G8, v. 1.0. Purdue Ag Air Quality Lab
12.10. SOP G11. 2007. Operation of the Environics Computerized Gas Dilution System. G11, v. 1.0. Purdue Ag Air Quality Lab.
12.11. SOP U4. 2008. Open Source Instrument Trailer. Standard Operating Procedure U4, v. 1.0. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
12.12. SOP U5. 2008. Installation of Open Source Measurements. Standard Operating Procedure U5, v. 1.0. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.

13. Contacts

13.1. TDLAS manufacturer: Boreal Laser, Inc., #13, 51127 RR 255, Spruce Grove, AB, Canada T7Y 1A8
  13.1.1. Phone: (780) 987-4382
  13.1.2. Fax: (780) 987-2418

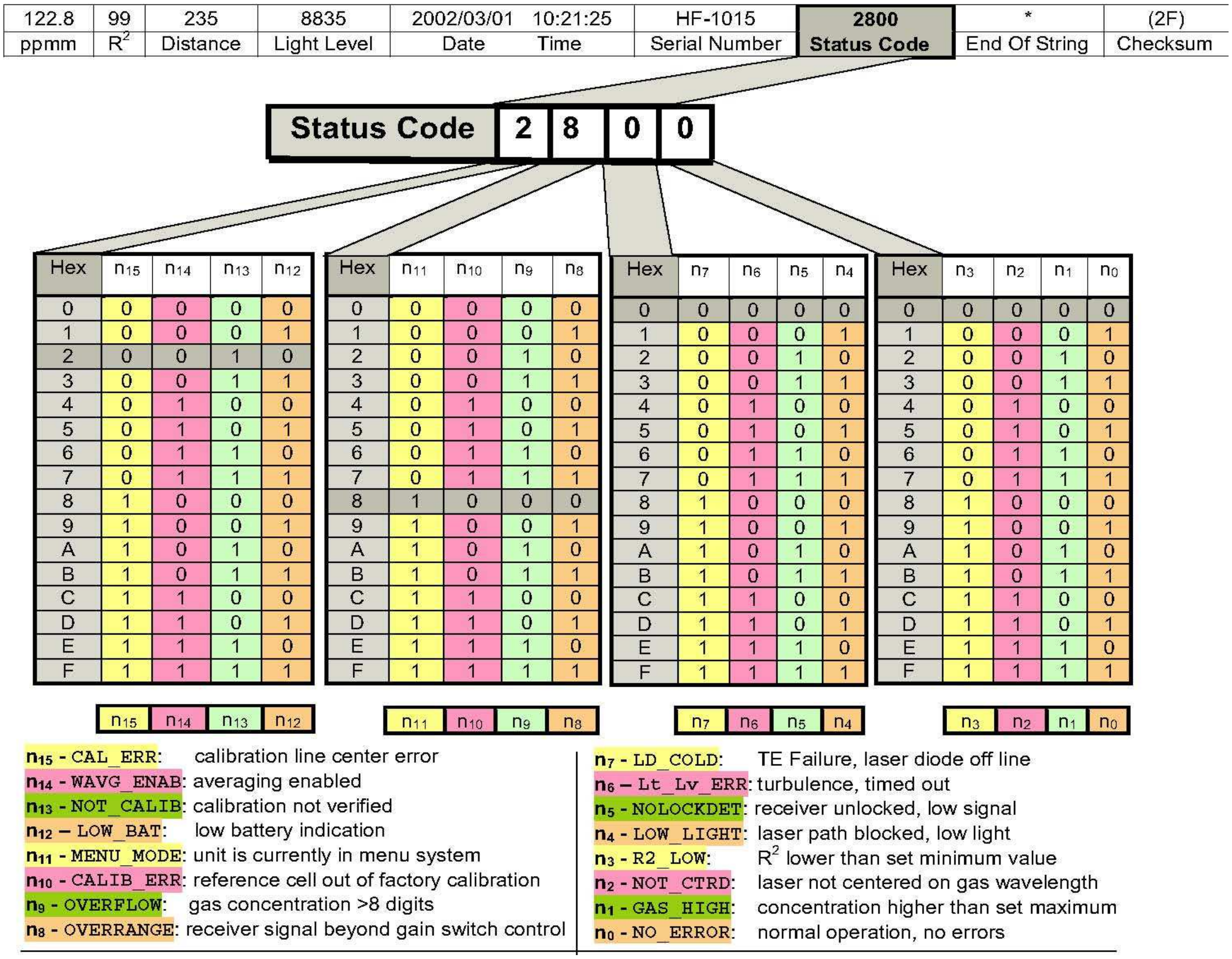
13.2. Modem manufacturer: Maxstream, Inc., 355 South 520 West, Suite 180, Lindon, UT 84042

- 13.2.1. Phone: (801) 765-9885
- 13.2.2. Fax: (801) 765-9895
- 13.2.3. email: rf-xperts@maxstream.net

Appendix A- GasFinder2.0 Status Codes

The GasFinder data stream output after the '$GFDTA,scanline#,' is as follows:

122.8,99,235,8835,2002/03/01 10:21:25,HF-1015,2800,*(2F)



122.8	99	235	8835	2002/03/01	10:21:25	HF-1015	2800	*	(2F)
ppmm	R^2	Distance	Light Level	Date	Time	Serial Number	**Status Code**	End Of String	Checksum

Status Code 2 8 0 0

Hex	n_{15}	n_{14}	n_{13}	n_{12}
0	0	0	0	0
1	0	0	0	1
2	0	0	1	0
3	0	0	1	1
4	0	1	0	0
5	0	1	0	1
6	0	1	1	0
7	0	1	1	1
8	1	0	0	0
9	1	0	0	1
A	1	0	1	0
B	1	0	1	1
C	1	1	0	0
D	1	1	0	1
E	1	1	1	0
F	1	1	1	1

Hex	n_{11}	n_{10}	n_9	n_8
0	0	0	0	0
1	0	0	0	1
2	0	0	1	0
3	0	0	1	1
4	0	1	0	0
5	0	1	0	1
6	0	1	1	0
7	0	1	1	1
8	1	0	0	0
9	1	0	0	1
A	1	0	1	0
B	1	0	1	1
C	1	1	0	0
D	1	1	0	1
E	1	1	1	0
F	1	1	1	1

Hex	n_7	n_6	n_5	n_4
0	0	0	0	0
1	0	0	0	1
2	0	0	1	0
3	0	0	1	1
4	0	1	0	0
5	0	1	0	1
6	0	1	1	0
7	0	1	1	1
8	1	0	0	0
9	1	0	0	1
A	1	0	1	0
B	1	0	1	1
C	1	1	0	0
D	1	1	0	1
E	1	1	1	0
F	1	1	1	1

Hex	n_3	n_2	n_1	n_0
0	0	0	0	0
1	0	0	0	1
2	0	0	1	0
3	0	0	1	1
4	0	1	0	0
5	0	1	0	1
6	0	1	1	0
7	0	1	1	1
8	1	0	0	0
9	1	0	0	1
A	1	0	1	0
B	1	0	1	1
C	1	1	0	0
D	1	1	0	1
E	1	1	1	0
F	1	1	1	1

n_{15} - CAL_ERR:	calibration line center error
n_{14} - WAVG_ENAB:	averaging enabled
n_{13} - NOT_CALIB:	calibration not verified
n_{12} – LOW_BAT:	low battery indication
n_{11} - MENU_MODE:	unit is currently in menu system
n_{10} - CALIB_ERR:	reference cell out of factory calibration
n_9 - OVERFLOW:	gas concentration >8 digits
n_8 - OVERRANGE:	receiver signal beyond gain switch control
n_7 - LD_COLD:	TE Failure, laser diode off line
n_6 – Lt_Lv_ERR:	turbulence, timed out
n_5 - NOLOCKDET:	receiver unlocked, low signal
n_4 - LOW_LIGHT:	laser path blocked, low light
n_3 - R2_LOW:	R^2 lower than set minimum value
n_2 - NOT_CTRD:	laser not centered on gas wavelength
n_1 - GAS_HIGH:	concentration higher than set maximum
n_0 - NO_ERROR:	normal operation, no errors

The above sample status code of "2800" corresponds to "0010_1000_0000_0000". This indicates an instrument status of: **n13 - NOT_CALIB, n11 - MENU_MODE**

The following is another example: Code "A42E" would correspond to "1010_0100_0010_1110" and indicate an instrument status of: **n15 -CAL_ERR, n13-NOT_CALIB, n10 -CALIB_ERR, n5 -NOLOCKDET, n3 - R2_LOW, n2 -NOT_CTRD, n1 -GAS_HIGH**

Under normal operating conditions, the most common status code is "1". This is actually 0001, but the software ignores the leading zeros and indicates a status code of "1". This corresponds to "0000_0000_0000_0001", and indicates an instrument status of: **n0 - NO_ERROR.**

Appendix B
TDLAS Single Point Calibration Verification Log

Trailer: (EAST/WEST/FIXED)
Site: _______________

TDLAS Serial number _____________ Operator________________________
Date (MM/DD/YYYY):______________________
Calibration verification filename ():________________________

Calibration cylinder# (_____________) concentration: _____________ppm
Diluted chamber calibration gas concentration: _______________ppm

Trail[1]	TEST	Valid measurement times[2]		TDLAS mean measurement			
		Start	End	Mean	SD	Bias	RSD
	Background						
1	Calibration gas						
	Background						
2	Calibration gas						
	Background						
3	Calibration gas						
	Background						
4	Calibration gas						
	Background						

1: Only one good trial is needed (comments needed if more that one is conducted)
2: Valid measurement times represent the time after 10 min of flow.

Comments:

OPERATION OF THE THERMO ELECTRON MODEL
146C MULTI-GAS DILUTER
Standard Operating Procedure (SOP) C3

OPERATION OF THE THERMO ELECTRON MODEL 146C MULTI-GAS DILUTER

Standard Operating Procedure (SOP) C3

Prepared by
Congna Li

Reviewed by
Bill W. Bogan and Richard H. Grant

Effective Date: November 6, 2006

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1. Scope and Applicability

1.1. Precise dilution of manufacturer-supplied gases is required during the calibration of the open-path monitoring instruments that are used to monitor emissions from confined animal feeding operations (such as corrals) and open sources (such as lagoons) at livestock facilities.

1.1.1. Typical dilution ratios needed to calibrate open-source monitoring instruments, such as UV-DOAS (SOP C1) and TDLAS (SOP C2) systems are approximately 100:1 to 1000:1.

1.2. This procedure describes the setup and operation of the Thermo Electron Corporation Model 146C Dynamic Gas Calibrator.

1.2.1. The Model 146C can handle zero air flows of 0.2-10 L/min, and span gas flows of 1.0-50 sccm.

1.2.2. The accuracy of each mass flow measurement is 2% of reading or 1% of full scale, whichever is less.

1.2.3. Linearity of mass flow measurement is 0.5% of full scale.

1.2.4. Repeatability of mass flow measurement is 2% of reading or 1% of full scale, whichever is less.

1.2.5. Permeation oven temperature stability is 0.1°C.

2. Summary of Method

The Thermo Environmental Model 146C Multigas Calibrator precisely dilutes calibration gases, which can then be used for precision and Level I span checks, audits, and multipoint calibration of various instrumentation. The Model 146C integrates mass flow controllers, an ozone generator, a permeation tube oven, power supplies, and solenoid valves into a single microprocessor-controlled unit, which can also be controlled remotely using a datalogger or computer. The calibrator uses either permeation tubes (optional add-on) or standard gases as calibration gas sources. The standard Model 146C hardware/plumbing configuration mixes gas and zero air flows. The basic unit can handle three gas standards, which are controlled by individual solenoids and regulated by mass flow controllers; the zero air controller is high-flow, while the gas flow controller is low-flow. The desired concentration is produced in a Teflon mixing chamber. When a permeation gas source is used in conjunction with the gas dilution system, permeation dilution concentration is calculated based on the permeation rate and the flow rate of zero air controlled by the gas dilution system. The permeation oven heats a permeation tube that supplies an internal source of calibration gas. A small amount of zero-air flows through the permeation tube oven and is mixed with the remainder of the zero-air flow in the mixing chamber. Accuracy is achieved through precise control of the release rate of the permeation tube gas and the flow of zero air through the zero-air mass flow controller, as well as maintenance of a stable permeation tube temperature. When only the permeation oven is activated, up to five gas concentration levels (ppm) can be set.

3. Definitions

3.1.	LED	Light-emitting diode
3.2.	MFC	Mass flow controller
3.3.	NIST	National Institute of Standards and Technology
3.4.	QAPP	Quality Assurance Project Plan
3.5.	sccm	Standard cubic centimeters per minute
3.6.	TDLAS	Tunable diode laser acoustic spectroscopy
3.7.	UV-DOAS	Ultraviolet differential optical absorption spectroscopy

4. Health and Safety

4.1. Be careful when connecting the precision standard and dilution gas cylinders to the diluter, especially in the case of toxic, reactive, or otherwise hazardous gases. See SOP G2 for more information on handling gas cylinders.
4.2. Avoid mixing gases that may produce an explosion or other hazardous reaction.
4.3. Be careful when making electrical connections, disconnecting and connecting connectors and making voltage measurements in the instrument as high voltage or current can injure or kill.
4.4. Disconnect power before servicing the unit.

5. Cautions

5.1. Verify that the scheduled calibration date is prior to the expiration date specified on the EPA protocol calibration gas cylinder. Do not use expired cylinders.
5.2. Leave the power on at all times when a permeation tube has been installed. The zero air-air supply should also be connected and active to allow a small flush flow across the permeation tube.
5.3. When determining the actual flow through the mass flow controller, use the LED reading, and not the value set on the potentiometer.

6. Interferences

6.1. Reactive compounds can be lost in this instrument. Follow the procedure to verify that reactive compounds are not lost in the gas dilution system, especially in Methods 15[*], 16, and, 25A and 25B (40 CFR part 60, appendix A) when using a gas other than propane.

7. Personnel Qualifications

7.1. Each operator must read and understand this SOP and SOP G2 before working with the instrument. Any operator using this instrument to calibrate the UV-DOAS and/or TDLAS instruments must also read and understand SOP C1 and/or C2, respectively.

7.2. Each operator must be trained in the use of the handheld multimeter, the barometer, water bath, and mass balance before initiating the procedure.

8. Equipment and Supplies

8.1. Model 146C Dynamic Gas Calibrator (Thermo Electron Corporation, Waltham, MA)
8.2. Permeation tube oven and permeation tube(s) containing appropriate gas(es) (optional)
8.3. Internal ozone generator (optional)
8.4. Teflon tubing
8.5. Zero air and span gas cylinders (EPA protocol standard, 1% certification)
8.6. Stainless steel dual-stage gas cylinder regulators as appropriate for the particular gas(es) (SOP G2)
8.7. Wrench to manipulate gas cylinder connections
8.8. NIST-traceable flow calibrator
8.9. NIST-traceable thermometer with a range of 0-45°C
8.10. Water bath (heatable to 45°C)
8.11. Barometric pressure sensor
8.12. Balance with 0.01 mg accuracy
8.13. Ambient air temperature sensor
8.14. 4.369-KΩ Resistor or resistance box capable of providing 4.369 KΩ
8.15. Wire leads with clips to connect to resistance box
8.16. Multimeter

9. Procedure

9.1. Instrument Setup
  9.1.1. Connect a source of zero air to the inlet port labeled “ZERO AIR” (Fig. 1).
  9.1.2. The zero air source must be capable of supplying the full-scale flow required by the zero air controller (up to 10 L/min) at a pressure between 10 and 40 psi.
  9.1.3. Connect the standard gas cylinders to the ports labeled A, B and C (Fig. 1).

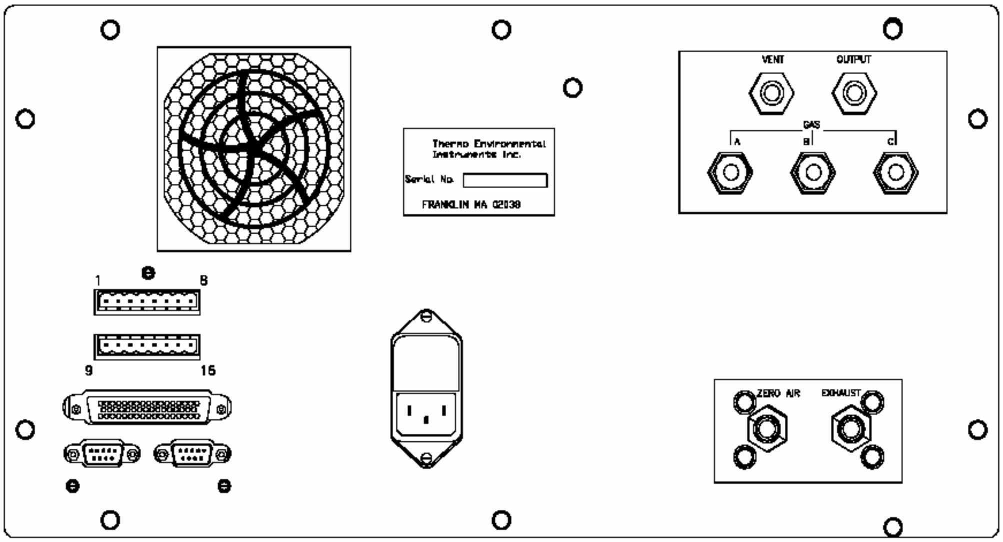


Figure 1. Model 146C Rear Panel.

9.1.4. Install the permeation tube using the following procedure (applicable only if the permeation option is installed):

9.1.4.1. Remove the instrument cover and locate the permeation oven (Fig. 2).

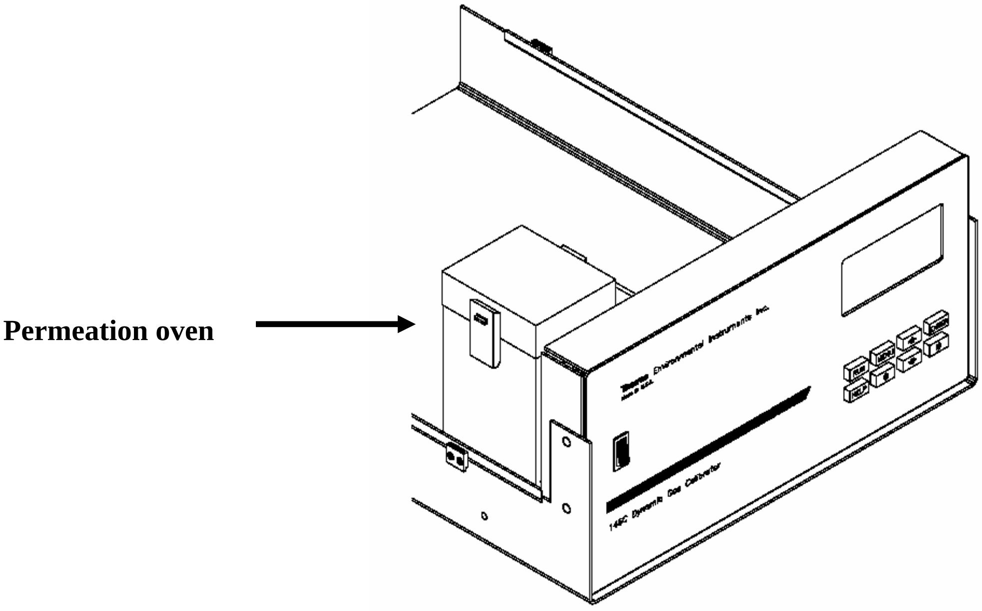


Figure 2. Removing cover of the TEI 146C to access the permeation oven.

9.1.4.2. Release the latches on the sides of the oven cover, and remove the cover.
9.1.4.3. Remove glass chamber assembly by loosening (not removing) knurled screw, located at the top of the chamber, and gently pulling assembly upward. Completely remove assembly from oven (Fig. 3).
9.1.4.4. Separate the glass chamber from the top assembly by twisting and gently pulling glass away from top. Keep glass clean by using Kimwipes or similar material to handle glass.
9.1.4.5. Place permeation tube in chamber.
9.1.4.6. Attach glass chamber to top assembly by gently pushing together with a slight twisting motion.
9.1.4.7. Replace glass chamber assembly into oven until top of assembly is flush or slightly below oven top (Fig. 4).
9.1.4.8. Tighten knurled screw with finger. Do not use tools to tighten.
9.1.4.9. Replace oven cover, being careful to place tubing and wire in slot of cover.

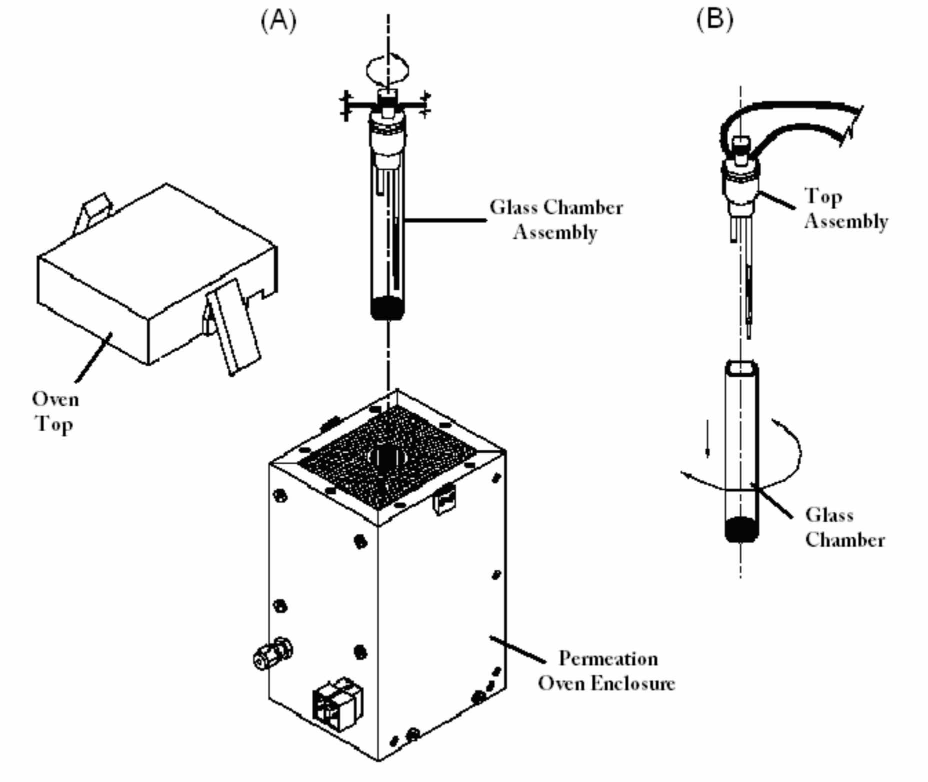


Figure 3. Removing glass chamber from permeation oven (A) and removing top assembly (B).

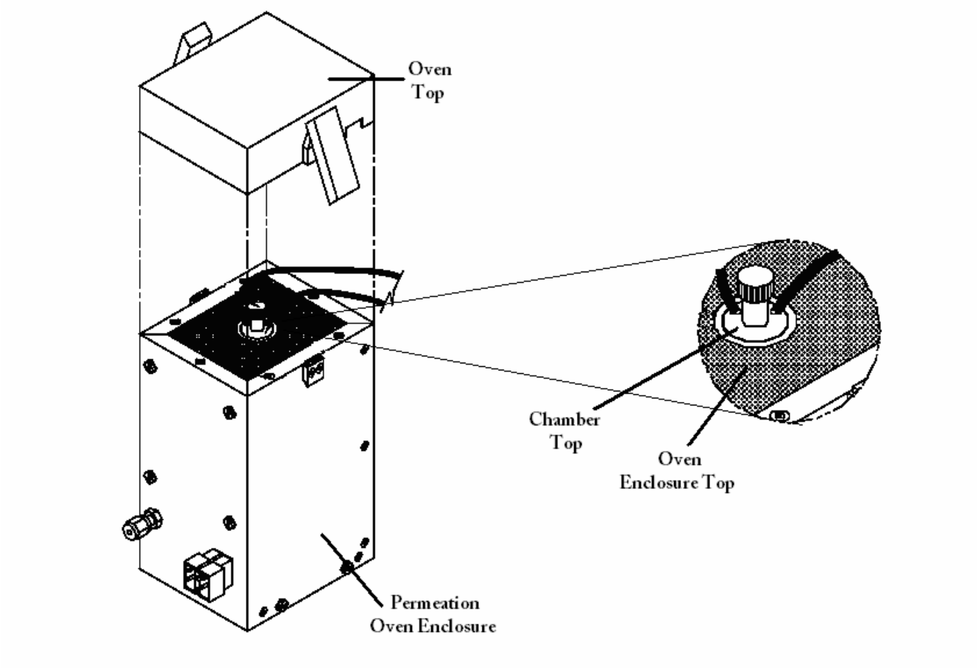


Figure 4. Replacing glass chamber. Inset shows chamber top flush with oven enclosure top.

9.2. Operation

9.2.1. Gas dilution using a gas cylinder

9.2.1.1. Press the “RUN” pushbutton, choose “Run Screen 1”, press the “MENU” pushbutton, select the “Local” mode using the ↑ and ↓ pushbuttons, and press “ENTER”. Press “MENU” to return to the Main menu.

9.2.1.2. From the “Main Menu”, move to Gas A, B, or C by using the ↑ and ↓ pushbuttons. Press “ENTER” to select.

9.2.1.3. Select “Name”, move the underscore to select a character, using the ← and → pushbuttons. Select a new character using the ↑ and ↓ pushbuttons. Press the “ENTER” pushbutton to save the selection.

9.2.1.4. Select “Tank Conc”, enter the gas concentration using the ← and → and the ↑ and ↓ pushbuttons. Press “ENTER” to save the selection.

9.2.1.5. Select “Span 0 Flow”, enter the value of the zero-air flow using the ← and → and the ↑ and ↓ pushbuttons. Press “ENTER” to save the selection.

9.2.1.6. Use the ← and → pushbuttons to select the desired span level (Span 1 to 5, or manual).

9.2.1.7. Select “Span”, select the span gas concentration and total flow rate (sum of zero-air and span gas flow) using the ↑ and ↓ pushbuttons. Press “ENTER” to switch to the corresponding screen.

9.2.1.8. Under the “Span” menu, select “Conc PPM”, enter the value of the diluted gas concentration using the ← and → and the ↑ and ↓ pushbuttons. Press “ENTER” to save your selection.

9.2.1.9. Under the “Span” menu, select “Span Flow”, enter the value of the span gas flow rate using the ← and → and the ↑ and ↓ pushbuttons. Press “ENTER” to save your selection.

9.2.1.10. To manually set the zero-air and gas flows, select “Manual” after selecting Gas A, B, or C, use the ↑ and ↓ pushbuttons to select zero-air and gas flow. Press “ENTER”. Then select “ZERO AIR SCCM” to display and modify the zero air flow setting manually, or select “Gas SCCM” to display and modify the span gas flow setting manually using the ← and → and the ↑ and ↓ pushbuttons. Press “MENU” to return to the upper menu or press “ALT” to return to the “Run” screen.

9.2.1.11. Computation of diluted gas concentrations

9.2.1.11.1.

$$\text{Model 146C output (ppm)} = \frac{F_{gas}}{F_{gas} + F_{zero\ air}} \times C_{cyl}$$

Where:
F_{gas} = Flow measured for cylinder gas, sccm.
$F_{zero\ air}$ = Flow measured for zero air, sccm.
C_{cyl} = Concentration of gas in cylinder, ppm

9.2.2. Gas dilution using the permeation tube oven

9.2.2.1. Allow 24 h for permeation tube temperature to stabilize after turning the unit on. If a permeation tube is purchased with a certified rate, R2, at a given temperature, T2, it is possible to calculate the approximate temperature T1 that is needed to achieve permeation rate R1, by using the following equation:

9.2.2.1.1.

9.2.2.1.2. $\text{Log } R1=\log R2-2950(1/T1-1/T2)$

Where:
R_1 = Permeation rate in ng/min at T_1,
R_2 = Permeation rate in ng/min at T_2.

9.2.2.2. From Run Screen 1, use the ↑ and ↓ pushbuttons to scroll to the ozone/perm line.

9.2.2.3. Use the ← and → pushbuttons to select the desired permeation level (Perm Levels 1 through 5). Press “ENTER”.

9.2.2.4. Set the flow of zero air in the manual settings screen of the flow controls menu.

9.2.2.5. Allow 24 h for the permeation tube temperature to stabilize.

9.2.2.6. Calculate the permeation rate, R, following Section 9.3.2.3.

9.2.2.7. Computation of diluted permeation gas concentrations

9.2.2.7.1. Model 146C output (ppm) = RK/Q_0

Where:

R = Permeation rate, ng/min
Q_o = Zero air flow rate, sccm
K = Constant for specific permeant = $24.45/MW$
MW = Molecular weight of specific permeant

9.3. Calibration

9.3.1. Calibration of mass flow controllers

9.3.1.1. Connect a source of clean, dry air to the inlet of the mass flow controller.

9.3.1.2. Measure barometric pressure and room temperature.

9.3.1.3. Connect a suitable flow meter (NIST-traceable calibrator) to the mass flow controller outlet.

9.3.1.4. Set the Model 146C to “Gas Drive” or “Zero Air Flow Calibration”.

9.3.1.5. Enter the flow meter reading using the flow input screen.

9.3.1.6. Repeat the above two steps for the remaining flow settings.

9.3.1.7. If difficulty is encountered due to a malfunction of the flow controller, contact Thermo Environmental Instruments.

9.3.2. Calibration of permeation tube oven

9.3.2.1. Setting water bath to the desired temperature

9.3.2.1.1. Unplug the connector at J3, from the Oven Controller Board (Fig. 5). Place a 4.369-KΩ resistor across pins 3 and 4 of J3 on the Oven Controller Board.

9.3.2.1.2. From the Permeation Oven Menu, select Cal Oven Thermistor. Adjust R2 on the Oven Controller Board until the oven drive voltage is 5.000 volts. Press MENU to return to the Permeation Oven Menu.

9.3.2.1.3. Remove the thermistor from the permeation tube oven. Leave the thermistor connected to the Oven Controller Board. Insert the thermistor into a water bath next to the NIST-traceable thermometer.

9.3.2.1.4. Turn on the power to the water bath. Using a NIST-traceable thermometer with a resolution of ±0.01°C, adjust water bath to 45.00±0.02°C.

9.3.2.1.5. From the Permeation Oven Menu, select Cal Gas Thermistor. Adjust R4 on the Oven Controller Board until the permeation gas temperature reading is 45.00°C.

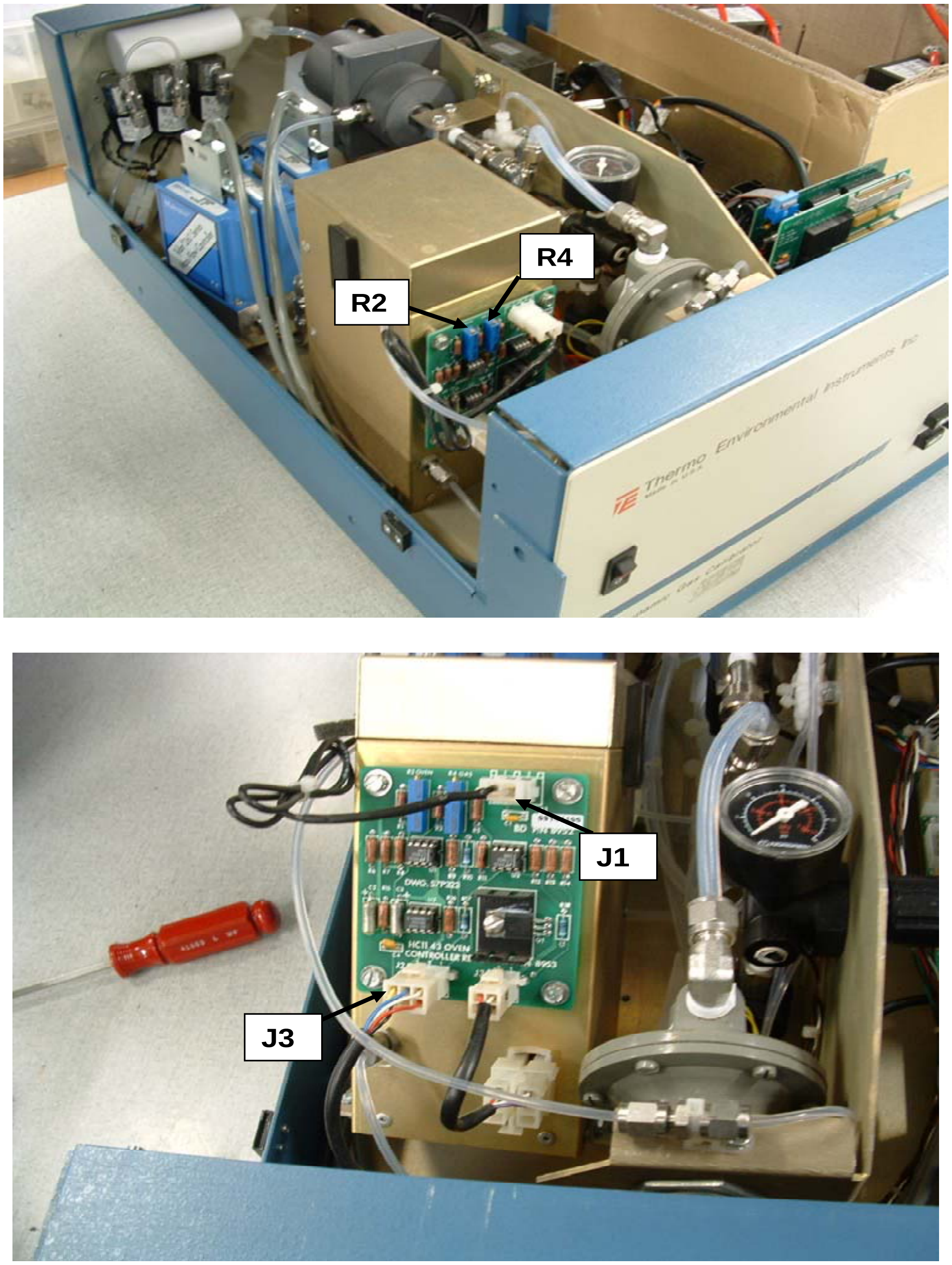
9.3.2.1.6. Remove thermistor from the water bath, dry, and replace into the permeation tube oven.

9.3.2.1.7. Wait for the permeation gas temperature reading to stabilize.

9.3.2.1.8. From the Permeation Oven Menu, select Set Gas Temperature. Adjust R2 until the Perm Gas reading displayed on the first line is 45.00°C. Wait at least 10 min between adjustments.

9.3.2.2. Setting measure temperature with accurately known oven temperature (as an alternative approach to that described in Section 9.3.2.1)

9.3.2.2.1. Unplug the connector at J3, from the Oven Controller Board. Place a 4.369 KΩ resistor across pins 3 and 4 of J3 on the Oven Controller Board.



J3 Pin Layout J1 Pin Layout

Figure 5. The permeation oven controller board, showing the board's location, the locations of R2, R4, J1 and J3 on the board, and the pin layouts of J1 and J3.

9.3.2.2.2. From the Permeation Oven menu, select Cal Oven Thermistor. Adjust R2 on the Oven Controller Board until the oven drive voltage is 5.000 volts. Press MENU to return to the Permeation Oven menu.

9.3.2.2.3. Unplug the thermistor from J1 on the Oven Controller Board.

9.3.2.2.4. Connect a resistance of 4.369 KΩ across pins 1 and 2 of J1.

9.3.2.2.5. From the Permeation Oven menu, select Cal Gas Thermistor. Adjust R4 on the Oven Controller Board until the permeation gas temperature reading is 45°C.

9.3.2.2.6. Reconnect the thermistor to J1.

9.3.2.2.7. Wait for the permeation gas temperature reading to stabilize.

9.3.2.2.8. From the permeation oven menu, select Set Gas Temperature. Adjust R2 until the Perm Gas reading displayed on the first line is 45.00°C. Since it takes several minutes for the permeation oven temperature to stabilize, wait 10 min between adjustments.

9.3.2.3. Determination of permeation rate by weight loss

9.3.2.3.1. Make sure the oven has been calibrated as described above.

9.3.2.3.2. Gently insert the permeation tube using clean tweezers or similar tool. Never touch the tube with your fingers.

9.3.2.3.3. Turn on the Model 146C.

9.3.2.3.4. Wait 48 to 72 hours for the permeation tube temperature to stabilize.

9.3.2.3.5. Carefully remove the permeation tube from the oven and weigh permeation tube to nearest 1 mg. Perform this measurement as quickly as possible.

9.3.2.3.6. Return permeation tube to the oven.

9.3.2.3.7. Allow permeation tube to permeate at constant temperature with gas flow over tube for a period sufficient to generate a measurable weight loss (usually a minimum of 72 h).

9.3.2.3.8. Reweigh permeation tube to nearest 1 mg.

9.3.2.3.9. Compute the permeation rate, R, in terms of ng/min as:

$$R = (\text{Initial weight} - \text{Final weight})/\text{Time}.$$

9.3.2.3.10. Use the permeation tube in the same oven that was used to determine the permeation tube's weight loss.

9.3.2.4. Determination of release rate by use of transfer standard

9.3.2.4.1. To perform this procedure, one Model 43C and two Model 146Cs are needed.

9.3.2.4.2. Ensure that the ovens in both of the Model 146Cs have been calibrated.

9.3.2.4.3. Determine permeation rate for permeation tube in Model 146C being used as calibration standard, or install a certified permeation tube.

9.3.2.4.4. Allow the permeation tubes in both Model 146Cs to stabilize at least 48 h.

9.3.2.4.5. Carefully calibrate an analyzer, such as a Thermo Environmental Model 43C, using the Model 146C with the calibrated permeation tube. Follow the instruction for calibration in the 43C instrument manual.

9.3.2.4.6. Connect calibrated analyzer to Model 146C with permeation tube whose permeation rate is to be determined.

9.3.2.4.7. Adjust flow of the Model 146C so that the analyzer reads close to full scale. Note flow and measured concentration.

9.3.2.4.8. Compute permeant concentration from the following equation:

$$\text{Model 146C output (ppm)} = \frac{R \times K}{Q_o}$$

Where:

R = Permeation rate, ng/min

Q_o = Flow rate, sccm

K = Constant for specific permeant $= \frac{24.45}{MW}$

MW = Molecular weight of specific permeant

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this instrument. Supplement this electronic record by a record book designated for this instrument, which should contain copies of the electronic record.

10.2. Manage all data according to SOP B5.

10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all such corrections.

10.4. A copy of the manufacturer' instructions for the operation of the instrument, as well as the most recent calibration documentation shall be made available for the project auditor's inspection upon request.

10.5. A label (Fig. 6) shall be affixed at all times to the gas dilution system listing the date of the most recent calibration, the due date for the next calibration, and the person or manufacturer who carried out the calibration.

Calibration date:__________________
Next calibration due date:__________
Calibrated by:____________________

Figure 6. Sample calibration label.

11. Quality Control and Quality Assurance

11.1. Check and recalibrate the mass flow controller, using a NIST-traceable flow calibrator, whenever the target and actual flow (within the range of 20 to 100% of full scale) deviates greater than ±2% of reading or 1% of full scale, whichever is less. The QAPP for the particular project may have additional requirements.

11.2. Calibrate the permeation tube oven whenever the temperature indicator deviates by 0.2°C (an error of about 0.1°C corresponds to an error of 1% in release rate).

11.3. Calibrate the flow calibrator, thermometer, water bath, barometric pressure sensor, balance and ambient air temperature sensor at the initiation of the study and annually thereafter, unless the QAPP specifies otherwise.

12. References

12.1. Thermo Electron Corporation. 2004. Model 146C Dynamic Gas Calibrator, Operating Manual.
12.2. U.S. Code of Federal Regulations, US 40 CFR 51 Appendix M, Method 205—Verification of Gas Dilution Systems for Field Instrument Calibrations. Available at: http://ecfr.gpoaccess.gov/cgi/t/text/text-idx?c=ecfr&sid=47660cf35a767ae7d9a8284226be43a7&rgn=div9&view=text&node=40:2.0.1.1.2.20.11.20.24&idno=40. Accessed 10 January 2006.
12.3. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
12.4. SOP C1. 2006. Open Path Measurement of Ammonia and Hydrogen Sulfide with the Cerex UV Sentry Ultraviolet Differential Optical Absorption Spectrometer. Standard Operating Procedure C1. Purdue Applied Meteorology Lab.
12.5. SOP C2. 2006. Measurement of Ammonia with the Boreal Laser GasFinder Tunable Diode Laser Absorption Spectrometer (TDLAS). Standard Operating Procedure C2. Purdue Applied Meteorology Lab.
12.6. SOP G2. 2006. Standard Operating Procedure for Compressed Gas Cylinders G2. Purdue Ag Air Quality Lab.
12.7. SOP G5. 2006. Measurement of Hydrogen Sulfide (H_2S) with the Thermo Electron Corporation Model 45C Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab.

SYNTHETIC OPEN-PATH SAMPLING SYSTEM
Standard Operating Procedure (SOP) C4

SYNTHETIC OPEN-PATH SAMPLING SYSTEM

Standard Operating Procedure (SOP) C4

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1. Scope and Applicability

1.1. This method is used for continuous real-time sampling of airborne gases, for example, those downwind of an open source area, and provides a path-integrated concentration, comparable to open-path techniques.

1.1.1. This method allows the use of analyzers that can measure gases not measurable by standard open-path techniques, e.g. TDLAS (SOP C2).

1.1.2. Any gas for which a suitable detector can be configured for real-time, unattended operation can be measured with this method.

1.1.3. Any gas for which adsorption/desorption in tubing does not prohibit real-time measurements given the sampling intervals of the technique can be measured with this method.

1.2. The maximum practical length of the path that can be sampled with this method is approximately 1000 ft (330 m).

1.3. The sensitivity of the measurement does not depend on path length.

1.4. The desired residence time for an air sample in the tube is approximately 20% of the sampling period. The flow rate of the sampling pump should be adjusted to achieve this.

1.5. This procedure applies only to single-tube sampling lines, with multiple inlets spaced along their length to sample across a plume.

1.6. The GSS draws air through long sampling lines from one or more sampling points, and provides a mixed sample for analysis by gas analyzer(s) or auxiliary manual (bags, sorbent tubes, impingers, and canisters) collection. Delivery of calibration gases to the analyzer(s) is also facilitated with the GSS.

1.7. The minimum detection limit (MDL) for this method is determined by the detection limit of the detector being used.

1.7.1. This method does not cover the analyzer or detector. Consult the relevant SOP for the detector in question for that information.

1.8. Data generated using this method is analyzed using the Backward Lagrangian Stochastic (bLs) method (SOP O1) or a ratiometric method referenced to a path-integrated concentration measurement.

2. Summary of Method

2.1. The synthetic open-path method consists of a long section of tube (Teflon or other suitable material) that samples along a line intersecting a plume resulting from gases emanating from a contaminant source. Multiple air inlets are spaced at equal distances along the tube to draw air samples from several discrete locations within the plume. The inlet flow rates through the air inlets are adjusted using flow restricting orifices and measured using a mass flow meter to ensure equal flows and avoid biased sampling of the plume. The tube functions as a combining manifold, blending the incoming sample streams as they are drawn through the tube by the sampling pump. Suitable flow rates are calculated based on a single diameter tubing and the assumption that the outlet of the system will be located at one extreme end of the main tube. The maximum inlet air residence time and the variance of inlet air residence times in the system can be reduced by using multiple tube diameters in the main line. After passing through the tube, the blended air sample is subsampled in the GSS for the target gas(es). The GSS and analyzers are generally contained in a suitable instrument shelter at the site to allow for long-term, continuous, unattended operation.

2.2. The GSS was developed by the Purdue Agricultural Air Quality Laboratory (PAAQL) in 2006, and supersedes the GSS described by Heber et al. (2006, in press). It has sampling and bypass manifolds that are arranged in a parallel configuration on a sloped mounting board (Fig. 4). Compared to the previous design, it has better gas leakage/blockage prevention, lower gas adsorption/desorption time, and more convenient operation and maintenance. A schematic diagram of the GSS, as it is integrated with the other components at a site (gas analyzers, DAC system) is presented in Fig. 5.

2.3. A sampling pump draws air from a S-OPS at 4-7 L/min, while a bypass pump composites all unsampled S-OPS lines and purges them simultaneously with a total flow of about 10 L/min. The sample air is continuously delivered to a slightly positive-pressure analyzer manifold, from which each gas analyzer draws continuous subsamples. The sample air is optionally delivered to a manually operated valve for collecting samples for offline analysis.

2.4. A data acquisition and control (DAC) system controls the automatic or manual sequential gas sampling through an array of independently-controlled 3-way solenoid valves in the GSS. Typically, air from only one sampling location at a time is provided to the sampling and analyzer manifolds. The GSS has built-in continuous sensors that measure pressure (positive or negative) in the sampling manifold and the flow rate of the sampling air. Sensor data is recorded by the DAC system.

2.5. This GSS is enclosed and is heated by thermal energy dissipated by the pumps and solenoids. The increased GSS temperature reduces adsorption/desorption of gases and helps to prevent condensation.

3. Definitions

3.1.	AI	Analog input
3.2.	bLs	Backward Lagrangian stochastic
3.3.	DAC	Data acquisition and control
3.4.	DAQ	Data acquisition
3.5.	DO	Digital output
3.6.	GSS	Gas sampling system
3.7.	TDLAS	Tunable diode laser absorption spectrometry
3.8.	M_a	Analyzer manifold
3.9.	M_b	Bypass manifold
3.10.	M_c	Calibration manifold
3.11.	MDL	Minimum detection limit
3.12.	M_s	Sampling manifold
3.13.	NC	Normally closed (air normally directed to bypass manifold)
3.14.	NO	Normally open (air normally directed to sampling manifold).
3.15.	PIC	Path-integrated concentration
3.16.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.17.	PAML	Purdue Applied Meteorology Laboratory

3.18.	PC	Personal computer
3.19.	PM	Particulate matter
3.20.	QAPP	Quality Assurance Project Plan
3.21.	SMP	Site Monitoring Plan
3.22.	S-OPS	Synthetic Open Path System

4. Health & Safety

4.1. Check with the producer to make sure the area is free of underground wires, pipes, etc before digging post holes or driving the support stakes for the sampling line.

4.2. The GSS has two 120-VAC vacuum pumps. Follow electrical safety rules when working on the pumps.

4.3. Air sampled from the open-area sources may contain air pollutants with high concentrations that could cause negative health effects. Properly exhaust excess sample air to the outside of the trailer, and keep the trailer well ventilated.

5. Cautions

5.1. Plugging of individual inlets by ice, dust, insects, or other debris will lead to biased sampling of the plume, and may produce inaccurate results. Install suitable in-line filters to avoid plugging and monitor inlet flow using a mass-flow meter.

5.2. Water in the sampling line can trap water-soluble gases in the sample stream, and lead to underestimates of these. The filter at the inlet has a large aperture and is pointed with its opening downward to shed water away from the inlet (Fig. 1).

5.3. Minimize exposure of the sampling line to external contamination (water, dust or other material) during the installation process by sealing each end of the tube, and the open ends of the tees that will connect to the inlets, properly with electrical tape (or equivalent). Remove the seal only when the inlet filters are in place, and the line is ready to be connected to the sampling pump.

5.4. Place the sampling line in an area where it will not interfere with animal, vehicle, or foot traffic.

5.5. Flow rates that are too high for the tube used in the sampling line may cause the tube to collapse. Consult the manufacturer’s specifications on any tube before setting the sampling flow rate.

5.6. The sampling line should be kept straight. Use multiple supports and anchors along the distance of the line.

5.7. Handle the GSS with care to avoid dropping it to the floor.

5.8. When transporting the GSS or storing it for a long time, seal all the openings of the solenoid valves and tubes to avoid contamination.

5.9. Only hand-tighten fittings inside the GSS to avoid over tightening them.

5.10. Each compression fitting inside the GSS has a gripper (seal) and a ferrule. Use these correctly to avoid air leakage.

5.11. Use a filter (Section 8.16) with about 1 mm x 1 mm mesh holes at the end of the GSS exhaust to prevent flies from entering the exhaust tubes. Make sure that the filter does not create excessive (>10 Pa) pressure drop in the exhaust.
5.12. Verify all power and signal connections and make sure that they are correct before using the GSS.
5.13. A 47-mm Teflon filter does not always stay flat when taking it out of the box. When inserting filters into the filter holders, carefully check the filter and make sure that it does not leave part of the surface area uncovered before tightening the holder. Always tighten the filter holder with the special filter holder wrenches.

6. Interferences

6.1. Off-gassing of the sampling tube could introduce interferents into the gas stream, depending on the target gas(es), the tube material, and the detection method. If off-gassing does occur from the sampling line, the magnitude of this interference will increase with longer residence times and temperature.
6.2. Any obstruction between the source and the sampling line (e.g. trees or bushes, uneven ground) can interfere with air flow, and cause biased sampling. Make sure no inlets are located directly downwind of any significant obstruction.
    6.2.1. Do not position the instrument shelter between the source and the sampling line, or within 4 m downwind from the sampling line.
6.3. Narrow “hotspots” within a plume could potentially pass between sampling inlets, and not be drawn into the line. This should be minimized over time due to fluctuations in wind direction making it unlikely that even a narrow hotspot would always pass entirely between the inlets. This problem is minimized with more inlets into the sampling line.
    6.3.1. If the source is significantly heterogeneous and hot spots within the plume are a concern, inlets should not be spaced further apart than 10% of the total path length.
6.4. Any specific interferences inherent to the detector that is used to quantify the gas(es) in the sample must also be addressed. Consult the SOP and instrument manual for the particular detector in question for this information (e.g. SOP G5 for hydrogen sulfide).
6.5. Air leaks into the GSS can dilute the sample gas. Maintain the GSS and perform leak checks when suspicions arise. Correct all problems discovered during leak checks.
6.6. Condensation in sampling air will not only introduce measurement error, but also may damage the analyzers. Stop the GSS if condensation is found in the tubes that provide sampling air to the GSS until the problem is solved.
6.7. Particulate matter in the sample can cause malfunctions of the solenoids and the mass flow meter. Verify proper installation of the in-line Teflon filter in all sampling inlets.
6.8. Improper conditioning of the Tedlar bags for the bag test (Section 9.19) can cause inaccurate results. Strictly follow the procedures in Section 9.19.4 to condition each bag,

7. Personnel Qualifications

7.1. Field staff members must read and understand this SOP, the SOP for any detector(s) used with the sampling system, and any additional documentation required by those SOPs, before performing this method.

7.2. The operator should be trained to change air filters, to check sensors (relative humidity, temperature, and pressure), and to perform leak checks.

8. Equipment and Supplies

8.1. 1500′ of 3/8″ OD Teflon sampling tubing
8.2. (2) 3/8″ Stainless steel unions
8.3. 3/8″ OD Teflon sampling tube, with 3/8″ tees for connecting inlets
8.4. Inlet tubings (Fig. 3)
8.5. 1/8″ OD Teflon tubing for making flow-restricting orifices for each inlet
8.6. Miniature Bunsen burner
8.7. 3/8″ 25-mm inlet filter holders for each inlet
8.8. 3/8″ – 1/4″ 47-mm in-line filter holders for each S-OPS
8.9. Storage/feeder device for extension tubing
8.10. #3 Rubber stoppers for plugging each sampling inlet
8.11. Gas sampling #3 rubber stopper with tubing/connector
8.12. 1.5-m high metal fenceposts. Enough fenceposts are needed to place one every 5 m (or less) along the sampling line.
8.13. Small sledge hammer to drive stakes.
8.14. Cable ties
8.15. ‘Reverse flow’ sampling pump
8.16. Teflon filters (1-µm pore size and 47-mm diameter) used in the in-line filter holders
8.17. Teflon filters (1-µm pore size and 25-mm diameter) used in the inlet filter holders
8.18. Electrical tape to seal the ends of the tubes to prevent their contamination during maintenance, testing, transportation or storage
8.19. Gas Sampling System
8.20. 0-10 L/min rotameter with 1/4″ connector (termed 10 L/min rotameter)
8.21. 100-1000 cc/min rotameter with 1/4″ connector (termed 1 L/min rotameter)
8.22. Approximately 10′ of Teflon tube (1/4″ OD x 1/8″ ID) for connecting the GSS to the 47-mm Teflon filters (Fig. 3)
8.23. Approximately 15′ of PVC tube (3/16″ ID) for connecting bypass exhaust air from the GSS to outdoors
8.24. Approximately 15′ of PVC tube (5/8″ ID) for connecting excess sample air from the GSS to outdoors
8.25. Digital signal cable (16-conductor 22-gage shielded or unshielded cable) for connecting the solenoid control receptacle in the GSS to the DO channels in the DAC hardware.
8.26. Analog signal cable (14-conductor 22-gage shielded cable) for connecting the sensor power/signal receptacle in the GSS to the AI channels in the DAC hardware.
8.27. Standard filter holders (2″ dia), with discs of air conditioner filter foam (“NaturalAire” filters, or equivalent) to keep flies out of sampling lines
8.28. Multimeter to check electrical connections
8.29. Tedlar bags for the bag test. The size of the Tedlar bag depends on the sampling flow rate. An 80-L Tedlar bag will work in most all cases.
8.30. DAC system (Campbell Scientific CR800 data logger)
8.31. Software programs for CR800 Data logger

8.31.1. Program for system testing ‘NAEMS_GSS-test.CR8’
8.31.2. Program for system operation ‘NAEMS_GSS.CR8’
8.32. Relay controller for solenoid control (Campbell Scientific A21Rel-12).
8.33. 24-VDC power supply for solenoid control (from TEC 450I Pulsed Florescence H_2S Analyzer).
8.34. 12-VDC power supply for DAC (Campbell Scientific CR800 and A21Rel-12).

9. Procedures

9.1. Consult applicable SMP to determine the location(s) for the sampling line(s), and for the posts and supports.
9.1.1. Check with landowner to be sure that posts/ support stake locations are free of underground wires, pipes, etc.
9.2. Setup of support stakes and synthetic open path sampling line (in field)
9.2.1. Cable and support stakes or posts
9.2.2. Supports
9.2.2.1. Using the cable as a guide to make sure all supports are in a line, drive support stakes every 5 m or less along the path of the sampling line
9.2.2.2. Determine coordinates of start and end of S-OPS using GPS
9.2.2.3. Record on “Open-Path Sensor Installation Report” (Appendix F).
9.2.3. Sampling line
9.2.3.1. Use a suitable feeder device to protect and guide the tubing from the reel, while laying it along the ground next to the fence posts, beginning with the closest one to the trailer.
9.2.3.1.1. Uncoil the tubing correctly when there is no feeder device available. Turn the coil while feeding the tubing. Do not pull the tubing from the coil when the coil lays flat on the ground.
9.2.3.2. If necessary, turn the tube so that the openings of the tees (taped) are facing upward
9.2.3.3. Insert the sampling tubes (Fig. 2) with the attached inlets and filter holders (and taped rubber stopper plugs) into the corresponding tees (the sampling tubes are numbered 1-10; 1 is closest to extension tubing connector). Remove tape covering on tee connections
9.2.3.4. Hand-tighten connections as tight as possible. Do not use wrench to tighten.
9.2.3.5. Using 4″ cable ties, secure the sampling tubes and inlets to the fence posts with the 180-degree bend in the tube pointing the inlet downwards (Fig. 1).
9.2.3.6. Keep all tees plugged with taped rubber stoppers while placing the tube.
9.3. Setup of sample line extension tubing (in field)
9.3.1.1. Use a suitable feeder device to protect and guide the tubing from the reel, while laying it along the ground outside the trailer to the end of the sampling line closest to the trailer. Leave at least 6 m at trailer end for feed-through into trailer and attachment to GSS.
9.3.1.1.1. Uncoil the tubing correctly when there is no feeder device available. Turn the coil while feeding the tubing. Do not pull the tubing from the coil when the coil lays flat on the ground.

            - 9.3.1.2. Two sections of 500′ extension tubing are needed for the most distant synthetic open-path sampling line. One section of 500′ extension tubing is needed for the near synthetic open-path sampling line.
                - 9.3.1.2.1. Connect extension tubing sections with stainless steel union connectors (Fig. 2) using wrenches.
                - 9.3.1.2.2. Connect the synthetic open-path sampling line with the extension tubing with stainless steel connectors (Fig. 2) using wrenches.
            - 9.3.1.3. Connect extension tubing sections to the GSS with Teflon connectors according to Fig. 2. Hand-tighten as tight as possible. Do not use wrench to tighten.
            - 9.3.1.4. Attachment of extension tubing to GSS with Teflon connector
                - 9.3.1.4.1. Feed extension tubing through tubing pass-thru in trailer floor
                - 9.3.1.4.2. Connect 500′ (corresponding to nearest S-OPS) S-OPS extension to 47-mm filter holder inlet feeding solenoid 2 (normally open).
                - 9.3.1.4.3. Connect 1000′ (corresponding to farthest S-OPS) S-OPS extension to 47-mm filter holder inlet feeding solenoid 3 (normally closed).
                - 9.3.1.4.4. Hand-tighten all connections as tight as possible. Do not use wrench to tighten..

- 9.4. Setup of S-OPS and GSS (in field)
    - 9.4.1. Connect each S-OPS to the S-OPS extension tubing with a Stainless steel union connector
        - 9.4.1.1. Connect nearest S-OPS (S-OPS #1) to 500′ S-OPS extension tubing.
        - 9.4.1.2. Connect farthest S-OPS (S-OPS #2) to 1000′ S-OPS extension tubing.
    - 9.4.2. Connect a piece of 3/16″ ID PVC tube to the bypass exhaust (Fig. 7), and lead the other end of the tube to outside the trailer trough tubing pass-thru on floor.
    - 9.4.3. Insert a piece of 5/8″ ID PVC tube through the sample exhaust hole (Fig. 7) and connect it to the barbed fitting on the top of the analyzer manifold (Fig. 7), and run the other end of the tube to outside the trailer through tubing pass-thru on floor.
- 9.5. Inlet flow verification (in field)
    - 9.10.1 Measure flow at each inlet using 1 L/min rotameter
    - 9.10.2 Check flow rate of line with each installed inlet and record on Inlet Flow Verification Form (Appendix D).
    - 9.10.3 Adjust inlet as necessary to maintain constant flow rate across all inlets (5% accuracy)
- 9.6. Power up GSS (in field)
    - 9.6.1. Verify that all the completed connections are correct.
    - 9.6.2. Plug the GSS power cord into a 115-VAC power outlet to power up the GSS.
    - 9.6.3. Turn on the sampling pump and the bypass pump.
- 9.7. Initial setup of single-inlet gas sampling system (GSS) (in lab)
    - 9.7.1. Cut a 3′ section of 1/4″ OD Teflon tubing with a sharp tube cutter.
    - 9.7.2. Connect one end of the 1/4″ Teflon tube to the outlet of the Teflon filter holder. Tighten it with a wrench (this is different type of compression fitting that does not have ferrules).

- 9.7.3. Place a 47-mm filter holder and 1-μm pore membrane filter at one end of each 3′ 1/4″ OD tube to be used to connect the S-OPS extension tubing to the GSS solenoids. This prevents PM from entering the air tubing, GSS, and gas analyzers.
  - 9.7.3.1. Install a Teflon filter with 1-µm pore size in the 47-mm in-line filter holder and tighten the holder with the special filter holder wrenches.
- 9.7.4. Insert the other end of the 3′ piece of 1/4″ OD Teflon tube through the tube holder on the top of the GSS (Fig. 4) and align it with the solenoid port that faces upwards (Fig. 4). Connect the tube to the Teflon adapter in the solenoid by hand tightening the nut (Fig. 4). Hand-tighten the adapter (tube holder) on the top panel so that the tube does not move.
- 9.7.5. Connect the inlet of the filter holder to the 3/8″ OD extension tubing for a S-OPS using Teflon connectors.
- 9.7.6. Repeat Sections 9.7.4 to 9.7.5 to connect all the sampling tubes to the GSS.
- 9.7.7. Connect the inlet tubes of the gas analyzers to the gas ports (Fig. 7), and hand-tighten the Teflon fittings.

9.8. Cable connections

- 9.8.1. There are two cable connection receptacles in the GSS. One has 16 pins, and is for solenoid control. Another has 14 pins, and is for sensor power and sensor signals.
  - 9.8.1.1. Connect the solenoid (digital) control cable to the A21REl-12 Relay controller (Table 1).
  - 9.8.1.2. Connect the analog cable to the DAC (CR800 Data logger) (Table 2).
- 9.8.2. Connections for the A21Rel-12 Relay Controller (Table 3)
  - 9.8.2.1. Connect the positive of the 24VDC power supply from the TEC 450i Terminal I/O connector terminal 24 to ‘REL 1 COM’ and the ground of the TEC 450I digital output connector terminal 24 to the ‘GRND’ on the relay controller.
  - 9.8.2.2. Connect the blue wire from the GSS digital cable to ‘REL 1-NC’ on the relay controller
  - 9.8.2.3. Connect the Black/spotted red and white wire from the GSS digital cable to ‘GRND’ on the relay controller .
  - 9.8.2.4. Connect the positive and ground/common of the 12-VDC power supply to the CR800 data logger. Power for the A21REL-12 Relay Controller comes from the data logger.
  - 9.8.2.5. Connect the Green wire from ‘C1’ on the CR800 data logger to ‘CTRL1’on the relay controller.

9.9. Construction of S-OPS sampling manifold (at laboratory)

- 9.9.1. Choose length of sampling manifold
- 9.9.2. Cut 3/8″ OD Teflon tubing into section no longer than 10% of the manifold length or 5 m, whichever is smaller
  - 9.9.2.1. The last 5-m section should have additional length for bending tubing to sample height of 1-2 m.
- 9.9.3. Connect lengths of tubing with 3/8″ Teflon Tees
- 9.9.4. Put stainless steel connector on tubing to connect with S-OPS extension tubing and seal with tape.

- 9.9.5. Cut inlet tubing to length corresponding to desired sampling height. Assume tees will be at ground level.
- 9.9.6. Connect inlet tubing to tees (last inlet tubing is bent section) and hand tighten
- 9.9.7. Connect inlet tubing to 25-mm filter holders and hand tighten tubing connectors.

9.10. Static balancing of S-OPS inlet flows (at laboratory)

- 9.10.1. Construct flow-restricting orifices out of 1/8″ OD Teflon tubing
- 9.10.2. Cut tubing into 5″ sections
- 9.10.3. While rotating tubing pieces, hold middle of tubing over small Bunsen burner and stretch tubing to decrease inside diameter to a very small hole
- 9.10.4. Cut stretched piece of tubing in half to make 2 orifices
- 9.10.5. Repeat procedure to make the required number of orifices (with many extras).
- 9.10.6. Initial flow testing
  - 9.10.6.1. Connect spare pump to a controlling flow valve to a section of tubing in which each orifice can be inserted (Fig. 3) for flow testing.
  - 9.10.6.2. Plug inlet of 25-mm filter holder (with 1-μm pore filter installed) with rubber stopper pre-fit with 1/4″ OD Teflon tubing section. Attach Gillian flow meter (or equivalent) to 1/4″ Teflon tubing extending from stopper.
  - 9.10.6.3. Insert flow restrictor in tubing at filter holder outlet, turn on pump, and measure flow with meter.
  - 9.10.6.4. Orifices should measure a flow of approximately 0.5 L/min

9.11 Dynamic balance inlets in S-OPS sampling manifold (at laboratory)

- 9.11.1 Attach 1000′ of 3/8″ Teflon tubing to S-OPS manifold to determine the flow rate (use 1000′ because this should be about the maximum amount of tubing needed to reach the S-OPS sampling manifold from the GSS)
- 9.11.2 Use a Gillian flow meter (or equivalent) to measure the flow rate at the end of the 1000′ of tubing (about 6.7 L/min).
- 9.11.3 Using a spare pump and ball valve, establish a flow rate equal to this flow (6.7 L/min) and attach the spare pump (with 1/4″ OD tubing to the S-OPS sampling manifold (with 3/8″ OD tubing) with reducing union. Lay out orifices in a straight line
- 9.11.4 Insert flow restrictors into sampling manifold at the filter holder inlet (with 1-μm pore filter installed) of each inlet tube.
- 9.11.5 Attach the rubber stopper with pre-fit 1/4″ tubing into the filter inlet of the inlet to be flow-tested. Keep all other inlets of the S-OPS open.
- 9.11.6 Turn on pump and measure flow at the 1/4″ OD Teflon tubing using a Gillian flow meter (or equivalent).
- 9.11.7 Progressing from inlet 1 to 10, balance inlets to 0.67 L/min (±5%) by cutting off small amounts of the ends of the orifices as needed.

9.12 Software configuration

- 9.12.1 The GSS is designed to have one solenoid normally open to the sampling manifold and the others normally open to the bypass manifolds (Fig. 4). This is to avoid choking the pumps in case that all solenoids shut off (e.g. computer shut-down or DAC power failure) while the pumps are still running.

9.12.2 The GSS is controlled by the DAC hardware (CR800 and A21REL-12 Relay Controller) and program in data logger.

9.13 GSS initial tests in field

9.13.1 Verify GSS sensor operation

9.13.1.1 Using the LoggerNet software, connect to the CR800 and upload the GSS program ‘NAEMS_GSS_test.CR8’. After uploading, access measurements at “Data displays/Numeric/1” by the radio button on the screen.

9.13.1.1.1 Add to the Numeric listing: PortStatus(1) from ‘Status’ Table and Pressure, Mass Flow, and Flow direction from ‘Public’ Table.

9.13.1.2 The pressure sensor should have values in the range between -20 kPa and -50 kPa at normal sampling. If it is not in this range, there may be either a leakage or a blockage in the sampling tube anywhere from the sampling location to the sampling pump.

9.13.1.3 The sampling flow rate should be between 4 and 7 L/min. If it is not in this range, check the sampling tubes, the sampling pump, and the mass flow meter.

9.13.1.4 Record readings under ‘Sample flow test’ on the GSS Acceptance Form (Appendix B).

9.13.2 Verify solenoid control

9.13.2.1 Listen for clicking sound from inside the GSS, indicating that the solenoids are switching. Check PortStatus(1), which will change from ‘True’ to ‘False’ in “Data displays/Numeric/1”

9.13.2.2 If clicking sound is heard, the solenoids are being controlled.

9.13.2.3 If no clicking sound is heard, check power to relay controller and signal lines between DAQ and relay controller and GSS to DAQ and relay controller according to Tables 1 and 3.

9.13.3 Verify sampling air flow in the GSS and gas sampling filter.

9.13.3.1 Check the airflow using the mass flow meters at the outlet of the sampling system.

9.13.3.2 Disconnect extension S-OPS tubing from 47-mm sampling filters.

9.13.3.3 Record readings under ‘Max flow test’ on the GSS Acceptance Form (see Appendix B).

9.13.4 Verify bypass air flow

9.13.4.1 Make sure that the bypass pump has about 4 L/m flow rate and is running.

9.13.4.2 Disconnect S-OPS manifold from S-OPS extension tubing and connect the 10 L/min rotameter to inlet of S-OPS extension tubing

9.13.4.3 Measure the airflow rate at the inlet of the S-OPS extension tubing when the line is not being sampled. An airflow rate of > 0.5 L/m should be detected, depending on the total sampling locations in the system.

9.13.4.4 If airflow rate is too low, check the flow restrictor inside the 1/4″ OD Teflon tube between the bypass manifold and the solenoid connected to this particular S-OPS extension line (Fig. 7). If necessary, pull out the flow restrictor from the 1/4″ OD Teflon tube and cut it a little shorter (enlarge the orifice) before inserting it back into the 1/4″ OD Teflon tube. Because there is a balance of bypass airflow among all sampling lines, be cautious not to enlarge the orifice of the flow restrictor too much.

9.13.4.5 Repeat 9.13.4.1 to 9.13.4.4 for each S-OPS extension line.

9.14 GSS leakage test (in field)

9.14.1 Connect the GSS to the DAQ hardware and software so that the solenoids can be controlled, and the pressure and airflow rate can be monitored.

9.14.2 Check all air passage connections. Hand-tighten fittings inside the GSS if loose.

9.14.3 Make sure that the GSS pressure and airflow rate measurement are correct.

9.14.3.1 When the sampling pump inside the GSS is shut off, both pressure and airflow rate should read near zero.

9.14.3.2 Record readings under ‘No flow test’ on the GSS Acceptance Form (see Appendix B). These values are used to ‘correct’ measurements for sensor offsets.

9.14.4 Plug inlet side of 47-mm filter at inlet of solenoid passing air into the sampling manifold.

9.14.5 Watch the readings of the sampling pressure and sampling flow rate in the data logger measurements. Both of them should drop down quickly.

9.14.6 Do not leave the vacuum status too long because the GSS is “over loading” during this test.

9.14.6.1 Record readings under ‘Leakage test’ on the GSS Acceptance Form (see Appendix B)

9.14.7 Repeat 9.14.4 – 9.14.6 for all actively-used solenoids

9.14.8 Correct all flow and pressure readings for sensor offsets by subtracting the no-flow values from the measured pressure and flow measurements. Record calculated values under “Corrected Mass flow” and “Corrected Pressure” on the GSS Acceptance Form (see Appendix B).

9.14.9 If the sampling airflow drops to less than 0.1 L/m within about one minute, the GSS is leak-free starting from the solenoids. Indicate a ‘Pass’ on the GSS Acceptance Form if this is true.

9.14.10 If the sampling airflow is more than 0.1 L/m Check integrity (tightness) of the filter holder, tube connections of the filter holder and in the GSS (in that order) and repair as needed. Indicate a ‘Fail’ on the GSS Acceptance Form if this is true and indicate in the comments the remediation and results of remediation.

9.15 Sampling system (GSS + S-OPS) leakage test (in field- requires two persons and a pair of walkie-talkies)

9.15.1 Plug all inlets of an S-OPS with rubber stoppers

9.15.2 Record the sampling pressure and sampling airflow rate from the data logger measurements.

9.15.3 If the sampling line is leak-free, the sampling airflow will drop down to about 0.7 L/min within several minutes, depending on the length of the tube.

9.15.3.1 Record the test results under “S-OPS/GSS Leak test” on the NAEMS open S-OPS Acceptance Form (Appendix C).

9.15.4 Do not leave the vacuum status too long because the GSS is “over loading” during this test

9.15.5 Correct all flow and pressure readings for sensor offsets by subtracting the “No-Flow” values on GSS Acceptance Form (Appendix B) from the measured pressure and flow measurements in 9.15.2. Record calculated values under

"Corrected Mass flow" and "Corrected Pressure" on the S-OPS Acceptance Form (Appendix C).

9.15.5.1 If the airflow is below 10% of the total sampling air flow (0.7 L/min for a 7-L/min sampling airflow), the sampling system should be considered normal. Indicate a 'Pass' on the S-OPS Acceptance Form if this is true.

9.15.5.2 If the airflow is more than 10% of the sampling air flow, the sampling system has a leak. Check integrity of the 25-mm filter holders, connections to 25-mm filter holder, tees in S-OPS manifold, extension S-OPS tubing to S-OPS connection, and connection of S-OPS extension tubing to 47-mm filter holder in trailer (in that order) and repair as needed. Apply or replace Teflon tape to nylon screw joints (especially 25-mm filter) to enhance seal.

9.15.5.3 If failure continues, indicate a 'Fail' on the S-OPS Acceptance Form, and indicate in the comments the attempted remediation and results.

9.16 Sampling system (GSS/ S-OPS) flow balance test (in field)

9.16.1 Requires two persons and a pair of walkie-talkies

9.16.2 Disconnect S-OPS manifold from S-OPS extension tubing and connect 10 L/min rotameter to inlet of S-OPS extension tubing.

9.16.3 Connect the outlet of the airflow meter or rotameter to the end of the S-OPS 3/8″ extension tubing using a short piece of 1/4″ tube coming from the suction-side of the meter inserted into the 3/8″ tubing.

9.16.3.1 The mass flow meter inside the GSS can serve as the outlet airflow meter. Use another airflow meter or a rotameter as the inlet measurement device. Make sure that the two meters are calibrated together before the test.

9.16.4 Record mass flow at end of extension tubing (from rotameter) and mass flow measured at the inlet to the Analyzer manifold (from mass flow meter measurement reported in GSS data logger data record).

9.16.5 Correct the mass flow meter measurements as in 9.15.5, and record under "Max Flow Test" on S-OPS Acceptance Form (Appendix C)

9.16.6 Compare the airflow readings from the inlet air flow meter with that from the mass flow meter in the GSS. A positive difference in readings (difference = inlet - outlet) indicates a possible air leakage.

9.16.6.1 The difference between the two mass flow measurements should be less than 0.2 L/min. Indicate a 'Pass' on the S-OPS Acceptance Form if this is true.

9.16.6.2 If the difference in flow is greater than 0.2 L/min, there is a leak in the sampling system. Check integrity of the extension S-OPS tubing, connections and the GSS (in that order) and repair as needed. Indicate a 'Fail' on the S-OPS Acceptance Form if this is true and indicate in the comments the remediation and results of remediation.

9.17 Initial GSS acceptance tests in laboratory

9.17.1 Verify GSS sensor operation

9.17.1.1 Using the LoggerNet software connect to the CR800 and upload the GSS program 'NAEMS_GSS_test.CR8'. After uploading, access measurements at "Data displays/Numeric/1" by the radio button on the screen.

9.17.1.1.1 Add to the numeric listing: PortStatus(1) from ‘Status’ Table and Pressure, Mass Flow, and Flow direction from ‘Public’ Table.

9.17.1.2 The pressure sensor should have values in the range between -20 kPa and -50 k Pa at normal sampling. If it is not in this range, there may be either a leak or a blockage in the sampling tube anywhere from the sampling location to the sampling pump.

9.17.1.3 The sampling flow rate should be between 4 and 7 L/min. If it is not in this range, check the sampling tubes, the sampling pump, and the mass flow meter.

9.17.1.4 Record readings under ‘Sample flow test’ on the GSS Acceptance Form (Appendix B).

9.17.2 Verify solenoid control

9.17.2.1 Listen for clicking sound from inside the GSS, indicating that the solenoids are switching and PortStatus(1) which will change from ‘True’ to ‘False’ in “Data displays/Numeric/1”

9.17.2.2 If clicking sound is heard, the solenoids are being controlled.

9.17.2.3 If no clicking sound is heard, check power to relay controller and signal lines between DAQ and relay controller and GSS to DAQ and relay controller according to Tables 1 and 3.

9.17.3 Verify sampling air flow balance to check for leaks in the GSS itself and in its immediate connections.

9.17.3.1 This method can be used prior to leak testing of the entire sampling system (Section 9.15). It requires two persons to work together.

9.17.3.2 Check the airflow balance using two airflow (mass flow) meters, one at the inlet and another at the outlet of the sampling system.

9.17.3.3 The mass flow meter inside the GSS can serve as the outlet airflow meter. Use another airflow meter or a rotameter as the inlet measurement device. Make sure that the two meters are calibrated together before the test.

9.17.3.4 Connect the outlet of the airflow meter or rotameter to the end of 500′ of S-OPS 3/8″ extension tubing using a short piece of 1/4″ tube coming from the suction-side of the meter inserted into the 3/8″ tubing.

9.17.3.5 Compare the airflow readings from the air flow meter with that from the mass flow meter in the GSS. A positive difference in readings (difference = inlet - outlet) indicates a possible leak.

9.17.3.6 Record readings under ‘Max flow test’ on the GSS Acceptance Form (Appendix B).

9.17.3.7 If there is leakage indicated, the leak should be identified and removed.

9.17.3.8 This procedure can be combined with that of solenoid testing (Section **Error! Reference source not found.**) to save testing time.

9.17.4 Test flow direction sensor

9.17.4.1 Plug all analyzer ports.

9.17.4.2 Be sure that sample and bypass pumps are on.

9.17.4.3 Note sample flow rate and flow direction sensor response and record on Flow Direction Sensor Calibration Form (Appendix G) for each solenoid used.

9.17.4.4 Turn off sample and bypass pumps and disconnect lower connection of tee between sample pump and analyzer manifold (Fig. 7).

9.17.4.5 Using a spare sampling pump (reverse flow pump) of the same design as the GSS pumps, connect 5/8″ ID PVC to 1/4″ Teflon reverse flow adapter to sample manifold exhaust to positive flow side of spare pump.

9.17.4.6 Start reverse flow pump and verify flow from open analyzer port (verify that flow is exhausting from the disconnected tubing at sample pump tee.

9.17.4.7 Note reverse flow rate and flow direction sensor response and record on Flow Direction Sensor Calibration Form (Appendix G) for each solenoid used. Value should be negative

9.17.4.7.1 If there is no difference in values, look for leaks in the analyzer manifold. Repair and retest.

9.18 GSS leakage test

9.18.1 Connect the GSS to the DAQ hardware and software so that the solenoids can be controlled, and the pressure and airflow rate can be monitored.

9.18.2 Check all air passage connections. Hand-tighten fittings inside the GSS if loose.

9.18.3 Make sure that the GSS pressure and airflow rate measurement are correct.

9.18.3.1 When the sampling pump inside the GSS is shut off and the analyzer manifold and exhaust are plugged, both pressure and airflow rate should read near zero.

9.18.3.2 Record readings under ‘No flow test’ on the GSS Acceptance Form (Appendix B). These values are used to ‘correct’ measurements for sensor offsets.

9.18.4 Disconnect 47-mm filter on inlet tube. Plug tube entering sampling solenoid.

9.18.5 Turn on pumps and watch the readings of the sampling pressure and sampling flow rate in the data logger measurements. Both of them should drop down quickly.

9.18.5.1 Do not leave the vacuum status too long because the GSS is “over loading” during this test.

9.18.5.2 Record readings under ‘Leakage test’ on the GSS Acceptance Form (Appendix B)

9.18.6 Repeat 9.16.1 – 9.16.5 for all actively-used solenoids

9.18.7 Correct all flow and pressure readings for sensor offsets by subtracting the no-flow values from the measured pressure and flow measurements. Record calculated values under “Corrected Mass flow” and “Corrected Pressure” on the GSS Acceptance Form (Appendix B).

9.18.8 If the sampling airflow drops to less than 0.1 L/min within about one minute, the GSS is leak-free starting from the solenoids. Indicate a ‘Pass’ on the GSS Acceptance Form if this is true.

9.18.9 If the sampling airflow is more than 0.1 L/min, check integrity/tightness of the filter holder, tube connections of the filter holder and in the GSS (in that order), and repair as needed. Indicate a ‘Fail’ on the GSS Acceptance Form if this is true and indicate in the comments the remediation and results of remediation.

9.19 Bag test (system adsorption test done in lab)

9.19.1 Conduct this test once for each gas sampled through the GSS

9.19.2 Connect 3 m (10′) of 1/4″ Teflon tubing to the output of the TEC 146i diluter or the cylinder regulator (as appropriate).

9.19.3 Program the diluter (SOP C3) to produce a concentration significantly different from that which is currently being measured at the sampling location, or utilize a calibration tank of concentration significantly different from that which is currently being measured at the sampling location.

- 9.19.3.1 Selecting a different concentration from that which is being measured allows the operator to easily see that a particular location is being tested, and to visualize differences from one sampling location to another during the bag test.

9.19.4 Conditioning of Tedlar bags

- 9.19.4.1 Check the Tedlar bags for damage that might cause leaks (holes, cuts, a bad or loose valve, etc.)
- 9.19.4.2 Using flexible (i.e. vinyl) tubing, connect the first Tedlar bag to a vacuum pump. Evacuate the bag, then disconnect it and evacuate the other 5 bags.
- 9.19.4.3 Fill 1/3 full with gas of a known concentration for the bag test.
- 9.19.4.4 Repeat Sections 9.19.4.2 - 9.19.4.3 two more times.
- 9.19.4.5 Completely fill the Tedlar bag with gas of a known concentration.
- 9.19.4.6 Repeat Sections 9.19.4.2 - 9.19.4.5 for the other 4 Tedlar bags

9.19.5 Test the integrity of each inlet on the S-OPS as follows:

- 9.19.5.1 Plug all inlets to the S-OPS with rubber stoppers
- 9.19.5.2 Sample each location on each S-OPS for a minimum of 5 min before attaching the Tedlar bag with the known concentration.
- 9.19.5.3 Change rubber plug at inlet of interest with stopper with tubing insert.
- 9.19.5.4 Using flexible (i.e. vinyl) tubing, attach Tedlar bag to the sampling location that is being measured. Use a reducing union, and make sure that the connection is a tight connection to avoid any leaks.
- 9.19.5.5 Open valve on Tedlar bag with 3 turns. Do not open any further than this, because the valve will come apart.
- 9.19.5.6 Sample the Tedlar bag for 10 min.
- 9.19.5.7 Monitor the data on the analyzer.
- 9.19.5.8 Save a print screen of the data logger in a PowerPoint file.
- 9.19.5.9 Record the sampling time (start & stop), sampling location, concentration readings from the diluter (cylinder specification) and the analyzer, and the analyzer serial number in the field notes, using the form in Appendix E.
- 9.19.5.10 Switch to the next location.
- 9.19.5.11 Repeat steps 9.19.5.3 to 9.19.5.10 until all sampling locations have been tested. If necessary, refill empty Tedlar bags with the known gas concentration again until all the locations have been tested.

9.19.6 Average analyzer concentrations

- 9.19.6.1 Go to computer data file containing measurements from the analyzer of interest.
- 9.19.6.2 Copy and paste the copy of the current 1-min data file in the data file
- 9.19.6.3 Open the copy of current 1-min data file with Excel.

9.19.6.4 Locate the start and stop times for each location during the bag test in the 1-min data file.
9.19.6.5 Average the concentration readings for the last two minutes of the Tedlar bag sampling.
9.19.6.6 Enter the average concentrations for each S-OPS into the form (Appendix E).
9.19.6.7 Close the copy of the 1-min data file without saving.
9.19.6.8 Calculate the percent difference from average concentration reading from all sampling locations for each sampling location.
9.19.6.9 If the percent difference is greater than 5 % for any sampling location, take the following corrective action(s), and retest that sampling location (repeat Sections 9.19.5.4 to 9.19.5.10), until the percent difference is less than 5 %.
  9.19.6.9.1 Check and tighten all connections for the sampling line that has a leak.
  9.19.6.9.2 Check for holes or damage to the sampling line.
  9.19.6.9.3 Check to make sure that the filter holder is closed tightly
  9.19.6.9.4 Check the GSS for leakage or loose connections
  9.19.6.9.5 Check Tedlar bag for leaks
  9.19.6.9.6 Check the connects between the GSS and the gas analyzers
  9.19.6.9.7 Check the connection between the sampling location and the Tedlar bag
9.19.6.10 Check the Tedlar bags for damage that might cause leaks (holes, cuts, a bad or loose valve, etc.).
9.19.6.11 Evacuate Tedlar bags and store in a safe place until needed again.

9.20 GSS operation

9.20.1 Using the LoggerNet software connect to the CR800 and upload the GSS program ‘NAEMS_GSS.CR8’. After uploading, access measurements at “Data displays/Numeric/1” by the radio button on the screen.
9.20.1.1 Add to the Numeric listing: PortStatus(1) from ‘Status’ Table and Pressure, Mass Flow, and Flow direction from ‘Public’ Table.
9.20.1.2 The pressure sensor should have values in the range between -30 kPa and -80 k Pa at normal sampling. If it is not in this range, there may be either a leak or a blockage in the sampling tube anywhere from the sampling location to the sampling pump.
9.20.1.3 The sampling flow rate should be between 4 and 7 L/min. If it is not in this range, check the sampling tubes, the sampling pump, and the mass flow meter.
9.20.1.4 If results differ from those on the GSS Acceptance Form, address problem following Section 9.21.

9.21 Troubleshooting

9.21.1 Contamination
9.21.1.1 If moisture is observed in the line, remove the end cap and flush the interior of the line with dry air for a minimum of 5 min.
9.21.1.2 If dust, PM or other debris is observed in the line, remove the end cap, plug the sampling inlets, and flush with water for 5 min, and then air for 30 min.

9.21.2 Air sampling tubing failure
9.21.2.1 While pumping air through the tubing with all inlets plugged except for the inlet at the opposite end of the tube, use rotameters or similar devices to

measure airflow rate at both ends. If the two flow rates do not agree, inspect the tube for damage or leaks.

9.21.2.2 While pumping air through the tubing with all inlets open, measure all inlet and exhaust flows with rotameters or mass flow meters. If the sum of the inlet flows disagrees with the total pump flow, inspect tubing for damage or leaks.

9.21.2.3 If tubing is found to be damaged or chewed through by rodents, cut out and replace the section (if necessary) and connect using stainless steel union(s). Flush the whole tube from the trailer with pressurized N_2 zero air, and inspect for trapped particles. Measure flow rates again to guard against leakage.

10. Data and Records Management

10.1. Maintain all records on appropriate worksheets of the electronic field notes designated for this method. Supplement this electronic record by documentation in the open site field notes.

10.2. Manage all data according to SOP D1.

10.3. On initial set-up for a given location, use the Synthetic Open-Path Placement Form (Appendix F) in conjunction with the SMP to record the position and configuration of each sampling line.

10.4. Document all data and information (e.g., sample collection method used) on field data sheets, and within site field notes with permanent ink, or in electronic field notes.

10.5. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

11.1. Inspect S-OPS and S-OPS extension tubing for physical damage, firm connections, and sampling line contamination at the beginning and end of each sampling period or every 20 d, whichever is less

11.2. Inspect filters at S-OPS inlets for physical damage, firm connections, and filter contamination at the end of each sampling period or every 20 d, whichever is less.

11.3. Test S-OPS and extension tubing for leakage (Section 9.15) at the beginning and end of each sampling period or every 20 d, whichever is less.

11.4. Test S-OPS inlets for flow rates at the end of each sampling period or every 20 d, whichever is less.

11.4.1. Use 1 L/min rotameter attached to 1/4″ OD tubing inserted through rubber stopper.

11.4.2. Insert rubber stopper in inlet leaving all other inlets un-obstructed. Test all 10 inlets of each S-OPS. Record flow results on Synthetic Open Path Inlet Flow Verification Form (Appendix D).

11.4.3. Calculate mean flow of all 10 inlets.

11.4.4. Calculate deviation of inlet from the mean flow of all other inlets. To avoid biased sampling of the plume, the flow through each inlet must be within 5% of the average flow through all inlets.

11.4.5. If an inlet has a flow deviation greater than 5% low, check inlet filter for accumulated dirt. Replace as needed and retest flow.

11.4.6. Mass flow meters measuring the air flow must be calibrated (SOP A9) at least once per year.

11.5. Keep minimum sampling airflow about 0.5 L/min higher than the maximum sample airflow drawn by all analyzers and the manual port. If additional analyzers are connected to the GSS, and cause increased sample air demand, adjust the flow restrictor between the sampling pump and the mass flow meter to increase the sample flow.

11.6. Review the daily measurement data for the GSS sampling pressure and flow rate to identify abnormal readings. Some common problems that can be identified by the pressure and airflow data are listed in Table 4.

12. References

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12.3. iPort Instruction Manual. 2007. Communications software for iSeries and C Series Instruments. Part#102606-00. Thermo Fisher Scientific, Franklin, MA.
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12.5. SOP A9. 2006. Measurement of Airflow Rate or Calibration of Air Sampling Instruments with Gilibrator 2 Calibration Systems. Standard Operating Procedure A9, v. 0.0. Purdue Ag Air Quality Lab
12.6. SOP C2. 2008. Measurement of Ammonia with the Boreal Laser GasFinder Tunable Diode Laser Absorption Spectrometer (TDLAS). Standard Operating Procedure C2, v. 1.0. Purdue Applied Meteorology Lab
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12.9. SOP G5. 2008. Measurement of Hydrogen Sulfide (H_2S) with the Thermo Electron Corporation Model 450C Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5, v. 1.0. Purdue Ag Air Quality Lab
12.10. SOP G7. 2008. Standard Operating Procedure for the Operation of the INNOVA 1412 Photoacoustic multi-gas analyzer. Standard Operating Procedure G7, v. 1.0. Purdue Ag Air Quality Lab.
12.11. SOP O1. 2006. Emissions Estimation Using the Thunder Beach Scientific Windtrax® Backward Lagrangian Stochastic Model. Standard Operating Procedure O1, v. 0.0. Purdue Applied Meteorology Lab.
12.12. SOP U5. 2008. Installation of Open Source Measurements. Standard Operating Procedure U5, v. 1.0. Purdue Applied Meteorology Lab

12.13. SOP U7. 2008. The Instrument Trailer and Installation of Open-Source Measurement Equipment (Corrals). Standard Operating Procedure U7, v. 1.0. Purdue Applied Meteorology Lab

Table 1. GSS digital solenoid control connections to A21REL-12 relay controller

Solenoid #	**Pin #**	**Cable wire color**	**A21REL-12 Terminal**	**Sampling location**
S2	2	Blue	REL1 NC	Near S-OPS
S3	3	Black/spotted red	NC	Far S-OPS
S5	5	White/spotted red	NC	
S7	7	Blue/spotted red	NC	
Ground	22	Blue/spotted black	GROUND	

Table 2. GSS sensor connections to DAC (CR800).

Sensor	**Pin #**	**Wire color**	**DAC Terminal #**
Pressure transducer, signal	1	Black	2H
Pressure transducer, ground	2	Green	2L
Mass flow meter, signal	5	Red	3H
Mass flow meter, ground	6	Black/White stripe	Ground ⏚
Flow direction +	7	Green/Black stripe	1H
Flow direction -	8	Orange	1L
Cooling fan, +24 DVC	22	Black	

Table 3. A21REL-12 Relay controller connections.

Cable/Instrument	**Terminal**	**Wire color**	**Signal**	**A21REL-12 Terminal #**
GSS digital cable		Black/spotted red&white	Ground	GRND
GSS digital cable		Blue	C2+	REL1-NC
TEC 450I digital output connector	24	White	+24VDC	REL1 COM
TEC 450I terminal I/O connector	24	Black	Ground	GRND
CR800	12V	Red	+12V	+12V
CR800	GND	Black	Ground	GRND
CR800	C1	Green	Digital 5VDC	CTRL1

Table 4. Possible sampling system problems related to GSS pressure and airflow data.

Pressure[1]	**Airflow**	**Number of locations to check for problems**	**Possible problem and solution**
Low	Low	One or several	An overloaded air filter or a blockage in the sampling line. Change the filter and clean the blockage.
High	Low	All locations	Sampling pump loses vacuum. Replace worn diaphragm and fix loose check valves inside pump. Check filters at inlets, and replace if heavily loaded.
High	High	One or a few	Leakage in the sampling line or GSS.
Low	High	All	Leakage in the GSS.
Unstable	Unstable	One	Solenoid not receiving stable power due to relay control problem. Check PortStatus(1) and power supply at connection terminals of solenoid.

1: Low= larger negative pressure

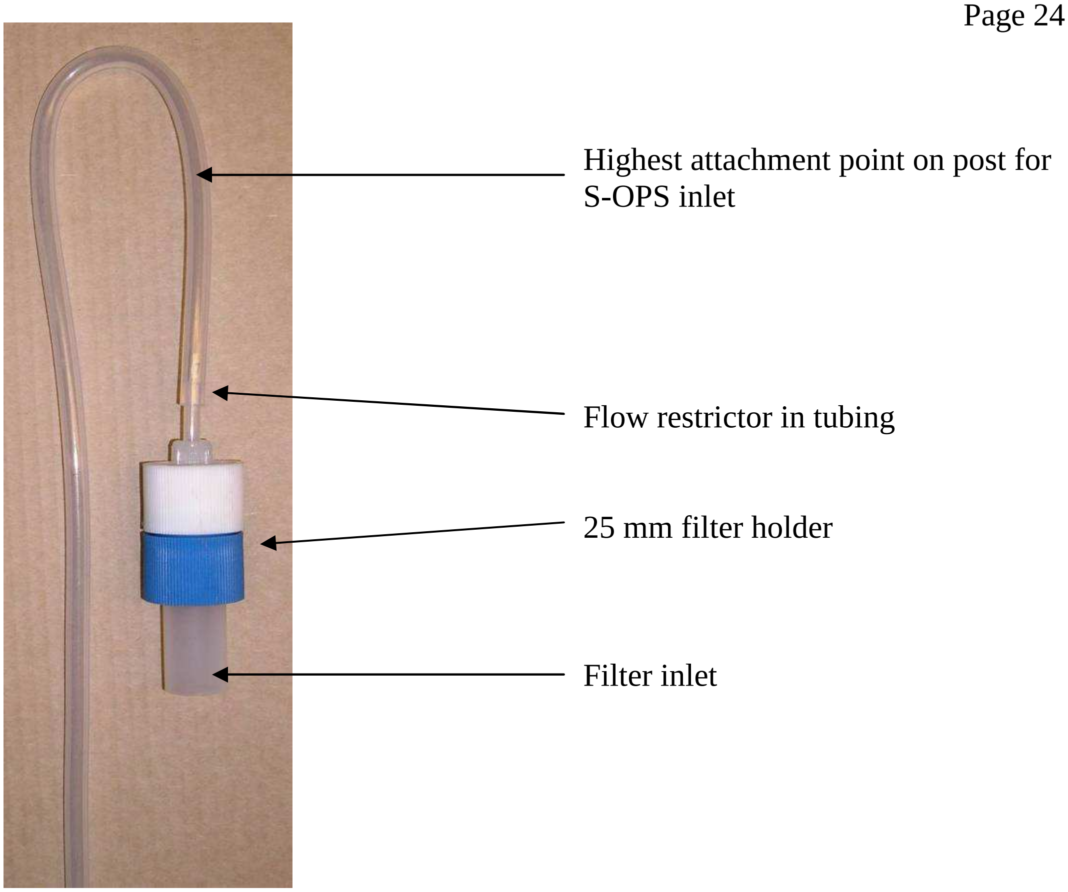


Figure 1. Synthetic open path sampling tube inlet construction.

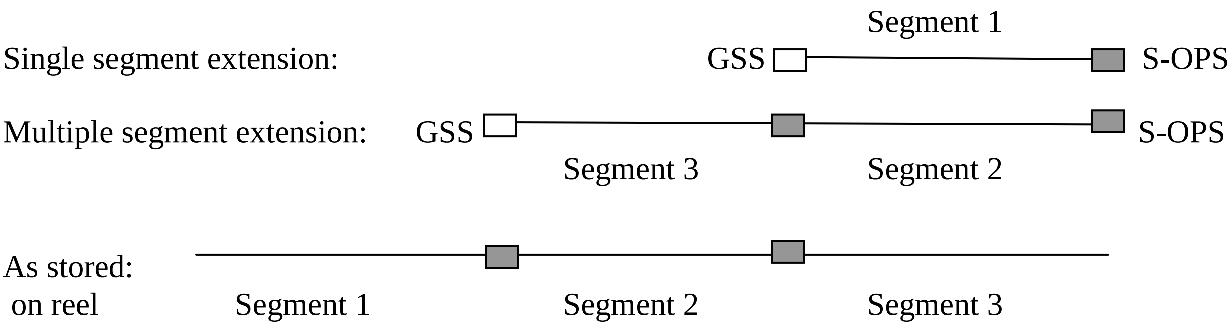


Figure 2. Extension tubing configuration.
Connector material: Stainless steel (■) and Teflon (□). Extension tubing is stored on the reel with Stainless Steel unions connecting segments together. An additional union is needed to attach both the single and multiple segment extension lines to the two S-OPS.

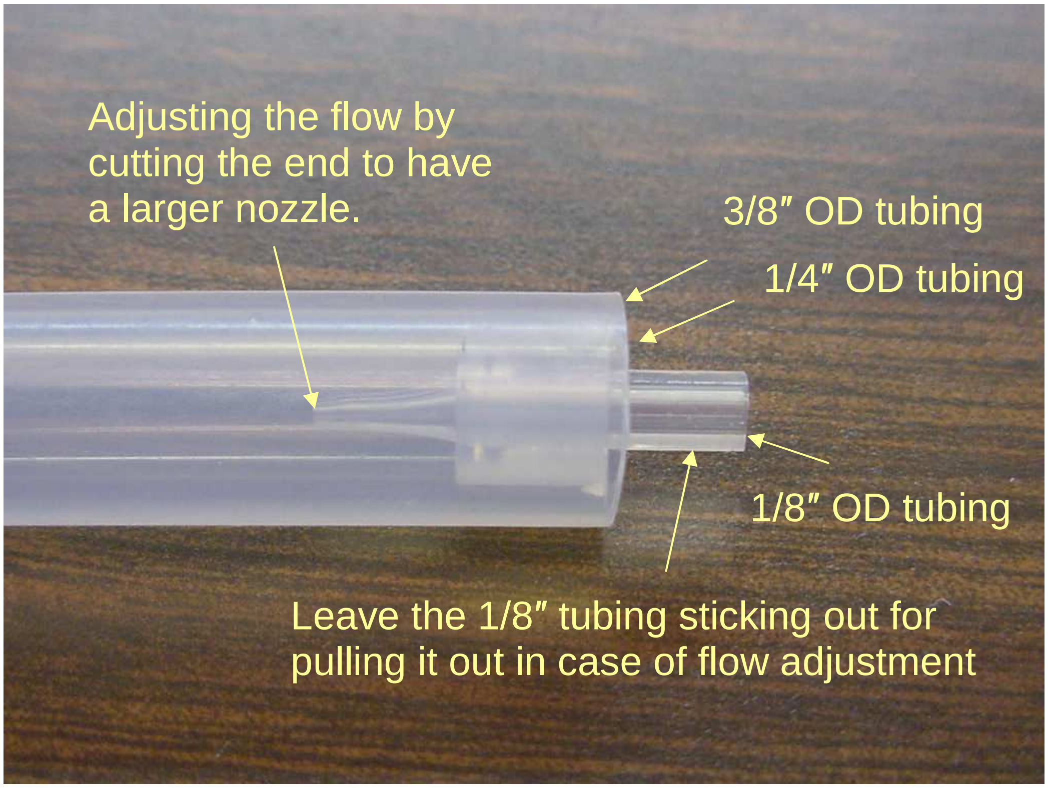


Figure 3. Configuration of flow restrictor in sampling inlet.

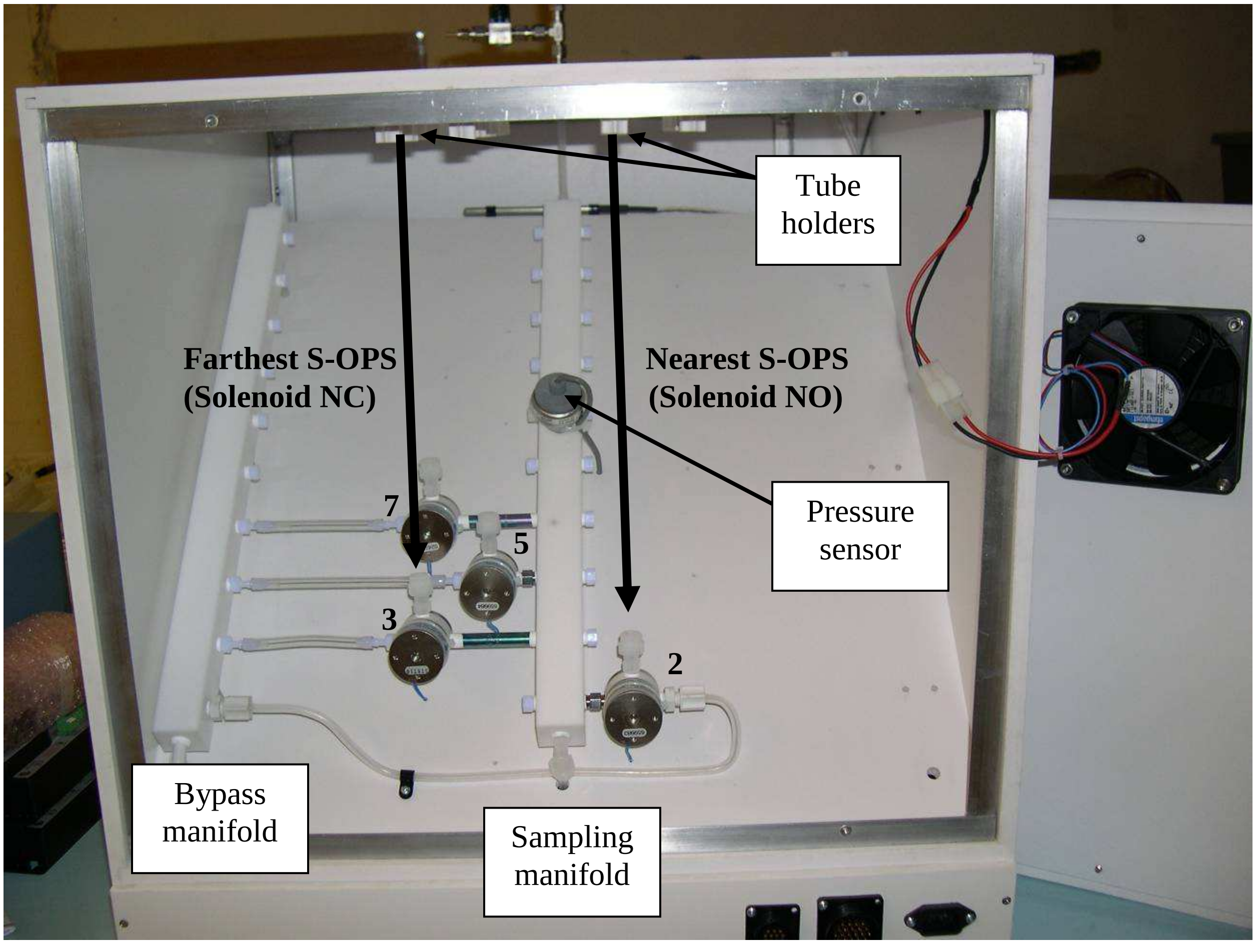


Figure 4. Open-cover view of the multi-point Gas Sampling System (front). Solenoid numbers are indicated as well as first two solenoids to be used with S-OPS configuration. NC= Normally closed (air normally directed to bypass manifold),NO=Normally open (air normally directed to sampling manifold).

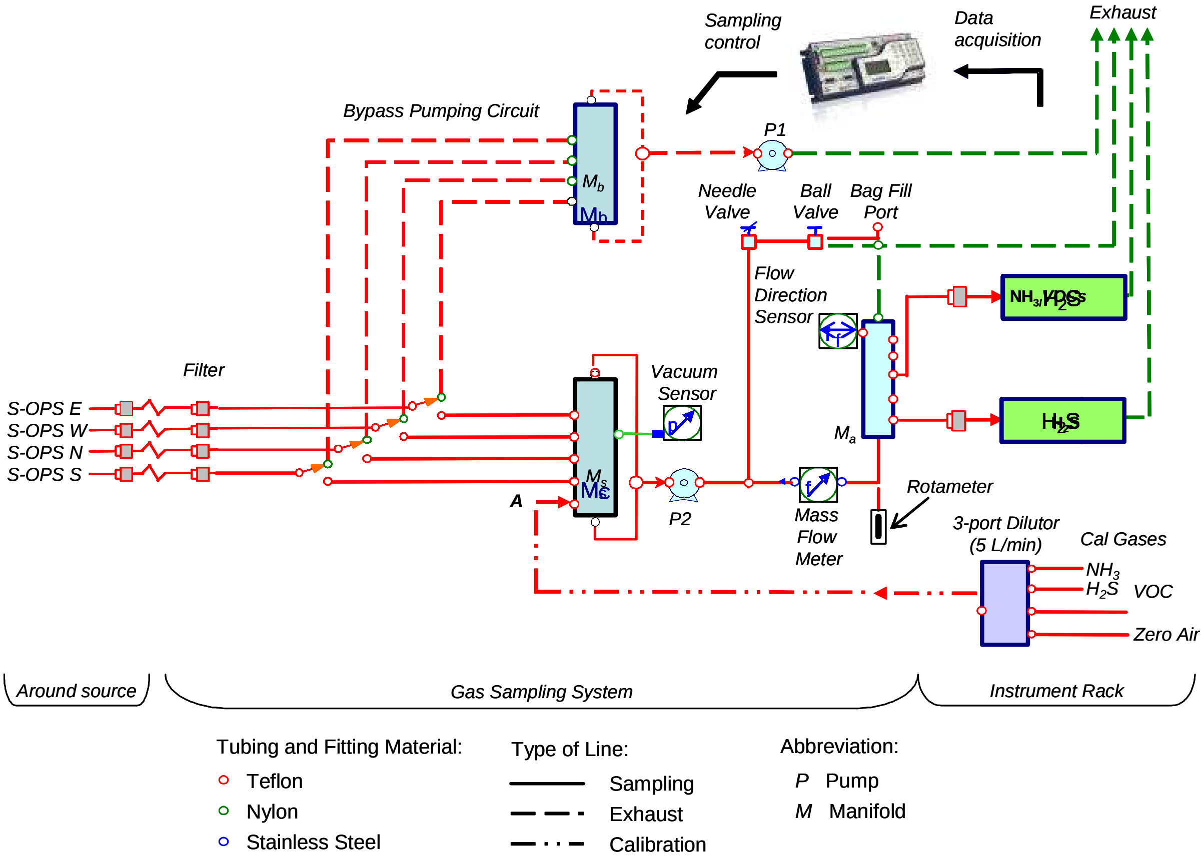


Figure 5. Schematic of Gas Sampling System.

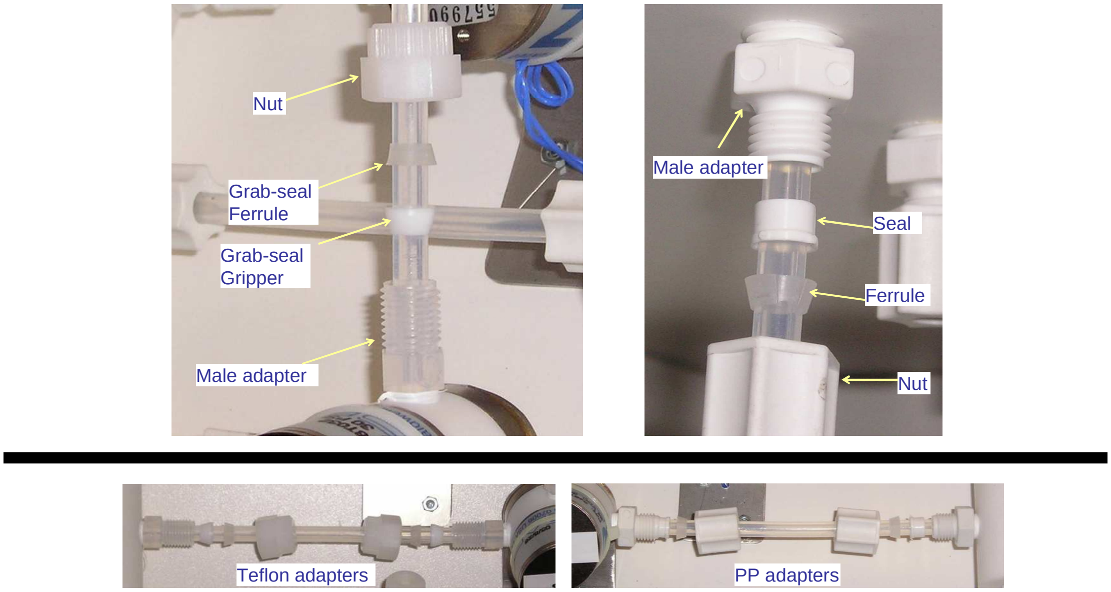


Figure 6. Connection of sampling tube to the Grab-seal Teflon fitting (left) at the solenoid and through the white polypropylene (right) fitting as the tube holder on top panel of the GSS.

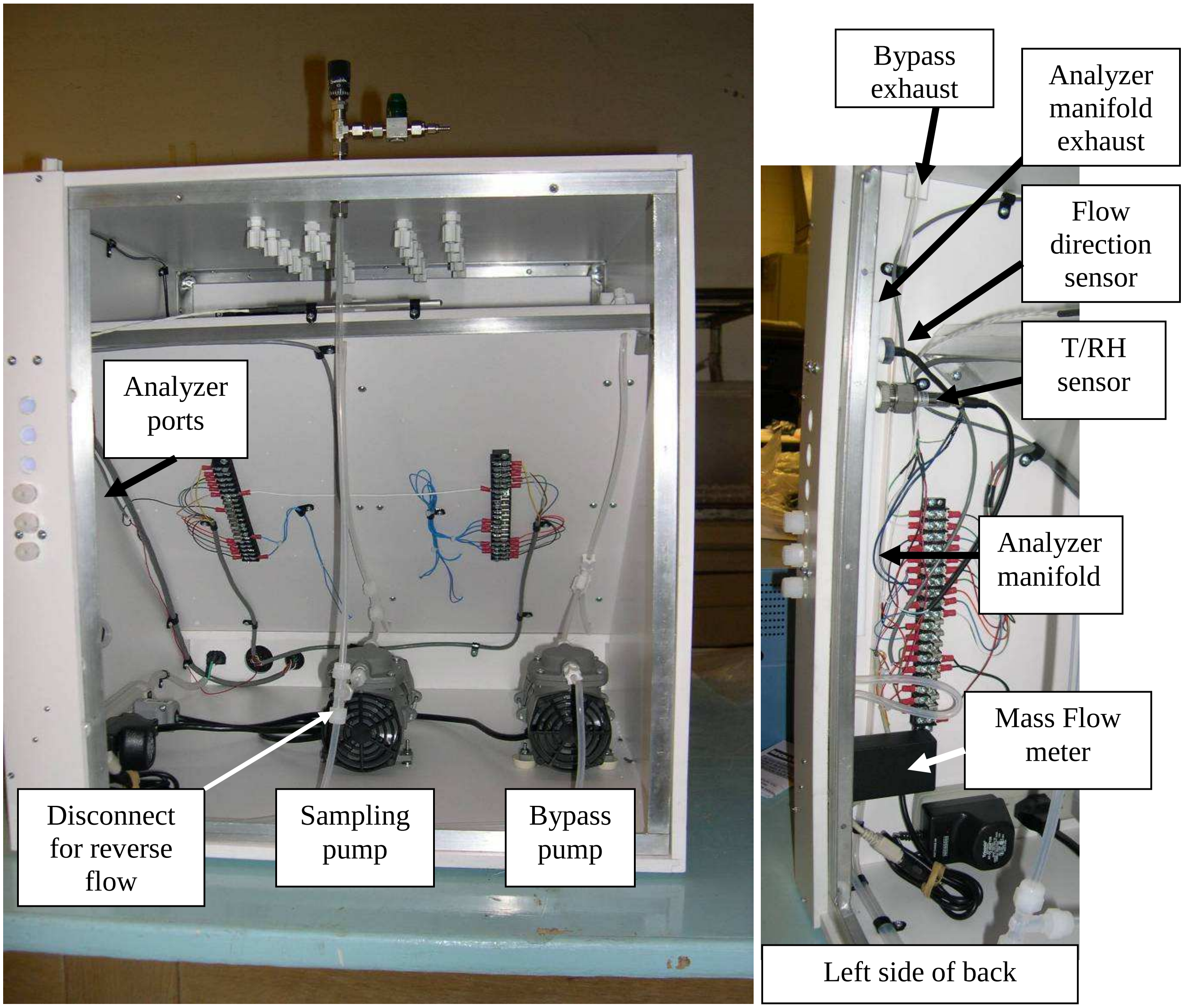


Figure 7. Open-cover view (back) of the multi-point gas sampling system.

Appendix A. Residence Time Calculations.

The residence time (ti) was calculated using the following equations:

$t_i = L_i / V_i$

Where:
L_i: length of tubing between inlet ni and next inlet.
V_i: velocity of gas in tubing segment Li.

$V_i = q_i/C_d * A_i$

Where:
q_i: gas flow in tubing segment L_i
A_i: internal cross area of tubing segment L_i.

Table A1 show the gas residence time and pressure loss as a function of the sampling volume per inlet . The pressure loss includes only the friction pressure loss, not the pressure drop due to the inlet filter and the needle valve used to regulate the inlet flow. Table A2 shows the residence time for a sample of air to travel through the S-OPS and S-OPS extension tubing given flow rates at the GSS.

Table A1. Gas residence time and pressure loss as a function of inlet sampling volume.

Inlet flow $q_{i,}$, L/min	Residence time, t_i, s		Pressure loss at 20°C	
	Maximum, i=1	Minimum, i=10	Pa	Psi
1	55	0.19	5,113	0.73
2	27	0.95	17,223	2.4
3	18	0.63	35,070	5.0
4	13	0.47	58,106	8.3
5	11	0.38	85,990	12.2

Tubing diameter: 0.63 cm (0.25 in.); tubing length: 100 m; number of inlets: 10; inlet i=10 is closest to the sampling pump.

Table A2. Gas residence time for sampling through S-OPS extension tubing.

	Residence time (s)	
Tube length, m	**5 L/min flow**	**10 L/min flow**
0 (GSS only)	1.9	1.0
50	21	20
100	40	20
200	78	39
300	116	58

Appendix B **NAEMS Open GSS Acceptance Form**

GSS S/N ____________________

Date: ________________________

Operator: _____________________

	Solenoid 2	**Solenoid 3**	**Solenoid 5**	**Solenoid 7**
Sample flow test				
Outlet mass flow (L/min)				
Pressure (kPa)				
Pass/Fail				
GSS leak test				
Mass flow (L/min)				
Corrected mass flow (L/min)				
Pressure (kPa)				
Corrected pressure (kPa)				
Pass/Fail				
No flow test				
Mass flow (L/min)				
Pressure (kPa)				
Max flow test				
Outlet mass flow (L/min)				
Pressure (kPa)				

Comments:

Appendix C

NAEMS Open S-OPS Acceptance Form

Date: _____/____/_____

GSS S/N _________________

Location: ____________________

FOS: ____________________

	Solenoid 2	Solenoid 3	Solenoid 5	Solenoid 7
Max flow test				
Inlet mass flow (L/min)				
Outlet mass flow (L/min)				
Pressure (kPa)				
Pass/Fail				
S-OPS/ GSS Leak test				
Mass flow (L/min)				
Corrected mass flow (L/min)				
Pressure (kPa)				
Corrected pressure (kPa)				
Pass/Fail				
S-OPS S/N				

Comments:

Appendix D
Synthetic Open-Path System Inlet Flow Verification Form

S-OPS S/N ______________________ Date: ______/______/______

FOS: _________________________

Inlet	**S-OPS inlet flow rate (L/min)**	**Deviation[1]**	**Action (clean, replace..)**
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
Mean			

1: Deviation = (Inlet flow-Mean flow)/Mean flow

Appendix E

S-OPS/ GSS Bag Test (System Adsorption)

S-OPS: __________________ Date: ____/_______/________

GSS S/N: ________________ FOS: ____________________

Gas: H2S Analyzer: TFS 450I s/n:						
			Diluter/cylinder	**Analyzer**	**Average from**	
Time		**Sampling**	**Reading**	**Reading**	**Data File**	**Percent**
Start	**Stop**	**Location**	**ppm**	**ppm**	**ppm**	**Difference**
		S-OPS 1				
		S-OPS 2				
		S-OPS 3				
		S-OPS 4				
		S-OPS 5				
		S-OPS 6				
		S-OPS 7				
		S-OPS 8				
		S-OPS 9				
		S-OPS 10				
		Ambient				
Average						

Gas: Methane Analyzer: Innova 1412 s/n:						
			Diluter/cylinder	**Analyzer**	**Average from**	
Time		**Sampling**	**Reading**	**Reading**	**Data File**	**Percent**
Start	**Stop**	**Location**	**ppm**	**ppm**	**ppm**	**Difference**
		S-OPS 1				
		S-OPS 2				
		S-OPS 3				
		S-OPS 4				
		S-OPS 5				
		S-OPS 6				
		S-OPS 7				
		S-OPS 8				
		S-OPS 9				
		S-OPS 10				
		Ambient				
Average						

S-OPS/ GSS Bag Test (System Adsorption) (Continued)

S-OPS: _________________ Date: _____/______/________

GSS S/N: _______________ FOS: ___________________

Gas: Methanol Analyzer: Innova 1412 s/n:

			Diluter/Cylinder	**Analyzer**	**Average from**	
Time		**Sampling**	**Reading**	**Reading**	**Data File**	**Percent**
Start	**Stop**	**Location**	**ppm**	**ppm**	**ppm**	**Difference**
		S-OPS 1				
		S-OPS 2				
		S-OPS 3				
		S-OPS 4				
		S-OPS 5				
		S-OPS 6				
		S-OPS 7				
		S-OPS 8				
		S-OPS 9				
		S-OPS 10				
		Ambient				
Average						

Gas: Ethanol Analyzer: Innova 1412 s/n:

			Diluter/cylinder	**Analyzer**	**Average from**	
Time		**Sampling**	**Reading**	**Reading**	**Data File**	**Percent**
Start	**Stop**	**Location**	**ppm**	**ppm**	**ppm**	**Difference**
		S-OPS 1				
		S-OPS 2				
		S-OPS 3				
		S-OPS 4				
		S-OPS 5				
		S-OPS 6				
		S-OPS 7				
		S-OPS 8				
		S-OPS 9				
		S-OPS 10				
		Ambient				
Average						

S-OPS/ GSS Bag Test (System Absorption) (Continued)

S-OPS: __________________ Date: _____/______/________

GSS S/N: ________________ FOS: ____________________

Gas: Propane Analyzer: Innova 1412 s/n:

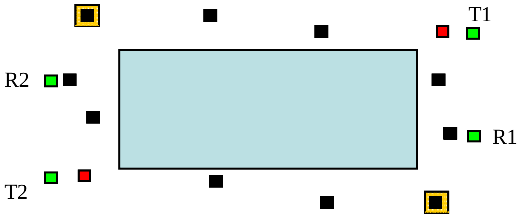
			Diluter/cylinder	**Analyzer**	**Average from**	
Time		**Sampling**	**Reading**	**Reading**	**Data File**	**Percent**
Start	**Stop**	**Location**	**ppm**	**ppm**	**ppm**	**Difference**
		S-OPS 1				
		S-OPS 2				
		S-OPS 3				
		S-OPS 4				
		S-OPS 5				
		S-OPS 6				
		S-OPS 7				
		S-OPS 8				
		S-OPS 9				
		S-OPS 10				
		Ambient				
Average						

Gas: Ammonia Analyzer: Innova 1412 s/n:

			Diluter/cylinder	**Analyzer**	**Average from**	
Time		**Sampling**	**Reading**	**Reading**	**Data File**	**Percent**
Start	**Stop**	**Location**	**ppm**	**ppm**	**ppm**	**Difference**
		S-OPS 1				
		S-OPS 2				
		S-OPS 3				
		S-OPS 4				
		S-OPS 5				
		S-OPS 6				
		S-OPS 7				
		S-OPS 8				
		S-OPS 9				
		S-OPS 10				
		Ambient				
Average						

Appendix F

Lagoon/Basin Site Open-path Sensor Installation Report



Location: ______________________

Date: ______/______/_______

FOS: ________________________________

NEAR S-OPS with 500’ extension tubing

S-OPS Path #1 start location (T1) ______Deg (N) ____.____Min Deg (W) ______.__ Min

S-OPS Path #1 end location (R1) ______Deg (N) ____.____Min Deg (W) ______.__ Min

Distance between Path #1 outlet tube and GSS manifold (extension tubing): _______ m

Height of Path #1 above lagoon berm : _______________________ m

---

FAR S-OPS with 1000’ extension tubing

S-OPS Path #2 start location (T2) ______Deg (N) ____.____Min Deg (W) ______.__ Min

S-OPS Path #2 end location (R2) ______Deg (N) ____.____Min Deg (W) ______.__ Min

Distance between Path #2 outlet tube and GSS manifold (extension tubing):________ m

Height of Path #2 above lagoon berm : _______________________ m

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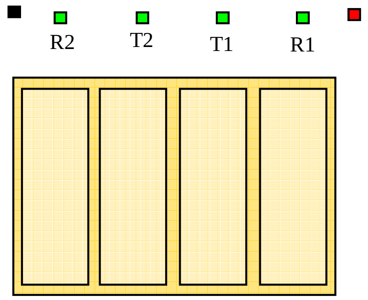
Notes:

Corral Site Open-path Sensor Installation Report

Location: ______________________

Date: ______/______/_______

FOS: ________________________________



NEAR S-OPS with 500’ extension tubing

S-OPS Path #1 start location (T1) ______Deg (N) ____.____Min Deg (W) ______.__ Min

S-OPS Path #1 end location (R1) ______Deg (N) ____.____Min Deg (W) ______.__ Min

Distance between Path #1 outlet tube and GSS manifold (extension tubing): _______ m

Height of Path #1 above corral: _______________________ m

FAR S-OPS with 1000’ extension tubing

S-OPS Path #2 start location (T2) ______Deg (N) ____.____Min Deg (W) ______.__ Min

S-OPS Path #2 end location (R2) ______Deg (N) ____.____Min Deg (W) ______.__ Min

Distance between Path #2 outlet tube and GSS manifold (extension tubing):________ m

Height of Path #2 above corral: _______________________ m

Notes:

Appendix G

Flow Direction Sensor Calibration

GSS S/N ____________________

Date: ________________________

Operator: ____________________

	Solenoid 2	Solenoid 3	Solenoid 5	Solenoid 7
Flow				
Sample flow rate (L/min)				
Reverse flow rate (L/min				
Δ Flow (L/min)				
Sensor Response				
Sample flow				
Reverse flow				
Δ response				
Pass/Fail				

MANAGEMENT OF OPEN-SOURCE, WEATHER, AND LAGOON-CHARACTERIZATION DATA
Standard Operating Procedure (SOP) D1

MANAGEMENT OF OPEN-SOURCE, WEATHER, AND LAGOON-CHARACTERIZATION DATA

Standard Operating Procedure (SOP) D1

Prepared by
Richard H. Grant

Reviewed by
Bill W. Bogan, Ji-Qin Ni, and Albert J. Heber

Effective Date: November 6, 2006

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
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Table of Contents

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1. Scope and Applicability

1.1. Careful management of open-source, weather, and lagoon-characterization data is required to ensure that these data are all accurately documented and preserved.

1.1.1. Data generated with the Tunable Diode Laser Absorption Spectrometer (TDLAS) is needed to properly apply the radial plume mapping method, and thus to document the changing ammonia concentrations of the air surrounding the open-air source.

1.1.2. Data generated by Ultraviolet Differential Optical Absorption Spectroscopy (UV-DOAS) is needed to properly apply the backward Lagrangian Stochastic emissions model, and thus to document the changing ammonia and hydrogen sulfide concentrations of the air surrounding the open-air source

1.1.3. Data generated by INNOVA Model 1412 Photoacoustic Multi-gas Monitor (SOP G7) to quantitate total and individual volatile organic compounds (VOCs) from the air surrounding the open-air source needs to be properly documented.

1.1.4. Data on meteorological and lagoon conditions are needed for valid modeling of the effect of weather and microclimate on emissions.

1.2. The data management regime must be capable of managing data collected during multiple measurement periods, and at multiple study locations.

2. Summary of Method

The objective of data management is to ensure data security, easy data identification and retrieval, and efficient publication. All project-related files and data must be classified and organized in the computerized research data storage system. The tools to deliver, backup, inspect, analyze and present data must be available and kept current with the latest updates.

3. Definitions

3.1.	bLS	Backward Lagrangian Stochastic
3.2.	CD	Compact disk
3.3.	DM	Data Manager
3.4.	DVD	Digital video disk
3.5.	Flash memory	Non-volatile rewritable memory
3.6.	FOS	Field operations staff
3.7.	HD	Computer hard drive
3.8.	ODBC	Open data base connectivity
3.9.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.10.	PAML	Purdue Applied Meteorology Laboratory
3.11.	PIC	Path-integrated concentration
3.12.	PM	Project Manager
3.13.	ppb	Parts per billion (by volume)
3.14.	QAM	Quality Assurance Manager
3.15.	QAPP	Quality Assurance Project Plan

3.16. RPM	Radial Plume Mapping method of emissions measurement
3.17. Site PI	Site Principal Investigator
3.18. SMP	Site monitoring plan
3.19. LAN Server	Local Area Network Sever computer
3.20. TDLAS	Tunable Diode Laser Absorption Spectrometer/Spectroscopy
3.21. UV-DOAS	Ultraviolet Differential Optical Absorption Spectrometer/ Spectroscopy
3.22. VOC	Volatile organic compounds
3.23. WXL	Weather and lagoon

4. Personnel Qualifications and Responsibilities

4.1. Most of these procedures assume a familiarity with the use of personal computers and general computer application software (e.g. MS Office), as well as the following:
  4.1.1. UV-DOAS procedures (SOP C1) and the WINDTRAX bLS software (SOP O1)
  4.1.2. The RPM software (SOP O2) and the TDLAS procedures (SOP C2)
  4.1.3. INNOVA 1412 Photoacoustic Multi-gas Monitor procedure (SOP G7)
  4.1.4. The data analysis software programs which are used to perform the transformations described in SOPs W1-W5
  4.1.5. The data acquisition and control software described in SOP W6
4.2. The site PI will follow an orderly system of data recording, organization, and safeguards, and will ensure that all members of the research team (including appropriate administrative personnel) understand and follow the system.
4.3. Data access management will be controlled according to Purdue University (Information Technology at Purdue) procedures defined for sensitive data (Appendix A). Those authorized to access research data will use strong passwords that are difficult to crack, according to Appendix B. Passwords will not be written down or shared with others. Passwords will be changed on a regular basis (every two months at a minimum).
4.4. Only those people explicitly authorized to access the research data will be allowed to write to the files (Appendix A).

5. Equipment and supplies

5.1. Data transfer pouch
5.2. UV-DOAS 4-Gb flash memory drives (2 per measurement period)
5.3. 64Mb CompactFlash® memory card (1 per measurement period)
5.4. 256Mb flash memory sticks (3 per measurement period)
5.5. Blank CDs
5.6. Fire-proof data vault
5.7. Innova Gas Monitor Software (Version 7304)

6. Procedures

6.1. Classification of electronic files
  6.1.1. Classify all electronic files according to the following four categories:

6.1.1.1. Research information and data. These files, if lost, cannot be rebuilt or recovered.
  6.1.1.1.1. Measurement data
  6.1.1.1.2. Data acquisition configuration data
  6.1.1.1.3. Calibration data forms
  6.1.1.1.4. Data logged by instruments (TDLAS and UV-DOAS) with internal data storage
  6.1.1.1.5. Calibration data recorded by TDLAS and UV-DOAS systems, and electronic calibration forms
  6.1.1.1.6. Data recorded by INNOVA 1412 Photoacoustic multi-gas monitor
  6.1.1.1.7. Field notes
  6.1.1.1.8. Email messages
  6.1.1.1.9. Image files (digital photos or graphs)

6.1.1.2. Project-specific configurations that could be rebuilt or recovered, if lost
  6.1.1.2.1. Hardware configuration files
  6.1.1.2.2. Configurations of firewalls, virus protection, etc
  6.1.1.2.3. Email accounts, address books, message rules
  6.1.1.2.4. Favorite lists

6.1.1.3. Project-specific software and documents
  6.1.1.3.1. Thunder Scientific WINDTRAX
  6.1.1.3.2. Boreal Laser GasView2
  6.1.1.3.3. Arcadis RPM
  6.1.1.3.4. INNOVA Gas Monitor Software 7304
  6.1.1.3.5. Campbell Scientific LoggerNet data logger programming and collection software
  6.1.1.3.6. National Instruments LabVIEW
  6.1.1.3.7. Files provided by vendors, such as instrument manuals and application notes
  6.1.1.3.8. Instrument communication software

6.1.1.4. General PC Software
  6.1.1.4.1. Microsoft Windows XP
  6.1.1.4.2. Microsoft Office
  6.1.1.4.3. WS_FTP server and ftp encryption software
  6.1.1.4.4. PC Anywhere
  6.1.1.4.5. Network Associates Enterprise
  6.1.1.4.6. Spybot S&D
  6.1.1.4.7. Genie Backup Manager

6.2. Data acquisition of UV-DOAS PIC spectra (SOP O1) for NH_3 and H_2S concentration calculation is conducted by the on-site ‘bLS’ computer (Fig. 1).

6.2.1. Data is stored in a 4Gb flash memory drive on each UV-DOAS receiver. After 20 days, memory used is approximately 500Mb.

6.3. Data acquisition of TDLAS PIC spectra (SOP O2) for NH_3 concentration calculations is conducted by each of the on-site ‘TDLAS/scanner’ computers (Fig. 2).

6.3.1. Data is stored in the HD of each TDLAS/scanner computer. After 20 days, memory used is approximately 3.3Mb.

6.4. Data acquisition using INNOVA 1412 Photoacoustic multi-gas monitor (SOP G7) for total and individual VOC concentrations stored directly in the HD of the LAN computer (Fig. 3).

6.5. Data acquisition of lagoon temperature, pH, and redox state (SOPs L1, L2 and L3), barometric pressure, air temperature, relative humidity, solar radiation, surface wetness, and data logger power supply voltage (SOPs E3, W1, W3, and W5) is conducted by a datalogger (SOP W6) (Fig. 4).

6.5.1. Data is stored in a first in first out (FIFO) memory, and on a 64Mb CompactFlash® memory card. After 20 days, memory used is approximately 64Kb.

6.6. Data acquisition of wind components at three heights (SOP W2) is conducted by the on-site LAN Server computer (Fig. 4).

6.7. Data processing for the parameters listed in Section 6.2 is conducted by the on-site ‘bLS’ computer, which is connected to the UV-DOAS system by radio communications (Fig. 1).

6.8. Data processing for the parameters listed in Section 6.3 is conducted by the on-site LAN server computer, which is connected to the two ‘TDLAS/scanner’ computers by Ethernet (Fig. 2).

6.9. Data processing for the parameters listed in Section 6.4, and control of the INNOVA Multi-gas Monitor, are conducted by the Gas Monitoring Software 7304 running in the on-site LAN Server computer, which is connected by RS 232 cable to the INNVOA 1412 (Fig. 3).

6.10. Data processing for the parameters listed in Section 6.5 is conducted by the on-site Network Server computer, which is connected to the datalogger by radio communications.

6.11. File organization

6.11.1. All project-specific files on computers will be in folders called *YY*farm*n*, where *YY* is a two-letter abbreviation for the state and *n* is the farm identifier within the state. For example, two farms in Missouri would be identified as MOfarm1 and MOfarm2.

6.11.2. The flash memory drive of each UV-DOAS instrument (Fig. 1) contains:

6.11.2.1. Raw 5-min average PIC spectra [MinMinHrHr, PIC NH_3, PIC H_2S]

6.11.2.2. Spreadsheet of 5-min PIC averages (filename ‘C’MMDDYY [MinMinHrHr] containing: background PIC, SignalStrength, IntegrationTime, PIC NH_3, NH_3 measurement error, PIC H_2S, H_2S measurement error, RMSnoise)

6.11.3. The datalogger flash memory (Fig. 4) contains:

6.11.3.1. Raw data files (named by MMDDYY, containing farm number, MMDDYY, HrHrMinMin, 5-min averages of lagoon temperature, pH, and redox state, barometric pressure, air temperature, relative humidity, solar radiation, surface wetness, and power supply voltage) (SOP W6)

6.11.4. In the on-site computer associated with both UV-DOAS systems (Fig. 1), termed the ‘bLS computer’, store all project date-specific measurement files in folders called MMDDYY where MM is numerical month, DD is numerical day in month, and YY is a two-letter abbreviation for the year in which the WINDTRAX bLS program was started. Organize the subfolders as:

6.11.4.1. Program. WINDTRAX bLS software

6.11.4.2. Config. Contains configuration files for bLS at location *YY*farm*n*.

6.11.4.3. Touchdown tables (See SOP O1) for *YY*farm*n*

6.11.4.4. 5-min 2-m height wind turbulence statistics (SOP W2) for *YY*farm*n* so that statistics coincide with periods of PIC measurements according to SOP O1.

6.11.4.4.1. Wind data is transferred from the LAN server computer to the UV-DOAS systems through the Ethernet connection.

6.11.4.5. UV-DOAS Instrument 1 data for location *YY*farm*n*

6.11.4.5.1. Raw 5-min average PIC spectra [MinMinHrHr, PIC NH_3, PIC H_2S

6.11.4.5.2. Spreadsheet of 5-min PIC averages (filename 'C'MMDDYY [MinMinHrHr] containing: background PIC, SignalStrength, IntegrationTime, PIC NH_3, NH_3 measurement error, PIC H_2S, H_2S measurement error, RMS noise)

6.11.4.6. UV-DOAS Instrument 2 data for location *YY*farm*n*

6.11.4.6.1. Raw 5-min average PIC spectra [MinMinHrHr, PIC NH_3, PIC H_2S

6.11.4.6.2. Spreadsheet of 5-min PIC averages (filename 'C'MMDDYY [MinMinHrHr] containing: background PIC, SignalStrength, IntegrationTime, PIC NH_3, NH_3 measurement error, PIC H_2S, H_2S measurement error, RMSnoise)

6.11.4.7. WINDTRAX input spreadsheet for location *YY*farm*n*

6.11.4.7.1. 5-min average difference in PICs and wind data (filename MMDDYY [MinMinHrHr]; containing: turbulence statistics, PIC NH_3, PIC H_2S)

6.11.4.8. WINDTRAX output file for location *YY*farm*n* (filename MMDDYY [MinMinHrHr]

6.11.5. In the on-site data acquisition computer associated with each TDLAS/scanner system (each of 2 computers and associated systems, located according to Fig. 2), The two TDLAS/scanner systems are designated TDLAS1 and TDLAS2. All date-specific measurement files are in folders called MMDDYY where MM is numerical month, DD is numerical day in month, and YY is a two-letter abbreviation for the year in which the RPM program was started. Organize the subfolders as:

6.11.5.1. Config1. Contains all files associated with first plane of measurement for RPM analysis of TDLAS

6.11.5.1.1. Config. Contains geometry configuration file

6.11.5.1.2. Input file. Includes PIC values for each time period of averaging

6.11.5.1.2.1. Format according to SOP O2.

6.11.5.1.3. Plume map graphs. Contains each averaging period plume map from the RPM program (See SOP O2) as a jpeg file (70Kb/image)

6.11.5.1.4. Flux output file. Contains flux measurements with QA/QC checks on the RPM measurement according to SOP O2

6.11.5.1.5. Valid raw wind data

6.11.5.1.5.1. Format according to SOP W2.

6.11.5.1.5.2. Data is transferred from the LAN server computer through the Ethernet connection.

6.11.5.1.6. Parsed wind data so that the measurement period of wind data coincides with the measurement period of each complete cycle of PIC measurements.

6.11.5.1.7. Raw PIC measurements from TDLAS

6.11.5.1.7.1. Beam1. PIC values along beamline 1 with QA/QC flags (SOP C2).

6.11.5.1.7.2. Beam2. PIC values along beamline 2 with QA/QC flags (SOP C2).

            6.11.5.1.7.3. Beam3. PIC values along beamline 3 with QA/QC flags (SOP C2).
            6.11.5.1.7.4. Beam4. PIC values along beamline 4 with QA/QC flags (SOP C2).
            6.11.5.1.7.5. Beam5. PIC values along beamline 5 with QA/QC flags (SOP C2).
    6.11.5.2. Config2. Contains all files associated with second plane of measurement for RPM analysis of TDLAS
        6.11.5.2.1. Config. Contains geometry configuration file
        6.11.5.2.2. Input file. Includes PIC values for each time period of averaging
            6.11.5.2.2.1. Format according to SOP O2.
        6.11.5.2.3. Plume map graphs. Contains each averaging period plume map from the RPM program (SOP O2) as a jpeg file (70Kb/image)
        6.11.5.2.4. Flux output file. Contains flux measurements with QA/QC checks on the RPM measurement according to SOP O2
        6.11.5.2.5. Valid raw wind data
            6.11.5.2.5.1. Format according to SOP W2.
            6.11.5.2.5.2. Data is transferred from the LAN server computer
        6.11.5.2.6. Parsed wind data so that the measurement period of wind data coincides with the measurement period of each complete cycle of PIC measurements
        6.11.5.2.7. Raw PIC measurements from TDLAS
            6.11.5.2.7.1. Beam6. PIC values along beamline 6 with QA/QC flags (SOP C2).
            6.11.5.2.7.2. Beam7. PIC values along beamline 7 with QA/QC flags (SOP C2).
            6.11.5.2.7.3. Beam8. PIC values along beamline 8 with QA/QC flags (SOP C2).
            6.11.5.2.7.4. Beam9. PIC values along beamline 9 with QA/QC flags (SOP C2).
            6.11.5.2.7.5. Beam10. PIC values along beamline10 with QA/QC flags (SOP C2).

6.11.6. In the on-site INNOVA data acquisition computer (which is also the on-site LAN server) organize the subfolders in the C drives as :
    6.11.6.1. C:\INNOVA (for all single point data acquisition and control)
    6.11.6.2. \Data acquisition program (Gas Monitoring Software7304)
    6.11.6.3. \Configuration file (common for all measurement locations)
    6.11.6.4. Data collected from the Gas Monitor Software 7304 (SOP G7) by the LAN server will be saved in the directory [C:\INNOVA\Farm number\MMDDYY\ODBC] (SOP G7).
    6.11.6.5. 5-min raw sample values will be stored on the LAN server in MS-access format (ODBC).
    6.11.6.6. Data includes: Measurement times, measurement sequence numbers, Error and user flags, atmospheric pressure, water vapor, NH_3 concentration, acetic acid concentration, methanol concentration, ethanol concentration.

6.11.7. In the <u>on-site weather data acquisition computer (which is also the on-site LAN Server)</u> organize the subfolders in the C drive as:
    6.11.7.1. LabVIEW files (for wind sensor data acquisition and control)
        6.11.7.1.1. Data acquisition program
        6.11.7.1.2. Configuration file (common for all measurement locations)
        6.11.7.1.3. Wind data files for *YY*farm*n* (See SOP W2)
            6.11.7.1.3.1. File with 30 min of raw 16-Hz data for spectral analysis (including U, V, W, T_v)
            6.11.7.1.3.2. 5-min average statistics

6.11.7.1.3.3. 30-min average statistics

6.11.7.2. LoggerNet® datalogger software directories and subdirectories according to LoggerNet manual (page 2-3) for data collection and datalogger control of weather and lagoon characterization sensors (Section 5.2 of manual).

6.11.7.2.1. Communication logs

6.11.7.2.2. System. Includes binary files, configuration files and raw data downloads collected by the data acquisition program named as [MMDDYY-*YY*farm*n*]

6.11.7.2.3. PakBusGraph. Contains daily plots of raw data.

6.11.7.2.3.1. Data files for *YY*farm*n* (named by date MMDDYY, containing HrHrMinMin, 5-min averages of lagoon temperature, pH, and redox state, barometric pressure, air temperature, relative humidity, solar radiation, surface wetness, and power supply voltage)

6.11.7.3. Datalogger configuration and program editors

6.11.7.4. Library directory containing compilers and definition files

6.11.7.5. Calibration. Contains calibration sheets, gas cylinder logs, control charts, and other records

6.11.7.6. QAPP. The latest copy of the Quality Assurance Project Plan

6.11.7.7. SOP. Latest copies of all relevant Standard Operating Procedures

6.11.7.8. SMP. Site monitoring plan

6.11.7.9. Certifications

6.11.7.10. Measurement period records. Indicates dates and locations

6.11.7.11. Field Notes

6.11.7.11.1. Typed logs of activities and observations (All notes must include name and date of the person making the note)

6.11.7.11.2. Producer Event forms and updated farm specific information (layout drawings etc) other farm lagoon management data (information on animals, weight, feed, etc) collected from the farm manager (SOP S7)

6.11.7.12. Photos. Digital pictures of field setup and observations

6.11.7.13. Email. Contains email messages and attachments. Name the email account yyfarmn@zzz.zzz where zzz.zzz is the internet service provider's domain. Outlook Express is the preferred email program.

6.11.7.14. Miscellaneous

6.11.7.14.1. Site equipment inventory including gas cylinders, etc

6.11.7.14.2. Transported equipment inventory

6.11.8. The administrative server at PAML contains the following folders and information, and operates various personal computers:

6.11.8.1. Weather data from outside sources for each day and every location that measurements are taken. Example of an outside source might be; http://www.wunderground.com/us/nc/elizabethtown.html.

6.11.8.2. Data ftped from *YY*farm*n*

6.11.8.3. UV-DOAS and bLS data and configuration files

6.11.8.4. TDLAS and RPM data and configuration files

6.11.8.5. Excel files of text data exported from bLS and RPM directories during processing

6.11.8.6. LoggerNet configuration files

6.11.8.7. INNOVA Gas Monitor Software 7304 configuration files
6.11.8.8. LabVIEW configuration files
6.11.8.9. Excel files of data exported from LoggerNet during processing
6.11.8.10. Excel files of data exported from LabVIEW during processing.
6.11.8.11. AutoCad drawings of lagoon and equipment
6.11.8.12. Data tables for reports and manuscripts
6.11.8.13. Farm production data
6.11.8.14. Reports
6.11.8.15. ftp transfer logs

6.12. Data security

6.12.1. The on-site computer associated with the UV-DOAS (Fig. 1) is linked through an Ethernet local area network to the Network Server computer.

6.12.2. Each on-site data acquisition computer linked with each TDLAS instrument is linked to the Network server computer in the trailer through an Ethernet local area network (SOP U4).

6.12.3. Datalogger

6.12.3.1. Three 5-digit security passwords are needed to change the data acquisition program, download data, or change the clock.

6.12.3.1.1. FOS will each have all security codes.

6.12.3.2. Replace 1Mb flash memory at the end of each measurement period (identified here as WXL).

6.12.3.2.1. Put flash memory into the zippered Data pouch.

6.12.4. LoggerNet will be operated as a service on the on-site computer, so that the program will restart after power failures.

6.12.5. Innova Gas Monitoring Software 7304 shortcut will be placed in the start up folder so the program will restart after power failure or restart.

6.12.6. Program Windows “Scheduled Tasks” to ftp the 24-h data files from the on-site weather data acquisition computer, Innova Gas Monitoring software, and the data acquisition computers linked to the TDLAS instruments and UV-DOAS instruments to the PAML computer at midnight.

6.12.6.1. All ftp file transfers are encrypted.

6.12.7. Collect archived raw data from flash drives in each UV-DOAS instrument (approximately 500MB of data on each of the 4GB flash drives) at the conclusion of the measurement period.

6.12.7.1. Replace the flash drive in each instrument.

6.12.7.2. Copy content of each UV-DOAS flash drive onto two CDs.

6.12.7.2.1. Identify CD with location (*YY*farm*n*), measurement period (MM-DD-YY to MM-DD-YY), “UVDOAS1” for “UVDFlash1” and “UVDOAS2” for “UVDFlash2” and label “vault” or “carry”.

6.12.7.2.2. Copy using LAN Server computer

6.12.7.2.3. Put one copy of each of the two CDs into the fire-proof data vault in the trailer.

6.12.7.2.4. Put the other copy of each CD in Data pouch.

6.12.7.3. Store all data-containing UV-DOAS Flash drives in Data pouch.

- 6.12.8. Field notes
  - 6.12.8.1. Notes are stored on the HD of the LAN server computer, and are (automatically) backed up daily on an external HD.
  - 6.12.8.2. Email field notes to PM and DM at the end of each work day in the field, before leaving the site.
  - 6.12.8.3. Download field notes onto flash memory stick.
  - 6.12.8.4. Store memory stick in the zippered Data pouch.
- 6.12.9. FOS copies of calibration data and control charts
  - 6.12.9.1. Email to PM, DM, and QAM after each 20-day measurement period.
  - 6.12.9.2. Retain one paper copy in trailer.
  - 6.12.9.3. Download onto flash memory stick with field notes.
  - 6.12.9.4. Store memory stick in the zippered Data pouch.
- 6.12.10. Send (by ftp) configuration files to DM whenever they are updated.
- 6.12.11. File backups by field staff
  - 6.12.11.1. Back up project folders and subfolders *YY*farm*n* of each onsite computer onto an external hard drive at 1:00 a.m. each night during the measurement period.
  - 6.12.11.2. Back up data files onto DVD at the conclusion of every measurement period.
    - 6.12.11.2.1. Two copies each of UVDOAS1, UVDOAS2 from LAN server.
    - 6.12.11.2.2. Two copies each of TDLAS 1 and TDLAS 2 from respective Data Acquisition computers.
    - 6.12.11.2.3. Two copies of INNOVA data files from LAN server
    - 6.12.11.2.4. Two copies of WXL data from LAN server
    - 6.12.11.2.5. Identify each CD with data type (above), location (*YY*farm*n*) and measurement period (MM-DD-YY to MM-DD-YY). Label one copy of each DVD as “vault”, one copy as “carry”.
    - 6.12.11.2.6. Put “vault” copy of DVD into the fire-proof data vault.
    - 6.12.11.2.7. Put “carry” copy of DVD in the data pouch.
  - 6.12.11.3. Manually back up the operating system of any computer onto an external HD whenever significant changes are made to the system.
- 6.12.12. Automatically back up filesystem of the LAN server onto an external HD every day at 2:00 a.m.
- 6.12.13. Carry Data pouch in hand luggage upon returning to PAML.
  - 6.12.13.1. Data pouch includes:
    - 6.12.13.1.1. For latest measurement period:
      - 6.12.13.1.1.1. One pair of 4-Gb UV-DOAS flash memory drives (UVDFlash1 and UVDFlash2)
      - 6.12.13.1.1.2. One pair of CDs with copies of UV-DOAS flash memory drives (UVDOAS1 and UVDOAS2)
      - 6.12.13.1.1.3. Two CDs with TDLAS data (TDLAS1 and TDLAS2)
      - 6.12.13.1.1.4. One CD with weather,and lagoon (WXL) and INNOVA data from recently-completed measurement period
      - 6.12.13.1.1.5. One 64-Mb flash memory chip of weather and lagoon (WXL) data from recently-completed measurement period
      - 6.12.13.1.1.6. One 256-Mb memory stick with Field notes, calibration data and control charts from recently-completed measurement period

6.12.13.1.1.7. One DVD with *YY*farm*n* backup of all *YY*farm*n* directories
6.12.13.1.2. For previous measurement period (material previously stored in data vault):
6.12.13.1.2.1. One pair of CDs with copies of UV-DOAS flash memory drives (UVDOAS1 and UVDOAS2)
6.12.13.1.2.2. Two CDs with TDLAS data from the previous measurement period (TDLAS1 and TDLAS2)
6.12.13.1.2.3. One CD with weather, lagoon (WXL) and INNOVA data from previous measurement period
6.12.13.1.2.4. One DVD with *YY*farm*n* backup of all *YY*farm*n* directories
6.12.13.1.3. Electronic data transfer forms for both recently-completed and previous measurement period (Appendix C) with upper section filled out.
6.12.13.2. Deliver Data pouch to DM on arrival.
6.12.13.2.1. Document delivery of data to DM with Electronic Data Transfer Forms (Appendix C).
6.12.13.2.1.1. Forms to be stored in the folder marked 'Electronic data Transmittal records' under the control of the DM.
6.12.14. DM archives files on the PAML computer onto an external HD drive every day at 5:00 a.m.
6.12.15. DM downloads all flash memory to a separate directory on PAML computer for backup.
6.12.16. DM stores all received CDs in an off-campus location.
6.12.17. DM archives files on the PAAQL server., which is backed up daily on the Purdue University backup system.
6.13. Retention of data
6.13.1. Keep all data files for at least six (6) years.

7. Quality Control and Quality Assurance

7.1. Verify that the following software are included in their respective computers' startup menus, for automatic recovery after power failure.
7.1.1. LoggerNet, Gas Monitor Software 7304, and LabVIEW (in the on-site, LAN server, computer)
7.1.2. WINDTRAX program (in the bLS computer)
7.1.3. RPM program (in each TDLAS/scanner data acquisition computer)
7.2. Verify that all data acquisition computers log power failures, and that emails are sent to the QAM and DM upon restart.
7.3. Check field computer and relevant software, and take immediate measures to solve related problems, if the daily data file ftp, and/or emails with other files are not received.
7.4. Multiple means of irreplaceable data transfer to PAML
7.4.1. Daily ftp of files
7.4.2. Hand delivery of data from each measurement period at end of measurement period and at the end of the next sequential measurement period (stored in the data vault)
7.5. Raw data stored in multiple places prior to transfer to PAML
7.5.1. UV-DOAS raw (Fig. 1)
7.5.1.1. Flash memory drive of each UV-DOAS instrument

    - 7.5.1.2.bLS computer HD
  - 7.5.2. TDLAS raw and RPM processed data (Fig. 2)
    - 7.5.2.1.TDLAS data acquisition computer HD
    - 7.5.2.2.External HD
  - 7.5.3. bLS computer filesystems (including wind measurements and bLS input files and results) (Fig. 1)
    - 7.5.3.1.bLS computer HD
    - 7.5.3.2.External HD
  - 7.5.4. INNOVA raw data (Fig. 3)
    - 7.5.4.1.LAN server HD
    - 7.5.4.2.External HD
  - 7.5.5. Datalogger raw data (excluding wind measurements) (Fig. 4)
    - 7.5.5.1. CompactFlash memory card
    - 7.5.5.2. On-site datalogger
    - 7.5.5.3. LAN server HD
    - 7.5.5.4. External HD
  - 7.5.6. LAN Server computer filesystems (including wind measurements) (Figs. 1-4)
    - 7.5.6.1. HD of on-site computer
    - 7.5.6.2. External HD
- 7.6. DM tracks all modifications to electronic files. This includes tracking the original data entered, tracking who and when an individual or system viewed or changed any data, and recording why data was changed.
  - 7.6.1. Save and archive all raw data in their original form.
  - 7.6.2. Use a “notes” worksheet in Excel files to describe modifications.
  - 7.6.3. Include date at the end of file name in the following format: MM-DD-YY, ex: 01-09-06. Leading zeros in day and month can be omitted, ex. 1-9-06.
  - 7.6.4. Utilize electronic audit trails as much as possible.

8. References

8.1. Campbell Scientific Inc. 2005. LoggerNet Instruction Manual, v. 3.2, Campbell Scientific Inc., Logan UT.
8.2. Campbell Scientific Inc. 2006. CR1000 Measurement and Control System Operators Manual. v. 3/06, Campbell Scientific Inc., Logan, UT
8.3. Information Technology at Purdue. 2006. Electronically Stored (Computer-based) Information. Available at: http://www.itap.purdue.edu/security/procedures/dataHandling/electrStored.cfm. Accessed 2-28-2006.
8.4. Information Technology at Purdue. 2006. Password Guidelines. Available at: http://www.itap.purdue.edu/security/procedures/passGuidelines.cfm. Accessed 2-28-2006.
8.5. SOP C1. 2006. Open Path Measurement of Ammonia and Hydrogen Sulfide with the Cerex UV Sentry Ultraviolet Differential Optical Absorption Spectrometer. Standard Operating Procedure C1. Purdue Applied Meteorology Lab.

8.6. SOP C2. 2006. Measurement of Ammonia with the Boreal Laser GasFinder Tunable Diode Laser Absorption Spectrometer (TDLAS) Standard Operating Procedure C2. Purdue Applied Meteorology Lab.
8.7. SOP E3. 2006. The Measurement of Solar Radiation with the Licor Model 200SL or 200SZ Silicon Pyranometer. Standard Operating Procedure E3. Purdue Ag Air Quality Lab.
8.8. SOP G7. 2006. Use of the INNOVA 1412 Photoacoustic Multi-gas Monitor. Standard Operating Procedure G7. Purdue Ag Air Quality Lab.
8.9. SOP L1. 2006. Measurement of Lagoon pH with Innovative Sensors Model CSIM11 Sensor. Standard Operating Procedure L1. Purdue Applied Meteorology Lab.
8.10. SOP L2. 2006. Measurement of Lagoon Temperature with Campbell Scientific Model 107-L Thermistor. Standard Operating Procedure L2. Purdue Applied Meteorology Lab.
8.11. SOP L3. 2006. Measurement of Lagoon Redox State with Campbell Scientific CSIM11_ORP Sensor. Standard Operating Procedure L3. Purdue Applied Meteorology Lab.
8.12. SOP O1. 2006. Emissions Estimation Using the Thunder Beach Scientific Windtrax Backward Lagrangian Stochastic Model. Standard Operating Procedure O1. Purdue Applied Meteorology Lab.
8.13. SOP O2. 2006. Emissions Estimation Using the ARCADIS Radial Plume Mapping Model. Standard Operating Procedure O2. Purdue Applied Meteorology Lab.
8.14. SOP S7. 2006. Producer Collaborations at Area Source Monitoring Sites. Standard Operating Procedure S7. Purdue Applied Meteorology Lab.
8.15. SOP U4. 2006. Open-source Instrument Trailer. Standard Operating Procedure U4. Purdue Applied Meteorology Lab.
8.16. SOP W1. 2006. Measurement of Atmospheric Temperature and Humidity with the Vaisala Model HMP45C Sensor and Solar Shield. Standard Operating Procedure W1. Purdue Applied Meteorology Lab.
8.17. SOP W2. 2006. Measurement of Wind with the RM Young Model 81000 3-Dimensional Sonic Anemometer. Standard Operating Procedure W2. Purdue Applied Meteorology Lab.
8.18. SOP W3. 2006. Measurement of Wetness with the Campbell Scientific Model Resistance Grid. Standard Operating Procedure W3. Purdue Applied Meteorology Lab.
8.19. SOP W5. 2006. The Measurement of Barometric Pressure with the Setra Model 278 (Campbell Scientific CS100) Barometer. Standard Operating Procedure W5. Purdue Applied Meteorology Lab.
8.20. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware.. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.

Figure 1. Data Processing and Management: UV-DOAS and bLS Data.

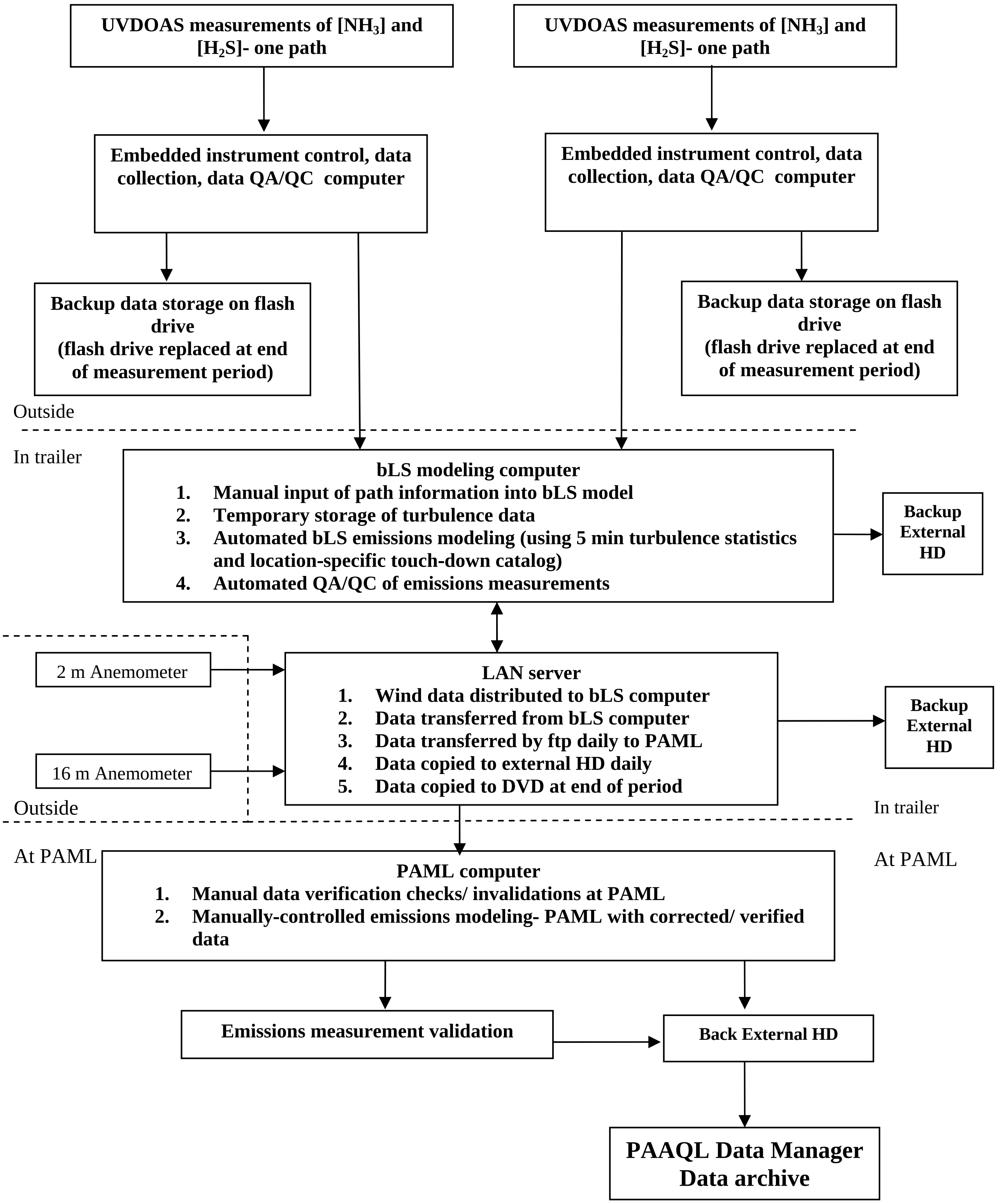


Figure 2. Data Processing and Management: TDLAS and RPM Data.

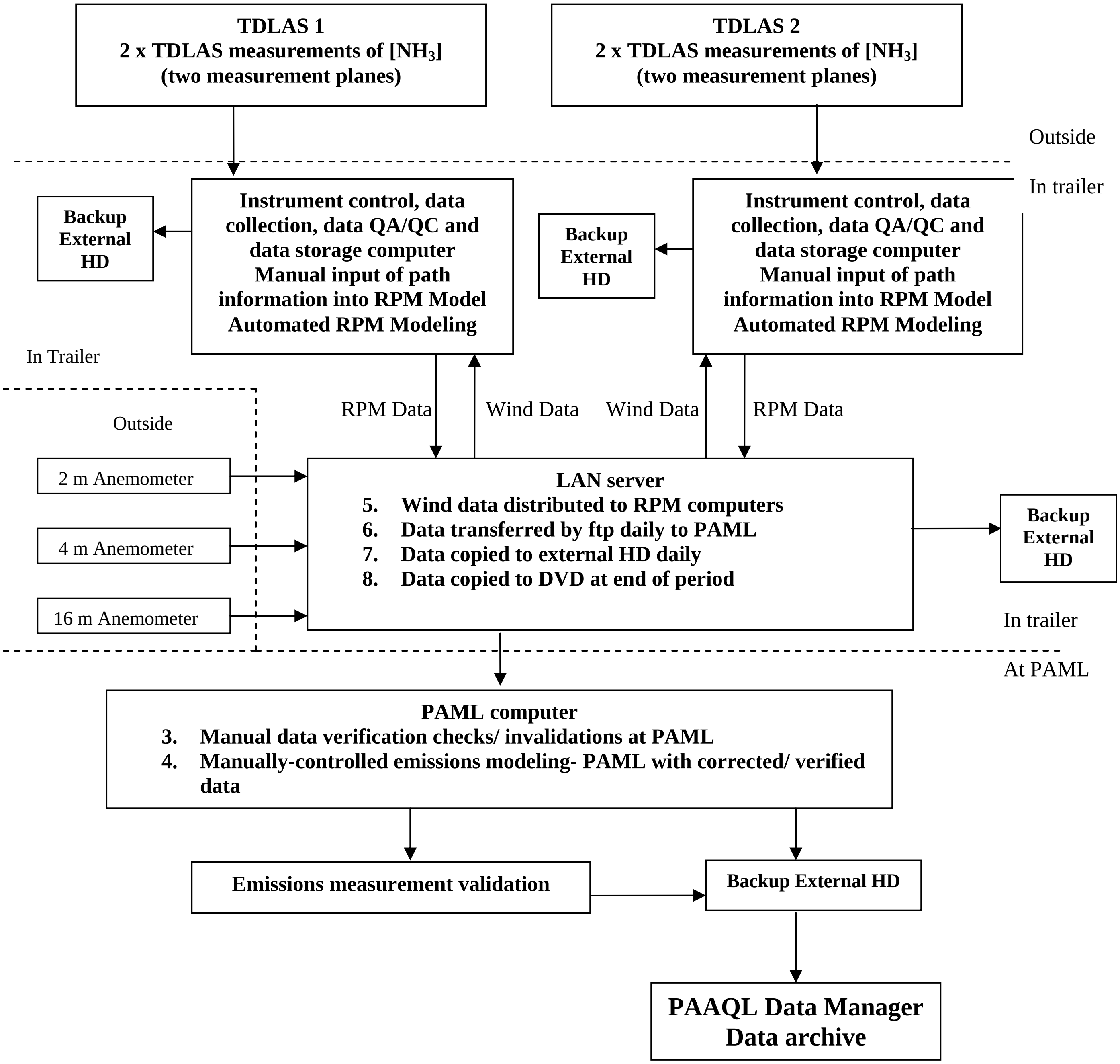
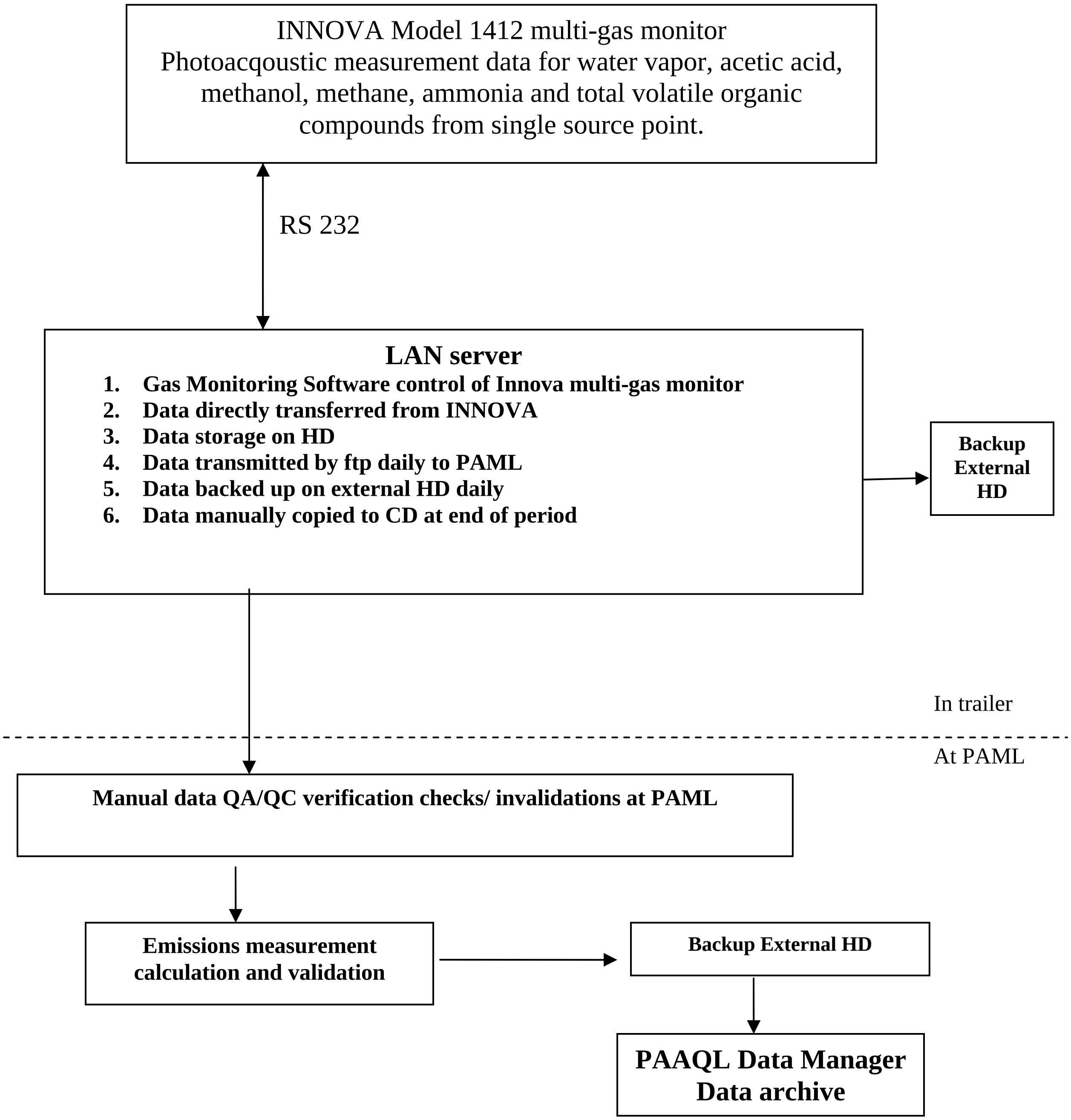


Figure 3. Data Processing and Management for INNOVA 1412.



INNOVA Model 1412 multi-gas monitor
Photoacqoustic measurement data for water vapor, acetic acid, methanol, methane, ammonia and total volatile organic compounds from single source point.

LAN server

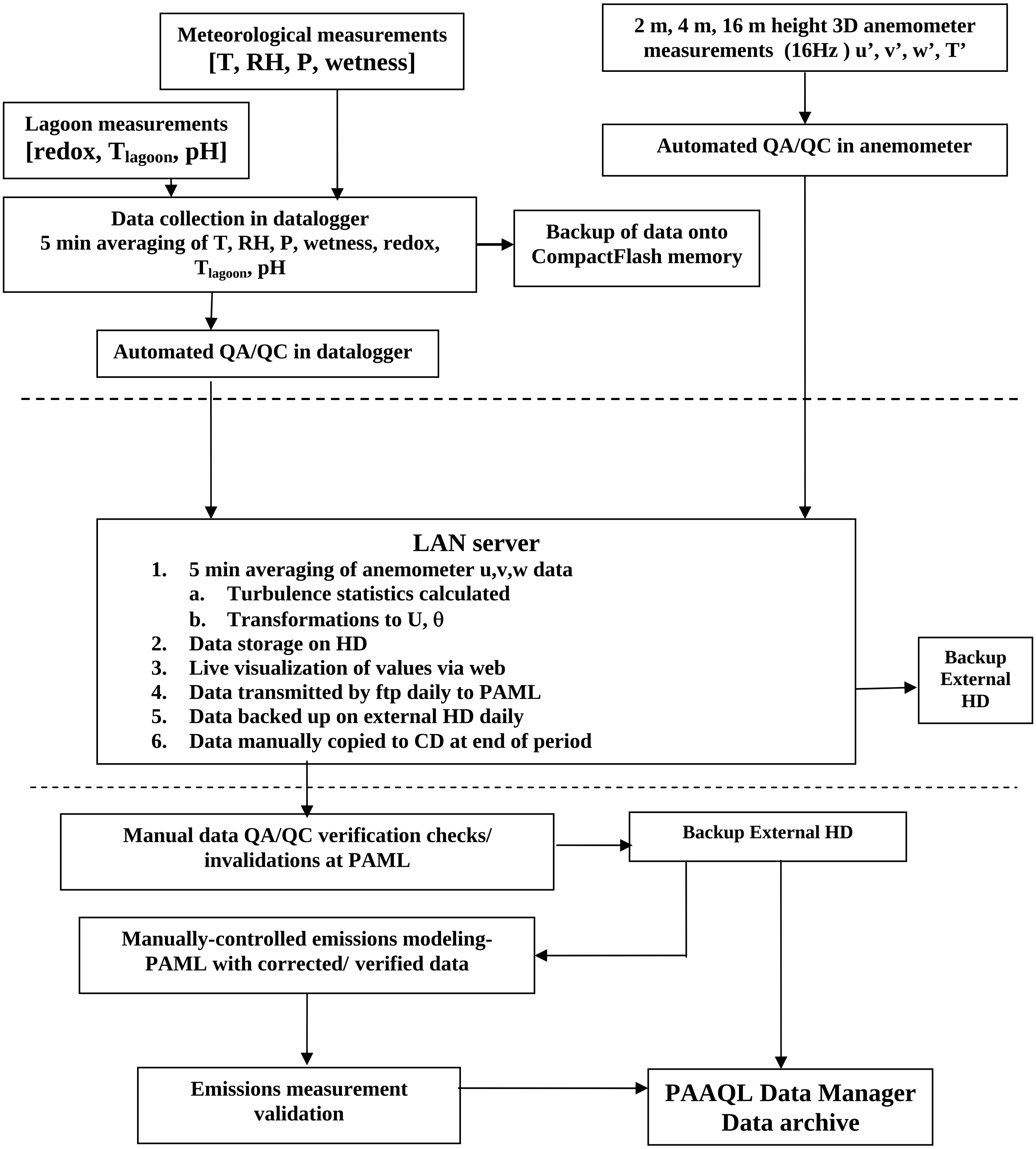
1. **Gas Monitoring Software control of Innova multi-gas monitor**
2. **Data directly transferred from INNOVA**
3. **Data storage on HD**
4. **Data transmitted by ftp daily to PAML**
5. **Data backed up on external HD daily**
6. **Data manually copied to CD at end of period**

Manual data QA/QC verification checks/ invalidations at PAML

Emissions measurement calculation and validation

PAAQL Data Manager Data archive

Figure 4. Data Processing and Management: Meteorological and Lagoon Data.



Appendix A
Guidelines for Managing Electronically-stored Sensitive Information[1]

Action	**Sensitive Information**
Storage on fixed media with access controls	No encryption required
Storage on fixed media without access controls, but accessible via the web	Not advised. If you must store data via this media, it must be encrypted.
Storage on fixed media without access controls, but not accessible via the web	No encryption required
Storage on removable media	No encryption required
Read access to information (includes duplication)	Access is based on roles defined by QAPP
Create / Update access to information	Access is based on roles defined by QAPP
Delete access to information	Access is based on roles defined by QAPP
Print hard copy report of information	Unattended printing permitted only if physical access controls are used to prevent unauthorized viewing.
Internal labeling of information at the application or screen/display level	No special requirement
Disposal of the physical electronic media device (diskettes, tapes, hard disks, etc.)	Physical destruction beyond ability to recover.
Disposal of information	Delete files through normal platform delete command, option or facility.
Data Manager & Information Owner review Data Confidentiality for continued applicability	Review annually

1: modified from Information Technology at Purdue:

http://www.itap.purdue.edu/security/procedures/dataHandling/electrStored.cfm,

Appendix B
Purdue University Password Requirements

- Passwords must contain at least 1 letter.
- Passwords must contain at least 1 number or punctuation mark.
- Passwords must be at least 8 characters long.
- Passwords must contain more than 4 unique characters.
- Passwords may not contain easily guessed words (e.g. Purdue, itap, boiler).
- Passwords may not contain a name or parts of the name (e.g., Bill, Julie, Bob, or Susan).
- New passwords must be different than the previous password (re-use of the same password will not be allowed for 180 days).
- Passwords never should be stored on your computer or written down and stored in plain sight (e.g., taped to the bottom of your computer keyboard). If a password must be written down, it should be locked up and stored in a place that is difficult for others to access.
- Passwords may be used only by the authorized user. Do not share your password with anyone.
- All users must change their password at least once every 30 days.
- If you suspect your password has been compromised, it should be changed immediately.

Appendix C
Electronic Data Transfer Form

Location of measurements: __________________________

Period represented: From Date (MM/DD /YYYY) _________ time (GMT: 24h clock) _____: _____

To Date (MM/DD/YYYY) _________ time (GMT: 24h clock) _____: _____

Data preparation/ copying done by: ______________________________________

Calibration conducted during period

UVDOAS1: s/n _______ Date (MM/DD/YYYY) _________ time (GMT: 24h clock) _____: _____

UVDOAS2: s/n _______ Date (MM/DD/YYYY) _________ time (GMT: 24h clock) _____: _____

TDLAS1: s/n ________ Date (MM/DD/YYYY) _________ time (GMT: 24h clock) _____: _____

TDLAS2: s/n ________ Date (MM/DD/YYYY) _________ time (GMT: 24h clock) _____: _____

Notes:

Data Receipt

Received: Date (MM/DD/YYYY) ______________________ time (GMT: 24h clock) _____: _____

Physical condition of TDLAS1 data CD	(Excellent / Good / Fair / Poor)
Physical condition of TDLAS2 data CD	(Excellent / Good / Fair / Poor)
Physical condition of UVDOAS1 data CD	(Excellent / Good / Fair / Poor)
Physical condition of UVDOAS2 data CD	(Excellent / Good / Fair / Poor)
Physical condition of WXL and INNOVA data CD	(Excellent / Good / Fair / Poor)
Physical condition of WXL and INNOVA flash memory chip	(Excellent / Good / Fair / Poor)
Physical condition of UVDFlash1 memory drive	(Excellent / Good / Fair / Poor)
Physical condition of UVDFlash2 memory drive	(Excellent / Good / Fair / Poor)
Physical condition of Backup DVD	(Excellent / Good / Fair / Poor)
Physical condition of memory stick	(Excellent / Good / Fair / Poor)

Received by: _______________________________________

THE MEASUREMENT OF SOLAR RADIATION WITH THE LICOR MODEL 200SL OR 200SZ SILICON PYRANOMETER

Standard Operating Procedure (SOP) E3

THE MEASUREMENT OF SOLAR RADIATION WITH THE LICOR MODEL 200SL OR 200SZ SILICON PYRANOMETER

Standard Operating Procedure (SOP) E3

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Effective Date: November 6, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
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Table of Contents

1. Scope and Applicability

1.1. The method described in this SOP is to use the silicon photodiode to measure solar incident radiation in the 300 to 1200 nm wavelength band.

1.2. This procedure applies only to the LiCOR Model LI-200SL Pyranometer for monitoring solar radiation during barn air emissions measurements, or for atmospheric stability class determination. By default, these instructions apply to both measurement scenarios. If there are any differences between the two applications, this will be specified.

1.3. Detection limit is 10 W/m^2. Ambient range is 10 to 1300 W/m^2.

2. Summary of Method

A pyranometer is an instrument for measuring solar radiation. The Model LI-200SL Pyranometer (LiCOR, Lincoln, NE) includes a Model LI-200SA pyranometer sensor and a millivolt converter. The sensor is installed on a mounting and leveling fixture to receive solar radiation (Figure 1). It uses a silicon-based photodiode semiconductor that converts the solar energy into a microamp current proportional to the solar radiation. The current signal is provided to a converter through a BNC connector. The converter contains a variable resistor that is unique to the LI-200SA sensor, and that converts the current signal to a standardized output voltage, 10 mV per 1000 W/m^2. The ultimate termination of the converter consists of bare leads to be connected to the data acquisition system, which converts the voltage signal to solar radiation in W/m^2, displays it on a computer monitor, and saves it into data files.

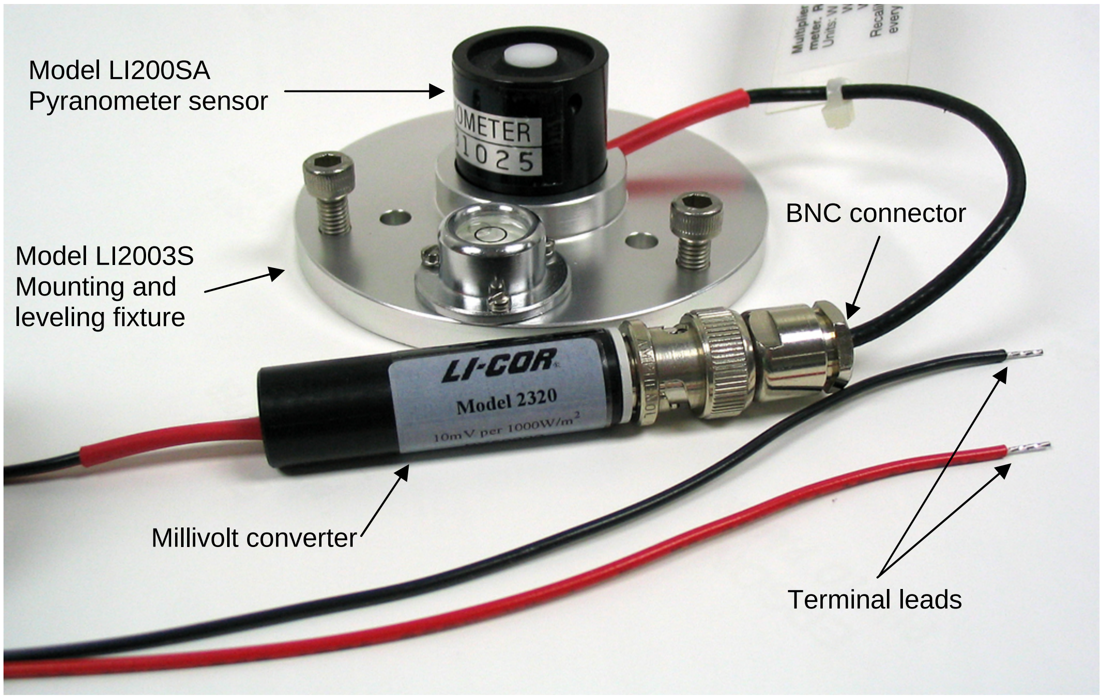

Figure 1. Model LI-200SL Pyranometer (including a LI-200SA Pyranometer sensor and a LI-2302 standardized millivolt adapter) and Model LI2003S Mounting and Leveling Fixture (adapted from LI-COR, 2006a).

3. Definitions

3.1.	BNC	Bayonet Neill Concelman connector (used with coaxial cables)
3.2.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.3.	QAPP	Quality Assurance Project Plan

4. Health and Safety

4.1. The pyranometer only produces weak electric signal that does not pose a health concern.

4.2. The pyranometer is installed outdoors, usually several meters above the ground. Follow all relevant safety precautions during installation and maintenance. Locate the sensor where it can be reached safely by the operator during the periodic clean up procedures.

5. Cautions

5.1. The acrylic material used in LI-COR light sensors can be crazed by exposure to alcohol or organic solvents, which adversely affects the response of the sensor. Clean the diffuser element of the sensor only with water and mild soap such as dishwashing soap. Do not use alcohol, organic solvents, abrasives, or strong detergents. LI-COR has found that vinegar can also be used to remove hard water deposits from the diffuser element, if necessary.

5.2. The spectral response of the LI-200 does not include the entire solar spectrum. Therefore, use the pyranometer in the same lighting conditions as those under which it was calibrated. Use it only to measure unobstructed daylight, not reflected sunlight.

5.3. Remove and save the red protective cap from the sensor. This is used to protect the sensor when it needs to be calibrated or stored.

5.4. Be careful when connecting the sensor cable to the data acquisition system. Make sure the connections are tight, and are made according to SOP B1.

5.5. Each pyranometer sensor is used only with the millivolt converter that is supplied with it and the serial numbers must match in the Model LI200SL. Be sure to change both the sensor and the converter if the LI200SL needs to be replaced.

5.6. The millivolt converter is not weatherproof. Install it inside the instrument shelter or protect it in a weatherproof enclosure.

6. Interferences

6.1. Incorrect siting of the solar radiation sensors can lead to inaccuracies over the period of data collection. One example is installing the sensor where it receives the shadow of a building or other object. Another example is installing the sensor where it receives the reflection of a clear painted wall or other object. It should not be used under vegetation.

6.2. Particulate matter and bird droppings on the optical head will cause low readings. The risk of this interference is greater near livestock buildings.

6.3. The signal provided by the converter to the data acquisition is a weak voltage. To prevent excess signal noise pickup, avoid using long cable between the converter and the data acquisition system or running the cable close to power cords.

7. Personnel Qualifications

7.1. Personnel must be trained in the use of the handheld millivolt multimeter before initiating the procedure. Training requires 10 minutes.
7.2. Each member of the field staff must read the pertinent sections of the sensor instruction manual (pp 1-5, 10-13) and this SOP before operating this instrument.

8. Equipment and Supplies

8.1. For barn measurement locations:
  8.1.1. Model LI-200SL Pyranometer (LiCOR, Inc., Lincoln, NE), which includes a LI-200 SA pyranometer, a BNC connector and a millivolt adapter. The LI-200SL comes with a 15-ft cable.
8.2. For open source measurement locations:
  8.2.1. Model LI-200SZ Pyranometer (no BNC connector) with Model 2220 millivolt adaptor (LiCOR, Inc., Lincoln, NE)
  8.2.2. Weatherproofed NEMA 4X box
  8.2.3. Data logger (SOP W6)
8.3. Model LI2003S Mounting and Leveling Fixture (LiCOR, Inc.)
8.4. Sensor stand and mounting arm (Fig. 2) if they are not available in the weather tower.
8.5. Machine screws with nuts to mount the Mounting and Leveling Fixture to the sensor stand.
8.6. 3/64″ hex wrench to mount the pyranometer sensor to the Mounting and Leveling Fixture, and 1/8″ screwdriver to fasten the screws
8.7. Plastic cable ties to fix the sensor cable
8.8. Multimeter to check the pyranometer signal output, with a resolution of ≤ 0.01 mV
  8.8.1. Multimeter must be pre-calibrated for open-source applications. This is not necessary for barn applications.
8.9. Soft cloth, soap, water and soft toothbrush if the sensor needs cleaning
8.10. Electronic parts cleaner
8.11. Heat shrink tubing
8.12. Heat blower
8.13. Unused LiCOR LI-200 (to calibrate the sensor)

9. Procedures

9.1. Acceptance
  9.1.1. Verify that the serial number on the calibration certificate matches the serial number on the instrument.
  9.1.2. Verify that the serial numbers of the pyranometer sensor and the converter match.
  9.1.3. Response check:

9.1.3.1. Place pyranometer outside facing towards the sky on a sunny day.
9.1.3.2. Remove and save the red protective cap from the sensor.
9.1.3.3. Connect the sensor and the converter with the BNC connector.
9.1.3.4. Measure sensor response across the positive and negative wires with a multimeter.
9.1.3.4.1. The multimeter resolution should be better than 0.01 VDC. For atmospheric stability classification uses, the multimeter should be a calibrated model.
9.1.3.5. If sensor voltage is less than 1 mV, return sensor to supplier for replacement.
9.1.4. Zero check:
9.1.4.1. Obstruct all light from entering the optical window using a non-reflective cup or other means and measure the voltage with a millivolt multimeter across the sensor wires.
9.1.4.2. Value should be less than 0.01 mV. If value is greater than 0.01 mV, return the instrument to supplier for replacement.
9.1.5. Installation
9.1.6. Mount the Mounting and Leveling Fixture to the horizontal platform, 2 ft away from the S side of the Weather Station Tower (or the S side of a 10-ft mast, in open-path measurements) using a mounting arm or a cross arm stand (Fig. 2), such that no obstruction can cast a shadow over the sensor, according to SOP E5 (barn installation), SOP U5 (lagoon/basin installation), or U7 (corral installation).

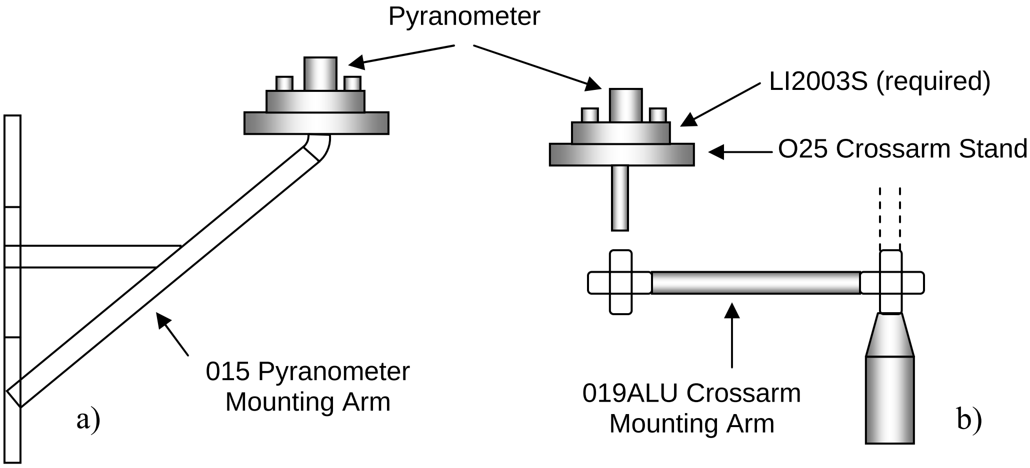

Figure 2. Pyranometer mounting arm (a) and cross arm stand (b) (LiCor Inc., 2006b).

9.1.7. Securely mount the pyranometer sensor onto the Mounting and Leveling Fixture using the 3/64″ hex wrench.
9.1.8. Adjust the level of the Mounting and Leveling Fixture.
9.1.9. If installing the sensor for open-source use, pass the coaxial cable through the appropriate knockout of the weatherproofed NEMA 4X box.

9.1.10. If installing the sensor for barn use, no NEMA 4X box is needed. Wrap the BNC connector, and its junction with the millivolt adapter, with electrical tape.

9.1.11. Run the cable from the pyranometer to the instrument shelter or trailer.

9.1.11.1. If a field datalogger is being used (i.e. during open-source applications), use the 1/8″slotted screwdriver to connect the positive signal wire to the single-ended measurement channel, and the black wire to ground according to SOP W6.

9.1.12. Connect the cable to the Millivolt Converter inside the instrument shelter.

9.1.13. Measure voltage across the two wires using a multimeter. On a sunny day, the voltage should be at least 1 mV.

9.1.14. Attach wires to data acquisition system according to SOP B1.

9.2. Calibration checks

9.2.1. Check the calibration of a pyranometer which is in use by comparing its readings with an unused pyranometer of the same model which is known to be accurate.

9.2.1.1. This can be done in the field or at Purdue. The QAPP for a particular project will state which option is to be used.

9.2.1.1.1. If these checks are to be done in the field, it is necessary to include an additional support and additional wiring for the collocated sensor, as well as the corresponding wiring, when installing the sensor. There must also be an available input channel in the data acquisition system for this collocated sensor.

9.2.1.1.2. This is not necessary if the instruments are to be sent to Purdue for calibration.

9.2.1.2. Measurements are made every 5 min for 2 d, using the same data acquisition system used in the study to collect data from both sensors.

9.2.1.3. The calibration check frequency will be specified in the QAPP.

9.2.1.3.1. The manufacturer recommends recalibrating this instrument every two years. A shorter interval can be chosen based on the QAPP's Quality Objectives.

9.2.2. Calculate the difference in each 5-min paired measurement, and compute the mean difference between the two sensors for the daylight hours over the two days.

9.2.3. If the mean difference between the sensor under calibration and the unused sensor is < ±5% of the unused sensor's reading, then the sensor being calibrated is assumed good. In this case, calculate a correction factor to apply to the in-field sensor.

9.2.4. If the mean difference between the sensor under calibration and the unused sensor is greater than ±5% of the unused sensor's reading, then check the cleanliness and the connections of the sensor that is being checked, and repeat the calibration check. If the difference persists, the sensor being checked is assumed to be defective, and must returned to the supplier for repair or replacement.

9.3. Troubleshooting

9.3.1. Low readings are often due to a dirty optical window. Clean surface of sensor with soap and water.

9.3.2. Erratic values are often due to poor connections. Check connections for tightness.

9.4. Data acquisition, calculations & data reduction requirements

9.4.1. The LI-200SL output is acquired by a data acquisition (DAQ) system (SOP B1 and SOP B2) at a frequency of 1 Hz.

9.4.2. The DAQ system converts the voltage signals to W/m^2, averages them every 15 s and 60 s, and saves the averaged data in data files.

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for the method.
10.2. Manage all data according to SOP B5 (for barn measurements) SOP D1 (for open-source meteorological measurements).
10.3. Document all data and information on field data sheets and in site logbooks with permanent ink, or in electronic notes.
10.4. Strike out errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

11.1. Do the following at the beginning and end of each measurement period, or as specified in the QAPP:
  11.1.1. Check physical cleanliness of the pyranometer's optical window. If dirty, clean with soap and a wet cloth. Always use lens cloth to avoid scratching the glass surface.
  11.1.2. Perform recalibration if the optical window shows any scratches, or discoloration that cannot be washed off.
  11.1.3. Check physical cleanliness of connectors. If corroded, clean with electronic cleaner and toothbrush.
  11.1.4. Check integrity of the plastic casing of the signal cable.
    11.1.4.1. If damaged slightly, repair with heat-shrink tubing.
    11.1.4.2. If damaged significantly, replace sensor and return sensor to manufacturer for repair.
  11.1.5. Perform zero checks (Section 9.1.4).
  11.1.6. Record results of each check in the calibration file (Appendix A) for open-source applications, or a similar table entry in the electronic field notes for barn applications.
  11.1.7. Calibration checks against a collocated sensor (Section 9.2).
11.2. Do the following daily:
  11.2.1. Compare the measurement with potential irradiance based on time of day, latitude and longitude of the location, and a solar constant of 1367 W/m^2 (Appendix B).
  11.2.2. Ratio of sensor measurement to potential irradiance must be less than 0.9.
  11.2.3. If ratio is greater than 0.9:
    11.2.3.1. Flag data value as invalid.
    11.2.3.2. Perform a calibration check against an unused pyranometer (Section 9.2). If the sensor fails this check (Section 9.3.4), replace sensor and return sensor to factory for repair.
  11.2.4. Check measurements between 10 AM and 2 PM.
    11.2.4.1. If measurements are less than 100 W/m^2:
      11.2.4.1.1. Flag data values for day as invalid
      11.2.4.1.2. Check wiring and optical window. Repair, clean, or replace as necessary.

12.

12. References

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12.8. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
12.9. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
12.10. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
12.11. SOP E5. 2006. Roof-mounted Weather Station Tower. Standard Operating Procedure E5. Purdue Ag Air Quality Lab.
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12.14. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
12.15. National Oceanic and Atmospheric Administration (NOAA). Solar Position Calculator. Available on http://www.srrb.noaa.gov/highlights/sunrise/azel.html. Accessed on 7/10/2006.
12.16. Free Zip Code Lookup. Available at http://zipinfo.com/search/zipcode.htm. Accessed on 7/10/2006

13. Contact Information

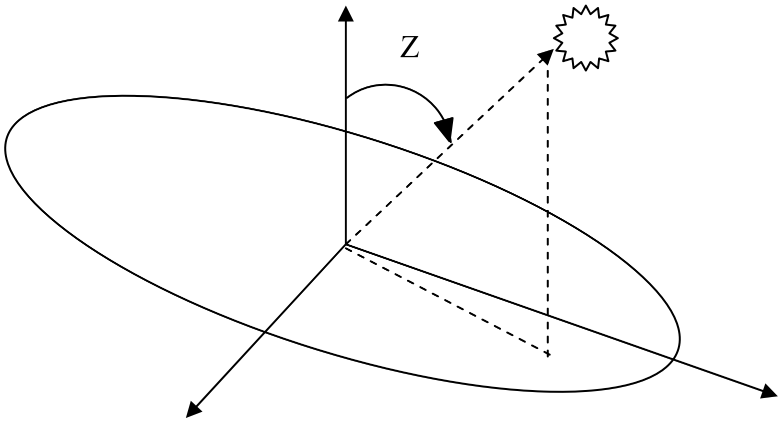
13.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, Utah, 84321. Technical support (435)-753-2342.

Appendix A. Calibration form: LI-200SL Solar Radiation Sensor

Instrument Serial Number: ___________________________________ Location: _______________________________________

Date (D-M-Y)	Time Begin	Time End	Mean irradiance (W/m^2)		Difference, (unused-measured)		Action Taken (Adjust, Replace)	Analyst
	AM/PM	AM/PM	Unused sensor	Sensor in use	W/m^2	%		

Appendix B. Procedure to Calculate the Potential Solar Radiation on a Horizontal Surface.



Calculate the vertical component of the potential solar radiation on a horizontal surface using the following equation:

$$\mathbf{Ho = Io * CosZ}$$

Where:

Ho = Potential extraterrestrial insolation on a horizontal surface (W/m^2)

Io = Extraterrestrial solar radiative flux (1367 W/m^2)

Z = zenith angle (from the vertical position to the position of the sun).

Obtain the site-specific Cosine of the Zenith angle (CosZ) from the National Oceanic and Atmospheric administration (NOAA) website (http://www.srrb.noaa.gov/highlights/sunrise/azel.html), by entering the geographic location of the monitoring site (latitude and longitude) and the time of day. The online calculator will determine the cosine of the zenith.

If unsure of the latitude and longitude of the specific monitoring site, this can be obtained through the website http://zipinfo.com/search/zipcode.htm, by checking the “latitude and longitude” box and the entering the ZIP code of the location.

COMPRESSED GAS CYLINDERS

Standard Operating Procedure (SOP) G2

COMPRESSED GAS CYLINDERS

Standard Operating Procedure (SOP) G2

Prepared by

Congna Li, Juan Carlos Ramirez, and Claude A. Diehl

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
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1. Scope and Applicability

1.1. Gas cylinders are used to transfer certified gas standards to research labs where gas concentration measurements are conducted. They are also used to supply fuel for flame ionization detectors, carrier gases for gas chromatographs, dilution gases for programmable diluters, and concentrated gases for gas injection systems. The following general facts about compressed gas cylinders are relevant to this SOP:

1.1.1. Gas standards typically have a defined, finite shelf life, and a specific expiration date.
1.1.2. Gas cylinders contain a finite volume of gas that may need to be replenished.
1.1.3. Gas cylinders are typically rented so money is saved by returning them promptly.
1.1.4. Gas cylinders generally look alike which challenges identification procedures.
1.1.5. Missing cylinders must be replaced at a significant replacement charge.
1.1.6. The time between purchase and delivery are relatively long.
1.1.7. The high pressure inside a gas cylinder represents a potential safety hazard.
1.1.8. The contents of a gas cylinder can range from very safe to extremely hazardous.
1.1.9. A gas-specific pressure regulator is required for each cylinder.
1.1.10. Managing gas cylinders requires a significant amount of labor.
1.1.11. They may be exposed to the extremes of indoor and outdoor temperatures.

2. Summary of Method

This SOP covers the necessary procedures, cautions and documentation for selecting, purchasing, receiving, transporting, storing, using, terminating, and returning gas cylinders. The compressed gas cylinder inventory will be scientifically selected, carefully controlled, and professionally handled to reduce cost, ensure safety, and improve data quality. Gas regulators will be properly selected and installed on each cylinder to prevent leakage and ensure safe delivery of the gases. Following this method will ensure proper documentation of the cylinders and the regulators that are used with the cylinders.

3. Definitions of Terms and Acronyms

3.1. ABE: Agricultural and Biological Engineering
3.2. CGA: Compressed Gas Association.
3.3. PAAQL: Purdue Agricultural Air Quality Laboratory
3.4. REM: Office of Radiation and Environmental Management at Purdue University
3.5. Compressed Gas: Any material or mixture having in the container either an absolute pressure exceeding 40 psia (3 bar) at 70°F (21°C).
3.6. Requested Concentration: The gas concentration requested by the customer when purchasing a standard gas.
3.7. Balance Gas: The material ("component") making up the majority of a mixture.
3.8. Blended Concentration: The concentration measured by the blending process. The numerical concentration assigned to a component based on the results of blending.

3.9. Blend Tolerance: The degree of agreement between the Blended Concentration and the Requested Concentration.
3.10. Crawl: Rise above a set delivery pressure during operation of a regulator.
3.11. Accuracy: The statistical agreement of a measured value with its true value
3.12. Expiration date: The stated date after which a gas concentration is no longer valid or legal to use for calibration purposes.
3.13. Traceability: Basis for establishing the true value of a standard or pathway back to the true value. For example, it is the relationship between a measured value and an established element of the National/International Measurement System.
3.14. Two-Stage Pressure Reducing Regulator: A regulator which reduces high-pressure to low pressure and controls the low or outlet pressure with two stages of pressure reduction. Used when more stability of operation is required.
3.15. Stability: The ability to maintain a constant concentration value over a defined time within statistical significance
3.16. Gas Mixture Grades
  3.16.1. EPA Protocol Grade: EPA Protocol Gases are manufactured and analytically certified in strict accordance with the most recent EPA traceability guideline document entitled “EPA Traceability Protocol for Assay and Certification of Gaseous Standards”. EPA protocol certified standards meet or exceed gas standards specified in U.S. regulations 40CFR60 and 40CFR75.
  3.16.2. Primary Grade: Specialty gas, high resolution, gravimetric mixture with certification accuracy of either 1% relative, or 0.02% absolute, whichever is less. Often referred to as NIST-Traceable By Weight.
  3.16.3. Certified Grade: Working standards prepared either by partial pressure or gravimetrically. Certification accuracies may range from ±2% to ±5%.
  3.16.4. Unanalyzed Grade: The unanalyzed grade is used when the minor component accuracy does not have to be less than 10%.

4. Health and Safety

4.1. Always use the smallest size cylinder required to perform the work.
4.2. Cylinders of compressed gases must be handled as high potential energy sources.
4.3. Cylinders on wheeled carts must be capped and secured by an approved cylinder support strap or chain. The cart must be an approved cylinder cart. Do not attempt to take a loaded cylinder cart up or down a stairway.
4.4. Secure uncapped cylinders independently to a solid element of the lab structure. Do not “gang” cylinders behind a single chain, and do not use carts to support uncapped or in-use cylinders.
4.5. Transporting cylinders
  4.5.1. Move cylinders by hand trucks or carts that are designed for this purpose. Always use a cylinder cart equipped with a chain restraint.
  4.5.2. During transportation, properly secure cylinders to prevent them from falling or striking each other.
  4.5.3. Do not move a cylinder with a regulator connected to it.
  4.5.4. Never transport a gas cylinder without its valve protection cap firmly in place.

4.5.5. Keep both hands on the cylinder cart during transport.

4.5.6. Follow applicable federal and state regulations when transporting cylinders on open roads. Provided that no more than 440 lbs (>220lbs of flammable gas and >220lbs of other gases including container weights) are being transported, the gases are classified as “Materials of Trade”, and are not subject to placards and other restrictions that would normally be associated with transporting larger quantities of compressed gas.

4.6. Securing cylinders

4.6.1. Place cylinder in an upright position against a cylinder rack located against the wall.

4.6.2. Place the straps around the cylinder.

4.6.3. Insert the strap through the buckle and pull tight.

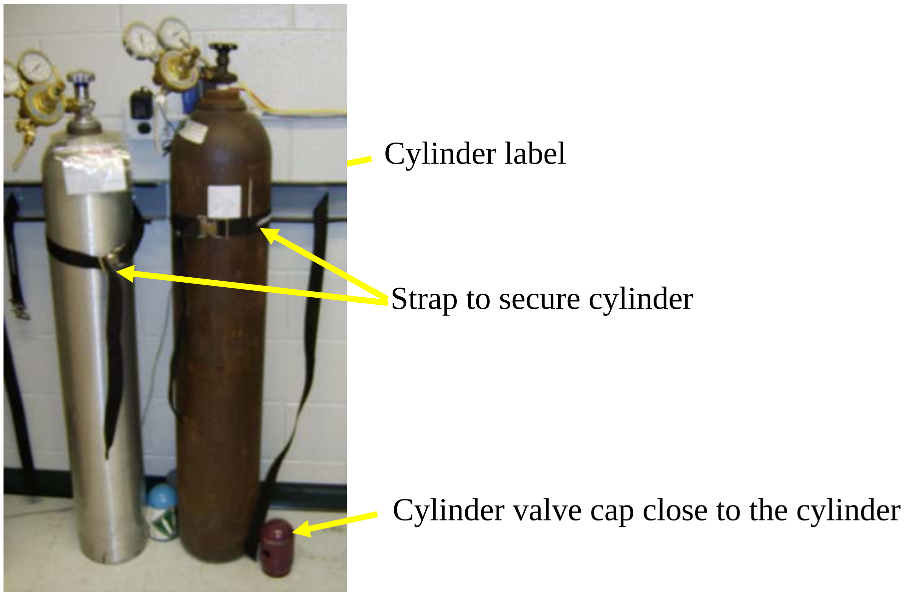


Figure 1. Gas cylinders safely located for use.

4.7. Storage of cylinders

4.7.1. Storage Area. Store gas cylinders in a ventilated and well-lit area, away from combustible materials. Store cylinders containing flammable gases away from oxygen cylinders and other oxidants. Separate by a fire-resistant wall, or locate them at least 20 feet apart from each other.

4.7.2. Storage Area Conditions. Storage areas should be located away from sources of excess heat, open flame or ignition, and not located in closed or subsurface areas. The area should be dry, cool and well-ventilated.

4.7.3. Securing Cylinders in Storage. Always hold cylinders in place with a chain or another type of fastener such as a bench or wall clamp. While in storage, cylinder valve protection caps must be firmly in place.

4.7.4. Cylinders may be stored flat on the ground, if vertical storage is not possible, provided they are secured as described in 4.7.3.

4.7.4.1. Note that this applies only to storage. Cylinders may not be transported or used while horizontal.

- 4.7.5. For cylinders at remote sites, the conditions in 4.7.1 and 4.7.2 will be followed as closely as possible. Sections 4.7.3 and 4.7.4 will be followed strictly, including at remote sites.

4.8. Never tamper with safety devices in valves or cylinders.
4.9. Use only a cylinder cap wrench to loosen and unscrew cylinder caps; do not strike cylinder caps when attempting to loosen them.
4.10. Oil or grease on the high pressure side of an oxygen cylinder can cause an explosion. Do not lubricate an oxygen regulator or use a fuel gas regulator on an oxygen cylinder.
4.11. Wear goggles or safety glasses with side shields when handling compressed gases.
4.12. Eyewash fountains, safety showers, gas masks, respirators, and/or resuscitators should be located nearby but out of the immediate area that is likely to become contaminated in the event of a large release of gas.
4.13. Fire extinguishers, preferably a combination of water- and CO_2-based types, should be kept close at hand, and should be checked periodically to ensure their proper operation.
4.14. Always use appropriate gauges, fittings, and materials compatible with the particular gas. Regulators must be compatible with gas cylinders (do not use adapters). Each cylinder and regulator have connection fittings that are designated by a CGA number. CGA numbers can be stamped on the cylinder and regulator and listed on the producer’s label. CGA numbers are typically (but not always) stamped on the regulator just above the threads of the cylinder connection (Fig. 2). Table 1 shows some examples of CGA connection numbers and some examples of the gases used with these connections.
4.15. MSDS information sheets will be kept in a 3-ring binder located near the cylinders.
- 4.15.1. MSDS information will also be kept on-site for any gases which are stored and/or used at remote locations.

Table 1. Cylinder Valve Outlets and CGA Connections.

Examples of Gases	CGA Connection #
Ammonia	705
Carbon Dioxide	320
Butane, ethylene oxide, propane, propylene	510
Air (industrial grade)	590
Carbon monoxide, ethylene, methane, hydrogen	350
Hydrogen sulfide, hydrogen chloride	330
Nitrogen, helium, argon, noble gases	580
Nitrogen dioxide, sulfur dioxide	660
Oxygen	540

Figure 2. Photo of a gas pressure regulator (CGA 580) showing the connector to the gas cylinder. The CGA number can be found on the side of the nut closer to the regulator.

4.16. When work is planned with toxic, corrosive, or reactive gases, consult the MSDS and/or institutional safety office for information concerning specific handling requirements for the gas involved. Generally, these gases will need to be used and stored with local exhaust ventilation, such as a lab hood, gas cabinet, or a ventilated OFIS that is designed for that purpose.

4.17. Check the regulator and cylinder valve for evidence of damage or contamination.

4.18. Do not apply Teflon tape between the regulator and the cylinder connections. Bits of Teflon tape can get blown into the regulator, causing a leak, valve malfunction or erroneous reading.

  4.18.1. If a leak-free connection cannot be established between the regulator and the cylinder, inspect the regulator and cylinders threads for possible damage or contamination. If regulator components (adapter threads, washers, and/or fittings) are damaged, they should be replaced. Any regulator which cannot be made leak-free should be replaced.

4.19. If an OFIS at a field site (farm) contains cylinders, notify the producer and/or appropriate staff that there are compressed cylinders within the OFIS and describe the contents of the compressed cylinders. This is especially important in case of a fire, so that the producer will stay away from the OFIS, and properly notify responding emergency personnel.

5. Cautions

5.1. Do not allow cylinder temperature to exceed 125°F (52°C), or to drop to -30°C or lower, since many types of steel will lose their ductility and impact strength at lower temperatures.

5.2. Store empty cylinders separately from full cylinders, and clearly identify them as such. Leave some pressure (>50 psi) in a depleted cylinder to prevent air backflow that would allow moisture and contaminants to enter the cylinder.

5.3. Flammable gases have reverse threads, meaning that the connection is tightened by turning the nut counterclockwise. You can always tell a reverse thread connection because the nut that you tighten has a line inscribed around its circumference.
5.4. Do not use an expired cylinder for a Level 1 Precision Check. In other words, never adjust an analyzer based on data obtained from an expired cylinder.

6. Personnel Qualifications

6.1. Personnel should be trained in the handling of cylinders and also in the updating of the web-based cylinder control file before initiating the procedure alone.
6.2. Personnel handling any compressed gas should be familiar with the potential hazards before using the gas.
6.3. Personnel should address potential emergencies and the safe and proper measures necessary to avoid these emergencies.
6.4. Personnel should identify several scenarios that could result in gas leaks or other emergencies to be totally prepared to respond adequately.

7. Equipment and Supplies

7.1. Gas cylinders
  7.1.1. EPA maintains a web site which documents the performance of major gas-cylinder vendors, based on the results of a round robin test conducted every two years.
  7.1.2. Consider the following parameters when selecting cylinders:
    7.1.2.1. Certification accuracy
    7.1.2.2. Blend tolerance
    7.1.2.3. Shelf life
    7.1.2.4. Delivery time
    7.1.2.5. Reanalysis tolerance
    7.1.2.6. Requested concentration
7.2. Gas regulators appropriate for each gas (Section 4.14)
7.3. Labels to identify each cylinder (Section 8.1.7)
7.4. An adjustable or combination cylinder wrench to connect and disconnect the regulator to the cylinder.
7.5. Cylinder hand truck
7.6. Cylinder brackets
7.7. Cylinder return labels (Section 8.8.4)

8. Procedures

8.1. Receiving gas cylinders (PAAQL)
  8.1.1. Cylinders are delivered to the ABE shop’s gas cylinder receiving station by Purdue’s Gas Cylinder Control Office.
  8.1.2. Upon delivery, the cylinders should be picked up by PAAQL staff, after verifying that there is a plastic seal on the cylinder valve. If there is no seal, make a note and contact the company.

8.1.3. Write the cylinder-stamped number on the form by the gas cylinder rack, and sign the form to indicate that the cylinder has been received.

8.1.4. A delivery sheet or packing slip is attached to every cylinder to verify that the proper cylinder is delivered (gas type, amount of gas, cylinder ID).

8.1.5. Sign and date the delivery sheet.

8.1.6. Place the original delivery sheet in the ABE Business Office's inbox and a copy of the delivery sheet in the loose leaf cylinder notebook in the section corresponding to the year received and the company that supplied the cylinder. This notebook is kept on top of the manual's file cabinet in room ABE-102E.

8.1.7. Attach a cylinder label (Fig. 3) to the cylinder upon receipt, with the following information filled out:

Gas ______________________________
Concentration _____________________
Initial Pressure _________________psi
CGA No. ________________________
Cylinder No. ______________________
Date Received ___________________
Expiration Date __________________
Date:___________Pressure:________psi
Date:___________Pressure:________psi
Date:___________Pressure:________psi
Date:___________Pressure:________psi
Date:___________Pressure:________psi

Figure 3: A cylinder label.

8.2. Receiving gas cylinders (Field sites)

8.2.1. Cylinders are delivered to a specified location (e.g. the field site itself, a subcontracting university or other entity, etc) by the supplier's local vendor/agent.

8.2.2. Upon delivery, the cylinders should be picked up by study personnel (PAAQL or otherwise), after verifying that there is a plastic seal on the cylinder valve. If there is no seal, make a note and contact the company.

8.2.3. If necessary, transport the cylinder to the field site, observing all cautions listed in Section 4.5.

8.2.4. Write the cylinder-stamped number on the form by the gas cylinder rack and sign the form to indicate that the cylinder has been received.

8.2.5. A delivery sheet or packing slip will be attached to every cylinder by the supplier to verify that the proper cylinder is delivered (gas type, amount of gas, cylinder ID).

8.2.6. Sign and date the delivery sheet.

8.2.7. File a copy of the delivery sheet on-site in a binder or loose-leaf notebook (Section 8.1.6).

8.2.8. Fill out a cylinder label (Fig. 3), and attach it to the cylinder.

8.3. Installing pressure regulators

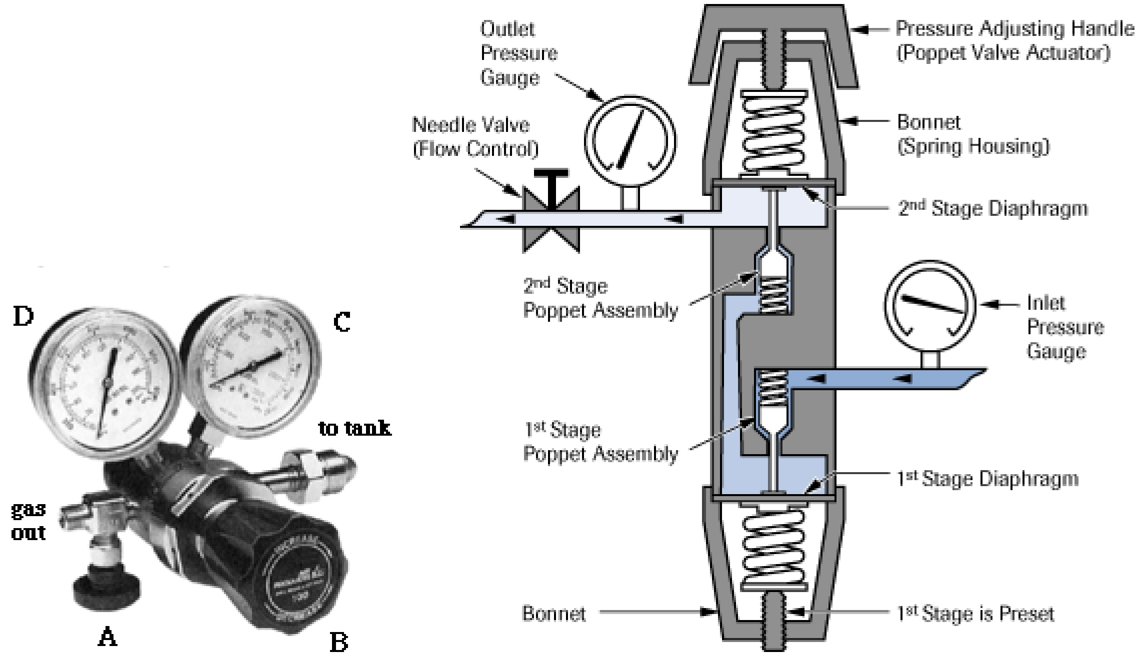
8.3.1. Make sure the cylinder is properly secured, and that the regulator is the correct one for the cylinder, and that any special hazards of the gas are known.

8.3.2. Remove the cylinder valve cap (counterclockwise). Place it somewhere nearby.

8.3.3. Some regulators (on lecture bottles and certain corrosive gases) require a Teflon washer to be inserted between the tank outlet and regulator. Check to see if this is required before continuing to the next step.

8.3.4. Make sure that the regulator's needle valve (A) (Fig. 4) is shut. Screw it clockwise until it seats. Do not over tighten it or you can damage the valve seat.

8.3.5. Make sure that the regulator's pressure adjusting handle (B) (Fig. 4) is shut. Turn it counterclockwise until it is almost completely unscrewed. If you unscrew it completely, just put it back in.



(a) (b)

Figure 4. (a) A two –stage pressure regulator and its components. A: Needle valve for outlet flow control. B: Pressure adjusting handle (Poppet Valve Actuator). C: Inlet pressure gauge. D: Outlet pressure gauge. (b) Schematic diagram of a two-stage pressure regulator.

8.3.6. Turn the regulator onto the tank by hand until it is almost finger tight.

8.3.7. Use an adjustable wrench or cylinder wrench to tighten the regulator onto the cylinder.

8.4. Operation of pressure regulator

8.4.1. Open the tank valve slowly (counterclockwise (CCW)). Watch the tank pressure on the regulator's inlet pressure gauge (C) (Fig. 4).

8.4.2. Slowly turn the pressure adjusting handle (B) on the regulator until the regulator's outlet pressure gauge (D) is at the desired pressure level (Fig. 4).
8.4.3. Open the regulator's needle valve (A) to regulate flow with this valve.
8.4.4. Check your system for leaks using Snoop (a commercial product) or some soapy water. Snoop is preferred since it leaves no residue.

8.5. FTIR validation of gas concentration

8.5.1. If the composition of the gas in a cylinder is to be checked by FTIR, this procedure should be conducted according to SOP G12 ("FTIR Verification of Gas Cylinder Composition").

8.6. Shut down of pressure regulator

8.6.1. Shut the tank valve on the gas cylinder.
8.6.2. Slowly open the needle valve (A) on the regulator.
8.6.3. Watch the pressure gauges C and D drop to zero.
8.6.4. Open the regulator's pressure adjusting handle (B) (turn it clockwise) to ensure that all pressure is released.
8.6.5. After relieving all the gas pressure, turn the regulator pressure adjusting handle (B) CCW as far as it will go to close the regulator, and also close the needle valve (A).

8.7. Disconnecting a pressure regulator

8.7.1. Use a wrench to disconnect the regulator from the gas cylinder.
8.7.2. Replace the protective cylinder cap immediately.

8.8. Cylinder return

8.8.1. Prior to returning the cylinder, place a return label on the cylinder and date it. (Fig. 5)
8.8.2. The cylinder should be returned to the cylinder collection rack in the ABE shop.
8.8.3. Place an "Empty Cylinder" ring on the cylinder.
8.8.4. Enter the returned cylinder number and date on the form.

Return To: General Stores

From: Al Heber
Department of Ag and Bio Engineering
1146 ABE

Date:

Figure 5: Return label for gas cylinders.

9. Troubleshooting

9.1. Check regulators periodically to ensure proper and safe operation. This periodic check will vary depending on gas service and usage. The procedure for checking out any regulator is as follows:

- 9.1.1. Be sure gauges read zero when all pressure is drained from the system.
- 9.1.2. Open the cylinder valve and turn the pressure adjusting handle counterclockwise; the inlet pressure gauge will read the cylinder pressure.
- 9.1.3. Starting at Section 9.1.1, close the cylinder valve after reading the cylinder pressure, if the pressure drops after one (1) hour of monitoring the inlet pressure, there is a leak between the inlet pressure gauge and the cylinder valve.
- 9.1.4. Starting at Section 9.1.1, close the regulator's outlet needle valve then wait 1 hour; the outlet pressure gauge should not indicate a pressure increase. A pressure increase would indicate leakage across the internal valve system.
- 9.1.5. Turn the pressure adjusting handle clockwise until a nominal delivery pressure is indicated. Inability to attain a proper delivery-pressure setting or abnormal adjustment of the screw indicates improper operation, which may be attributed to blockage of the gas passage or a leak in the low-pressure side of the regulator. Continued wear on a regulator valve-and-seat assembly will cause a rise above a set delivery pressure, termed *crawl*. A regulator exhibiting crawl should not be used.
- 9.1.6. Close cylinder valve and observe pressure on both inlet and delivery sides of the regulator after 1 h. A drop in the pressure reading after this period of time may indicate a leak in the system, possibly at the inlet or through the needle valve, safety devices, or diaphragm.
- 9.1.7. An excessive fall in delivery pressure under operating conditions and normal flow indicates an internal blockage.
- 9.1.8. Any deviation from the normal in the preceding checkout will require servicing by reputable repair personnel.

10. Data and Records Management

10.1. Use the information on the delivery sheet to update the PAAQL Cylinder Inventory webpage. To update this webpage you must:

- 10.1.1. Open the "odor" drive (permission to access this drive is required).
- 10.1.2. Open the "public-web" folder.
- 10.1.3. Open the "cylinder" folder
- 10.1.4. Open the "PAAQL Cylinder Inventory.htm" file.
- 10.1.5. To edit the "PAAQL Cylinder Inventory.htm" webpage:
  - 10.1.5.1. Click on "File".
  - 10.1.5.2. Click "Edit with Microsoft Excel".
  - 10.1.5.3. Add the following data to the PAAQL Cylinder Inventory web page:
    - 10.1.5.3.1. Gas name
    - 10.1.5.3.2. Balance gas
    - 10.1.5.3.3. Gas analyzer that cylinder is assigned for calibration
    - 10.1.5.3.4. Cylinder number (this is the engraved number only)

10.1.5.3.5. Cylinder style
10.1.5.3.6. Cylinder volume
10.1.5.3.7. Supplier of the gas cylinder
10.1.5.3.8. Purchase order number
10.1.5.3.9. Person who requested the order
10.1.5.3.10. Date issued
10.1.5.3.11. Date received
10.1.5.3.12. Date tested
10.1.5.3.13. Date returned
10.1.5.3.14. Cylinder expiration date
10.1.5.3.15. Initial pressure, psi
10.1.5.3.16. Final pressure, psi
10.1.5.3.17. CGA number (this can be found on the cylinder valve)
10.1.5.3.18. Gas concentration,
10.1.5.3.19. Certified accuracy, %
10.1.5.3.20. Project
10.1.5.3.21. Location
10.1.5.3.22. Cylinder cost
10.1.5.3.23. Monthly cost
10.1.5.3.24. Total cost to date

10.1.5.4. Update the following data each month on the PAAQL Cylinder Inventory:
10.1.5.4.1. Location
10.1.5.4.2. Pressure test
10.1.5.4.3. Pressure test date
10.1.5.4.4. Monthly cost (see demurrage report)
10.1.5.4.5. Total cost to date (see demurrage report)

10.1.5.5. The PAAQL Cylinder Inventory web page is color coded.
10.1.5.5.1. The cylinders that have been recently returned have a purple color.
10.1.5.5.2. Cylinders that are in ABE are in yellow.
10.1.5.5.3. Cylinders that are on site are in dark blue.
10.1.5.5.4. Cylinders not currently in the inventory but still on the demurrage report are in red.
10.1.5.5.5. Cylinders that were apparently returned but yet cylinder rental charges are accruing are in green.

10.2. Initiate an order for a replacement cylinder well in advance of a cylinder’s running out (i.e. dropping below 150 psi, as per Section 5.4).

10.2.1. Each time a cylinder is used, record the pressure remaining at the end of use. Keep a graph tracking pressure vs. date to allow projection of when pressure will reach the 150 psi minimum limit.

10.2.2. Allow sufficient time to meet the following requirements:
10.2.2.1. Replacement cylinders will be compared to the old cylinder using the applicable gas analyzer. Leave sufficient gas in the old cylinder to conduct this test.
10.2.2.2. If the replacement cylinder is outside the acceptable specifications, there must be sufficient gas remaining in the old cylinder to operate until an acceptable cylinder is received.

10.2.2.3. In general, a new purchase order should be initiated two months in advance before the current cylinder is empty or expires, if such gas is needed on a continuous basis.

10.2.3. Email notification of new order will be sent to the personnel responsible for the project.

11. Quality Control and Quality Assurance

11.1. Personnel handling any compressed gas will be trained to be aware of potential hazards before using the gas, to address potential emergencies and the safe and proper measures necessary to avoid these emergencies, and to identify several scenarios that could result in gas leaks or other emergencies to be totally prepared to respond adequately.

11.2. Regulators and cylinder valves should be checked periodically to ensure proper and safe operation.

11.3. The PAAQL Cylinder Inventory webpage will be used to update the information for all the cylinders in the current inventory and the most recent cylinders that have been returned.

11.4. Do not calibrate with a span gas if its cylinder pressure is below 150 psi.

12. References

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MEASUREMENT OF HYDROGEN SULFIDE (H2S) WITH THE THERMO ELECTRON CORPORATION MODEL 450I PULSED-FLUORESCENCE ANALYZER

Standard Operating Procedure (SOP) G5

MEASUREMENT OF HYDROGEN SULFIDE (H_2S) WITH THE THERMO ELECTRON CORPORATION MODEL 450I PULSED-FLUORESCENCE ANALYZER

Standard Operating Procedure (SOP) G5

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PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

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Table of Contents

1. Scope and Applicability

- 1.1. The purpose of this procedure is to present the methodology and instrumentation used to automatically and sequentially measure hydrogen sulfide (H_2S) emissions from barns, other buildings or open sources at confined animal feeding operations (CAFO).
- 1.2. This procedure applies only to the Model 450I Pulsed Fluorescence H_2S/SO_2 Analyzer (Thermo Electron Corporation, Franklin, MA).
- 1.3. The pulsed fluorescence method is useful for measuring hydrogen sulfide (H_2S) in air, due to its suitable detection limit, response time, and measurement range.
  - 1.3.1. The detection limit of the method is:
    - 1.3.1.1. 6 ppb (when using a 10-s averaging time)
    - 1.3.1.2. 2 ppb (60-s averaging time)
    - 1.3.1.3. 1.5 ppb (300-s averaging time)
  - 1.3.2. The 90% response time to a step change in H_2S concentration is:
    - 1.3.2.1. 80 s (10-s averaging time)
    - 1.3.2.2. 110 s (60-s averaging time)
    - 1.3.2.3. 320 s (300-s averaging time)
  - 1.3.3. The measurement range of the Model 450I is from 0 to 10,000 ppb (10 ppm).
- 1.4. The stated precision of the 450I is 1% of reading or 1 ppb (whichever is greater).

2. Summary of Method

Hydrogen sulfide in sample air entering the 450I is converted to sulfur dioxide (SO_2) in a converter operating at 300-400°C (1).

$$H_2S \rightarrow SO_2 \qquad (1)$$

The sample flows directly to the hydrocarbon kicker when the 450I measures $SO_{2,}$ or flows through the converter first, then to the hydrocarbon kicker when the 450I measures H_2S (Fig. 1) Any hydrocarbon present in the sample permeates through the tube wall of the hydrocarbon kicker, and is removed from the sample. SO_2 passes through the hydrocarbon kicker into the fluorescence chamber (the L-shaped box at the center right of Fig. 1).

The sample containing SO_2 is pulsed with ultraviolet (UV) light by a high-intensity xenon lamp. SO_2 molecules absorb at one wavelength and become excited (SO_2^*); they then decay to a lower energy state by emitting UV light at a different wavelength (2).

$$SO_2 + hv_1 \rightarrow SO_2^* \rightarrow SO_2 + hv_2 \qquad (2)$$

Emitted UV light is proportional to SO_2 concentration and is detected and converted to an electrical current by a PMT detector.

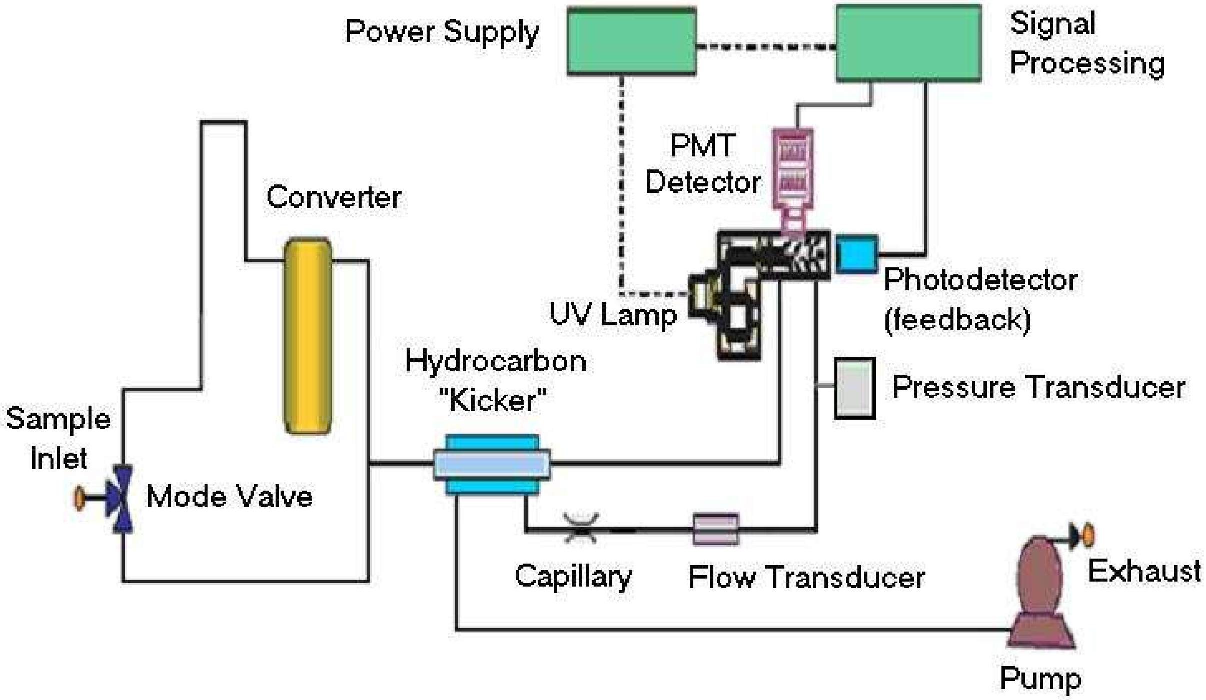

Figure 1. Flow schematic of the 450I.

3. Definitions of Terms and Acronyms

AirDAC	Data acquisition and control software for barn applications (SOP B2)
CS	Combined sulfur (H_2S + SO_2)
CAFO	Confined animal feeding operation
GSS	Gas sampling system (SOP G1)
Hydrocarbon kicker	A membrane tube in the 450I that removes aromatic hydrocarbons based on their ability to pass through the membrane. SO_2 molecules pass through the hydrocarbon kicker unaffected.
QAPP	Quality Assurance Project Plan
OFIS	On-farm instrument shelter (SOP U1)
PAAQL	Purdue Agricultural Air Quality Laboratory
PMT	Photomultiplier tube

4. Health and Safety

4.1. Be careful when working with the electrical power connection.
4.2. The converter of the Model 450I contains high-temperature elements (300-400°C), and should not be touched.
4.3. Properly vent analyzer exhaust to avoid exposure to noxious gases in the sample.
4.4. Prevent leaking of the calibration gases into the working environment.

5. Cautions

5.1. Use antistatic wrist strap or discharge your body's electrostatic charge to a grounded metallic frame before touching any internal components of the 450I to avoid damage of the electronics by static discharge. See Chapter 7 ("Servicing") of the 450I manual.

6. Interferences

6.1. Compounds that interfere with fluorescence-based H_2S measurements are as follows:

Interferences	**Effect on H_2S Readings**
Methyl mercaptan	0.8 ppm increase per 1 ppm of CH_3SH
Dimethyl disulfide	0.5 ppm increase per 1 ppm of CH_3SSCH_3
Dimethl sulfide	< 0.1 ppm increase per 1 ppm of CH_3SCH_3
NO	Increase of < 3 ppb at 500 ppb NO
m-xylene	Increase of < 1 ppb at 200 ppb *m*-xylene
Water	< 2% of reading per 2% humidity ratio

6.1.1. The effect of humidity will typically be less than 3% of reading. For example, the difference in humidity ratio between saturated air at 30°C (86°F) and saturated air at 0°C (32°F) is approximately 2.3%

6.1.2. H_2S is expected to be present at one or two orders of magnitude higher than dimethyl sulfide or dimethyl disulfide; thus, the relative magnitude of these interferences should be minor. Dimethyl sulfide and dimethyl disulfide can be monitored with the Innova Model 1412 Multi-Gas Monitor (SOP G7).

6.1.3. Concentrations of NO and *m*-xylene are expected to be negligible.

7. Personnel Qualifications

7.1. Personnel should be trained in the operation, maintenance and calibration of the analyzer before initiating the procedure. Training requires about three hours.

7.2. Each analyst must read and understand the entire analyzer manual and this SOP before operating the Model 450I.

7.3. Personnel should also be trained to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

8. Equipment and Supplies

8.1. Hydrogen sulfide analyzer (Model 450I, Thermo Electron Corporation)

8.2. H_2S-to-SO_2 converter (Model 340, Thermo Electron Corporation)

8.3. Keep the following spare parts at each site:

8.3.1. 20-mil capillary, Part No. 4126

8.3.2. O-ring capillary, Part No. 4800

8.3.3. Teflon tubing (1/4″ OD x 1/8″ ID) for connecting analyzer to GSS and exhaust port

8.3.4. 4-conductor cable (shielded, 22-gauge)

8.4. Keep the following spare parts in a central stock at PAAQL:
  8.4.1. 20-mil capillary, Part No. 4126
  8.4.2. O-ring capillary, Part No. 4800
  8.4.3. Pump, 110 VAC, Part No. 8550
  8.4.4. Pump repair kit, Part No. 8606
  8.4.5. Flash lamp, Part No. 8666
  8.4.6. Particulate filter assembly, Part No. 60100
  8.4.7. Converter assembly, 120 VAC, Part No. 450P702-1
  8.4.8. Power supply board, Part No. 10570
  8.4.9. Trigger pak, Part No. 8774
  8.4.10. Flash intensity board, Part No. 8884
  8.4.11. Flasher supply board, Part No. 9681
  8.4.12. Temperature control board, Part No. 8765
  8.4.13. PMT high voltage power supply, Part No. 9901
  8.4.14. Temperature control board (converter), Part No. 9889
  8.4.15. Teflon tubing (1/4″ OD x 1/8″ ID) for connecting analyzer to GSS and exhaust port
  8.4.16. 4-conductor cable (shielded, 22-gauge)

8.5. Calibration gas cylinders: Hydrogen sulfide (H_2S), sulfur dioxide (SO_2), zero gas (see the QAPP for exact specifications and compositions), with appropriate regulators (SOP G2)
  8.5.1. In projects where SO_2 is not a primary measurement, it may still be used to check the converter efficiency (Section 9.3).

9. Procedure

9.1. Model 450I Analyzer setup
  9.1.1. Check the exterior of the analyzer (Fig. 2) carefully to assure it is free of visible defects or damage.
    9.1.1.1. Remove the instrument cover to expose the internal components (Fig. 3).
    9.1.1.2. Remove any packing material, check for possible shipping damage and ensure that all connectors and circuit boards are firmly attached.
    9.1.1.3. Replace the instrument cover.
  9.1.2. Securely mount the analyzer in the instrument rack.
  9.1.3. Connect the analyzer manifold in the GSS to the “SAMPLE” bulkhead (Fig. 2) on the instrument’s rear panel using ¼” OD (1/8” ID) Teflon tubing less than 10’ long. Allow enough slack to permit movement of the instrument rack.
  9.1.4. For barn applications:
    9.1.4.1. Connect the “EXHAUST” bulkhead to the OFIS air exhaust with 1/4″ OD (1/8″ ID) tubing less than 10′ long, which is unimpeded and free of kinks or blockages.
    9.1.4.2. Connect a 4-conductor, 20- or 22-gage shielded cable to the designated channels of the FieldPoint analog input module (SOP B1). Properly ground the shield of the analog output cable.
    9.1.4.3. Connect the other end of the 4-conductor cable to the analyzer’s analog outputs for SO_2 and H_2S (not to the CS analog output).
      9.1.4.3.1. Remove about 1.8 cm of insulation from the other end of the cable. Fold back the shielding. If necessary, use electrical tape or shrink tubing to hold

shielding in place. Expose a minimum of 1.5 cm of shielding. Strip each signal wire and connect them to the terminals (1) through (4) of the 8-position header of the 450I (Fig. 4) according to the wire connection to the data acquisition hardware

9.1.5. For open source applications:

- 9.1.5.1. Connect the “EXHAUST” bulkhead to the GSS bypass air exhaust with 1/4″ OD (1/8″ ID) tubing such that total tubing length is less than 10′ long. Tubing must be unimpeded and free of kinks or blockages.
- 9.1.5.2. Connect a null modem RS232 cable from the RS232 connector of the data-collecting computer to the “RS232/485” connector at the back of the instrument.

Figure 2. Front (top) and back (bottom) views of the TEC 450I.

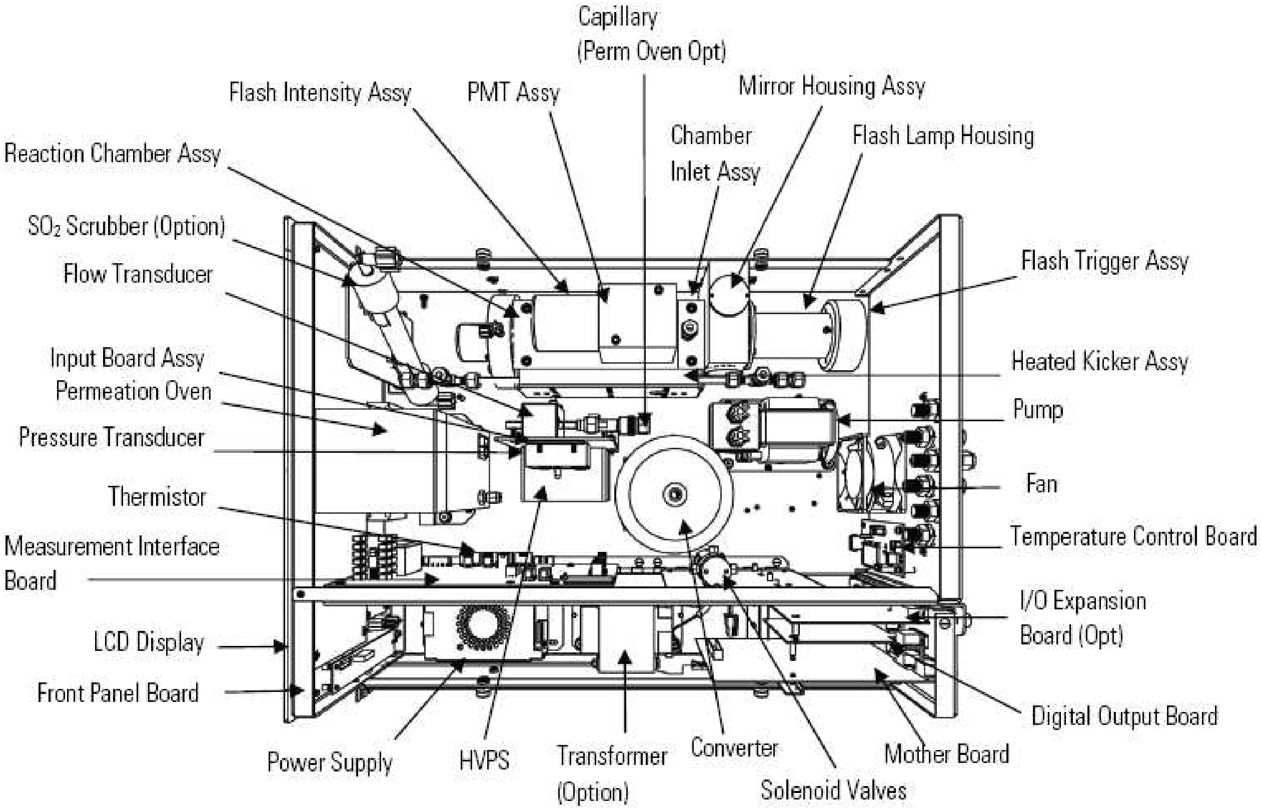

Figure 3. Internal schematic of the TEC 450I analyzer.

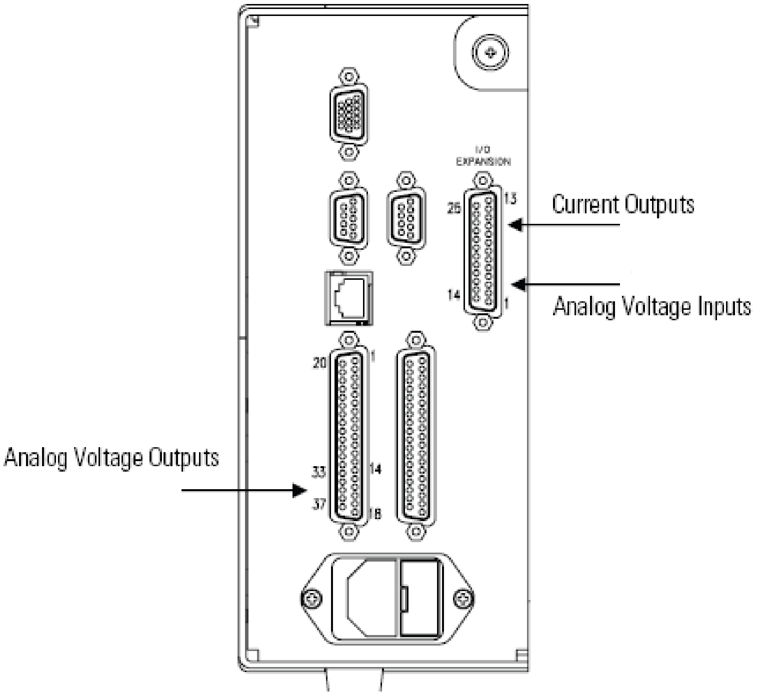

Figure 4. Connectors on the 450I rear panel (left side of Fig. 2 lower panel).

9.1.6. Plug the 450I power cord into a 110VAC power source, turn on its power, and allow a minimum of 90 min for the unit to warm up and stabilize before calibrating.

9.1.7. Operate the instrument menu (Fig. 5) to navigate to the screens necessary to set the instrument control settings (Table 1) and to check the operating parameters and ranges (Table 2), as described in detail in Chapter 3 of the instrument manual. An alarm message will appear in the screen on the front panel of the instrument when any of these parameters is operating outside the set limits.

Table 1. TEC 450I Instrument settings.

Parameter	**Setting**
Gas Units	ppb
Measurement Mode	Automatic Mode
Averaging Time	60 s
Temperature Correction	ON
Pressure Correction	ON
Flash Lamp	ON
Baud Rate	9600
Service Mode	OFF
Time	Current
Date	Current
Converter Temperature	352°F

Table 2. TEC 450I instrument operating parameters and ranges.

Parameter	**Range**
Pressure (mm Hg)	400 – 1000
Sample Flow (L/min)	0.350 – 1.400
Lamp Voltage (V)	500 – 1200
Lamp Frequency (Hz)	10,000 – 50,000
Converter Temperature (°C)	300 – 400
Internal Temperature (°C)	15 – 45
Chamber Temperature (°C)	43 – 47

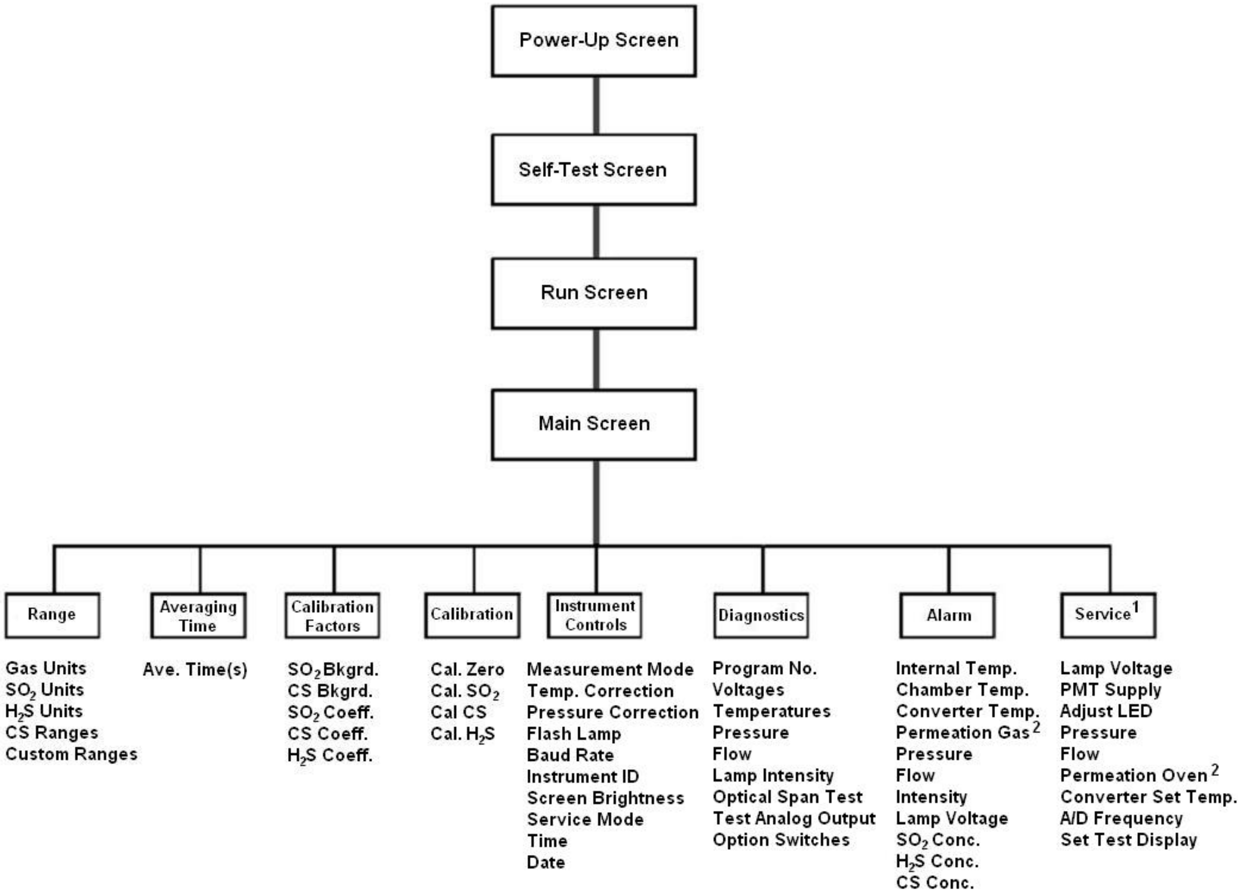

Figure 5. Partial flow chart of the TEC 450I menu from the instrument manual. Notes: 1 – Option only appears when the Service Mode is ON. 2 – Appears only when optional Permeation Oven is installed.

9.1.8. For barn measurements:
  9.1.8.1. The analyte (H_2S, SO_2, CS) measurement ranges for the 450I are from 0 to 10,000 ppb. The analyte measurement range settings for each site are listed in the QAPP.
  9.1.8.2. Enter the analyzer’s analog signal ranges (0 to 10 VDC) and H_2S and SO_2 measurement ranges into the DAQ table in the AirDAC software (SOP B2) so that concentration reading can be displayed, converted and recorded.
9.1.9. For open source measurements:
  9.1.9.1. Using iPort software, set data logging settings in instrument
    9.1.9.1.1. Under “Main Menu->Instrument Controls->Datalogging settings”:
      9.1.9.1.1.1. Set “Select SREC/LREC” to “LREC”

9.1.9.1.1.2. Under “Select Content”, make sure the following appears

“FIELD 1	H2S”	
“FIELD 2	SO2”	
“FIELD 3	INTT”	(corresponds to INT TEMP)
“FIELD 4	RCTT”	(corresponds to CHAMBER TEMP)
“FIELD 5	CONVT”	(corresponds to H2S CNV TEMP)
“FIELD 6	PRES”	(corresponds to CHAMBER PRES)
“FIELD 7	SMPLFL”	(corresponds to SAMPLE FLOW)
“FIELD 8	PMTV”	(corresponds to PMT VOLTS)
“FIELD 9	LMPV”	(corresponds to FLASH VOLTS)
“FIELD 10	LMPI”	(corresponds to FLASH REF)
“FIELD 11	NONE”	
……….		
“FIELD 32	NONE”	

9.1.9.1.1.3. Select any fields that are incorrect and modify as necessary. The item type for fields 1 and 2 is “CONCENTRATIONS” while that for the remaining 8 fields is “OTHER MEASUREMENTS”

9.1.9.1.1.4. *If changes were made to any of the above fields and* ***if all data on the instrument have been saved to the LAN:*** Go to “Main Menu->Instrument Controls->Datalogging Settings->Commit Content”; You will be asked to confirm that you want to commit content. Only say “YES” if you are positive that all data logged on the analyzer have been saved

9.1.9.1.1.5. Check configuration of the data logging at “Main Menu->Instrument Controls->Datalogging Settings->Configure Data logging”
-LOGGING PERIOD, MIN should be set to 1 MIN
-MEMORY ALLOCATION SHOULD BE SET TO 100%
-DATA TREATMENT should be set to AVG

9.1.9.1.1.6. Check the time zone setting; “Main Menu->Instrument Controls->Timezone”; should be set to UTC(GMT).

9.1.9.1.1.7. Set Ethernet communications at “Main Menu->Instrument Controls->Communication Settings->TCP/IP Settings->”
->USE DHCP: OFF
->IP ADDR: 192.168.0.15
->NETMASK: 255.255.255.0
->GATEWAY: 192.168.0.1
->NTP Server: 132.239.1.6

9.1.9.1.1.8. If any value is modified, turn the instrument off and back on again.

9.1.9.2. Using the iPort software, prepare instrument for transmission and storage of the analyzer’s quality assurance and gas concentration measurements

9.1.9.2.1. Under “File->Preferences>instrument”:

9.1.9.2.1.1. Set “size” to FULL

9.1.9.2.1.2. Set “Data Record Loading” to “Save to File” and “Create”

9.1.9.2.2. Under “Comm->Connect”, assure settings are:
Location: H2Sanalyzer
Com: (set to correct value)

Baud Rate: 115200
Len: 8
Stop: 1
Parity: N
Handshake: None
Polling Config: 45 (this is the instrument ID)

9.1.9.2.3. Click on “OK” and respond “YES” to attach instrument.

9.1.9.2.4. Real-time data file save is set up under “Instrument->Load Records”: Click on “Real time” and click “Save to file”. Remove dash and spaces from the default file name (before the date and before the time compoents) and add the path and site code before the filename (e.g. C:\NAEMS\Data\OK4A\Period3\H2S\OK4A), and click “OK”

9.1.9.3. Verify that the output file is being produced in the desired location and check the contents by right clicking and opening with ‘Windows Notepad’. File should appear as:

```
;;
;; C:\NAEMS\Data\450I0103082156.dat Thu Jan 03 21:57:05 2008
;;
;; Model 450I- (prog iSeries 450I 01.05.06.165)
;;
Time   Date    Flags      h2s      so2     intt     rctt    convt     pres   smplfl     pmtv     lmpv     lmpi
14:53  01-03-08 LL------    1.052  9.36528  10.8132  32.8277   322.29  661.186  1.06992 -647.131  808.951       89
14:54  01-03-08 LL------  1.48701  8.50626  10.9028  33.1631  324.389  656.023  1.06526 -647.131   809.76       88
14:55  01-03-08 LL------  1.40748  7.61518  11.0148  33.4469  327.276  663.312  1.07233 -647.131  809.356       87
14:56  01-03-08 LL------ 0.247904  7.33171  11.1044  33.8854  326.751  658.149  1.06647 -647.131  810.973       88
14:57  01-03-08 LL------ 0.142362  7.49943  11.2164   34.066  323.865  663.312  1.07233 -647.131  808.143       88
14:58  01-03-08 LL------ 0.912892  7.91842  11.2612  34.1692  322.552  656.023  1.06492 -646.761  809.356       89
14:59  01-03-08 LL------ 0.957739  8.52361  11.3956  34.4788  325.177  661.186  1.07026 -647.131  808.547       89
15:02  01-03-08 LL---LL-        0        0   11.642   35.132  323.602  661.489  1.07164 -647.131  808.547       88
15:03  01-03-08 LL------        0        0   11.754   35.352  323.077  655.719  1.06544 -646.761  807.738       88
```

9.2. Gas Analyzer Calibration

9.2.1. Reference calibration gases

9.2.1.1. Sulfur Dioxide (SO_2) in N_2 - exact composition as specified in the QAPP.

9.2.1.2. Hydrogen Sulfide (H_2S) in N_2 or air - exact composition as specified in the QAPP.

9.2.1.3. Zero air, also referred to as Continuous Emission Monitoring (CEM) Zero Air.

9.2.2. For barn measurements, set AirDAC to “Gas Cal” (Gas Calibration) and calibrate the 450I following SOPs G8 and G9

9.2.3. For open source measurements:

9.2.3.1. Start the 450I gas analyzer and let it warm up for at least 30 minutes.

9.2.3.2. Use Environics Series 4040 Gas Dilution System or TEC 146I dilutor to dilute H_2S levels if necessary.

9.2.3.2.1. Install gas line from exhaust of 450I to vent into trailer or outside

9.2.3.2.2. Install gas line from output port of dilutor to sample port of 450I, with a tee in the line to vent excess to outside through a flowmeter; this prevents over-pressurizing the 450I and verifies sufficient flow for 450I sampling.

9.2.3.2.3. Open both gas cylinders (calibration gas and zero air) to a pressure of 20 psi, then

9.2.3.2.4. Initial calibration gas is 0 ppm H_2S.

9.2.3.2.5. If using the Environics dilutor:

9.2.3.2.5.1. Attach gas lines of H_2S cylinder and Zero Air cylinder to ports 2 and 1 (respectively) of Environics dilutor.

9.2.3.2.5.2. Set the H_2S concentration in the Environics dilutor program to the chosen concentration according to SOP G11.

9.2.3.2.6. If using the TEC 146I dilutor:

9.2.3.2.6.1. Attach gas lines from calibration cylinder to port A (or other programmed Port), and from the zero air cylinder to Zero Air Port.

9.2.3.2.6.1.1. Connect gas line from zero air cylinder (for 0 ppm H_2S) to sample port of 450I, with a tee in the line that vents exhaust (this prevents over-pressurizing the 450I).

9.2.3.2.6.2. Set the H_2S concentration on the diluter (not 0 ppm) to the chosen concentration according to SOP C3.

9.2.3.2.7. Open flow valve of regulator. Flow calibration gas through the 450I and wait 5-10 min. for the H_2S concentration reading on the 450I to stabilize.

9.2.3.2.8. Repeat sections 9.2.3.2.4, 9.2.3.2.6 (Environics diluter) or 9.2.3.2.5, 9.2.3.2.6 (TEC 146i diluter) using calibration gas at appropriate concentration.

9.2.3.2.8.1. Record pressure, flow rate, lamp intensity, and lamp voltage from 450I on a form similar to Table 3.

9.2.3.2.8.2. Once stabilized record H_2S output concentration from dilutor and H_2S concentration reading on the 450I.

9.2.3.3. If multipoint calibration, repeat section 9.2.3.2 for each target concentration, running a zero test in between span checks (the four concentrations are 3ppm, 2ppm, 1ppm, and 0.5ppm) in decreasing concentration and record results on form similar to Table 3.

9.2.3.3.1. Calculate linear regression of instrument response to concentration and apply to records as a post-process.

9.2.3.4. If calibration verification check, repeat section 9.2.3.2 for zero and 0.5 ppm concentrations respectively.

Table 3 – Example Multi-point calibration form for Open source instruments

450I Gas Analyzer Multi-Point Calibration

Serial #	Time	Pressure	Flow	Lamp Int.	Lamp Volt.	H2S cyl. Conc.	Dilutor Conc.	450I H2S reading
						0		
						3 0		
						0		
						2 0		
						0		
						1 0		
						0		
						0 5		
						0		

9.2.4. Recording information during a calibration or precision check

9.2.4.1. Record the analyzer's responses to both zero and reference (precision check) gases before and after adjusting the analyzer. The recorded responses before adjustment (as-found responses), are used to correct the data since the previous calibration or previous precision checks.

9.2.4.2. Record 450I pressure, sample flow, lamp intensity, and lamp voltage in the electronic field notes in a table like Table 4.

9.2.4.3. For barn measurements: Record the start / stop (read) times, analyzer, calibration gas, calibration gas concentration, analyzer reading, AirDAC concentration reading, and AirDAC signal in the field notes like in Table 5 (Precision Checks) or Table 6 (Calibration)

9.2.4.4. For open source measurements: Record the start / stop (read) times, analyzer, calibration gas, calibration gas concentration, analyzer reading, data file concentration reading in the field notes like in Table 7 (Calibration Verification/Precision Checks).

9.2.5. Introduce reference gas. The number of points and gas concentrations for multipoint calibrations and calibration verification/ precision checks are specified in the Quality Objectives of the QAPP.

9.2.5.1. Start flow of reference gas to the analyzer. See SOP G1 for delivery options

9.2.5.2. Record date and time in the electronic field notes in a table like Table 5.

9.2.5.3. Record pertinent gas delivery parameters (SOP G1).

9.2.5.4. Wait a minimum of 10 min for the display to stabilize (The minimum of 10 min corresponds with the sampling time used for each location).

9.2.5.5. Record the time and concentrations shown on the 450I display, and in AirDAC (barn measurements), on provided form and Excel® spreadsheet form (open area measurements) and in the electronic or paper field notes. Update the control chart in accordance with SOP Q1.

Table 4. Recording of TEC 450I operating parameters.

Date	TEC 450I Operating Parameters			
	Pressure,	Sample Flow	Lamp Intensity	Lamp Voltage
	mm Hg	L/min	Hz	V
3/07/2006	558.6	0.996	21173	1095
3/12/2006	557.5	1.040	20998	1094
"	"	"	"	"
"	"	"	"	"

9.2.5.6. If the stabilized concentration during a precision check deviates from the control chart baseline calibration value by 10% or more, conduct a multipoint calibration in accordance with SOP G9 (barn measurements) or Section 9.2.3.3 of this SOP (open area measurements).

9.2.5.7. Stop gas flow and record the time.

9.2.6. Repeat the steps in Section 9.2.5.for each reference gas.

9.2.7. Return analyzer's display back to the run screen by pressing run.

9.2.8. For barn measurements, return the GSS back to normal sampling mode by setting AirDAC to auto sample.

9.3. Converter Efficiency Check

9.3.1. Following Section 9.2, calibrate the SO_2 and CS channels with the SO_2 calibration gas. Then, calibrate the H_2S channel with the H_2S calibration gas.

9.3.2. Navigate to the “Calibration Factors” Menu (Fig. 5). The H_2S coefficient shown here is the converter efficiency.

9.3.2.1. For example, if the H_2S coefficient is 0.905, the converter efficiency is 90.5%.

9.4. Maintenance

9.4.1. Preventive maintenance is covered in Chapter 5 of the 450I Instrument Manual.

9.4.1.1. Clean the dust filters on the back of the analyzer and converter on a regular basis. This can range from weekly to monthly, depending on the dust level in the OFIS.

9.4.1.2. If the lamp voltage is equal to or greater than 1200 V, lower the voltage by adjusting the potentiometer (R28) on the lamp power supply board (See Chapter 7 of the Instrument Manual for Servicing), or replace the lamp.

9.4.1.3. Run a leak test if the sample flow rate, as reported by AirDAC (barn measurements) or iPORT (open area measurements), is < 0.9 L/min.

9.4.1.3.1. Block the bulkhead labeled “SAMPLE” on rear panel with a leak-tight cap then wait 5 to 10 min.

9.4.1.3.2. Check the sample flow and pressure readings. There should be zero flow and less than 160 mm Hg pressure. If not, check to see that none of the fittings or threads of the input lines are cracked or broken.

9.4.1.4. Check the capillary tubes for particulate deposits within the bore. Clean or replace as necessary.

9.4.1.5. Conduct a zero & precision span check before and after any maintenance, except for cleaning of the dust filter.

9.5. Troubleshooting

9.5.1. Troubleshooting guidelines are in Chapter 6 of the 450I manual.

9.5.1.1. Check wiring, power connections, and fuses if there is no signal or power.

9.5.1.2. Check flow path integrity if span concentration cannot be attained, or if flow rate is low.

9.5.1.3. If there is no or low flow, check the capillary tube or internal pump.

9.5.1.4. Replace lamp if the lamp intensity is varying widely (ranging quickly from 10,000 to 50,000 Hz).

9.6. Data acquisition, calculations & data reduction requirements

9.6.1. Barn measurements: Monitor a 0-10 VDC analog output signal using AirDAC (SOP B2).

9.6.2. Open area measurements: Monitor measurements using iPort software.

9.6.3. Report all data with 3 significant digits.

9.6.4. Follow SOP B4 to correct gas concentrations for calibration and air density.

9.7. Computer hardware & software

9.7.1. Barn measurements:

9.7.1.1. DAQ hardware (SOP B1)

9.7.1.2. AirDAC (SOP B2)

9.7.1.3. ARDIS (SOP B3)

9.7.1.4. CAPECAB (SOP B6)
9.7.2. Open area measurements:
9.7.2.1. LAN server (SOP U4)
9.7.2.2. iPort software

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
10.2. Manage all data according to SOP B5 (barn measurements) or SOP D1 (open area measurements).
10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
10.4. Overstrike all errors in writing with a single line, and initial and date all such corrections.
10.4.1. Open area measurements:
10.4.1.1. LAN server (SOP U4)
10.4.1.2. iPort software

11. Quality Control and Quality Assurance

11.1. Conduct zero and one or more precision checks at the QAPP-specified frequency.
11.2. Conduct multipoint calibrations at the QAPP-specified frequency
11.3. Conduct a multipoint calibration whenever a precision check indicates a drift of > ±10% from the baseline calibration value, unless otherwise stated in the QAPP for a particular project.
11.4. Conduct a multipoint calibration whenever the zero check is > ±5% of full scale different than the baseline zero, unless otherwise stated in the QAPP for the particular project.
11.5. Check response time every month for barn measurements, and every six months for open area measurements.
11.6. In cases where SO_2 is not a primary measurement, weekly zero/precision checks with SO_2 are not necessary. In these cases, the QAPP will dictate the frequency of converter efficiency checks, and the converter efficiency that must be maintained.
11.7. Perform preventative maintenance every six months, according to the schedule provided in Chapter 5 of the 450I manual.
11.7.1. Visually inspect for obvious visible defects (loose connections and/or fittings, cracked or clogged Teflon lines, and excessive dust accumulation.
11.7.2. Inspect the fan filter located on the back of the instrument weekly; clean as needed.
11.7.3. The schedule for replacing the sample particulate filter is found in the QAPP.
11.7.4. Measure the gas analyzer inlet flow rate every six months.
11.7.5. Check the lamp voltage under the diagnostics menu. If the lamp voltage is 1200 V or higher, replace the lamp or adjust its voltage.
11.7.6. Leak check the sample line and flow components and fittings every six months.

11.8. If troubleshooting of the analyzer reveals particulate deposits in the capillaries, these must be replaced.

12. References

12.1. iPort Instruction Manual. 2007. *i*Port Instruction Manual. Communications software for iSeries and C Series Instruments. Part#102606-00. Thermo Fisher Scientific, Franklin, MA.
12.2. TFS 450I Manual. 2006. Model 450I Pulsed Fluorescence SO_2- H_2S-CS Analyzer Instruction Manual. Part #103258-00. Thermo Fisher Scientific, Franklin, MA.
12.3. SOP B1. 2007. Data Acquisition and Control Hardware. Standard Operating Procedure B1, v. 1.0. Purdue Ag Air Quality Lab.
12.4. SOP B2. 2007. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B, v. 1.02. Purdue Ag Air Quality Lab.
12.5. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3, v. 0.0. Purdue Ag Air Quality Lab.
12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5, v. 0.0. Purdue Ag Air Quality Lab.
12.7. SOP B6. 2007. Data Processing Software (CAPECAB). Standard Operating Procedure B6, v. 1.0. Purdue Ag Air Quality Lab.
12.8. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1, v. 0.0. Purdue Ag Air Quality Lab.
12.9. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2, v. 0.0. Purdue Ag Air Quality Lab.
12.10. SOP G7. 2008. Use of the INNOVA 1412 Photoacoustic Multi-Gas Monitor. Standard Operating Procedure G7, v. 1.0. Purdue Ag Air Quality Lab.
12.11. SOP G8. 2007. Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8, v. 1.0. Purdue Ag Air Quality Lab.
12.12. SOP G9. 2006. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure G9, v. 0.0. Purdue Ag Air Quality Lab.
12.13. SOP G11. 2007. Operation of the Environics® Computerized Gas Dilution System. Standard Operating Procedure G11, v. 1.0. Purdue Ag Air Quality Lab.
12.14. SOP Q1. 2007. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1, v. 1.0. Purdue Ag Air Quality Lab.

13. Contact Information

13.1. Manufacturer: Thermo Fisher Scientific (www.thermo.com), 8 West Forge Parkway, Franklin, MA 02038, Technical Support: 1-866-282-0430
13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, http://www.AgAirQuality.com.

Table 5. TEC 450I Analyzer Precision Check - Barn Measurements.

Precision Checks							
3/12/2006				Concentration	Instrument	Lab View	Signal
Started	Stopped	Instrument	Gas	ppb	Reading	ppm /ppb	vdc or mA
4/28/2006 14:05	4/28/2006 14:15	TEC 450I (H2S)	CEM Zero	0	-3.1	2.55	0.0005
4/28/2006 14:05	4/28/2006 14:15	TEC 450I (SO2)	CEM Zero	0	0	0.98	0.0008
4/28/2006 14:16	4/28/2006 14:26	TEC 450I (H2S)	H2S	2080	2090	2101.22	2.090
4/28/2006 14:16	4/28/2006 14:26	TEC 450I (SO2)	H2S	2080	10	10.78	0.011
4/28/2006 14:27	4/28/2006 14:37	TEC 450I (H2S)	SO2	1900	-106	-107.74	-0.109
4/28/2006 14:27	4/28/2006 14:37	TEC 450I (SO2)	SO2	1900	1907	1904.71	1.912

Table 6. TEC 450I Analyzer Calibration Check- Barn Measurements.

Calibration (H2S)							
8/9/2005				Concentration	Instrument	Lab View	Signal
Started	Stopped	Instrument / Comments	Gas	ppb	Reading	ppb	vdc
8/9/2005 8:55	8/9/2005 9:10	TEC 450I	CEM Zero	0	-33	-38	0.059
8/9/2005 9:11	8/9/2005 9:22		H2S	4370	4570	4551	9.091
8/9/2005 9:23	8/9/2005 9:35	Recorded reading	CEM Zero	0	-25.7	-33	-0.049
	8/9/2005 9:37	Zero-ed instrument		0			
	8/9/2005 9:39	Recorded reading (9:39:19)		0	0	0	0.001
8/9/2005 9:11	8/9/2005 9:22	Recorded reading	H2S	4370	4540	4536	9.071
	8/9/2005 9:52	Adjusted instrument to H2S span of 4370 ppb					
	8/9/2005 9:53	Recorded reading (9:53:08)			4370	4371	8.741
8/9/2005 9:54	8/9/2005 10:06		CEM Zero	0	3.6	5.0	0.011
8/9/2005 10:06	8/9/2005 10:17		H2S	4370	4370	4376	8.751
8/9/2005 10:17	8/9/2005 10:28		CEM Zero	0	0.4	0	0.001
8/9/2005 10:28	8/9/2005 10:39		H2S	4370	4420	4416	8.831
8/9/2005 11:19	8/9/2005 11:29	Baseline	CEM Zero	0	-1.6	-2	0.001
8/9/2005 11:29	8/9/2005 11:39	Baseline	H2S	4370	4380	4361	8.771

Table 7. TEC 450I Analyzer Calibration Verification Check- Open Area Measurements.

3/12/2006			Concentration	Instrument Reading	Deviation from baseline	Site FOS
Started	Stopped	Gas	ppb			
4/28/2006 14:05	4/28/2006 14:15	CEM Zero	0	-3.1		
4/28/2006 14:05	4/28/2006 14:15	CEM Zero	0	0		
4/28/2006 14:16	4/28/2006 14:26	H2S	2080	2090		
4/28/2006 14:16	4/28/2006 14:26	H2S	2080	10		

USE OF THE INNOVA 1412 PHOTOACOUSTIC MULTI-GAS MONITOR

Standard Operating Procedure (SOP) G7

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Standard Operating Procedure (SOP) G7

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1. Scope and Applicability

1.1. The purpose of this procedure is to present the methodology and instrumentation used to automatically and sequentially measure concentrations of various gases at confined animal feeding operations (CAFO), specifically, within the barns and other buildings, and around open sources such as corrals, manure lagoons, or storage basins.

1.2. The method described in this SOP is suitable for measurement of any gas which absorbs infrared (IR) radiation. Gas selectivity is achieved through the use of optical filters. Gases which can be measured through this method, and which are relevant to CAFOs, include:

  1.2.1. Ammonia (NH_3)
  1.2.2. Ethanol/methanol (total alcohols)
  1.2.3. Acetic acid
  1.2.4. Acetaldehyde
  1.2.5. Dimethyl sulfide
  1.2.6. Carbon dioxide
  1.2.7. Methane
  1.2.8. Phenol
  1.2.9. Total VOC

1.3. The QAPP for a particular project will specify which gases will actually be measured.

1.4. This procedure applies only to the INNOVA Model 1412 Photoacoustic Monitor.

  1.4.1. The minimum detection limit (MDL) of this instrument is gas-dependent, but is typically in the ppb region. It is often below 100 ppb, with the exception of ammonia, which has a minimum detection limit of 200 ppb.

  1.4.2. The range is also gas-specific. The linear range for a given gas is typically from 1 to 10,000 times the MDL.

    1.4.2.1. For example, the Model 1412's linear range for ammonia is from 0.2 ppm (the MDL) to 2000 ppm.

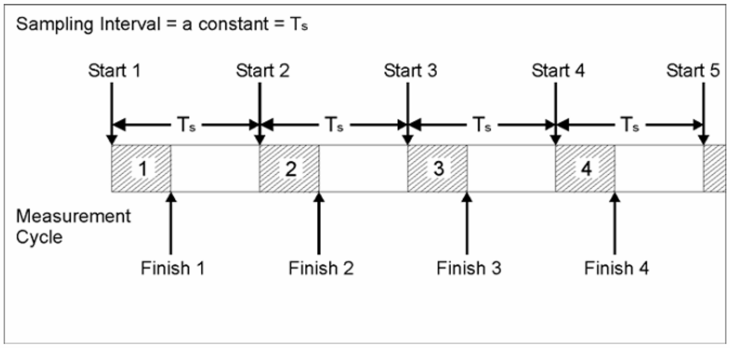
  1.4.3. The stated precision of the Model 1412 is 1% of measured value.

  1.4.4. The instrument's stated response time (97%) to a step change in concentration is 12 s.

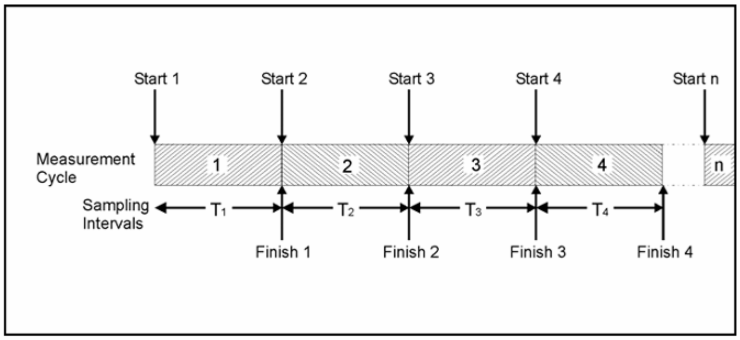
2. Summary of Method

The multi-gas monitor (INNOVA Model 1412, Innova AirTech Instruments, Ballerup, Denmark) is based on the photoacoustic infrared detection method. As shown in the schematic diagram in Fig. 1, a gas sample is drawn at a constant flow rate (in the case of the barns, from the GSS analyzer manifold), and introduced into an acoustic cell where it is exposed to pulsing infrared light of specific wavelength. If the sample contains the gas of interest, it will absorb an amount of infrared light that is proportional to the analyte gas concentration in the sample. When a gas absorbs infrared light, its temperature rises as its molecules increase in kinetic energy; this causes a pressure wave inside the photoacoustic cell. Pressure pulses, which are also proportional to the concentration of the analyte gas, thus correspond to the light pulses, and are detected by two sensitive microphones located inside the chamber. Almost any gas that absorbs infrared light can be measured by this method.

Gas analysis with the Model 1412 can be conducted either using set sampling intervals, or in continuous mode. Sampling interval is a measure of the time between the start of one measurement cycle and the start of the next measurement cycle. Therefore, if a measurement cycle (including readings of all gases being measured) takes 27 s, and the sampling interval is 1 min, then there will be 33 s before the next measurement cycle starts, as follows:



In continuous sampling mode, each measurement cycle is followed immediately by another similar measurement cycle, as below:



In either mode, a measurement cycle consists of the following steps:

- Measurement of the gas sample with each chosen individual optical filter (up to 5)
- Measurement of the gas sample with the water vapor optical filter
- Flushing the chamber & tubing and filling the chamber with the next gas sample

Gas selectivity is achieved through the use of optical filters. The Innova 1412's optical filter carousel can hold 5 optical filters for target gases, plus one for water vapor, thus allowing measurement of up to 5 gases and water vapor in a given air sample. Each individual optical filter rotates in front of the photoacoustic chamber, and pauses for the set sample integration time (SIT) before moving to the next filter. All filters rotate through once every measurement cycle. The MDL is gas-specific and filter wavelength-specific; examples include:

Acetic acid	40 ppb	Total TOC (reference propane)	20 ppb
Ammonia	200 ppb	Methane	100 ppb
Carbon dioxide	1500 ppb	Sulfur dioxide	300 ppb

Measurement accuracy is ensured by the Model 1412's ability to compensate for temperature and pressure fluctuations, water-vapor interference and interference from other IR-absorbing gases that are known to be present. Reliability of measurement results can be ensured by regular self-tests. The analysis time is dependent on the physical properties of the gas of interest, the number of gases being measured, the set-up of the instrument, the gas sampling system, and the sampling location. When a gas or a gas mixture is introduced to the analyzer, the response time can vary from approximately 13 s for one gas (or water vapor) to approximately 150 s if five gases plus water vapor are measured. The following table indicates in more detail the effect of analyzer configuration on analysis time:

Sample integration time, s	1	1	5	5	20
Water vapor measured?	no	yes	no	yes	yes
Number of gases	1	5	1	5	5
Flushing time, s	4	4	Auto (tube 1 m)	Auto (tube 1 m)	Auto (tube 1 m)
Analysis Time, s	**~13**	**~27**	**~25**	**~60**	**~150**

3. Definitions of Terms and Acronyms

AirDAC	Data Acquisition and Control software (SOP B2)
Analysis Time	The total time required to measure each gas's response to infrared light (SIT) and to flush the photoacoustic cell.
GSS	Gas sampling system (SOP G1)
IR	Infrared
LAN	Local area network
MDL	Minimum detection limit
NIST	National Institutes of Science and Technology
NMi	Netherlands Measurement Institute
PAAQL	Purdue Agricultural Air Quality Laboratory
PAML	Purdue Applied Meteorology Laboratory
PC	Personal computer
ppb	Parts per billion
ppm	Parts per million
QAPP	Quality Assurance Project Plan
SIT	Sample integration time

4. Health and Safety

4.1. Be careful when working with the analyzer's electrical power connection.
4.2. The IR source and analysis cell are very hot when the analyzer is operating. Do not attempt to service any internal components without allowing adequate cool-down time.
4.3. If used indoors (such as inside the OFIS). Properly vent the analyzer's exhaust to avoid exposure to noxious gases in the sample.

5. Interferences

5.1. Water absorbs IR light at most wavelengths, and is nearly always present in air samples. Thus, water will essentially always contribute to the total acoustic signal. The Model 1412 is permanently fitted with a special water-selective filter, which enables it to compensate for this interference.

5.2. By selecting different filters, this technique can also be used to cross-compensate for known interferents (i.e. gases which are not target analytes, but which are known to absorb IR at the wavelength(s) used to quantify one or more target compounds.

5.2.1. Possible interferent gases relevant to livestock barns, anaerobic treatment lagoons, manure storages, and related facilities (Schmidt et al, 2005; Summers, 2005) include the following (levels are based on detection limits in Innova AirTech Instruments' "Detection Limits Wall Chart" (Reference 12.3) for these gases at the same wavelength used for quantifying the given primary analyte):

5.2.1.1. **Ammonia** can be quantified at either of two wavelengths – 974 µm or 976 µm. The stated detection limit at each of these wavelengths is 200 ppb. Some of these compounds will interfere more at one wavelength (indicated in parentheses) and less (but still possibly significantly) at the other wavelength. If VOC characterization of the site indicates that any of these compounds are present above the indicated thresholds, it may be necessary to compensate for them in order to obtain accurate NH_3 data.

5.2.1.1.1. 2-butanone ≥ 500 ppb (976 µm)
5.2.1.1.2. Ethanol ≥ 60 ppb (974 µm)
5.2.1.1.3. Ethanolamine ≥ 110 ppb (974 µm)
5.2.1.1.4. Methanol ≥ 80 ppb (974 µm)
5.2.1.1.5. Phenol ≥ 400 ppb (974 µm)

5.2.1.2. **Ethanol/methanol** is optimally measured at 974 µm, with a detection limit of 80 ppb. Interferents at this wavelength include:

5.2.1.2.1. Ammonia ≥ 200 ppb
5.2.1.2.2. Ethanolamine ≥ 110 ppb
5.2.1.2.3. Phenol ≥ 400 ppb

5.2.1.3. **Acetic acid** is optimally measured at 970 µm. Its stated detection limit is 40 ppb. Interferents at this wavelength include:

5.2.1.3.1. Methanol ≥ 500 ppb

5.2.1.4. **Acetaldehyde** is optimally measured at 986 µm, with a detection limit of 80 ppb. Interferents at this wavelength include:

5.2.1.4.1. Dimethyl sulfide ≥ 400 ppb
5.2.1.4.2. Dimethylamine ≥ 40 ppb
5.2.1.4.3. Formaldehyde ≥ 40 ppb
5.2.1.4.4. Trimethylamine ≥ 20 ppb

5.2.1.5. **Dimethyl sulfide** is optimally measured at 936 µm. Its detection limit is 600 ppb at this wavelength. Interferents at this wavelength include:

5.2.1.5.1. Methanol ≥ 80 ppb

5.2.1.6. No obvious relevant interferents for **CO_2** (quantified at 983 µm) are evident. The stated diction limit for CO_2 is 3.4 ppm

5.2.1.7. **Methane** can be quantitated at 968 µm or 969 µm; however, the former of these is much more sensitive to interference from water vapor. When methane is quantified at 969 µm, it has a detection limit of 400 ppb, with the following possible interferents:
  5.2.1.7.1. $H_2S \geq 14$ ppm
  5.2.1.7.2. Acetone ≥ 70 ppb
5.2.1.8. **Phenol** is optimally measured at 971 µm. Its detection limit is 8 ppb. Interferents at this wavelength include:
  5.2.1.8.1. m-cresol ≥ 50 ppb
  5.2.1.8.2. Dimethylamine ≥ 200 ppb
  5.2.1.8.3. Methyl methacrylate ≥ 20 ppb
  5.2.1.8.4. $SO_2 \geq 400$ ppb
  5.2.1.8.5. Trimethylamine ≥ 200 ppb
5.2.1.9. Total VOC is best measured at 987 µm, with a detection limit of 20 ppb. Since it is intended to measure all volatile organic hydrocarbons, no individual hydrocarbons can be considered interferents. Some oxygenated VOC will absorb at this wavelength; others will not. Ammonia and H_2S have negligible absorptions at 987 µm.

5.3. Certain gases require humidification in order to generate accurate calibration readings. This is needed to speed the relaxation (energy release) of the analyte molecules after they are excited by the IR light, by mediating the dispensation of energy to bulk fluid (gas). Without humidification, there will be significant downward drifting of the reading of the calibration gas, leading to inaccurate readings. An inline Nafion-based humidifier (Section 8.5) must be used (Section 9.4.3.3.1.3) with these gases.
  5.3.1. Humidification is required for any calibration or span check with CO_2.
  5.3.2. Humidification is required for methane if air is used as the diluent.

6. Cautions

6.1. When installing the RS-232 cable to the back of the gas analyzer monitor, ensure that both the monitor and the PC are switched off at the mains. Failure to do so may result in equipment being damaged.
6.2. Blown fuses in the monitor can indicate a serious fault in the analyzer; therefore, it is advisable to contact the Innova representative before changing the fuse.
6.3. Do not pressurize the instrument above 250 Pa (1 in. of water).
6.4. Avoid water condensation in the analyzer.
6.5. Do not allow any liquid to enter the analyzer's photoacoustic chamber.

7. Personnel Qualifications

7.1. Personnel should be trained in the use of the analyzer before initiating the procedure. Training requires about three hours.
7.2. Each analyst must read and understand the entire analyzer manual and this SOP before operating the Model 1412.

7.3. Personnel should also be trained to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

8. Equipment and Supplies

8.1. Multi-gas Photoacoustic Monitor (Model 1412, Innova AirTech Instruments, Ballerup, Denmark), with appropriate optical filters (as defined in the particular project's QAPP):
  8.1.1. UA0976 or 974 for ammonia
  8.1.2. UA0974 for ethanol/methanol
  8.1.3. UA0970 (or UA0971) for acetic acid
  8.1.4. UA0986 for acetaldehyde
  8.1.5. UA0936 for dimethyl sulfide
  8.1.6. UA0983 for carbon dioxide
  8.1.7. UA0968 for methane
  8.1.8. UA0971 for phenol
  8.1.9. UA0985 for nitrous oxide
  8.1.10. UA0984 for carbon monoxide
  8.1.11. UA0987 for total VOC (general hydrocarbon filter)
8.2. Rack and rack mounts
8.3. Data acquisition and control
  8.3.1. Barn applications: PAAQL data acquisition and control hardware (SOP B1), and AirDAC (SOP B2) and proprietary custom software to allow communication between INNOVA and AirDAC (Nexus Solutions, London, Ontario, Canada)
  8.3.2. Open-source applications: LAN Server computer in open-source trailer (SOP U5 or U7 for lagoon or corral sites, respectively), and LabVIEW software (National Instruments Corporation, Austin, TX)
8.4. Mounting post and post driver (for open-source applications – SOP U5 or U7)
8.5. Nafion humidifier (California Analytical Instruments Part # CAI-110-03-34)
8.6. Keep the following spare parts at each remote site and in central stocks at PAAQL and PAML:
  8.6.1. Threaded nuts (2x YM 0652)
  8.6.2. Swagelok stainless steel ¼" nuts and ferrules
  8.6.3. Gas Monitoring Software 7304 for Photoacoustic Multi-gas monitors 1412
  8.6.4. External filters
    8.6.4.1. (Recommended) stainless steel filter frit (10 µm, 25 mm)
    8.6.4.2. External fine air filters (CAII-F01) 100/pkg. (Millipore: Mitex Membrane Filter, PTFE, hydrophobic, 10 µm, 25 mm, white, plain; catalog number: LCW02500)
  8.6.5. Cables
    8.6.5.1. Modem cable (9 pin)
    8.6.5.2. RS-232 null modem cable (25 pin female/9 pin male)
    8.6.5.3. Power cable (cord) for the analyzer
  8.6.6. Tubing
    8.6.6.1. Teflon tubing (¼″ OD)
    8.6.6.2. UD 5037 Nafion tubing

8.6.7. Spare slow-blow (T) fuses with a rating of 2.5 A, 250 VAC, 5x20 mm, UL-approved (Innova Order Number: VF0087A)

8.6.8. Instruction manual, menu tree, and field guide for the Innova 1412 Photoacoustic Monitor

8.7. Keep the following spare parts in central stocks at PAAQL and PAML:

8.7.1. Optical filter locking springs (8x DL 3322)

9. Procedure

9.1. Installation

9.1.1. Check analyzer carefully to assure it is free of visible defects or damage.

9.1.2. Mounting options

9.1.2.1. For barn applications, securely mount the analyzer horizontally on the instrument rack in the OFIS.

9.1.2.2. For open-source applications, mount the instrument securely to a mounting post at a specified location downwind of the lagoon or other open source which is being monitored. See the Site Monitoring Plan to locate the exact location.

9.1.3. Install the gas monitoring software 7304 for the control of photoacoustic gas monitors 1412 and 1314, if it is not already installed.

9.1.4. Connect the gas analyzer monitor to the data acquisition PC.

9.1.4.1. Connect the 25-pin female cable (RS-232) to the RS-232 port on the Innova 1412. Connect the 9-pin modem cable to the RS-232 cable from the Innova 1412. Connect the null modem cable into the COM 1 port of the DAC computer.

9.1.4.2. Leave sufficient extra length to the tubing and cables such that the instrument rack can be moved a few feet to get access to the back of the analyzer.

9.1.5. Connect tubing to the Innova 1412

9.1.5.1. Connect the analyzer inlet to the gas source (e.g. the GSS analyzer manifold for barn applications) with the minimum length of ¼″ OD Teflon tube.

9.1.5.2. Connect the analyzer exhaust with a 3″ length of 4-mm tube that is connected to ¼″ ID PVC or other tubing. Connect the exhaust tube from the analyzer to the air exhaust of the OFIS.

9.1.6. Power up the analyzer, turn on its internal pump, switch to measurement mode and allow the analyzer to warm up for a minimum of 60 – 90 min before analysis. This is necessary to warm up the analysis cell, as conditions within the cell tend to stabilize more quickly (allowing stable readings) once the temperature inside the analysis cell is 15°C above the ambient room temperature.

9.2. Setting up Communication Parameters between the Model 1412 and PC

9.2.1. Using the push buttons on the front of the monitor and following the operation manual, set the communication parameters for the serial interface as follows:

Baud rate	9600
Stop bits	1
Data bits	7
Parity	Even
Hardwire mode	Leased line
Handshake type	Hardwire

9.2.2. Using the push buttons on the front of the monitor and follow the operation manual, set the text line terminator, print data log and print error log as follows to prevent communication errors:

Text line Terminator	CR-LF
Print Data Log	NO
Print Error Log	NO

9.2.3. Select the PC communication port
  9.2.3.1. Start any one of the gas monitoring software 7304 options: Online, Offline, or Calibration.
  9.2.3.2. Pull down the Task menu. Click on Communication. The analyzer can use COM port 1 through COM port 4.
  9.2.3.3. Click on the radio push-button next to the correct port name. Refer to the PC manual if not sure which port the cable is connected to. COM Port 1 is used most of the time.

9.3. Set up and start a Monitoring Task using the gas monitor software 7304, and set the instrument parameters according to Table 1.
  9.3.1. Measurement parameter set-up
    9.3.1.1. Pull down the Task menu. Click on New to create a new database, or click on Open to use an existing database.
    9.3.1.2. For a new database, click in the “Enter Name For” field of the database window and type in a desired name. For existing databases, click on the desired database name.
    9.3.1.3. Click OK.
    9.3.1.4. Pull down the Sequence menu. Click on Settings and the Setup window appears.
    9.3.1.5. Click on the Sampling tab.
    9.3.1.6. Click on the radio-button for the desired sampling mode. If Sampling Interval is selected, set the time to the correct value.
    9.3.1.7. Click on the radio-button for the desired flushing mode.
      9.3.1.7.1. If Auto is selected, set the correct tube length.
      9.3.1.7.2. If Fixed Time is selected, set the desired chamber flushing time and the tube flushing time (recommended value: chamber flushing time, 8 s; tube flushing time, 3 s).
    9.3.1.8. If water vapor and cross interference are required, check the appropriate box.
    9.3.1.9. If average values are to be displayed on the gas monitor screen, click in the Average check box, and set the interval to the correct value.
    9.3.1.10. Click on the Gas tab.
    9.3.1.11. Click in the check box to the left of the desired filter.
    9.3.1.12. Click in the field to the right of the selected filter and select the correct gas.
    9.3.1.13. Repeat the above two steps until all the filters which are required for the measurements are selected and the correct names are displayed.
    9.3.1.14. Click in the Sample Integration Time field and select the desired option.
    9.3.1.15. Click on the Alarm tab and check to make sure that alarms are set to default.
    9.3.1.16. Click on OK to complete the system setup.

Table 1. Appropriate settings for various instrument parameters for the INNOVA Model 1412.

Instrument Parameters	
Units	
Concentration Unit:	ppm
Length Unit:	ft
Temperature Unit:	°C
Pressure Unit:	mm Hg
Humidity Unit:	Tdew
Normalization Temperature	20.0°C
Sampling Mode	
Sample Continuously	Yes
Sampling Interval (If selected)*	1 min (barns), 200 s (open-source)
Flushing	
Auto	No
Tube Length	10 ft (barns), 0.5 ft (open-source)
Fixed Time	Yes
Chamber	8 s (barns), 13 s (open-source)
Tube	3 s
Compensation	
Water Interference	Yes
Cross Interference	Yes
Gas Monitor Display	
Average	No
Time (HH:MM)	00:10
Current Air Pressure	
Pressure	Measured by Instrument
Memory Use	
Store History	Yes
Gas Setup	
Gas	S.I.T.
Ammonia	2 s
Ethanol / Methanol	1 s
Acetic Acid	2 s
Acetaldehyde	2 s
Dimethyl Sulfide	1 s
Carbon Dioxide	1 s
Methane	1 s
Phenol	2-5 s
Total VOC	1s
Alarms	
All gases are set to “Default”	No Alarms Set

* Note: If “Sampling Interval” Mode is selected, set “Sample Continuously’ to “No”.

- 9.3.2. Start a monitoring task
  - 9.3.2.1. Pull down the Sequence menu and click on Start.
  - 9.3.2.2. In the "Enter Sequence Description" field, type in the name of the monitoring sequence. If no name is entered, the default name "Sequence 1" is used.
  - 9.3.2.3. To start the measurement immediately, click on the Now radio-button in the Start Time group.
  - 9.3.2.4. To delay the start, click on the Start At: radio-button, and define the start time in the Start Time group.
  - 9.3.2.5. To manually stop the measurement later, click on the None radio-button in the Stop Time group.
  - 9.3.2.6. To automatically stop the measurement by defining a stop time, click on the Stop at: radio-button in the Stop Time group and enter the time to stop.
  - 9.3.2.7. Press OK to start the monitoring task.
- 9.3.3. Stop a monitoring task
  - 9.3.3.1. If no pre-set monitoring period is defined, pull down the Sequence menu and click on Stop.
- 9.3.4. Displaying measurement results while monitoring
  - 9.3.4.1. Using the Online program, measurement data are displayed on screen as soon as they are available from the monitor. The data can be displayed in both a graphic window and a numeric window, simultaneously. Refer to the manual for detailed procedures for defining the graphic window and numeric window.
- 9.3.5. Displaying measurement results from a stored monitoring task
  - 9.3.5.1. Click on the Presentation program, and pull down the Task menu. Click on Open to view an existing database.
  - 9.3.5.2. In the database window, click on the desired database name.
  - 9.3.5.3. Click on OK. A window opens showing all the measurement values for the complete monitoring task. Refer to the manual for detailed procedures for defining the graphic window and numeric window.
- 9.3.6. Export measurement data as comma-delimited ASCII files
  - 9.3.6.1. Click on the Presentation program. Open the database to be exported.
  - 9.3.6.2. Pull down the Task menu and click on Export.
  - 9.3.6.3. Use the standard Windows browser, which appears on screen, to define the destination for the exported data.
  - 9.3.6.4. Type in the desired file name. All files have a .TXT extension.
  - 9.3.6.5. Click on Save.
  - 9.3.6.6. Use Microsoft Excel to open and process the saved file.

9.4. Gas Analyzer Calibration

- 9.4.1. The exact composition for each reference calibration gas is dependent upon the gases being monitored in a given project, and the concentrations expected at the site. Calibration gas compositions for a particular project will be specified in the QAPP for that project.
- 9.4.2. Calibrate Innova 1412 following SOPs G8 (multipoint calibrations) and G9 (zero/precision checks).

9.4.3. Calibrate the optical filters.
  9.4.3.1. Calibrate the optical filters with the following steps: zero point calibration, humidity interference calibration, span calibration of CO_2 (if that filter is in the instrument), and span calibration of any other gases after the instrument has been dried with zero air or a clean dry source of nitrogen.
  9.4.3.2. Before calibrating the optical filters:
    9.4.3.2.1. Warm up the monitor for a period of 60-90 min in the measurement mode before a calibration task is started to reduce the time required for calibration.
    9.4.3.2.2. Detach the air inlet tube from the existing sampling manifold. Close the gas sampling manifold with a plug.
    9.4.3.2.3. Obtain a print-out of calibration data in the monitor by using the "Print Current Calibration" function. This will compare the "old" calibration factors with the "new" ones.
    9.4.3.2.4. Check the calibration of each installed optical filter following the procedures described in the manual.
    9.4.3.2.5. Verify that the installed optical filters have been entered correctly in the active set-up of the monitor.
  9.4.3.3. Basic setup for calibration
    9.4.3.3.1. Set up the calibration equipment according to Fig. 2. Connect three different lengths of Teflon tubing to a "Y"-piece.
      9.4.3.3.1.1. Connect the tube attached to point 1 to the analyzer's air inlet.
      9.4.3.3.1.2. Connect the tube attached to point 2 to a gas flow meter.
      9.4.3.3.1.3. Connect the tube attached to point 3 to the cylinder of gas which is to be used during calibration. This connection should be direct if humidity is not to be included, or through a water bath (as shown in Fig. 3) if humidity interference calibration is being conducted. If the calibration is being done for CO_2 or methane, use a Teflon tube which has been cut into two segments, and with a Nafion humidifier inserted into it using the tubing couplers provided.
  9.4.3.4. Preparation for humidity interference, zero, or span calibration
    9.4.3.4.1. Verify the settings of the Model 1412's communication parameters are set to the values described in Section 9.2.1.
    9.4.3.4.2. Make sure the text line terminator, print data log and print error log are set to the values described in Section 9.2.2.
    9.4.3.4.3. Open the "Calibration" program and create a new calibration task.
  9.4.3.5. Zero gas calibration
    9.4.3.5.1. Close the regulator valve on the zero gas cylinder.
    9.4.3.5.2. Open the main valve on the zero gas cylinder.
    9.4.3.5.3. Open the "Calibration" program and choose "New" to create a new task.
    9.4.3.5.4. Type in the desired task name and click "OK".
    9.4.3.5.5. Pull down the "Sequence" menu and click "Settings".
    9.4.3.5.6. Click on the "Calibration" tab, if it is not already at the front. Select the "Zero point" button.
    9.4.3.5.7. Click on the "Gas" tab, click in the "Sample Integration Time" field, and make sure that SIT is set to the same as that used for monitoring (Section 9.3).

9.4.3.5.8. Click on the Sampling tab and set the flushing time. The flushing type and values should be set to the same as those used when monitoring. Click OK.

9.4.3.5.9. Insert the 1/4"-OD tubing from the zero gas cylinder into the flow meter mounted on the side of the instrument rack. Open the regulator valve to allow gas flow. Adjust regulator valve until vent airflow is about 2.5 L/min. Use the flow meter to measure the flow rate.

9.4.3.5.10. Pull down the "Sequence" menu, and click "Start". Record time and analyzer display in field notes.

9.4.3.5.11. After display has stabilized (typically 10 to 15 min), pull down the "Sequence" menu and click "Stop".

9.4.3.5.12. Close regulator and remove tubing from the zero gas cylinder.

9.4.3.6. Humidity interference calibration

9.4.3.6.1. Run the Teflon tube from the zero gas cylinder into a temperature-controlled water bath. The outlet tube from the water bath should flow into a separate, empty flask, which acts as a safety trap. The outlet line from this flask then enters the "Y" connector, and is split between the flow meter and monitor. See Fig. 3 for a diagram of this setup.

9.4.3.6.1.1. The temperature of the water bath and the safety flask should be at least 2°C below the ambient temperature of the room.

9.4.3.6.2. Pull down the "Sequence" menu and click "Settings".

9.4.3.6.3. Click on the "Calibration" tab, if it is not already at the front.

9.4.3.6.4. Click to select "Humidity Calibration" radio-button.

9.4.3.6.5. Only select Humidity Interference Calibration (All Filters). Do not select Perform Water Vapor Span Calibration.

9.4.3.6.6. Click on the "Gas" tab, click in the "Sample Integration Time" field, and select the desired time (Fast 1 s).

9.4.3.6.7. Click "OK".

9.4.3.6.8. Open the regulator valve to allow gas flow through the water bath and safety flask (Do not allow any liquid into the instrument).

9.4.3.6.9. Adjust regulator valve until vent airflow is about 2.5 L/min using a flow meter to measure the flow rate.

9.4.3.6.10. Pull down the "Sequence" menu and click "Start".

9.4.3.6.11. Allow the readout to stabilize (typically 10 to 15 min). Record time and analyzer display in field notes.

9.4.3.6.12. Pull down the "Sequence" menu; click on "Stop".

9.4.3.6.13. Close regulator and main valve and remove tubing from the zero gas cylinder.

9.4.3.7. Span gas calibration of CO_2 (if applicable)

9.4.3.7.1. Pull down the "Sequence" menu, and click "Settings".

9.4.3.7.2. Click on the "Calibration" tab, if it is not already at the front. Select the "Gas Span Calibration" button.

9.4.3.7.3. Click in the "Active Filter" field, select "Carbon Dioxide", or other gas as appropriate. Check "Perform Cross Interference Calibration". Input the concentration of the analyte in the gas which is being used.

9.4.3.7.4. Click on the "Gas" tab, click in the "Sample Integration Time" field, and select the desired time (the same as that used when monitoring). Click "OK".

9.4.3.7.5. Insert the ¼″ OD tubing from the gas cylinder into the flow meter mounted on the side of the instrument rack. Open the regulator valve to allow gas flow. Adjust regulator valve until vent airflow is about 2.5 L/min, using the flow meter to measure the flow rate.

9.4.3.7.6. Pull down the "Sequence" menu, and click "Start"**.** Record time and analyzer display in field notes.

9.4.3.7.7. After the display has stabilized (typically 10 to 15 min), pull down the "Sequence" menu and click "Stop".

9.4.3.7.8. Close regulator and remove tubing from the gas cylinder.

9.4.3.8. Span gas calibration for other gases

9.4.3.8.1. Set the analyzer to measure water vapor only

9.4.3.8.2. Flow dry nitrogen or CEM zero air through the analyzer until the water vapor measurement is reading -58 to -60 for Tdew°C.

9.4.3.8.3. Pull down the "Sequence" menu, and click "Settings".

9.4.3.8.4. Click on the "Calibration" tab, if it is not already at the front. Select the "Gas Span Calibration" button.

9.4.3.8.5. Click in the "Active Filter" field, select "Ammonia", or other gas as appropriate. Check "Perform Cross Interference Calibration". Input the concentration of the analyte in the gas which is being used.

9.4.3.8.6. Click on the "Gas" tab, click in the "Sample Integration Time" field, and select the desired time (have to be the same as those used when monitoring). Click "OK".

9.4.3.8.7. Insert the ¼″ OD tubing from the gas cylinder into the flow meter mounted on the side of the instrument rack. Open the regulator valve to allow gas flow. Adjust regulator valve until vent airflow is about 2.5 L/min using a flow meter to measure the flow rate.

9.4.3.8.8. Pull down the "Sequence" menu, and click "Start"**.** Record time and analyzer display in field notes.

9.4.3.8.9. After display has stabilized (typically 10 to 15 min), pull down the "Sequence" menu and click "Stop".

9.4.3.8.10. Close regulator and remove tubing from the gas cylinder (Repeat Sections 9.4.3.7.1 through 9.4.3.7.8 for the other gases).

9.4.3.9. Calculating and downloading Calibration Factors

9.4.3.9.1. While the raw measurement data are still displayed on the screen, open the "Cursor Values" dialogue box.

9.4.3.9.2. Use two cursors and the statistical data (displayed in the Cursor Values dialogue box) to locate a suitable range of data. All values in the selected interval should be very stable, and the temperature should be above 40°C.

9.4.3.9.3. When the desired region is between the cursors, pull down the "Sequence" menu, and click on "Mark Interval".

9.4.3.9.4. Select and mark all the intervals for every calibration.

9.4.3.9.5. Pull down the "Value" menu, and click on "Calculate". When the calculation(s) is complete, a "Calculation Finished" dialogue box is displayed.

9.4.3.9.6. Pull down the "Values" menu, and click on "Download". The "Download" dialogue box is displayed.

9.4.3.9.7. Select the “Zero Point” tab, and set ticks in the appropriate check boxes, depending on which gas (and/or water vapor) calibrations were just run.
9.4.3.9.8. Repeat the above two steps in the “Humidity” and “Gas” tabs.
9.4.3.9.9. Click “OK” to download the calibration factors to the monitor.
9.4.3.10. After calibration is complete, reattach the Model 1412 to the GSS, and close the main valves on all gas cylinders.
9.4.4. Data acquisition, calculations & data reduction requirements
9.4.4.1. Report data with three significant digits.
9.4.4.2. Follow SOP B4 to correct gas concentrations for calibration and air density.
9.4.5. Computer hardware & software
9.4.5.1. Barn applications:
9.4.5.1.1. AirDAC (SOP B2)
9.4.5.1.2. AirDAC Data Pre-Processing Software (SOP B3)
9.4.5.1.3. CAPECAB (SOP B6)
9.4.5.2. Open-source applications (SOPs D1, U4, and U5/U7):
9.4.5.2.1. LabVIEW
9.4.5.2.2. LAN Server computer

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement his electronic record with a bound record book designated for the method, which should contain copies of the electronic record.
10.2. Manage all data according to SOP B5 or SOP D1 (barn or open-source applications), as appropriate.
10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
10.4. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

11.1. Conduct gas analyzer checks according to the QAPP for the particular project. This will include zero gas and one or more reference span concentrations.
11.2. Conduct gas analyzer calibration when the precision check is >±10% of baseline, unless the QAPP for the particular project specifies a different value.
11.3. Conduct gas analyzer calibration when the zero check is >±5% of full scale different than the baseline, unless the QAPP for the particular project specifies a different value.
11.4. The calibration gas should be certified using EPA protocol (1% certification), if this grade is available, unless the QAPP for the particular project specifies a different value.
11.5. Check the response time every six months, unless the QAPP for the particular project specifies a different value.
11.6. Measure the gas analyzer’s inlet flow rate every six months.
11.7. Perform leak check in sample line and flow components and fittings every six months by measuring the inlet and exhaust flow rates with a flow meter.

11.8. Change the fine air-filter paper in the internal and external air-filtration units monthly. For analyzers that have the stainless steel 10 µm frit for the internal filter, clean the frit monthly. After cleaning or replacing filters, perform a leak check at the inlet sample port and the exhaust port by measuring the inlet and exhaust flow rates with a flow meter, to ensure that no leaks were introduced.
11.9. Clean the filter in the ventilation unit monthly.
11.10. Record any maintenance in the Instrument Performance Record File's worksheet labeled maintenance.

12. References

12.1. Model 1412 Photoacoustic Field Gas-Monitor Product Data. Innova AirTech Instruments. Online at www.innova.dk/uploads/media/Product_Data_1412_PW.pdf. Accessed 2/15/2006.
12.2. Model 1412 Photoacoustic Field Gas-Monitor Details. Innova AirTech Instruments. Online at http://www.innova.dk/1412_details.gas_monitoring4.0.html. Accessed 2/15/2006.
12.3. Innova AirTech Instruments. 2000. Detection Limits Wall Chart. Online at www.innova.dk/uploads/media/Wall_Chart_PW_01.pdf. Accessed 2/15/2006.
12.4. Schmidt, C.E., T. Card, and P. Gaffney. 2005. Assessment of reactive organic gases and amines from a Northern California dairy using the USEPA surface emission isolation flux chamber. Presented at the California Air Resources Board Livestock Emissions Symposium. January 26, 2005. Online at http://www.arb.ca.gov/ag/caf/lersymp.htm. Accessed 2/17/2006.
12.5. Summers, M.D. 2005. Emissions from California poultry production. Presented at the California Air Resources Board Livestock Emissions Symposium. January 26, 2005. Online at http://www.arb.ca.gov/ag/caf/lersymp.htm. Accessed 2/17/2006.
12.6. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
12.7. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
12.8. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
12.9. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
12.10. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
12.11. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
12.12. SOP G8. 2006. General Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.
12.13. SOP G9. 2006. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.

12.14. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.

12.15. SOP U7. 2006. The Instrument Trailer and Installation of Open-source Measurement Equipment (Corrals). Standard Operating Procedure U7. Purdue Applied Meteorology Lab.

13. Contact Information

13.1. Distributor:
California Analytical Instruments
Tel: (714) 974-5560
Email: info@gasanalyzers.com

13.2. Manufacturer:
Innova AirTech Instruments (www.innova.dk)
Energivej 30, DK-2750 Ballerup, Denmark
Tel.: (+45) 44 20 0100, Fax: (+45) 44 20 0101, innova@innova.dk

13.3. PAAQL: odor@purdue.edu, heber@purdue.edu, http://www.AgAirQuality.com.

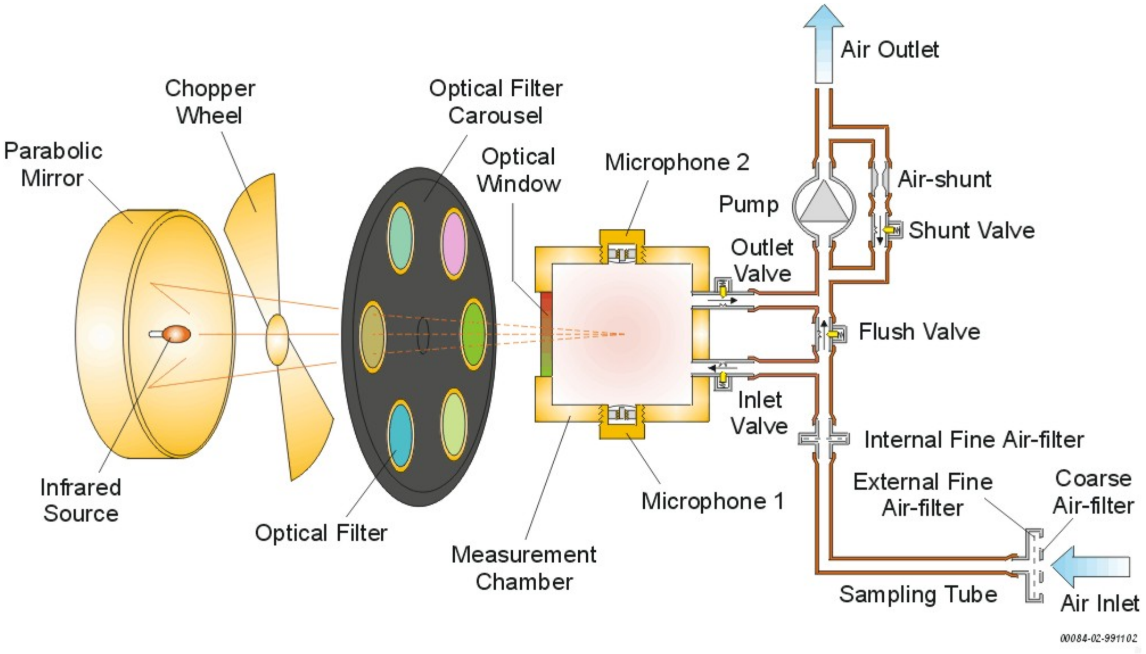


Figure 1. Schematic diagram of the major internal components of the INNOVA Model 1412 Photoacoustic Multi-gas Monitor.

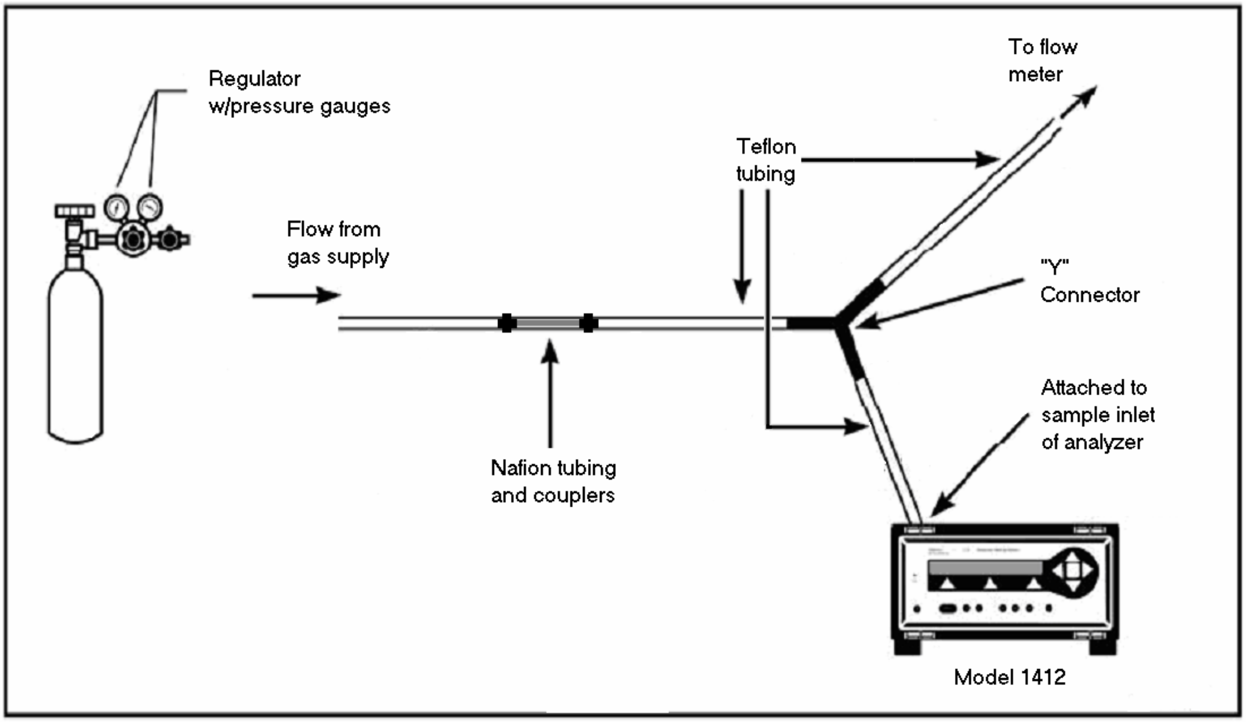


Figure 2. General equipment required for calibration of the Model 1412. Nafion tubing is coupled into the Teflon line from the cylinder during calibration of CO_2 or methane.

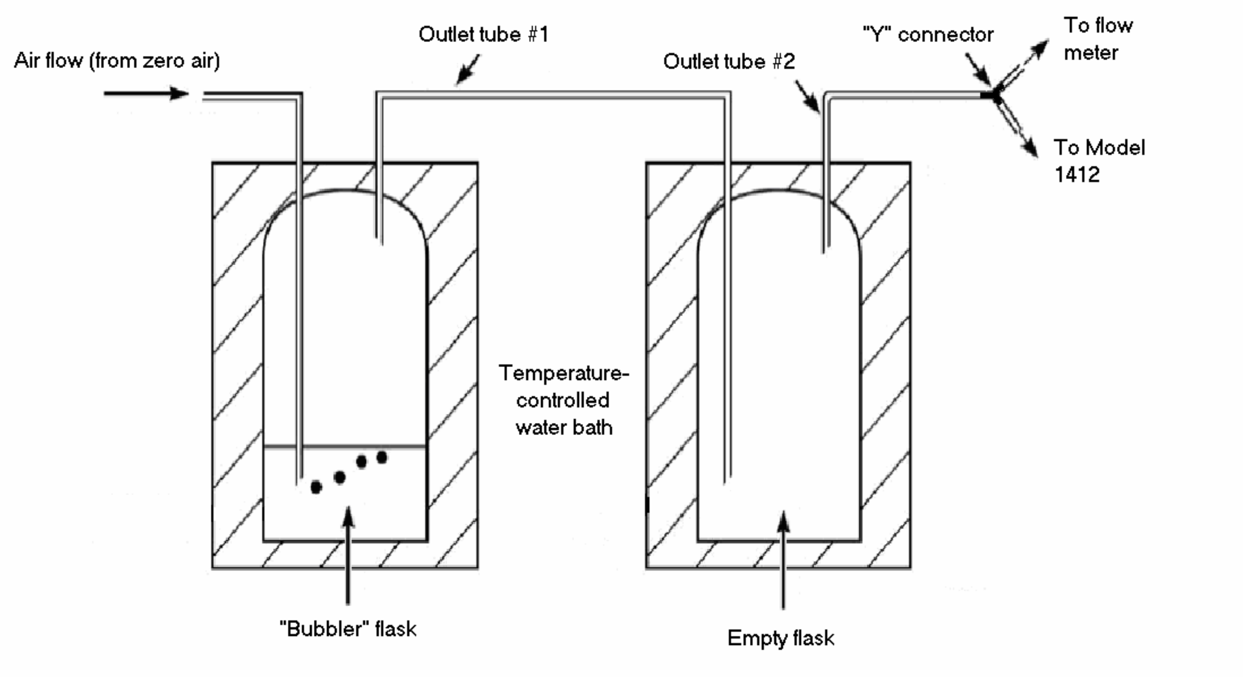


Figure 3. Schematic diagram of the equipment necessary to produce a supply of clean, wet air for humidity interference calibration.

OPERATION OF THE ENVIRONICS® COMPUTERIZED GAS DILUTION SYSTEM

Standard Operating Procedure (SOP) G11

OPERATION OF THE ENVIRONICS® COMPUTERIZED GAS DILUTION SYSTEM

Standard Operating Procedure (SOP) G11

Prepared by
Sam M. Hanni and Juan Carlos Ramirez

Reviewed by
Congna Li, Bill W. Bogan, and Albert J. Heber

Effective Date: December 28, 2007

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Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907

Table of Contents

1. Scope and Applicability

- 1.1. This method dilutes a compressed gas standard with a balance gas, allowing the resulting mixture to be used for calibrating the continuous gas analyzers which are used in monitoring barn emissions.
- 1.2. This method applies only to the Environics Series 4040 Computerized Gas Dilution System and its accompanying computer software.
  - 1.2.1. The maximum flow rate of the gas mixture is 5 L/min.
  - 1.2.2. The gas standard is diluted to concentrations that range from 10 to 99% of its original concentration.
  - 1.2.3. The minimum concentration can be reduced to 1% of the original with the addition of one mass flow controller (MFC).
  - 1.2.4. The minimum concentration can be reduced to 0.1% of the original with the addition of two MFCs.
  - 1.2.5. The accuracy of each MFC is ±1% of its set flow rate.
  - 1.2.6. The accuracy of the overall dilution ratio is an additive function of the errors of all MFCs involved in the dilution. For example, if two MFCs are used, the maximal error (if the individual errors of the two MFCs are in the same direction) is ±2%. If the errors of the individual MFCs are in the opposite directions, the overall dilution ratio error will be considerably lower than this.
  - 1.2.7. The dilutor can be programmed to automatically deliver diluted standards on a schedule.
  - 1.2.8. The diluter described and used in this SOP has all its gas-accessible internal surfaces passivated with a non-reactive (RESTEK) coating, which makes it suitable for use with reactive gases.

2. Summary of Method

The Environics® Series 4040 is a computerized gas dilution system that automatically generates precise gas calibration standards for single or multi-point calibration of analyzers. The system consists of two components: the Series 4040 Diluter and the user's PC. The user interface is a Microsoft® Windows application that communicates with the Environics system via a RS232 serial interface connected to any available COM port of the personal computer. The Series 4040 instrument has a single chassis supporting up to four mass flow controllers (MFC) having ranges of 5, 5, 0.5, and 0.05 L/min, respectively. This set of flow controllers allows accurate dilution of gas standards over a very wide range of dilution ratios, by selection of the appropriate flow controllers. The MFCs are factory-calibrated using a primary flow standard traceable to the National Institute of Standards and Technology (NIST). Each flow controller utilizes an 11-point calibration table with linear interpolation, to increase accuracy and reduce flow controller nonlinearity. During operation, the protocol gas concentration, inlet port, desired output concentration, and desired output flow rate are entered by means of the keypad of the personal computer used to operate the 4040 diluter, and the diluter then sets the required standard and diluents flow rates to produce the desired mixture. The 4040 diluter indicates on the computer's display the actual concentration being produced, which in some cases differs very slightly from the nominal concentration requested. In all cases the actual concentration produced is recorded as the concentration provided to the analyzers undergoing testing. To calibrate this instrument, it is necessary to choose a pre-calibrated gas analyzer to test the concentrations produced by the diluter.

3. Definitions

3.1.	FTIR	Fourier transform infrared spectroscopy
3.2.	GC/MS	Gas chromatography/mass spectrometry
3.3.	GSS	Gas sampling system
3.4.	K-factor	Gas correction factor
3.5.	MFC	Mass flow controller
3.6.	NIST	National Institute of Standards and Technology
3.7.	OGC	Output gas concentration
3.8.	PC	Personal computer
3.9.	QAPP	Quality Assurance Project Plan

4. Health and Safety

4.1. Avoid mixing gases that may produce an explosion or other hazardous reaction. Follow all other health and safety precautions related to gas cylinders (SOP G2).
4.2. Disconnect power before servicing the unit.
4.3. Make sure that replacement fuses are the correct size and rating (115 V).
4.4. Use only grounded outlet electrical outlets.

5. Cautions

5.1. Verify that scheduled calibration date is prior to the expiration date of the standard gas.
5.2. Verify that the instrument ambient temperature conditions are in the manufacturer’s recommended range of 0°C to 50°C.
5.3. If the Environics software is not connected to the LabView (AirDAC) data acquisition software (SOP B2), adjust its time to agree with the time of the LabView computer.
5.4. Note all other cautions related to gas cylinders (SOP G2).
5.5. In any case where the Model 4040 is not under the control of the AirDAC program, make sure that the computer clock (used by the Environics system) is synchronized with the time in AirDAC. If the times are not synchronized, any task programmed on the Environics dilutor could be out of phase with other tasks controlled by the AirDAC program.
5.6. Use of any USB adapter other than the one described in Section 8.3 may cause communications problems between the PC and diluter. If such problems are suspected with a different adapter, replace with this one as a first measure.

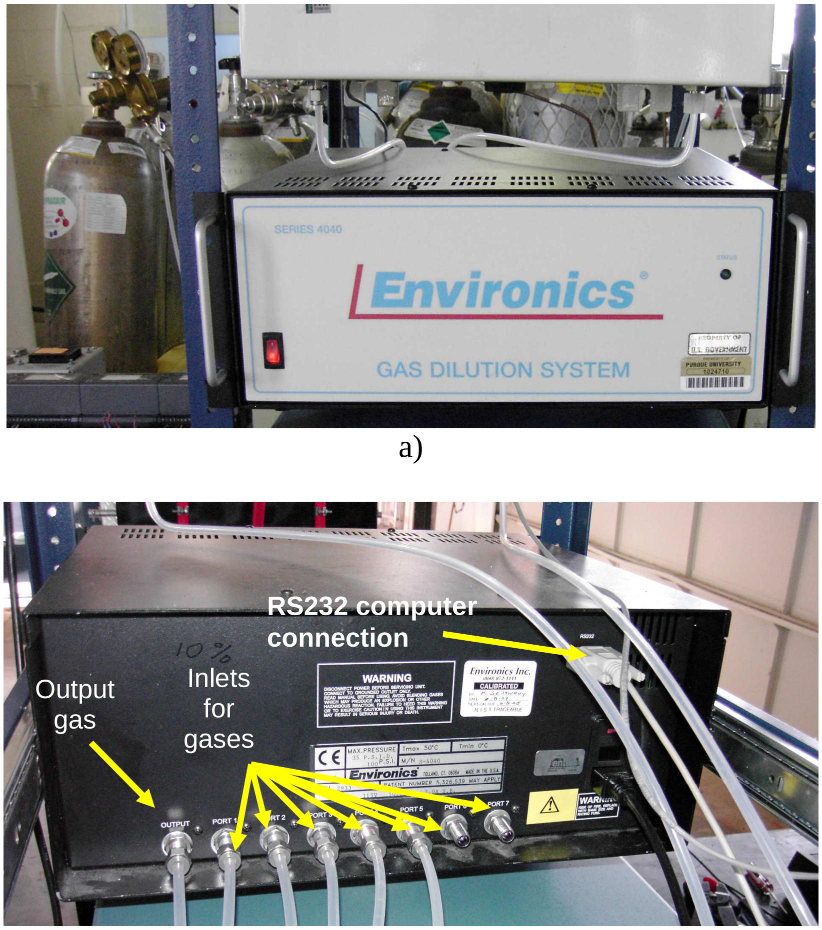
6. Interferences

6.1. Do not operate the diluter at a flow lower than 10% of the working range of the flow controller selected (this instrument has four flow controllers for different working ranges), which is a flow region where flow control errors might be enhanced. The 4040 diluter provide warnings if a flow controller is being operated at this low range. Switching to another flow controller, if available, will minimize these uncertainties.

7. Personnel Qualifications

7.1. Each operator must read and understand this SOP, SOP G2, and the instrument manual before working with the instrument.

8. Equipment and Supplies



a)

b)

Figure 1. Front (a) and back (b) of the Environics Model 4040 Gas Diluter.

8.1. Environics Series 4040 Gas Dilution System

8.2. PC. Minimum recommended: 66-MHz 486 PC with 8 MB of RAM.

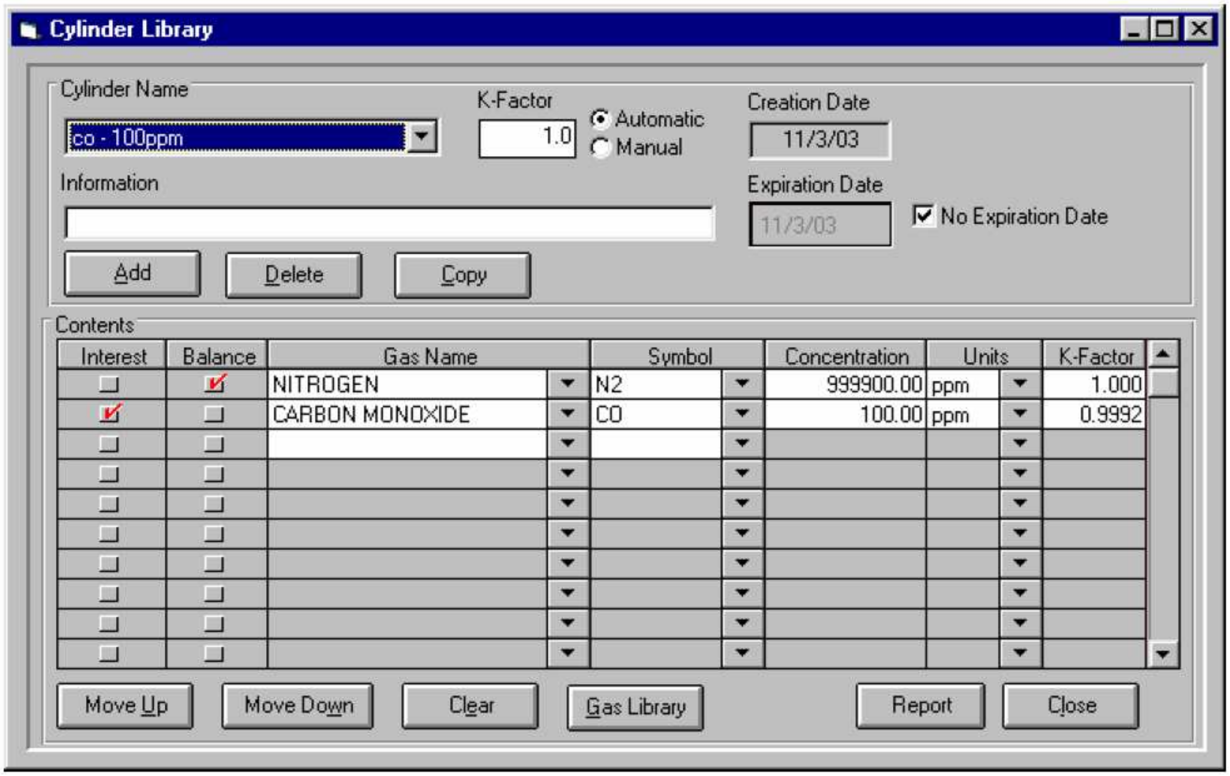
8.2.1. Operating system: Windows 3.1 or newer (including Windows 95/98/Me and Windows NT/2000/XP).

8.3. Communications cable/adapter: USB to Serial adapter DB9m, G-uc232a, Cyberguys.com.
8.4. Mass flow meter
8.5. Teflon tubing
8.6. 9/16″ box wrench

9. Procedure

9.1. Instrument setup
  9.1.1. Hardware setup
    9.1.1.1. Connect the diluter to the computer via an RS232 cable. Connect the cable to one of the COM ports (1-4) on the computer. COM 1 is the system's default port.
  9.1.2. Software installation
    9.1.2.1. Close all running window applications.
    9.1.2.2. Install the Environics Series 4000 Software from the CD included with the instrument.
    9.1.2.3. To start the software, double-click the Environics Series 4000 icon or select Environics Series 4000 from the START menu.
  9.1.3. Data disk installation
    9.1.3.1. Instrument Data Disk Installation installs instrument specific information into the Environics Instrument Data base.
    9.1.3.2. Start the Environics Series 4000 Software.
    9.1.3.3. From the Main Menu, select Configure – Instrument – Install.
    9.1.3.4. Insert the Instrument Data Disk in the appropriate floppy disk drive.
    9.1.3.5. When the Install Instrument box appears, select the appropriate Drive Icon (A or B) from the pull down box and click OK.
    9.1.3.6. The instrument is now ready to use.
  9.1.4. Creating desktop
    9.1.4.1. From the Environics Series 4000 main window select File and then New.
    9.1.4.2. In the DESKTOP box, enter a name for the Desktop.
    9.1.4.3. In the INSTRUMENT box, click the drop down arrow to display the list of available instruments and select the serial number of the instrument you are using.
    9.1.4.4. Click OK to save the new file.
  9.1.5. Cylinder Configuration
    9.1.5.1. Open Series 4000 main window click on Configure and then Cylinder to open the cylinder library. This will bring up the Cylinder Library form (Fig. 2).
    9.1.5.2. Click Add and enter a name for the new cylinder at the prompt. The name of the cylinder will usually be the name of the gas of interest in the cylinder.
    9.1.5.3. Click the OK button. This name will appear in the Name box.
    9.1.5.4. K-Factor should be set to automatic.
    9.1.5.5. Creation Date should be the current date.
    9.1.5.6. If there is no expiration date on the cylinder then click on the box next to No Expiration Date. If there is an expiration date then unclick the box next to No Expiration Date and enter in the expiration date on the cylinder.
    9.1.5.7. Type the cylinder serial number in the Information box.

9.1.5.8. Enter contents of the cylinder beginning with the balance gas. Using the pull-down box under Gas Name or Symbol in the Contents section, select the desired balance gas. The concentration will be automatically set at 1 million ppm.



Cylinder Name: co - 100ppm
K-Factor: 1.0
● Automatic
○ Manual
Creation Date: 11/3/03
Information:
Expiration Date: 11/3/03
☑ No Expiration Date

Contents

Interest	Balance	Gas Name	Symbol	Concentration	Units	K-Factor
☐	☑	NITROGEN	N2	999900.00	ppm	1.000
☑	☐	CARBON MONOXIDE	CO	100.00	ppm	0.9992
☐	☐					
☐	☐					
☐	☐					
☐	☐					
☐	☐					
☐	☐					
☐	☐					
☐	☐					

Figure 2. The Cylinder Library Screen.

9.1.5.9. Continue adding the remaining component gases by selecting them from the Gas Name or Symbol pull down box.

9.1.5.10. Select default gas of interest by clicking the Interest box next to the desired gas.

9.1.5.11. Once all gases have been added, enter the concentration of each component gas under the concentration box. The units can be changed from ppm to % using the pull down box under units. The concentration of each gas and the unit of gas will be identified on the side of the cylinder. If the gas is read in ppb then will need to be converted to ppm. The balance gas concentration will automatically be filled in as the difference so that all gases add up to 1 million ppm (100%).

9.1.5.12. As data is entered, the cylinder K-factor will automatically be computed based on the concentration of the individual gases in the cylinder.

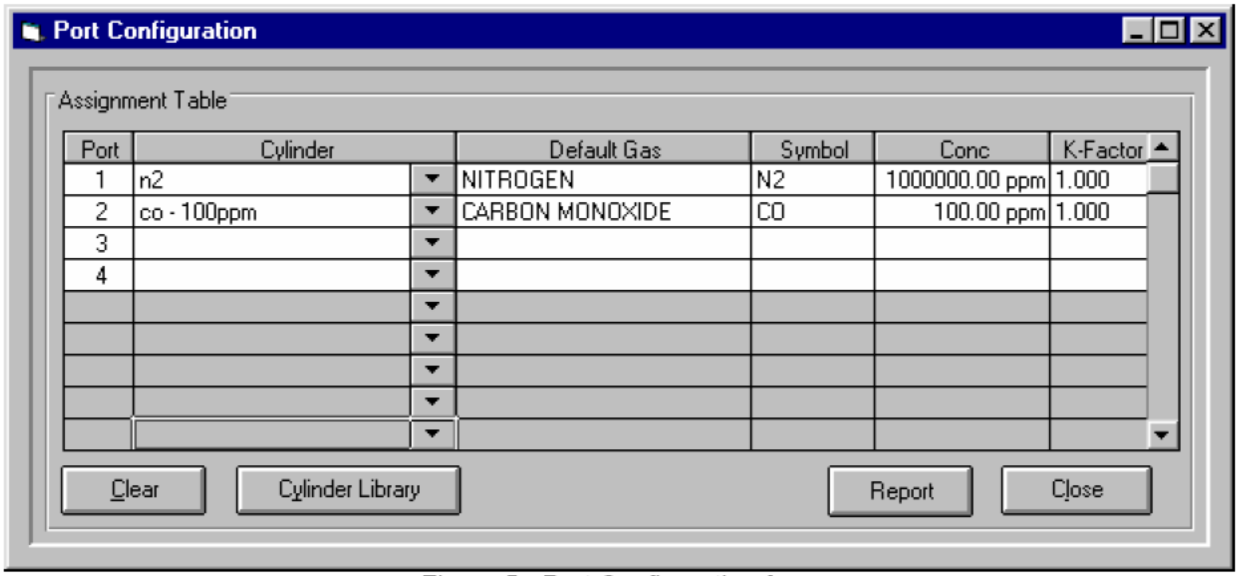
9.1.5.13. When done, click Close to save the cylinder information and exit the Cylinder Library. The Program will display a prompt to allow the new cylinder to be saved. Click the Yes button to save.

9.1.5.14. Alternately, click Add to add a new cylinder without exiting the Cylinder Library.

9.1.6. Editing cylinder gas information

9.1.6.1. To move or delete a gas, click the name of the gas. The name box will turn red.

9.1.6.2. To move a gas, click on the Move Up or Move Down button to move the gas down one position on the list. Continue to click on the Move Down button until the gas is in the desired location on the list.
9.1.6.3. To delete the selected gas from the cylinder, click the Clear button.
9.1.7. Port configuration
9.1.7.1. To open the Port Configuration screen, select Configure – Port from the main menu. The desktop must be open to change the port configuration. If a desktop is not open, then Configure – Port will not be accessible.
9.1.7.2. The current cylinder assignments for the port configuration are displayed in the port configuration assignment table (Fig. 3). To assign a different cylinder to the port, uses the pull down box under cylinder, and select the desired cylinder from those listed in the cylinder library.
9.1.7.3. Once the cylinder is assigned, the default gas, symbol, concentration, and K-factor are automatically placed in the table from the cylinder library. It cannot be edited within the port configuration table. To edit this information, click on Cylinder Library and edit from within the library.



Port	Cylinder	Default Gas	Symbol	Conc	K-Factor
1	n2	NITROGEN	N2	1000000.00 ppm	1.000
2	co - 100ppm	CARBON MONOXIDE	CO	100.00 ppm	1.000
3					
4					

Figure 3. The Port Configuration Table.

9.1.7.4. 9.1.7.4. To save the cylinder/port assignments, click on Close, then click YES.
9.1.7.5. Using ¼″ Teflon tubing with a Gyrolok fittings provided with the instrument connect one end of the tubing to the Environics configured port and, with a Swagelok fitting connect the other end of the Teflon tubing to the regulator on the desired cylinder.
9.1.7.6. To remove cylinders from a port, select the cylinder by clicking on the cylinder name in the assignment table, and click the Clear button to remove the cylinder from the port configuration table.

9.2. Operating the instrument

9.2.1. Concentration Mode

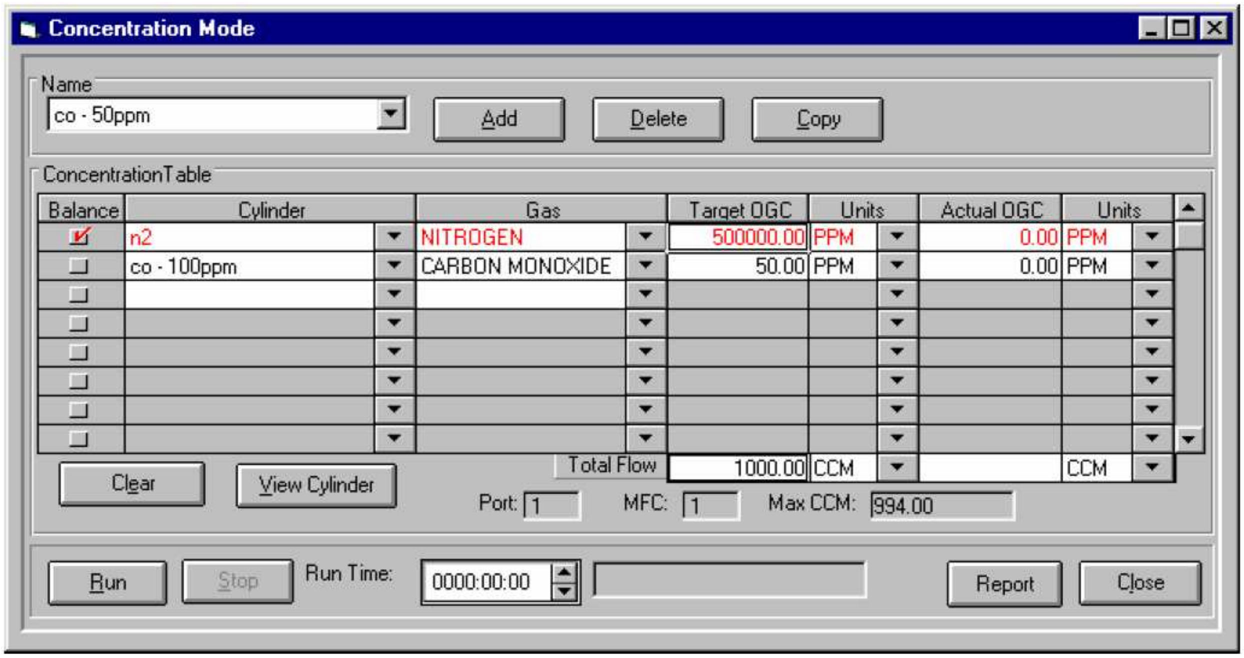
9.2.1.1. To open the Concentration Mode (Fig. 4) screen, select Run – Concentration from the Series 4000 main window.

9.2.1.2. To add a Concentration Mode file click on Add.

9.2.1.3. Enter the file name and click OK.

9.2.1.4. A blank Concentration Mode table will appear.

9.2.1.5. Specify each desired source cylinder by using the pull down box in the concentration table under Cylinder. Select the cylinder by clicking on it. The port is automatically selected based on the current cylinder/port assignments. The port number is displayed in the Port box.



Name: co - 50ppm

Balance	Cylinder	Gas	Target OGC	Units	Actual OGC	Units
☑	n2	NITROGEN	500000.00	PPM	0.00	PPM
☐	co - 100ppm	CARBON MONOXIDE	50.00	PPM	0.00	PPM
☐						
☐						
☐						
☐						
☐						
☐						
		Total Flow	1000.00	CCM		CCM

Port: 1 MFC: 1 Max CCM: 994.00

Run Time: 0000:00:00

Figure 4. The Concentration Mode Screen.

9.2.1.6. Next, specify the gas of interest by using the pull down box under Gas next to the cylinder name. Select the component gas of interest by clicking on it. To see the contents of the cylinder, click on View Cylinder.

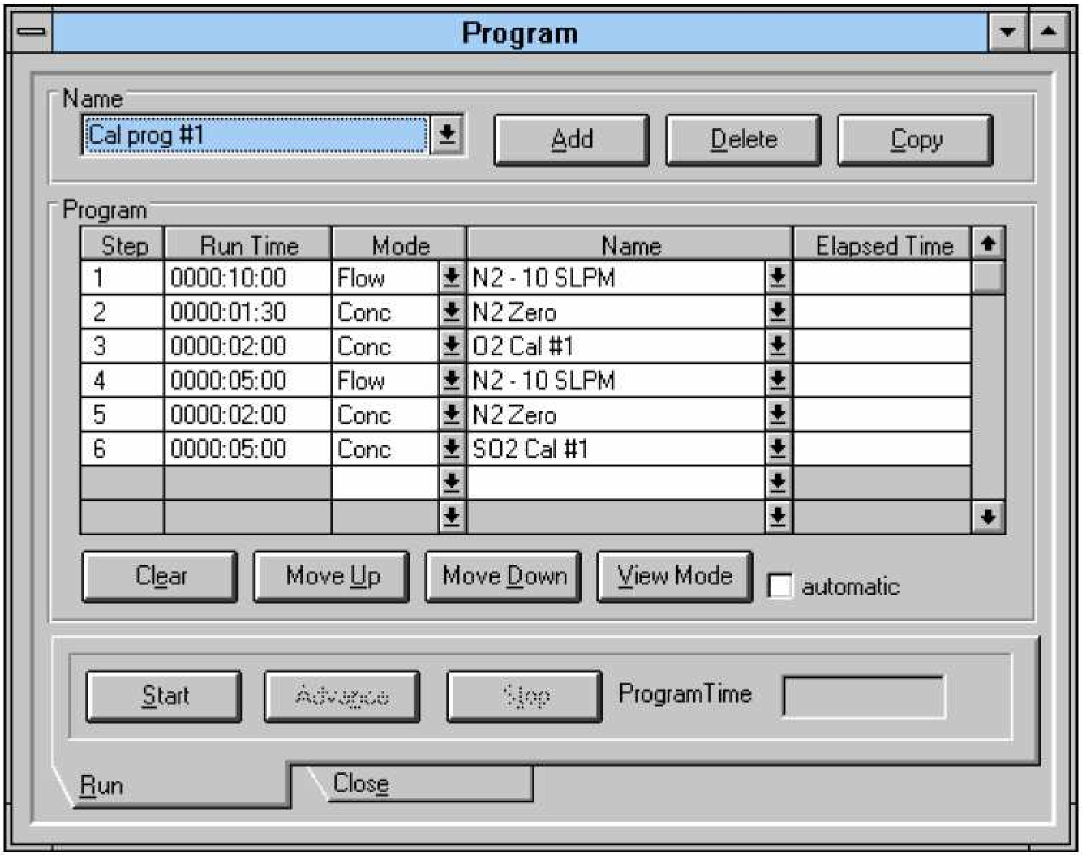
9.2.1.7. Once all of the cylinders are entered, select the balance gas by clicking the balance box beside the cylinder name.

9.2.1.8. Specify the desired output concentration for each cylinder. Type the value in the Target Output Gas Concentration (OGC) box. If desired, the units can be changed between ppm and % by using the pull down box under units.

9.2.1.9. Finally, enter the total flow by typing in its value. The units can be changed from ccm to L/min by using the pull down menu under units. The value entered is used by the system to compute the target concentration of the balance gas and the required flow of each controller.

9.2.2. Program Mode

9.2.2.1. To open the Program Mode screen (Fig. 5), select Run – Program from the Series 4000 main window.
9.2.2.2. To add a Program Mode file click on Add.
9.2.2.3. Click the Mode pull down box in the row of the desired step. Select the desired run mode which will be Conc.



Name: Cal prog #1

Program

Step	Run Time	Mode	Name	Elapsed Time
1	0000:10:00	Flow	N2 - 10 SLPM	
2	0000:01:30	Conc	N2 Zero	
3	0000:02:00	Conc	O2 Cal #1	
4	0000:05:00	Flow	N2 - 10 SLPM	
5	0000:02:00	Conc	N2 Zero	
6	0000:05:00	Conc	SO2 Cal #1	

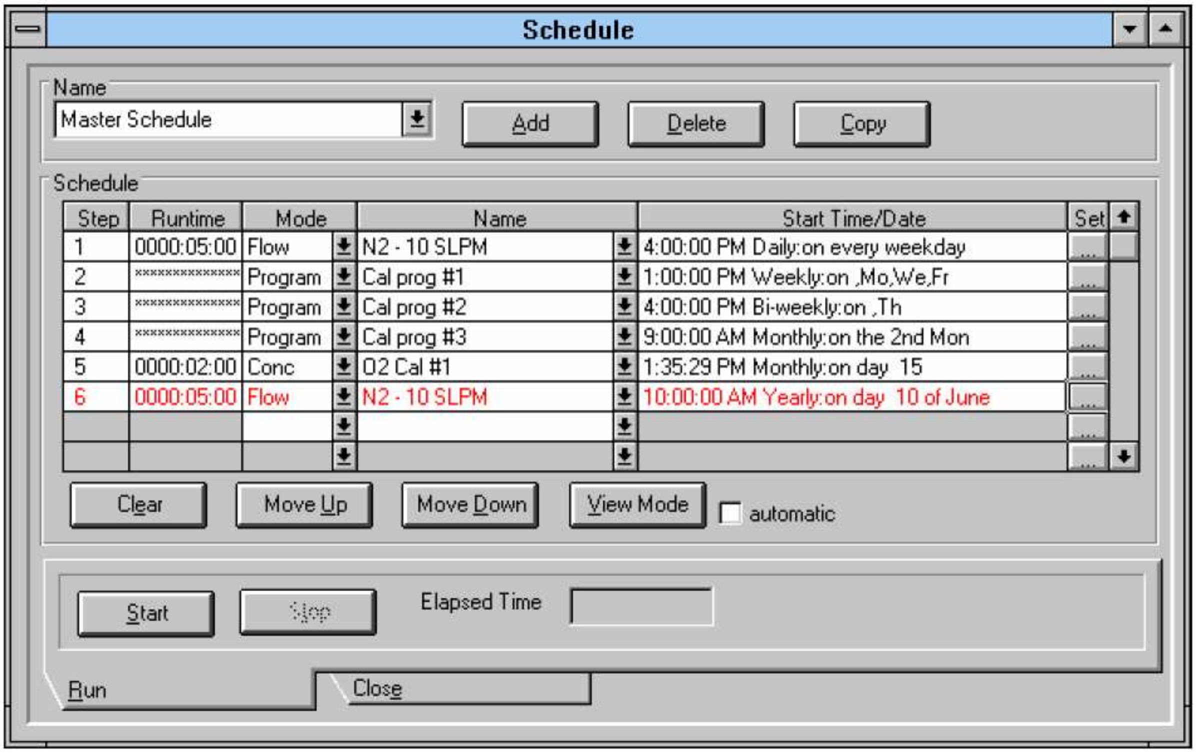
Figure 5. The Program Mode Screen.

9.2.2.4. The available saved mode names will appear in the Name box. Open the pull down box under Name; select the desired run mode file.
9.2.2.5. Click in the Run Time box to set the run time for the mode (HH:MM:SS).
9.2.2.6. Repeat until all desired steps have been entered.
9.2.2.7. Press the Start button to begin the current program. While the program is running, the Elapsed Time box indicates the elapsed time for the current step. In addition, the Program Time box indicates the total elapsed time.
9.2.2.8. To abort the current step and proceed to the next, press the Advance button.
9.2.2.9. To stop the program before completion, click the Stop button.

9.2.3. Schedule Mode

9.2.3.1. To open the Schedule Mode screen (Fig. 6), select Run – Schedule from the Series 4000 main window.
9.2.3.2. Click Add, enter a name for the file and click OK.

9.2.3.3. A blank Schedule Mode table will appear. Fill in the table with the desired values.



Name: Master Schedule

Schedule

Step	Runtime	Mode	Name	Start Time/Date	Set
1	0000:05:00	Flow	N2 - 10 SLPM	4:00:00 PM Daily:on every weekday	...
2	**************	Program	Cal prog #1	1:00:00 PM Weekly:on ,Mo,We,Fr	...
3	**************	Program	Cal prog #2	4:00:00 PM Bi-weekly:on ,Th	...
4	**************	Program	Cal prog #3	9:00:00 AM Monthly:on the 2nd Mon	...
5	0000:02:00	Conc	O2 Cal #1	1:35:29 PM Monthly:on day 15	...
6	0000:05:00	Flow	N2 - 10 SLPM	10:00:00 AM Yearly:on day 10 of June	...
					...
					...

☐ automatic

Figure 6. The Schedule Mode Screen.

9.2.3.4. Click the Mode pull down box in the row of the desired step. Select the desired run mode which will be Conc.

9.2.3.5. The available saved mode names will appear in the Name box. Open the pull down box under Name; select the desired run mode file.

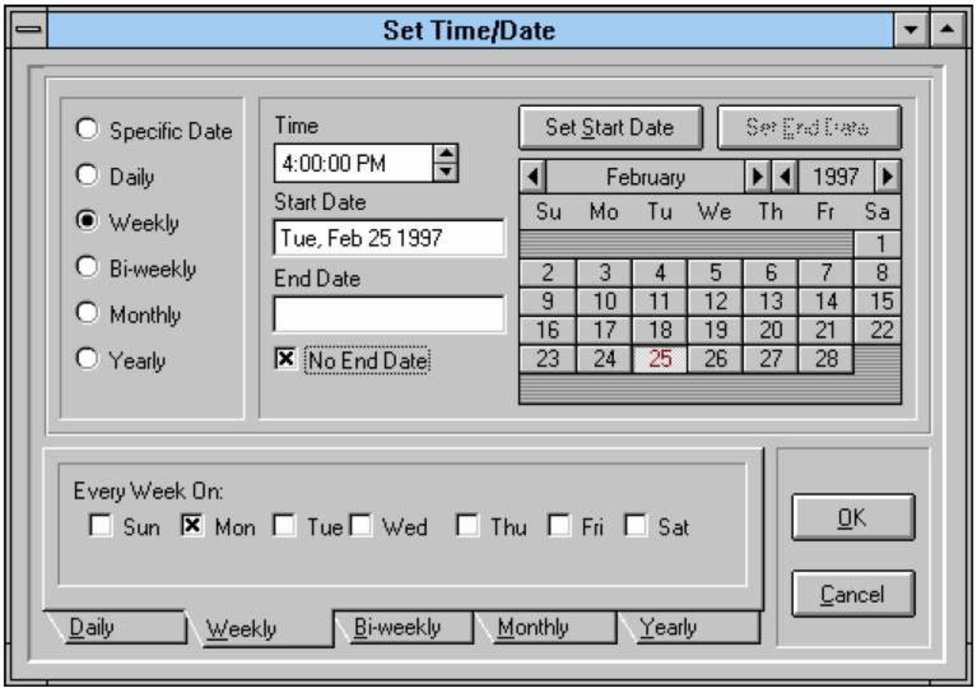
9.2.3.6. Click in the Run Time box to set the run time for the mode (HH:MM:SS).

9.2.3.7. Click the SET button to set the time/date for this item. This will bring up the Time/Date editor (Fig. 7).

9.2.3.7.1. Select the type of event using the buttons on the left side of the screen. For an item that occurs only once, click the Specific Date button. For a repeating (Daily, Weekly, etc.) event, click the button for the desired frequency.

9.2.3.7.2. For a repeating event, fill out the appropriate options at the bottom of the screen. The options displayed will vary, depending on the type of repeat chosen. If an item is repeated weekly or biweekly, select the day(s) of the week that it is to be run by clicking in the appropriate square(s). For items repeated monthly, select the day of the month on which to repeat the item (such as every first of the month or every third Monday). For items repeated yearly, select the day and month on which to repeat the item (such as every January 1 or every third Monday in May).

9.2.3.7.3. Enter the desired start time, by clicking on the Start Time box and typing in the time, or by clicking the up/down arrows.



○ Specific Date
○ Daily
● Weekly
○ Bi-weekly
○ Monthly
○ Yearly

Time: 4:00:00 PM
Start Date: Tue, Feb 25 1997
End Date:
☒ No End Date

Every Week On:
☐ Sun ☒ Mon ☐ Tue ☐ Wed ☐ Thu ☐ Fri ☐ Sat

Figure 7. The Time/Date Editor within Schedule Mode.

9.2.3.7.4. Select the desired start date from the calendar by clicking on the day and then clicking the SET START DATE button. Use the left/right buttons to change the month or year. For a Specific Date event, this is the date the item will be run. For repeating events, it is the date the event will start to repeat.

9.2.3.7.5. For repeating events, select the desired end date from the calendar by clicking on the day and then clicking the SET END DATE button. This allows the repeating event to occur within a specific range of dates. Alternately, if this item is to be repeated indefinitely, click the NO END DATE box. End Date is not used for a Specific Date event.

9.3. 9.3. Generating reports

9.3.1. From the Main Menu, select Report and then the desired report type, or click the Report button directly from the Configure/Run Mode that is currently in use.

9.3.2. In the Reports dialog box (Fig. 8), click the Select Report dropdown box to select the desired report type. The types of available reports are given in Table 1.

9.3.3. Select the desired "Create Report For" and "Selection" options desired.

9.3.3.1. To limit the report to a specific item, click the "Current desktop, selection only" option, and select the desired item in the "Selection" box.

9.3.3.2. To limit the report to all items in the current desktop, click the "Current desktop, all items" option. The item shown in the "Selection" box is ignored.

9.3.3.3. To report on all items in all desktops, click the "All items" option. The item shown in the "Selection" box is ignored.

Select Report: Divider Modes

Create Report For
● All items
○ Current desktop, all items
○ Current desktop, selection only

Selection: co divider

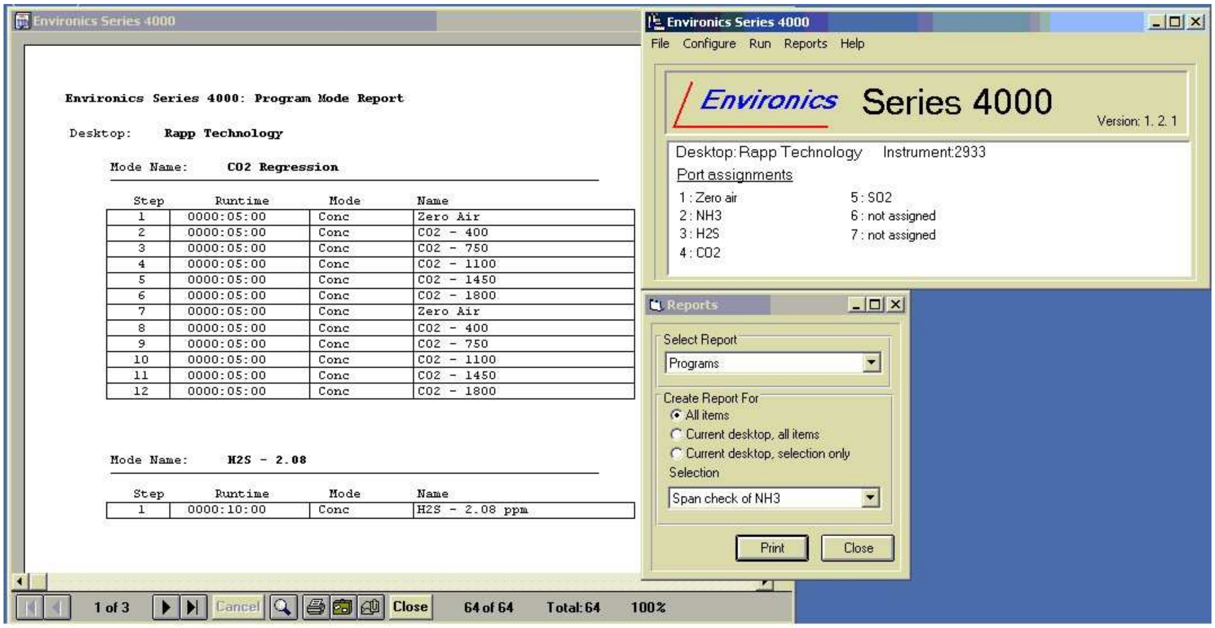
Figure 8. The Reports dialog box.

Table 1. Available report formats in the Environics software.

Gas Library	Lists all gases in the gas library
Cylinder Library	Shows cylinder name(s) and parameters (compositions and concentrations), for one or all cylinders
Cylinder Audit	Gives names, creation and expiration dates, and days until expiration for all cylinders
Physical Configuration	Gives configuration details for the MFCs, ports, and solenoids in the instrument
Instrument Calibration	Gives calibration tables for the MFC
Port Configuration	Gives cylinder assignments (name, gas and concentration) currently made to each port
Concentration Mode	Details one or more Concentration Mode files (cylinders, gas concentrations, total flow rate)
Programs	Details one or more Program Mode files
Schedules	Details one or more Schedule Mode files

9.3.4. Click Print to view the report on the screen. A sample Program Mode report is shown in Fig. 9.
9.3.5. Print the report and store it according to SOP B5.
9.3.6. Click the arrow buttons to move between pages. Click the Magnifying Glass Icon to change the view size.
9.3.7. Click the Printer icon to send the report to the printer.

9.3.8. When finished, click the Close button to return to the Reports menu.
9.3.9. Repeat Sections 9.6.2 through 9.6.8 to generate additional reports.
9.3.10. When done, click Close to return to the Main Menu.



Environics Series 4000: Program Mode Report

Desktop: **Rapp Technology**

Mode Name: **CO2 Regression**

Step	Runtime	Mode	Name
1	0000:05:00	Conc	Zero Air
2	0000:05:00	Conc	CO2 - 400
3	0000:05:00	Conc	CO2 - 750
4	0000:05:00	Conc	CO2 - 1100
5	0000:05:00	Conc	CO2 - 1450
6	0000:05:00	Conc	CO2 - 1800
7	0000:05:00	Conc	Zero Air
8	0000:05:00	Conc	CO2 - 400
9	0000:05:00	Conc	CO2 - 750
10	0000:05:00	Conc	CO2 - 1100
11	0000:05:00	Conc	CO2 - 1450
12	0000:05:00	Conc	CO2 - 1800

Mode Name: **H2S - 2.08**

Step	Runtime	Mode	Name
1	0000:10:00	Conc	H2S - 2.08 ppm

1 of 3 64 of 64 Total: 64 100%

Version: 1. 2. 1

Desktop: Rapp Technology Instrument:2933

Port assignments

1 : Zero air	5 : SO2
2 : NH3	6 : not assigned
3 : H2S	7 : not assigned
4 : CO2	

Select Report: Programs

Create Report For
● All items
○ Current desktop, all items
○ Current desktop, selection only

Selection: Span check of NH3

Figure 9. Screen capture showing a sample Program Mode report.

9.4. Troubleshooting
9.4.1. Perform the steps in Section 9.1.3 if a "no instruments registered" warning appears.
9.4.2. When opening a desktop, the software will try to communicate with the assigned instrument. If it is unable to communicate, a warning message will be displayed. There are two possible reasons for this:
9.4.2.1. The desktop's assigned serial number does not match the instrument being used.
9.4.2.2. The selected PC serial port on the Environics software might be configured to a COM different from the actual COM that is connected to the PC.
9.4.3. The only available cylinders listed are those assigned to ports. If the desired cylinder is not listed, modify the port settings.
9.4.4. The Program Mode cannot be set up unless either the Concentration, Flow, or Divider mode is set up.
9.5. Computer hardware & software
9.5.1. Store all applicable software in the data acquisition computer.
9.5.2. The dilutor can be controlled by, and input data to, the AirDAC program (SOP B2).

10. Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.

10.2. Manage all data according to SOP B5.

10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

10.4. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

11.1. Flow Calibration (Initial)

11.1.1. Mass flow controller zero check

11.1.1.1. Some MFCs require the cover to be removed to make adjustments

11.1.1.2. Connect the (-) lead from a voltmeter to PC402 GROUND (TP2).

11.1.1.3. Connect the (+) lead to each MFC Response Test Point: MFC1 (TP9), MFC2 (TP11), MFC3 (TP13), and MFC4 (TP15).

11.1.1.4. Verify that the voltage at each test point is 0 V ±.025 V for a Tylan MFC and 0.01 V ±0.005 V for a Hastings MFC. If the "zero" is out of spec, adjust the MFC “zero” pot to bring it within these specified ranges.

11.1.1.5. After adjusting the “zero” pot, replace system cover.

11.1.1.6. After "zero" adjustment, wait at least one hour (1 h) for temperature to stabilize before proceeding to MFC calibration (Section 11.1.2).

11.1.2. Mass flow controller calibration

11.1.2.1. Connect calibration gas to the appropriate input ports for the MFCs being calibrated. The input pressure should be approximately 25 psig.

11.1.2.2. Connect a primary standard flow calibration device to the output of the system.

11.1.2.3. Be sure the instrument is connected to the PC and powered up.

11.1.2.4. Run the Environics Series 4000 software.

11.1.2.5. From the menu screen, select Configure.

11.1.2.6. Select Instrument.

11.1.2.7. Select Calibrate.

11.1.2.8. In the Reference Gas box, click the down arrow button and set this to match the calibration gas for that MFC.

11.1.2.9. In the Controller Instrument box, click the down arrow button, and select the serial number of the instrument to be calibrated.

11.1.2.10. In the MFC box, click the down arrow button, and select the desired Mass Flow Controller to enter data (start with MFC 1).

11.1.2.10.1. If the MFC has been changed, select Init Points, enter appropriate size in Max Flow, Table will initialize.

11.1.2.11. Click on the 100% calibration point and press Start. Wait at least 10 min for flow to stabilize before continuing.

11.1.2.12. Using a mass flow meter, take a flow measurement. Measured flow value must be between 100% and 110% of the MFCs rated full-scale flow. If flow value is out of this range, adjust the MFC “span” pot.

11.1.2.12.1. Some MFCs require the cover to be removed to make adjustments. In this case, after performing “span” adjustment, replace MFC cover. After adjusting “span” pot, replace system cover and wait at least 1 h for temperature to stabilize before continuing.

11.1.2.13. If measured flow value is acceptable, enter the measured value in the True Flow column. If not, repeat Step 9.3.11.
11.1.2.14. Click on the next calibration point, and press the Start button. Wait at least 2 min for flow to stabilize before continuing.
11.1.2.15. Take a flow measurement, and enter the value in the True Flow column.
11.1.2.16. Repeat Steps 9.3.14 to 9.3.15 until all calibration points have been completed.
11.1.2.17. Press Stop, then press Save to save the MFC calibration data. This will download the new calibration files to the instrument.
  11.1.2.17.1. If a warning appears “CAL TABLE ERROR -3”, click OK, then save again before exiting.
11.1.2.18. Repeat steps 9.3.10-9.3.17 until all MFCs have been calibrated.
11.1.2.19. Click Close to return to the main menu screen.

11.2. Flow Calibration (Periodic)
11.2.1. The FRM 205 recommends that the gas dilution system shall be recalibrated once per calendar year using NIST-traceable primary flow standards with an uncertainty of ≤ 0.25%. The QAPP may specify a more frequent calibration schedule. The manufacturer will conduct this recalibration.
11.2.2. Keep a label (Fig. 10) affixed at all times to the gas dilution system listing the date of the most recent calibration, and the due date for the next calibration.

Calibration date:_________________
Next calibration due date:_________

Figure 10. Sample calibration label.

11.2.3. Before sending a unit to the manufacturer for re-calibration, record the existing (as-found) flow rate (if this is not automatically recorded by AirDAC), and the resultant dilution ratio based on that flow rate.
11.3. At each use of the Model 4040, document its delivered flow rate to ensure that it is at least 0.5 L/min higher than the GSS flow rate (SOP G1).
11.4. In any case where the Model 4040 is not under the control of the AirDAC program, make sure that the computer clock (used by the Environics system) is synchronized with the time in AirDAC.

12. References

12.1. Environics Model 4040 Manual. 2003. Series 4000/4040 Gas Mixing System/Gas Dilution System. Environics, Inc, Tolland, CT.
12.2. Environics Inc. 2003. Computerized Gas Dilution System Series 4040, Online at http://www.environics.com/4040.html. Accessed 10 January 2006.
12.3. SOP B2. 2006. Data Acquisition and Control Software (AirDAC) Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
12.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue

Ag Air Quality Lab.
12.5. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
12.6. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
12.7. SOP G12. 2006. FTIR Verification of Gas Cylinder Concentration. Standard Operating Procedure G12. Purdue Ag Air Quality Lab.
12.8. SOP V6. 2006. GC/MS Analysis of VOCs Collected in Sampling Canisters. Standard Operating Procedure V6. Purdue Ag Air Quality Lab.
12.9. SOP V7. 2006. Cleaning, Certification and Pre-sampling Preparation of Sampling Canisters. Standard Operating Procedure V7. Purdue Ag Air Quality Lab.

13. Contact Information

13.1. Manufacturer:
Environics, Inc.
69 Industrial Park Road East
Tolland, CT 06084-2805
Phone: (860) 872-1111
Fax: (860) 870-9333
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FTIR VERIFICATION OF GAS CYLINDER CONCENTRATION

Standard Operating Procedure (SOP) G12

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Standard Operating Procedure (SOP) G12

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1. Scope and Applicability

1.1. A method is needed which will accurately analyze the concentrations of certified standard gases in cylinders purchased from outside vendors. Such a method is needed to ensure that the gas cylinders which are used for calibrations and/or performance checks of gas analyzers and other analytical instruments do, in fact, contain gases at their stated concentrations.

1.2. Fourier Transform Infrared (FTIR) Spectroscopy provides a reliable method for verifying gas cylinder concentrations for vapor-phase organic or inorganic compounds that absorb energy in the mid-infrared spectral region, about 800-4000 cm-1. Compounds that can be checked using this method include ammonia, carbon dioxide, methane, hexane, n-butanol, acetone, ethylene, carbon monoxide, propane, and nitrous oxide.

1.3. Typically, the FTIR minimum detection limit is as low as 2-10 ppb. Analytical range and sensitivity depend on the frequency-dependent component absorptivity, instrument configuration, data collection parameters, and gas stream concentration.

1.3.1. Instrument factors that affect performance (range, sensitivity, etc.) include:

1.3.1.1. Spectral resolution

1.3.1.2. Interferometer signal averaging time

1.3.1.3. Detector sensitivity and response

1.3.1.4. Absorption path length

1.4. This procedure applies only to the Nexus 670 FTIR gas spectrometer (Thermo Electron Corporation, Palatine, IL).

2. Summary of Method

The FTIR gas spectrometer (Nexus 670, Thermo Electron Corporation, Palatine, IL) allows accurate analysis of concentrations of compounds that are delivered from gas cylinders. The FTIR analyzer consists of a KBr beamsplitter, a mercury cadmium telluride High D* (MCT-High D*) detector (cooled with liquid nitrogen), an IR source, a heated stainless steel gas absorption cell, an electronic package, and a computer. The sample gas can be delivered to the gas absorption cell either directly from the gas cylinder (continuous purge mode), or from a sample bag to the vacuumed gas cell (batch mode). The latter is used if the gas cylinder is not available onsite with the FTIR. The optical bench measures the intensity of a specially encoded infrared beam after it has been passed through a sample. The resulting signal, called an “interferogram”, contains information about all frequencies present in the beam. The computer reads the interferogram, uses Fourier transformation to decode the intensity information for each frequency, and presents the spectrum. Absorbance band intensities in the spectrum are related to sample concentrations by what is commonly referred to as Beer’s Law. The frequency-dependent relationship between absorptivity and concentration can be determined by taking standard spectra of samples of compounds at known concentrations. Unknown compounds can be detected by matching sample absorbance bands with bands in standard spectra, and their concentrations can be calculated by comparing sample band intensities with standard band intensities. Using OMNIC QuantPad software (Thermo Nicolet Instrument Corporation, Madison, WI), quantitative analysis methods can be created which allow analysis of spectra from gas- or condensed-phase samples using the Classical Least-Squares (CLS) quantitative analysis algorithms.

3. Definitions of Terms and Acronyms

3.1.	FTIR	Fourier Transform Infrared (FTIR) spectrometer
3.2.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.3.	Analyte	A compound that the method is required to measure.

4. Health and Safety

4.1. When filling the detector's Dewar flask, be careful not to contact the liquid nitrogen with your skin. Wear protective gloves and goggles, and follow standard laboratory safety practices.

4.2. Do not touch the infrared source housing; it may be very hot. It stays hot for up to 15 min after the spectrometer is turned off.

4.3. Always follow the safety information that is displayed on the spectrometer itself. Do not operate a spectrometer, or any accessory, without following the safety precautions described in the manual and the documentation that came with the spectrometer.

4.4. Laser light exposure is possible when a beam-deflecting accessory (for example, a diffuse reflectance accessory) is installed. Never stare into the sample compartment when a beam-deflecting accessory is installed.

4.5. Avoid exposure to hazardous compounds when delivered from gas cylinders. Ensure that the gases are properly vented and that the gas delivery system is leak-free.

5. Interferences

5.1. Background interference. This results from a change in throughput relative to single beam background. In routine sampling, throughput may degrade over several hours. Periodically a new background spectrum must be collected.

5.2. Interference occurs when other compounds in the sample have spectral features that overlap with those of the analyte(s). Water vapor and CO2 are common spectral interferents. The extent of interference depends on the interferent concentration, analyte concentration, and the degree of band overlap. Spectral interference can be minimized or avoided by choosing an alternate spectral region for analysis.

5.3. If the temperature in the gas cell is too low, condensation of the analytes and/or water vapor can occur. Set the temperature of the gas cell at 50°C to minimize condensation.

5.4. In addition to being a spectral interferent, condensed moisture removes soluble compounds from the gas phase by allowing them to dissolve into the separate liquid phase.

5.5. Ambient air can mix with the sample if the gas absorption cell valves are not leak-free when delivering the sample in batch mode from a sample bag.

5.5.1. When introducing sample from a sample bag, minimize the length of inlet tubing to reduce errors in gas concentrations.

5.6. Samples stored in sample bags should be analyzed as soon as possible (preferably within 24-48 hrs) to avoid potential contamination of chemicals released from the sample bag itself and the loss/uptake of gases via diffusion though walls.

5.6.1. If possible, controlled tests with sample bags should be performed prior to their use to collect samples, to establish that the analyte(s) of interest are not lost from the bags. See Section 11.9.

6. Cautions

6.1. Always ensure that the detector is cooled by liquid nitrogen before taking a spectrum.

6.2. The manufacturer recommends that the spectrometer power remain on, even when not in operation. Allow the spectrometer to warm up for one hour if the power has just been turned on, then align it before collecting spectra.

6.3. Collect a new background spectrum regularly, preferably once every 4 h during the operation of the FTIR.

6.4. Collect a new background spectrum if the software parameters have been changed.

6.5. Align the spectrometer daily for optimum performance. The spectrometer should also be aligned if the ambient temperature has changed by at least 5°C, or in such situations such as when a laser has been replaced, a beamsplitter has been installed, the spectrometer has just been moved, or the detector signal intensity has dropped significantly from its usual level.

6.6. Never use a flammable gas to purge a spectrometer bench. The purge gas must be free of moisture, oil and other reactive materials. Use dried air or nitrogen to purge the instrument. Other gases, even inert gases such as argon (Ar), can damage the spectrometer laser.

6.7. The FTIR operates reliably at ambient temperatures between 16°C and 27°C. Temperature changes may result in drift in the system response.

6.8. The sample spectra to be analyzed using a particular method must have the same data point spacing and the same spectral range (frequency limits) as the spectra used to calibrate the method.

6.9. The calibration spectra must be of pure components or pure components diluted with a solvent or gas that either doesn’t absorb infrared energy or absorbs in a different region of the infrared spectrum.

7. Personnel Qualifications

7.1. Personnel should be trained in the use of the instrument before initiating the procedure. Training requires about three days.
7.2. Each analyst must read and understand the entire instrument manual and this SOP before operating it.
7.3. Personnel should also be trained in verifying the instrument is functioning properly, performing routine maintenance, making a calibration method for analyzing a specific component, and interpreting the analyzer output signal.

8. Equipment and Supplies

8.1. FTIR gas spectrometer (Model Nexus 670, Thermo Electron Corp., Palatine, IL).
8.2. Stainless steel gas absorption cell (Mars Model 8L/40V, Gemini Scientific Instruments, Buena Park, CA)
8.3. Purge gas generator (Model PCDA1112-122, MTI-Puregas, Westminster, CO).
8.4. Absolute pressure gauge (Automation Products Group, Logan, UT)
8.5. Temperature controller (Model 689-0005, Barnant Co., Barrington, IL).
8.6. Heat Jacket and Thermo Controller
8.7. Vacuum pump
8.8. Nitrogen gas
8.9. Liquid nitrogen, Dewar flask, and funnel
8.10. Teflon tubing
8.11. Calibration standard gas
8.12. Sample bags (if gas cylinder is offsite)
8.13. Diluter

9. Procedures

9.1. Take triplicate bag (#1, 2, and 3) samples from a gas cylinder, if the gas cylinder is offsite.
  9.1.1. Flush each bag thoroughly at least 3 times with ultra-high purity (UHP) grade nitrogen and vacuum completely before use. Bag volume should be ≥ 40 L.
  9.1.2. Turn on the gas cylinder following SOP G2.
  9.1.3. Connect Teflon tubing that is connected to the gas cylinder regulator to the hose connection of bag (#1) fitting (stem protruding from the side of the bag fitting).
  9.1.4. Open the shut-off valve of the bag (#1) by holding the side stem and turning the entire upper portion of the fitting counterclockwise one revolution. Gas from cylinder begins to fill the bag. Fill the bag to no more than 80% of its maximum volume.
  9.1.5. Turn the gas cylinder off and close the shut-off valve of the sampling bag (#1) by holding the side stem and turning the entire upper portion of the fitting clockwise until it is snug.
  9.1.6. Detach the Teflon tubing from the sample bag (#1).

- 9.1.7. Finish sampling for bags #2, and 3 by repeating the Sections 9.1.1 to 9.1.6.
- 9.1.8. Pack the filled sample bags in a rigid carton to prevent damage or discharge during shipping. Ship the samples to the laboratory by overnight delivery, but do not ship sample bags by air unless the cargo cabin of the aircraft is pressurized. Time shipping dates so that samples are received by the laboratory when someone is there to accept them.

9.2. Turning on the system components

- 9.2.1. Connect external power supply.
- 9.2.2. Turn on accessories such as the temperature controller.
- 9.2.3. Turn on the spectrometer by pressing the power switch (I/O).
- 9.2.4. Turn on the printer (if available).
- 9.2.5. Turn on the computer.
- 9.2.6. Allow at least 1 h after turn on of the spectrometer and wait until the temperature inside the gas cell stabilized at 50°C before collecting spectrum.
- 9.2.7. Keep spectrometer on at all times for stable operation.
- 9.2.8. If the system components are to be turned off, do so in the following order:
  - 9.2.8.1. Turn off computer.
  - 9.2.8.2. Turn off printer.
  - 9.2.8.3. Turn off spectrometer.
  - 9.2.8.4. Turn off accessories.

9.3. Checking the purge gas generator

- 9.3.1. Set the pressure regulator of the purge gas generator between 20 and 40 psi.
- 9.3.2. Set the flow meter to deliver around 30 scfh.
- 9.3.3. Use only nitrogen or compressed air to purge the system.

9.4. Starting OMNIC software

- 9.4.1. Double-click the OMNIC shortcut on the windows desktop.

9.5. Cooling the detector with liquid nitrogen

- 9.5.1. Before running any samples, check whether liquid nitrogen needs to be added.
  - 9.5.1.1. Open OMNIC.
  - 9.5.1.2. Select “Experiment Setup” from the “Collect” Menu.
  - 9.5.1.3. Go to the tab labeled “Diagnostic”.
  - 9.5.1.4. If the detector signal is a flat line, liquid nitrogen must be added to the system before spectra are collected.
  - 9.5.1.5. Wait 15 min, or until the signal intensity is satisfactory before collection (the magnitude of signal changes as a function of resolution being used). A minimum signal intensity of 30% of maximum voltage is required if resolution is set at 1.
  - 9.5.1.6. Liquid nitrogen will evaporate over time, even if the FTIR does not scan. As a rule of thumb, it should be replenished after 8-10 h, even if the system has been idle during that time.
- 9.5.2. Add liquid nitrogen
  - 9.5.2.1. ALWAYS wear protective eye wear and gloves.
  - 9.5.2.2. Fill the small liquid nitrogen Dewar cylinder 2/3 full from a large Dewar.
  - 9.5.2.3. Open the lid on the left hand side of FTIR, and pour liquid nitrogen from the small Dewar into the top hole using the special funnel provided.

9.5.2.4. Close the lid and return gloves/goggles to the drawer.

9.6. Quant Pad set up prior to collecting spectrum

9.6.1. Open Quant Pad.

9.6.2. Click “Method” button on the Quant Pad button panel.

9.6.2.1. Click the “Select Method” button.

9.6.2.2. Select the file of the quant method you want to use for the gas cylinder concentration check and then choose “OK”. An analysis method can be made following the procedures described in Section 9.11. If the gas concentration being checked is outside the analysis range, dilute the gas (SOP G11).

9.6.3. Click “Collect” button on the Quant Pad button panel.

9.6.3.1. Set the number of scans for both background and sample at 64.

9.6.3.2. Click the “More” button at the bottom of the above dialog box to check the settings of the data collection parameters.

9.6.3.2.1. Make sure that Resolution, Apodization, Zero Filling, Final Format, and File Handling parameters are set properly according to Table 1.

9.6.3.2.2. If a new background is needed before taking the sample spectra (e.g., when the sample is going to be taken more than 4 h after the last background spectrum is taken), make sure the background setting is placed at the third option (“collect background after x minutes”).

9.6.3.2.3. Otherwise, make sure the background setting is placed at the fourth option (“use specified background file”), and that the latest background file is loaded.

9.6.3.2.4. Click “OK” when finished setting the collection parameters.

9.6.4. Align the optical bench by clicking “Align” in the Quant Pad button panel.

9.7. Perform FTIR gas absorption cell leak-check. Follow the procedures described in Section 11.8.

9.8. Collecting a background spectrum

9.8.1. Check the settings of the data collection parameters by clicking the “Collection” button on the Quant Pad button panel. Make sure the background setting is placed at “collect background after xx minutes”.

9.8.2. Flush the FTIR chamber with pure nitrogen, delivered directly from gas cylinders, for at least 10 min. Control delivery rate at 5-10 L/min. Continue to deliver nitrogen while the background spectrum is being collected.

9.8.3. Click the “Background” button on the Quant Pad button panel.

9.8.4. Click “OK”.

9.8.5. Wait until the specified number of scans has been collected. A typical background spectrum is depicted in Fig. 1.

9.8.6. Click “Yes” to add the background spectrum to the indicated spectral window.

9.8.7. Use the “Save As” command in the OMNIC File menu to save the background spectrum on the hard disk.

9.8.8. A new background should be taken at least once at the beginning of the day’s work, or preferably every 4 h during continuous scanning.

9.9. Collecting and analyzing a sample spectrum

- 9.9.1. Check the settings of the data collection parameters by clicking the “Collection” button on the on the Quant Pad button panel. Change the background setting to “use specified background file” option, and load the file where the most recent background spectrum is saved. When collecting sample spectra, use the same parameter settings that were used to collect the standard spectra (Table 1). Collect the sample and standard spectra at the same pressure and approximately the same temperature.
- 9.9.2. Introduce sample
  - 9.9.2.1. Batch Sampling (offsite cylinder): Load bag sample into FTIR chamber using vacuum/fill step.
    - 9.9.2.1.1. Purge the FTIR gas cell with nitrogen at 5-10 L/min for at least 10 min.
    - 9.9.2.1.2. Close inlet valve on FTIR.
    - 9.9.2.1.3. Attach vacuum pump to the outlet tubing connected to the FTIR.
    - 9.9.2.1.4. Turn on the vacuum pump, open outlet valve on FTIR, and wait until the pressure drops to stable level (usually around 80-200 torr).
    - 9.9.2.1.5. Close the outlet valve. Turn off vacuum pump.
    - 9.9.2.1.6. Attach sample bag to the inlet tubing. Open the bag valve. Minimize the length of inlet tubing to reduce errors in gas concentrations.
    - 9.9.2.1.7. Open the FTIR inlet valve partially and let the bag sample deliver to the FTIR gas cell, close the inlet valve until the pressure reading stabilizes to ambient pressure. Close the bag valve.
    - 9.9.2.1.8. Repeat Steps 9.9.2.1.3 to 9.9.2.1.7 to deliver and purge the sample 3 times. Use the third-time sample as the representative sample delivered from the bag. Wait for at least 2 min for stabilization of gas delivered to the FTIR gas cell before taking the sample spectrum.
    - 9.9.2.1.9. If the gas concentration from the gas cylinder is greater than the maximum analytical range of the current method, then a new method has to be established by incorporating a standard spectrum with a certified gas concentration greater than the gas cylinder concentration.
  - 9.9.2.2. Continuous Sampling (onsite cylinder): Load sample directly from cylinder into FTIR.
    - 9.9.2.2.1. Follow the procedures described in SOP G2 (Standard Operating Procedure for Compressed Gas Cylinders) to deliver gas directly from the gas cylinder.
    - 9.9.2.2.2. If the gas concentration from the gas cylinder is greater than the maximum analytical range of the method, use Environics (See SOP G11) or TEI 146 diluter (See SOP C3) to dilute the gas concentration to below the maximum concentration level that the method can analyze accurately before taking the spectrum.
    - 9.9.2.2.3. Flush the FTIR gas cell with nitrogen beforehand. Vacuum the FTIR gas cell before delivering standard gas to reduce the time before a stable concentration can be reached. Deliver the sample gas at a flow

rate of 5 L/min for at least 10 min to ensure delivery of a representative sample of the gas to be checked.

- 9.9.3. Click “Sample” button on the Quant Pad button panel. Scanning of the sample will begin immediately. The final spectrum displays when the specified number of scans have been collected.
- 9.9.4. Click “Save As” in the OMNIC File menu to save the displayed sample spectrum.
- 9.9.5. Click the “Quantify” button on the Quant Pad button panel. The Quantify window shows the title of the selected quantitative analysis method and the path length, temperature and pressure values it used to calculate the results, followed by the concentration value and confidence range for each component.
- 9.9.6. To analyze a spectrum already saved on the disk, click the “Open” button on the Quant Pad button panel. Click the “Quantify” button on the Quant Pad button panel to automatically display the gas sample concentration.
- 9.9.7. To save the concentration data in a file on the disk, click the “Save” button in the “Quantify” window. The concentration data will be saved in a file named with the current base name and the extension “CNC”.

9.10. Calculating the gas cylinder concentration analyzed by the FTIR

- 9.10.1. Following procedures described in Section 9.9, collect spectra for samples from at least three sample bags filled with the specified gas to be checked, if the gas cylinder is offsite. If the sample is continuously delivered directly from a gas cylinder, collect at least three spectra, at an interval of every 2-3 min.
- 9.10.2. Average the concentration data. Apply dilution factor if diluted sample is used. Compare the averaged value with the specified span gas concentration. The accuracy of the gas cylinder is confirmed if the average measured concentration is within 5% of the specified span gas concentration.

9.11. To make a Single Gas Method:

- 9.11.1. Collect calibration standard, a known concentration sample spectrum, using Quant Pad (select any method file) following the procedures described in Steps 9.2 to 9.9. Examples of standard spectra for representative compounds are displayed in Fig. 2.
- 9.11.2. Click the “Save as Standard” button on the Quant Pad button panel. Click “OK” after entering the spectrum information displayed on the window.
- 9.11.3. Enter a file name for this standard in the “File Name” box and select the drive and directory where the standards should be stored.
- 9.11.4. Open Quant Setup.
- 9.11.5. Choose “New Method” from the “File” menu
  - 9.11.5.1. Select your sample phase as gas and concentration units as ppm.
  - 9.11.5.2. Click “OK”.
- 9.11.6. Choose “Components” from the “Edit” Menu
  - 9.11.6.1. Type in the component name.
  - 9.11.6.2. Type an abbreviated name in the component code box.
  - 9.11.6.3. Click “OK”.

- 9.11.7. Choose “Standards” from the Edit Menu
  - 9.11.7.1. Click “Browse”.
  - 9.11.7.2. Find the file of the standard spectrum and click “OK”.
  - 9.11.7.3. Once the file is specified to be used in a method, it can not be moved out of its specified directory or the method will fail. If you choose to move the file, the method must be modified to match.
  - 9.11.7.4. Check that the concentration, temperature, and pressure are correct for your standard.
  - 9.11.7.5. Click “OK”.
- 9.11.8. Choose “Regions/Windows” from the Edit Menu
  - 9.11.8.1. Carefully study the calibration spectra and choose frequency ranges in which the component peak are clearly identifiable and can be used to quantify the component in a method. Avoid using regions which show high absorption for water vapor or CO_2. Do not choose a frequency range in which the absorbance units are greater than 1 for the component to be quantified. The recommended regions for certain representative compounds are listed in Table 2.
  - 9.11.8.2. Click the “Region Add” button to add regions.
  - 9.11.8.3. If you want Quant Setup to use only certain windows in this analysis region to calculate component concentrations, click in the “Window” box and then click the “Window Add” button to add windows.
  - 9.11.8.4. Repeat the above two steps until all regions and windows have been specified.
- 9.11.9. Choose “Calculations” from the Edit menu
  - 9.11.9.1. Type an “S” for the region of the spectrum that you wish to use to calculate the concentration of this component. For the other regions, type “I”.
  - 9.11.9.2. Click “OK”.
- 9.11.10. Choose “Save Method As” from the File Menu
  - 9.11.10.1. Enter a title for the method.
  - 9.11.10.2. Click “OK”.

9.12. Validating the Method:

- 9.12.1. Once Quant Pad is running, select any method and collect at least one sample spectrum from a sample containing a known concentration of the component, using Quant Pad and the method described above. The concentration should be about one-tenth of the concentration of the corresponding standard used to make the above method.
- 9.12.2. Save spectrum as a standard with Quant Pad.
- 9.12.3. Create a validation sample set
  - 9.12.3.1. From Quant Setup, choose “Sample Sets” from the Analyze menu.
  - 9.12.3.2. Click “New”.
  - 9.12.3.3. Click “Add” button.
  - 9.12.3.4. Select file names of spectra you wish to add.
    - 9.12.3.4.1. To change the information associated with the file, click the file name, then click in the appropriate text box and enter the correct value.

      - 9.12.3.4.2. To display a spectrum, click “View Spectra” in the Samples dialog box.
    - 9.12.3.5. Click “OK”.
    - 9.12.3.6. Click “Save Set”.
    - 9.12.3.7. Enter a unique title for the sample set.
    - 9.12.3.8. Click “New” to add this sample set to the list of sample sets.
    - 9.12.3.9. Click “OK” to close the Samples dialog box.
- 9.12.4. Quantify the validation sample set
  - 9.12.4.1. Be sure the correct method is open.
  - 9.12.4.2. Choose “Quantify” from the Analyze menu.
    - 9.12.4.2.1. This will open a dialog box containing information about the sample set based on the method selected.
    - 9.12.4.2.2. The calculated component concentrations will be displayed.
    - 9.12.4.2.3. Click the next button to see results for the next sample in the set.
  - 9.12.4.3. When finished click “OK”.
  - 9.12.4.4. If the calculated component concentrations differ from the actual concentrations by more than an acceptable amount (usually 1 to 5%, depending on the application), and if the errors tend to change regularly as a function of concentration, the absorbance vs. concentration plot for that component may be curved instead of linear. In this case, follow the instructions below to calculate a correction curve for that component.
- 9.12.5. Calculate a correction curve:
  - 9.12.5.1. Use Quant Pad Application to collect spectra from different known concentrations (preferably 4 to 6 different concentration levels) and save as standards using Quant Pad.
  - 9.12.5.2. Open “Quant Setup” and click “Open Method” from the File menu.
  - 9.12.5.3. Select the title of the method to be corrected in the drop down list box and choose “OK”.
  - 9.12.5.4. Create the calibration sample set:
    - 9.12.5.4.1. Choose “Sample Sets” from the analyze menu.
    - 9.12.5.4.2. Click “New”.
    - 9.12.5.4.3. Click the “Add” button.
    - 9.12.5.4.4. Select file names of the spectra you want to add.
    - 9.12.5.4.5. Check to be sure information for each spectrum is correct.
    - 9.12.5.4.6. Click “OK”.
    - 9.12.5.4.7. Click “Save Set”.
    - 9.12.5.4.8. Enter a unique title for your new sample set.
    - 9.12.5.4.9. Click “New” to add this sample set to the list of sample sets.
    - 9.12.5.4.10. Click “OK” to close the Sample dialog box. This set automatically becomes the active sample set.
- 9.12.6. Calculate the correction curve coefficients and save the calibration sample set.
  - 9.12.6.1. Click “Calibration” from analyze menu.
  - 9.12.6.2. Select the component you want to correct by clicking its name in the “Component” list.

- 9.12.6.3. Press the correction button in the calibration dialog box to analyze the calibration sample set using the primary standard for the selected component.
  - 9.12.6.3.1. To force the curve through zero, turn on the "Force Zero Intercept" Option. The accuracy of the method can be improved by forcing the correction curve through zero if the concentration of the analyte in the samples may approach zero. Do not force the curve through zero if the lowest expected concentration in the samples is higher than the lowest standard in the calibration set, especially when all of the spectra in the calibration set are taken at high concentration range.
- 9.12.6.4. Calculate the correction curve coefficient.
  - 9.12.6.4.1. Click on the "Calculate" button in the calibration dialog box to calculate the correction curve coefficients.
  - 9.12.6.4.2. To exclude a selected calibration spectrum from the curve calculation (in case the spectrum is suspected to be an outlier or out of the desired calibration concentration range), select its file name and then turn off the "Include This Sample" option. If necessary, re-run a standard spectrum at such concentration to guarantee the accuracy of the standard spectrum concentration, and reintroduce the new spectrum into the calibration sample set.
  - 9.12.6.4.3. Click "Display" in the Correction Curve Selection dialog to see a graph of the actual vs. measured values.
  - 9.12.6.4.4. Click "Results" in the Correction Curve Selection dialog to see detailed information about the fit of the selected polynomial function to the spectral data.
  - 9.12.6.4.5. Click the up or down arrow button next to the selected order to examine a higher- or lower-order function.
  - 9.12.6.4.6. Select the lowest curve order that fits the actual concentration to the calculated concentrations with the required degree of accuracy specified in the QAPP.
- 9.12.6.5. Transfer curve coefficients to the current method.
  - 9.12.6.5.1. Display the Correction Curve Selection dialog box and make sure the correct polynomial order is selected.
  - 9.12.6.5.2. Click the "Use" button. This transfers the current set of coefficients into the current method for use in correcting the concentrations of the selected component.
- 9.12.6.6. Save the Calibration sample set.
  - 9.12.6.6.1. Press the "Return" button in the Correction Curve Selection dialog box. Calibration Data dialog box appears on screen.
  - 9.12.6.6.2. Press "Save Set", then either click "Replace" or enter new title and click "New".
  - 9.12.6.6.3. Press "Return" to get to Quant Setup menu.

9.12.7. Save the corrected method by clicking "Save Method" from File menu, or clicking "Save Method As" and enter a title for the method, and then choose "OK".

9.12.8. To run the corrected method, open Quant Pad and select the method. Then either open, quantify, or collect and quantify a spectrum.

9.13. Troubleshooting

9.13.1. Troubleshooting measures for software, hardware, and application problems are listed in the last three chapters of the ThermoNicolet Nexus 670 User's Guide.

9.13.2. If the above action does not solve the problem, call Thermo Nicolet Service office at 1-800-642-6538, 608-276-6100, or email them at techinfo@thermonicolet.com.

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a record book designated for the method, which should contain copies of the electronic record.

10.2. Manage all data according to SOP B5.

10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

10.4. Maintain gas certification records.

10.5. Store the quantitative analysis method and spectra (standard, background, and sample spectra) on a computer disk. Store a second backup copy on a separate disk.

11. Quality Control and Quality Assurance

11.1. The accuracy of any FTIR quantitative analysis is limited by the accuracy of the concentrations of the gas standards used in establishing the calibration method. Preferably the calibration gas should be certified using EPA protocol (1% certification).

11.2. The calibration spectra must be of pure components, or pure components diluted with a solvent or gas that either doesn't absorb infrared energy or absorbs in a different region of the infrared spectrum.

11.3. Check the FTIR calibration on a monthly basis by comparing the concentrations of the spectra taken from a reference span gas cylinder with its specified gas cylinder concentration that is guaranteed of accuracy. Recalibrate the instrument if the average measured concentration deviates more than 5% from the specified span gas concentration.

11.4. New background spectra should be collected regularly, preferably once every 4 h during the operation of the FTIR.

11.5. Align the spectrometer daily for optimum performance.

11.6. Keep the room temperature where the FTIR is installed at temperatures between 16°C and 27°C.

11.7. Verify that the detector is cooled by liquid nitrogen before taking any spectrum.

11.8. Perform FTIR gas absorption cell leak-check before taking sample spectra. Leak check the FTIR cell under both vacuum and pressurized (greater than ambient) conditions.

- 11.8.1. For the batch sampling technique, close the inlet valve to the FTIR cell, open the outlet valve and connect to a vacuum pump. Evacuate the absorption cell to the minimum absolute pressure P_{min}. Close the outlet valve, and determine the change in pressure P_v after 2 min.
- 11.8.2. For both the batch sampling and continuous purging techniques, pressurize the system to about 100 mmHg above atmospheric pressure. Determine the change in pressure P_p after 2 min.
- 11.8.3. Determine the percent leak volume $\%V_L$, calculated as P_{max} divided by atmospheric pressure and then multiplied by 100%, where P_{max} is the larger of P_v or P_p.
- 11.8.4. Leak volumes in excess of 4% of the FTIR system volume are unacceptable, and the tubing connection should be re-checked.

11.9. Prior to use of sample bags for storage of gas samples for subsequent FTIR analysis, perform tests to evaluate sample recoveries and/or contamination from sample bags under controlled conditions. This could be accomplished by comparing FTIR responses to gases in the purge mode and in the batch mode simulating bag materials (walls and fittings), storage times and storage temperatures. Choose the type of sample bag that has the least amount of gas loss and contamination.

12. References

12.1. EPA Test Method 320. Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy. www.midac.com/apnotes/Ap-139.PDF
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12.5. Nicolet Instrument Corporation. 1995.QuantPad User’s Guide. Madison, WI.
12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
12.7. SOP G11. 2006. Operation of the Environics® Computerized Gas Dilution System. Standard Operating Procedure G11. Purdue Ag Air Quality Lab.
12.8. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.

13. Contact Information

13.1. Technical Support: 1-800-Nicolet (1-800-642-6538), 1-608-276-6373.
13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, http://www.AgAirQuality.com.

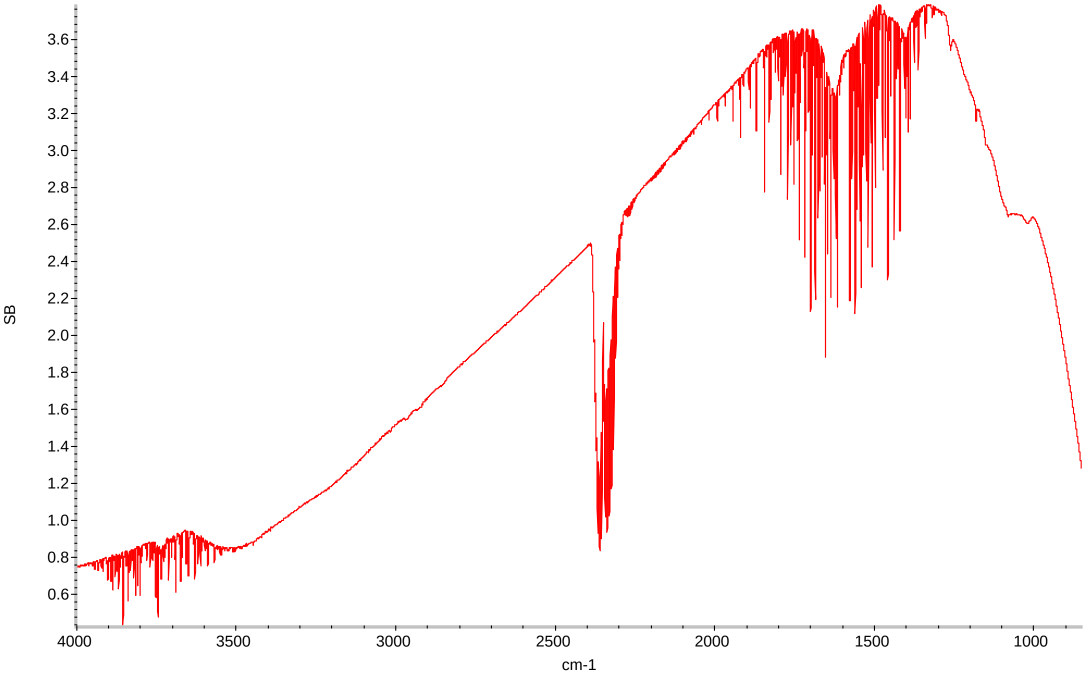

Figure 1. Example of a background spectrum.

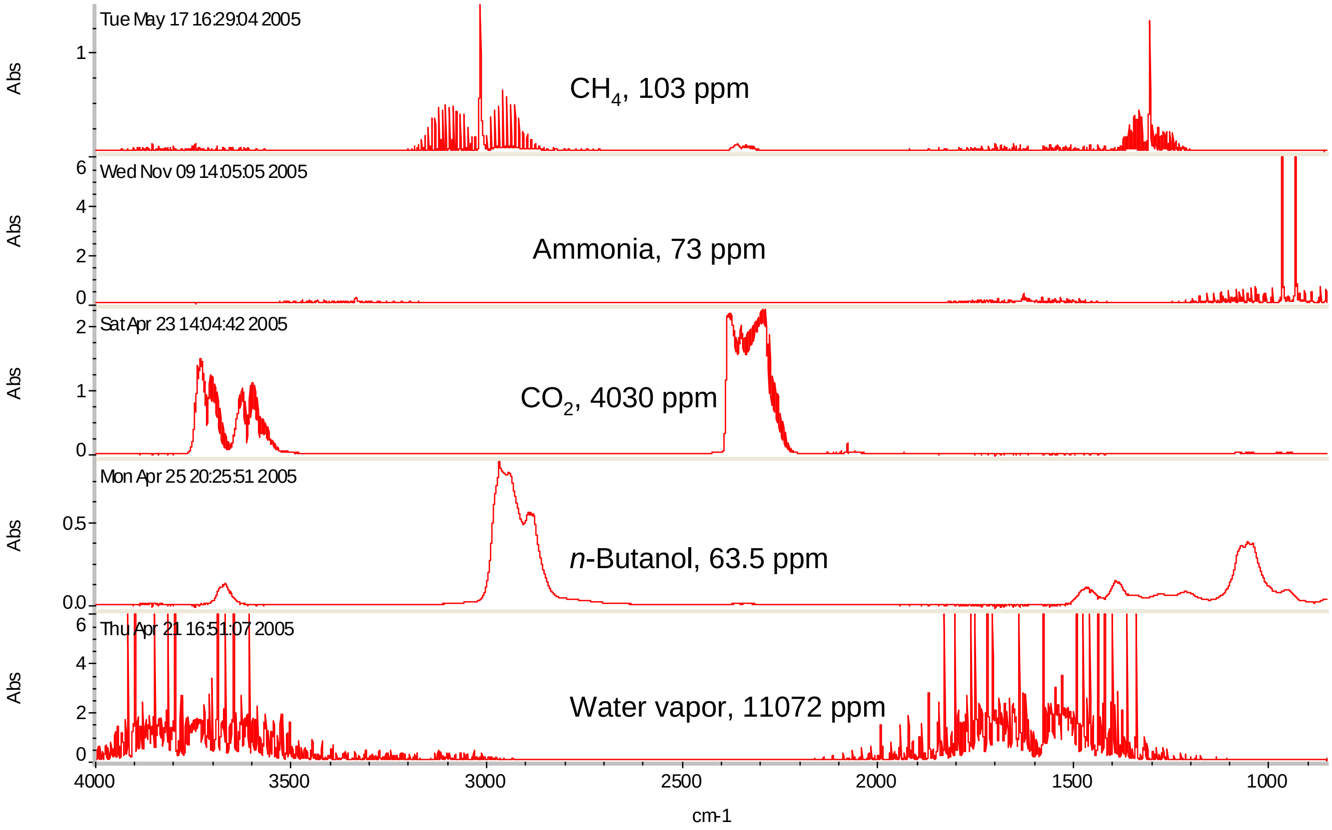

Figure 2. Standard spectra for representative compounds.

Table 1. Data collection and optical bench setup parameter for standard and sample spectra collection.

Parameter	Setting
Number of sample scans	64
Number of background scans	64
Resolution	1
Data spacing	0.482 cm-1
Spectral range	4000-850 cm-1
Mirror velocity	1.8988
Gain	1
Aperture	34
Level of zero filling	0
Apodization	Happ-Genzel
Phase correction	Mertz

Table 2. Preferred frequency ranges to be used as calculation regions for making the analysis methods for each compound.

Compound Name	Frequency Ranges (cm^{-1})
Carbon dioxide	2075.7-2080
Water vapor	3372.7-3405
Methane	3008.2-3019.6
n-Butanol	2809-3019
Ammonia	961.3-969

MEASUREMENT OF LAGOON pH WITH THE INNOVATIVE SENSORS MODEL CSIM11-pH SENSOR
Standard Operating Procedure (SOP) L1

MEASUREMENT OF LAGOON pH WITH THE INNOVATIVE SENSORS MODEL CSIM11-pH SENSOR

Standard Operating Procedure (SOP) L1

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1. Scope and Applicability

1.1. A range of pH-dependent chemical and biological interactions and processes can affect the odor of, and overall chemical emissions from, manure stored in a lagoon.
  1.1.1. Emissions of ammonia (NH_3) increase as pH increases.
  1.1.2. Emissions of hydrogen sulfide (H_2S) decrease as pH increases.
1.2. Ion-specific probes with built-in preamplifiers are useful for accurate pH measurements.
  1.2.1. The pH measurement range of these probes is from 0 to 14.
  1.2.2. The pH in anaerobic lagoons typically ranges from 6.8 to 8.3.
1.3. This SOP applies only to the Campbell Scientific Model CSIM11 pH probe.

2. Summary of Method

The activity of hydrogen ion (H^+) in solution is measured as pH using an ion-selective probe with built-in preamplifier (Innovative Sensors Model 11 Process pH sensor purchased as Model CSIM11, Campbell Scientific). A porous Teflon liquid junction resists fouling while the probe is deployed in the lagoon, and a double-junction reference cell increases the service life of the probe. The sensor voltage output is proportional to pH. The probe will be 0.3 m below the lagoon surface and 9 m away from the lagoon berm. The probe will be connected to a styrofoam float, held in place with two nylon ropes and a ½” PVC raceway, which keeps the float in place and protects its lead cable. The raceway will pivot off two steel posts that support a weatherproof NEMA box, which will house the data logger.

3. Definitions

3.1.	Ag	Silver
3.2.	AgCl	Silver chloride
3.3.	APHA	American Public Health Association
3.4.	BNC	Bayonet Neill Concelman Connector
3.5.	CS	Campbell Scientific, Inc
3.6.	H^+	Hydrogen ion
3.7.	KHP	Potassium biphthalate
3.8.	QAPP	Quality Assurance Project Plan
3.9.	QCCS	Quality Control Check Sample
3.10.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.11.	PAML	Purdue Applied Meteorology Laboratory
3.12.	pH	Hydrogen activity, defined as $-log[H^+]$
3.13.	PVC	Polyvinylchloride
3.14.	NEMA	National Electrical Manufacturers Association
3.15.	SOP	Standard operating procedure

4. Health and Safety

4.1. Be careful when working with solutions. Buffers may cause eye and skin irritations and may be harmful if inhaled or swallowed. Maintain proper lab procedures when filling electrodes or calibrating. Wear eye protection, as eye damage may result from splash.
4.2. Wear gloves and face shields when handling manure (including lagoon) samples.

5. Cautions

5.1. Be careful when connecting the sensor cable to the datalogger, being sure that the connections are tight and are made according to SOP W6.
5.2. Avoid crimping electrical cables and connectors.
5.3. Stray sources of ions such as tap water, soil, dust etc. can contaminate the reference solution. Maintain good lab cleanliness when refilling probe.
5.4. Take proper care to avoid damaging the probe's glass reservoir
  5.4.1. Never wipe the pH bulb, since dust may scratch the delicate gel layer.
  5.4.2. Do not drop the probe.

6. Interferences

6.1. The depth of measurement must be maintained at 0.3 m.
6.2. Algae and other biological organisms will foul the probe, and/or physically block porous liquid junctions.

7. Personnel Qualifications

7.1. Each field staff member must read and understand the sensor instruction manual, the reference pH sensor manual, and this SOP before operating this instrumentation.
7.2. Personnel must be trained in the use of the handheld multimeter before initiating the procedure. Training requires 10 min.

8. Equipment and Supplies

8.1. #2 common head screwdriver
8.2. 1/8″ slotted screwdriver
8.3. Low-temperature oven (capable of temperatures to at least 125^{o}C)
8.4. Deionized water
8.5. Mild (dish) soap for cleaning probe
8.6. Protective gloves and face shields
8.7. Stands and clamps for sensor and pH reference electrodes
8.8. Safety glasses
8.9. Teflon pipe tape
8.10. NIST- traceable pH buffers
  8.10.1. 4.01 pH (potassium biphthalate)
  8.10.2. 7.00 pH (potassium phosphate)

- 8.10.3. 10.00 pH (sodium borate/sodium carbonate)
- 8.11. Potassium biphthalate (KHP)
- 8.12. Thymol ($C_{10}H_{13}OH$)
- 8.13. Non-mercury thermometer
- 8.14. 50-mL beakers
- 8.15. High-input impedance multimeter.
- 8.16. Standard glass-body reference electrode (Ag/AgCl) with porous liquid junction and
  - 8.16.1. BNC connector
- 8.17. 5-VDC power supply for preamplifier
- 8.18. Analytical balance (200 g range , 0.01 g resolution)
- 8.19. pH preamplifier kit for BNC connector-equipped electrodes, with leads to connect to
  - 8.19.1. multimeter
- 8.20. pH probe. Model CSIM11, Campbell Scientific, North Logan, UT
- 8.21. Measuring tape
- 8.22. Reference solution
- 8.23. Drying oven

9. Procedures

- 9.1. Probe preparation
  - 9.1.1. Electrodes are shipped with a wetting cap covering the measuring end. The cap contains a pH 4 buffer, which is saturated with potassium chloride (KCl) for single-junction combination electrodes and potassium nitrate (KNO_3) for double-junction electrodes.
    - 9.1.1.1. Gently remove wetting cap by unscrewing the bottle from the cap and then sliding the cap and o-ring off the electrode body. Save the cap for storage.
    - 9.1.1.2. During shipping or transit, some air may have moved into the bulb from inside the probe. Grasp the electrode near the cable and gently swing it through an arc to force the internal electrolyte into the pH bulb. Visually inspect to ensure that the internal surface of the porous junctions in the electrodes is in contact with internal reference solutions.
    - 9.1.1.3. Rinse the electrode with distilled water.
- 9.2. Acceptance
  - 9.2.1. Physically inspect pH probe, referring to the figure in Appendix D. Verify that the glass sensing bulb is intact and not scratched. Verify that the porous junction is not visibly leaking, and that the area around the porous junction is not cracked or otherwise damaged. Reject the probe if it fails any of these tests. Record assessment on Acceptance Log (Appendix B).
  - 9.2.2. Prepare the pH Quality Control Check Sample (QCCS) solution inside the trailer.
    - 9.2.2.1. Dry 10.5 g of KHP for 2 h at 110 °C. Dissolve 10.21 g of dried KHP in 1 L distilled/deionized water. Add a single thymol crystal per L for preservation. The pH of this solution is 4±0.05 pH units .
  - 9.2.3. Connect the green lead from pH probe to one terminal of the high-input impedance multimeter. Connect the clear-cased reference lead from preamplifier kit for standard glass-bodied reference electrode to other terminal of multimeter. Connect black

ground lead from preamplifier (attached to the standard glass-bodied reference electrode) to the ground (brown) wire of the pH probe (Fig. 1).

9.2.4. Connect the three-wire preamplifier connector to the 5-VDC power supply. Connector cable has red to positive, black to negative, green to ground (Fig. 1).

9.2.4.1. Conduct this procedure inside trailer, where the temperature is maintained at 21˚ ±3˚C (SOP U4).

9.2.4.2. Place both pH probe and glass-bodied reference electrode into fresh QCCS solutions and measure voltage difference between the reference electrodes. Ideally, the measured voltage should be 0 ± 4 mV for two Ag/AgCl reference electrodes; however, internal construction differences, as well as liquid junction potentials, may result in a non-zero reading.

9.2.4.3. Record the voltage reading (millivolts) on the Acceptance Log (Appendix B) for future reference in assessing pH probe performance.

9.2.4.3.1. If the voltage reading is unstable after one min (±1 mV), the porous junction on the probe is likely either leaking or partially obstructed. According to the pH probe manual change internal reference solutions for the pH probe, and repeat measurements of the pH probe with standard glass-bodied reference electrode.

9.2.4.3.2. If the reading is still unstable, follow the procedure for cleaning clogged liquid junctions outlined in the manual (Section A.1.6 “Electrode Cleaning”), and repeat measurements of the pH probe with standard glass-bodied reference electrode.

9.2.4.3.3. If the readings are still unstable, reject pH probe and replace with a new one.

9.2.5. Calibrate the probe (Section 9.4).

9.2.6. Probe stability

9.2.6.1. Conduct procedure inside trailer, where the temperature will be maintained at 25˚ ±3˚C (SOP U4).

9.2.6.2. Place calibrated probe in fresh QCCS solution. Using Table 1 record its pH readings after 30 s, and after 2 min on the Acceptance Log (Appendix B). Both readings should agree within ±0.05 pH units or ±3mV.

9.2.6.2.1. If the 30-s reading is not 4.00±0.05 pH units, or if the 30-s and 2-min readings do not agree within ± 0.05 units, this may also indicate problems with the probe.

9.2.6.3. Check probe response with fresh QCCS solution. If the readings are still outside the acceptable range, clean the electrode, check for leakage at the junction and/or damage to the glass bulb. If neither is found, repeat Step 9.2.3. Retest with fresh QCCS solution; if the probe is still not functioning properly, replace it.

9.3. Installation (SOP U5, Section 9.6.7)

9.3.1. Locate the position on the lagoon corner (away from inlet) in which the sensor should be placed. Refer to the QAPP and/or Site Monitoring Plan.

9.3.2. Position the probe so that the sensor will be 0.3 m below the waterline of the float, and install all sensors in tri-sensor float.

9.3.3. Connect center of nylon guide rope to tri-sensor float and lift using rope. Rope has markers to indicate position to ensure the float will be 3 m from the edges of the lagoon in the corner.

- 9.3.4. Connect the probe cable from NEMA box to the indicated hole in the lagoon probe float.
- 9.3.5. Verify the waterline of the float matches to the 0.3 m mark on the Styrofoam float.
- 9.3.6. Attach wires to datalogger in NEMA box using the ⅛″ slotted screwdriver. Connect brown wire to ground and red and green wires to the differential-ended analog channel

9.4. Calibration (Beginning/end of measurement period or 20d, which ever comes first)

- 9.4.1. Conduct procedure inside trailer, where temperature will be maintained at 25˚±3˚C (SOP U4).
- 9.4.2. Based on APHA Standard Method 2580
- 9.4.3. Probes and buffers should be at temperature equilibrium before calibration.
- 9.4.4. Measure and record temperature from bulb thermometer on the Quality Assurance Log (Appendix C).
  - 9.4.4.1. Calculate the Nernst temperature compensation (-0.2 pH/mV per ºC).
- 9.4.5. Connect red wire to positive post and green wire to negative post of multimeter.
  - 9.4.5.1. Set multimeter to mV readings.
- 9.4.6. Rinse probe three times in distilled water
- 9.4.7. Place probe in pH 7.0 buffer
  - 9.4.7.1. Record buffer pH, temperature compensation of probe, expected buffer mV (from Table 1), and measured mV on the Quality Assurance Log (Appendix C).
    - 9.4.7.1.1. If the measured value, corrected for temperature, differs from the expected value by ±0.3 pH units (17.7 mV) or more, repeat Sections 9.4.5 and 9.4.6
      - 9.4.7.1.1.1. If the error is still ≥ ±0.3 pH units (17.7 mV), contact vendor for replace/repair decision (Section 13.2).
- 9.4.8. Repeat Sections 9.4.6 and 9.4.7 using pH 4.01 buffer.
- 9.4.9. Repeat Sections 9.4.6 and 9.4.7 using pH 10.0 buffer.

9.5. Troubleshooting

- 9.5.1. If the probe reads pH 14 or higher:
  - 9.5.1.1. Check for damaged cable. If damaged, replace or repair.
  - 9.5.1.2. Check for loose connections at datalogger. Tighten if necessary.
  - 9.5.1.3. Check for coating on bulb. If needed, clean according to Appendix A (Page A-3) of the probe manual.
  - 9.5.1.4. Check for clogging of reference probe. If needed, clean according to manual. Check performance as specified in Section 9.6.
  - 9.5.1.5. Check that bulb is full of solution. If not, gently swing it through an arc to force the internal electrolyte into the pH bulb. If still low, follow instructions in the probe manual (Pages 4-5) to refill.
- 9.5.2. Noisy or erratic readings
  - 9.5.2.1. Check for coating or clogging of glass or reference electrode. Clean as needed according to Appendix A (Page A-3) of the probe manual. Check performance as specified in Section 9.2.3.
- 9.5.3. No response to changes in pH
  - 9.5.3.1. If electrode reads between 5.8 and 6.2 in all pH solutions, visually inspect bulb for cracks. Replace probe if cracks are found.

9.5.3.2. If pH electrode reads pH 7 (or 0.0 mV) in all pH solutions, check cable for damage that might cause a short circuit. If damaged, replace and send for repair.

9.6. Conduct performance assessment and preventive maintenance at the beginning and end of each measurement period.

9.6.1. Remove pH probe from float and visibly inspect glass sensing bulb and liquid junction for damage or blockage. Record assessment on the Inspection Log Form (Appendix A).

9.6.1.1. If damaged, replace sensor and send damaged sensor to manufacturer for repair.

9.6.2. Rinse pH probe with copious amounts of distilled water to remove lagoon effluent.

9.6.2.1. Calibrate sensor (Section 9.4), indicating beginning or end of the measurement period on Quality Assurance Log (Appendix C).

9.6.2.2. If the temperature-corrected measured pH is within ±0.3 pH units (17.7 mV) of the buffer solution pH, the probe function is considered acceptable.

9.6.2.3. If the temperature-corrected measured pH differs from the buffer solution pH by > 0.3 pH units, the probe function has degraded. If this occurs at the beginning of a measurement period, do not use the probe, but replace it with a new one. If it occurs at the end of the measurement period, flag or correct the data collected during the period as follows:

9.6.2.3.1. If the temperature-corrected measured pH differs from the buffer solution pH by ≥ 0.5 pH units, flag the measurements during the period as questionable.

9.6.2.3.2. If the temperature-corrected measured pH differs from the buffer solution pH by < 0.5 pH units, age-correct the measurements made during the measurement period, based on a linear interpolation of the difference in measured and theoretical values across the time of the measurement period:

$$\text{Corrected pH measurement} = \text{pH measurement} + TM * (pHF - pHI)/(TP)$$

Where:
TM = Elapsed time into the measurement period when pH reading was taken, h
pHF = Probe pH reading at the end of the measurement period
pHI = Probe pH reading at the beginning of the measurement period
TP = Total length of the measurement period, h

9.6.3. Clean pH probe and sensor cable with mild soap and water, following Appendix A of the probe manual.

9.6.4. Inspect cable for damage and record assessment on the Inspection Log (Appendix A).

9.6.4.1. Repair or replace as necessary to maintain cable integrity and wire continuity.

9.6.5. Repeat Section 9.2.

9.6.5.1. If sensor meets acceptance criteria, store according to manufacturer’s guidelines in preparation for future use.

9.6.5.2. If sensor fails acceptance criteria, replace sensor and send to supplier for repair.

9.7. Data acquisition, calculations & data reduction requirements are given in the probe manual (Pages 3-4 and Appendix B)

9.7.1. Sensor output (-0.413 to 0.413 VDC) is monitored by the data logger.

9.7.1.1. Sensor response is 59 mV per pH unit at 25oC, with pH 7 equal to 0 mV.

9.7.2. Measurements will be recorded by the data logger every 5 min.

9.7.2.1. Temperature correction of pH measurement (Page 3 of probe manual) is:
1/(TempC+273)/298 * 59)
where TempC is the measured lagoon temperature (SOP L2) in oC.

9.7.2.2. Temperature correction is made in data logger, using the program described in SOP W6).

9.7.3. Report data to three significant digits

9.8. Computer hardware & software

9.8.1. Attach the sensors with electrical wires to a data logger (Section 9.3.5), which will extract data from the instruments and store it (SOP W6).

9.8.2. Data logger operates using on-board BASIC-like programming.

9.8.2.1. The general program for measurements is described in Appendix B of the probe manual

10. Data and Records Management

10.1. Maintain all lab records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for meteorological sensors.

10.2. Manage all data according to SOP D1

10.3. At the end of this SOP are forms to record the inspection and calibration information.

10.4. Document all data and information on field data sheets, sensor logs and in site logbooks with permanent ink, or in electronic notes.

10.5. Overstrike errors in writing on logs with a single line. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Check to be sure the probe is mounted 0.3 m below the waterline of the lagoon probe float.

11.2. Abrupt changes in the pH reading may indicate that the probe float has turned over. Daily checks of the data will provide a check against sudden changes that might indicate that the float has turned over or encountered other problems.

11.3. Replace reference solution in probe every six months (or as needed).

11.3.1. Clean the probe tip according to Appendix A of the probe manual.

11.3.2. Note how far the red refill plug is screwed into the probe body. Remove plug and place it where it will stay clean.

11.3.3. Rinse reservoir with deionized water repeatedly to remove old solution. Drain out all remaining water.

11.3.4. Completely fill the reservoir with the new reference solution. Make sure to keep the bottle tip clean, and replace bottle cap immediately after use.

11.3.5. Replace the red plug and apply new pipe tape. Make sure the plug is screwed back in as far as it was originally.

11.3.6. Because the reference solution is viscous, it may be necessary to invert the probe and wait for the reference solution to fill the glass bulb.

11.3.6.1. If necessary, take the probe in hand and GENTLY swing it in a downward arc to speed the flow of solution into the bulb.

11.4. Conduct the acceptance protocol (Section 9.2) and calibration (Section 9.4) on all new units, and at the beginning and end of each measurement period.

11.5. Conduct any preventative maintenance during the performance assessment (Section 9.6) at the beginning and end of each measurement period.

12. References

12.1. pH Probe Manual. 2003. Using CSIM11 pH and ORP Probes with Campbell Scientific Dataloggers, Campbell Scientific, Inc., North Logan, UT, 14 p.
12.2. APHA. 1995. Standard Methods for the Examination of Water & Wastewater, 19th Edition, Method 4500-H^+, A.D. Eaton, L.S. Clesceri, A.E. Greenberg (Eds.), AWWA, WEF.
12.3. Aneja, V.P.; B. Bunton, J.T. Walker, and B.P. Malik. 2001. Measurement and analysis of atmospheric ammonia emissions from anerobic lagoons. Atm. Env. 35:1949-1958.
12.4. Miller, W.J. 2003. Redox Chemistry of Six Anaerobic Swine Lagoons in Eastern North Carolina. MS Thesis, North Carolina State University, Raleigh, NC 167 p.
12.5. SOP D1. 2006. Management of Open-source, Weather and Lagoon Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
12.6. SOP L2. 2006. Measurement of Lagoon Temperature with the Campbell Scientific Model 107-L Thermistor. Standard Operating Procedure L2. Purdue Applied Meteorology Lab.
12.7. SOP U4. 2006. Open-source Instrument Trailer. Standard Operating Procedure U4. Purdue Applied Meteorology Lab.
12.8. SOP U5. 2006. Installation for Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.
12.9. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.
12.10. http://www.sensorex.com/support/education/pH_education.html

13. Contact Information

13.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, Utah, 84321
  13.1.1. Technical support: 435-753-2342
13.2. Manufacturer: Innovative Sensors, Inc, 4123 E. La Palma Ave #200, Anaheim, California 92807
  13.2.1. Telephone: 714-577-5600, 800-835-5474
  13.2.2. Fax: 714-577-5688

Figure 1. Quality Control Check Sample (QCCS) connections.

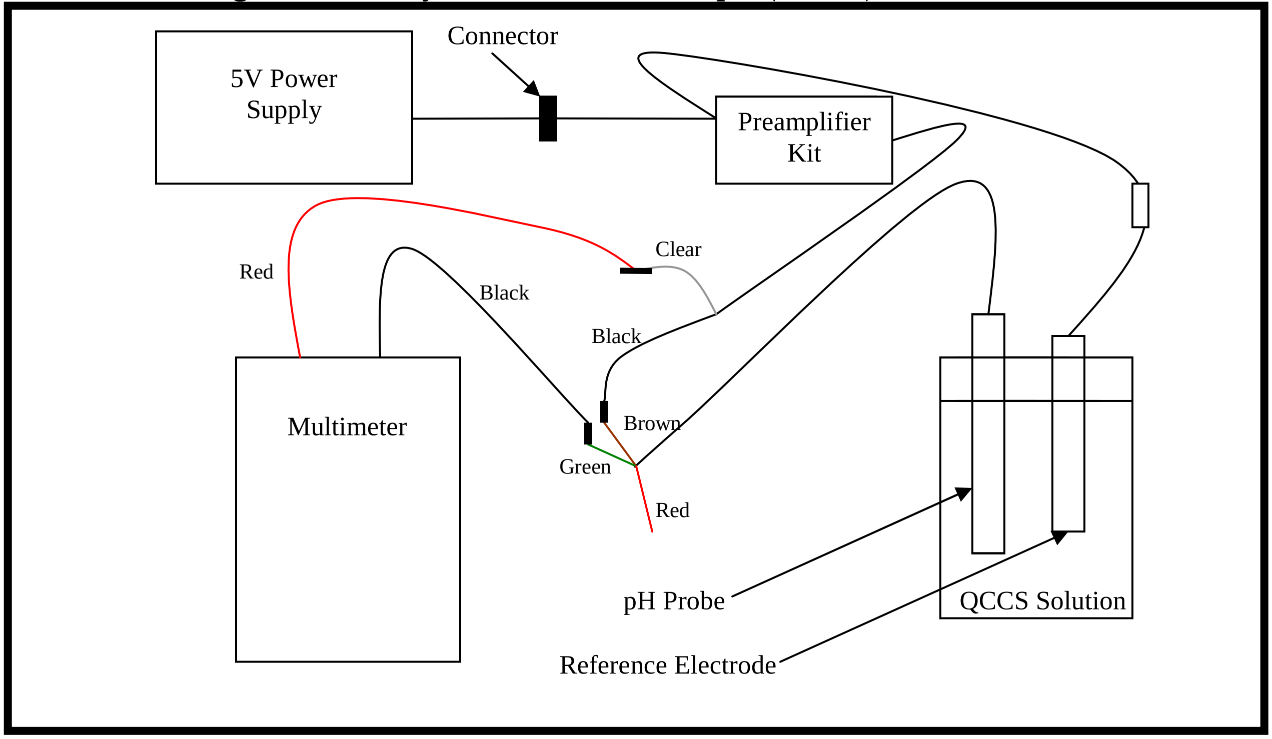


Table 1. pH units Vs mV

0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
+414	+355	+296	+237	+177	+118	+59	00	-59	-118	-177	-237	-296	-355	-414

Zero mV = pH 7
59.2mV per pH unit change
mV are + for pH < 7 and mV are – for pH >7

Table 2.pH vs Temperature Error Chart

	pH2	pH	pH	pH5	pH6	pH7	pH8	pH9	pH10	pH11	pH12
5º	0.30	0.24	0.18	0.12	0.06	0	0.06	0.12	0.18	0.24	0.30
15º	0.15	0.12	0.09	0.06	0.03	0	0.03	0.06	0.09	0.12	0.15
25º	0	0	0	0	0	0	0	0	0	0	0
35º	0.15	0.12	0.09	0.06	0.03	0	0.03	0.06	0.09	0.12	0.15
45º	0.30	0.24	0.18	0.12	0.06	0	0.06	0.12	0.18	0.24	0.30
55º	0.45	0.36	0.27	0.18	0.09	0	0.09	0.18	0.27	0.36	0.45
65º	0.60	0.48	0.36	0.24	0.12	0	0.12	0.24	0.36	0.48	0.60
75º	0.75	0.60	0.45	0.30	0.15	0	0.15	0.30	0.45	0.60	0.75
85º	0.90	0.72	0.54	0.36	0.18	0	0.18	0.36	0.54	0.72	0.90

Note: Values in light blue are less than 0.1 errors, and may not require temperature compensation. Values in gray are temperature/pH combinations at which there is no error in pH from temperature.

Appendix A. Inspection and Maintenance Log

INNOVATIVE SENSORS MODEL CSIM11 pH SENSOR

Serial Number _____________

Date (DMY)	**Condition**		**Action taken**	**Calibration done?**	**Acceptance done?**	**Person assessing**
	Glass electrode	**Cable**				

Appendix B. Acceptance Log

INNOVATIVE SENSORS MODEL CSIM11 pH SENSOR

Serial Number _____________

Date (DMY)	Condition		T (ºC)	QCCS reference check (mV)	Time to stable QCCS reading (min)[1]	pH reading in QCCS check[3]		Calibration done?	Action taken	Analyst
	Glass electrode	Porous junction				30-s	2-min			
						mV pH units	mV pH units			
						mV pH units	mV pH units			
						mV pH units	mV pH units			
						mV pH units	mV pH units			
						mV pH units	mV pH units			
						mV pH units	mV pH units			
						mV pH units	mV pH units			

[1] Should be ±1 mV in less than 1 min

[2] Both 30-s and 2-min readings should be 4.00±0.05 pH units (at 25°C) and agree within ±0.05 pH units.

[3] pH readings in mV and pH units (according to Table 1)

Appendix C. Quality Assurance Log

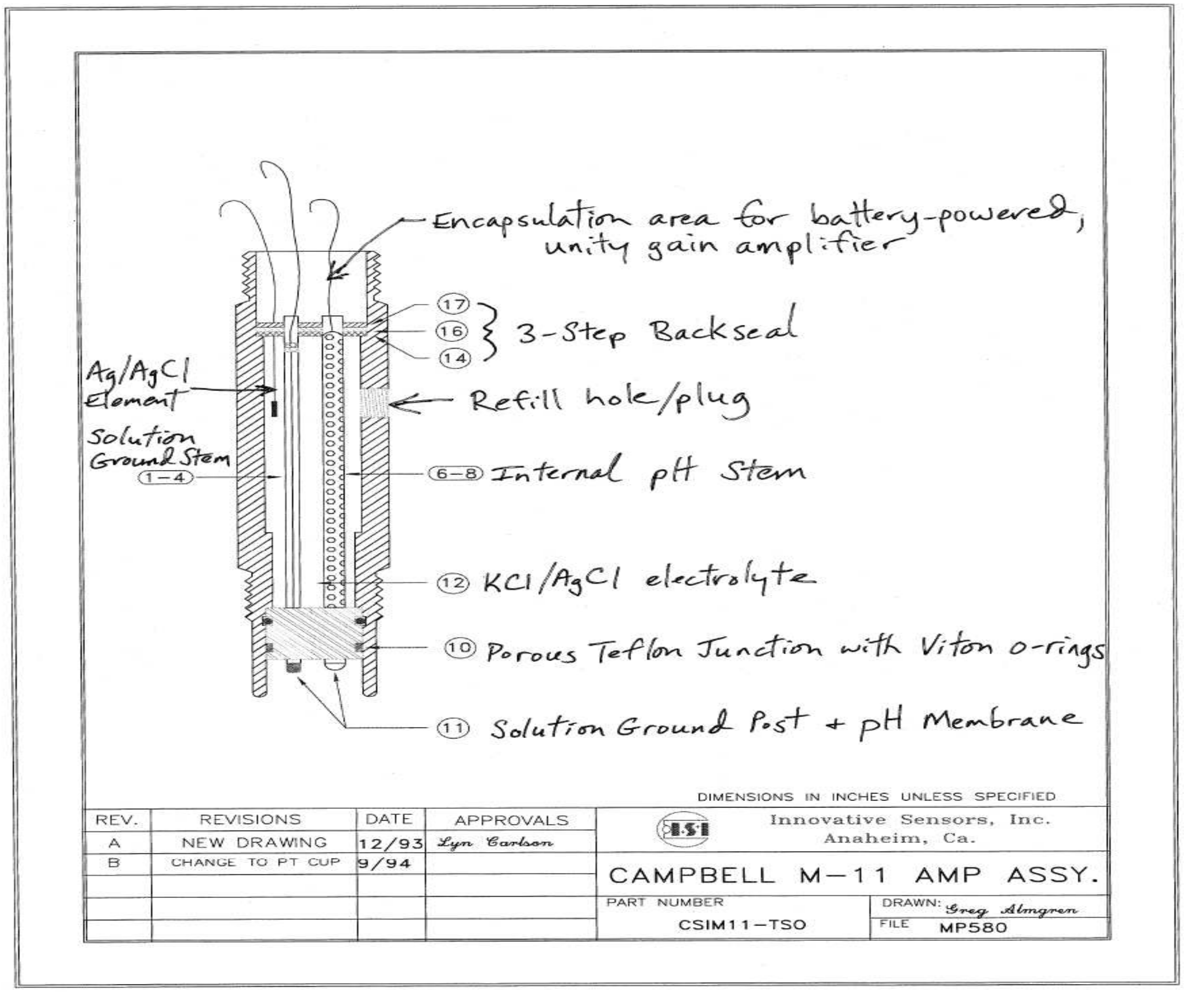
INNOVATIVE SENSORS MODEL CSIM11 pH SENSOR

Serial number ____________________

Beginning or end of measurement period?	Date (DMY) & Time	T (^{o}C)	Buffer pH	Nernst temp comp. (mV)	Expected potential of pH buffer (mV)	Probe measurement		pH Error (Expected vs. calculated)	Action (Adjust, Replace)	Analyst
						mV	Calculated pH			
			7							
			4							
			10							
			7							
			4							
			10							
			7							
			4							
			10							

Appendix D. pH Probe Diagram

INNOVATIVE SENSORS MODEL CSIM11 pH SENSOR



MEASUREMENT OF LAGOON TEMPERATURE
WITH CAMPBELL SCIENTIFIC MODEL 107-L THERMISTOR
Standard Operating Procedure (SOP) L2

MEASUREMENT OF LAGOON TEMPERATURE WITH CAMPBELL SCIENTIFIC MODEL 107-L THERMISTOR

Standard Operating Procedure (SOP) L2

Prepared by
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Reviewed by
Wayne P. Robarge (N.C. State Univ.), Albert J. Heber and Bill W. Bogan

Effective Date: November 6, 2006

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
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1. Scope and Applicability

1.1. Lagoon temperature is a factor influencing emissions. For example, ammonia emissions vary with temperature because the acid-dissociation constant for ammonium (NH_4^+) is temperature-dependent (De Visscher et al 2002). However, lagoon temperature is not needed to measure the emission, and is therefore a secondary measurement.

1.2. Thermistors are useful for the accurate measurement of temperature over long distances without the need for reference measurements.

1.3. This procedure applies only to the Campbell Scientific Model 107 Temperature probe.

- 1.3.1. The temperature range of this sensor is -38°C to +53°C.
- 1.3.2. The accuracy is ±0.4°C over the range of -24°C to 48°C and ±0.9°C over the ranges of -38°C to -25°C and 48°C to 53°C.
- 1.3.3. The time constant of the sensor is less than 30 s.

2. Summary of Method

The Model 107 thermistor measures lagoon water temperature using a negative resistance coefficient semiconductor. The sensor is linearized by bridge resistors. The bridge resistors and thermistor are excited by a 2-VAC voltage, and the measured voltage is further linearized with a fifth-order polynomial (Model 107-LC Temperature Probe Manual, 2003). The probe will be located 0.3 m below the surface of the lagoon and 9 m from the where the liquid meets the berm of the lagoon. The probe will be connected to a styrofoam float, which will be held in place with nylon ropes, as described in SOP U5. A weatherproof NEMA box will house a datalogger that will store data from the probe.

3. Definitions

3.1.	AC	Alternating current
3.2.	ARM	Atmospheric Radiation Monitoring Network
3.3.	CS	Campbell Scientific, Inc
3.4.	mVDC	Millivolts direct current
3.5.	NEMA	National Electrical Manufacturers Association
3.6.	NIST	Natioanl Institute of Standards & Technology
3.7.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.8.	PAML	Purdue Applied Meteorology Laboratory
3.9.	PVC	Polyvinylchloride
3.10.	QAPP	Quality Assurance Project Plan
3.11.	Time constant	The amount of time required for a thermistor to indicate 63% of a new temperature value

4. Health and Safety

4.1. Be careful when working with electrical connections.

5. Cautions

5.1. Be careful when connecting the sensor cable to the datalogger, and make sure the connections (SOP W6) are tight.
5.2. Be careful not to overcrimp the sensors' electrical cables and connectors.
5.3. Be careful not to puncture the lagoon liner when driving in the post for the NEMA box.

6. Interferences

6.1. The probe should be 0.3 m below the surface of the lagoon to prevent being pulled to the surface by waves on windy days.

7. Personnel Qualifications

7.1. Each field staff member must read and understand the sensor instruction manual and this SOP before operating the sensor.
7.2. Personnel must be trained in the use of the handheld multimeter.

8. Equipment and Supplies

8.1. Model 107-LC temperature probe (Campbell Scientific, North Logan, UT) with 30 m of lead cable
8.2. #2 common head screwdriver
8.3. ⅛″ slotted screwdriver
8.4. Temperature calibrator
8.5. Multimeter
8.6. Dishwashing soap for cleaning sensor and cable
8.7. Styrofoam float (1′ x 1′ x 4″)
8.8. Post and post driver
8.9. NEMA box
8.10. NIST-traceable thermometer, accurate to within 1°C
8.11. Datalogger (SOP W6)
8.12. Measuring tape

9. Procedures

9.1. Initial inspection/acceptance of probe
  9.1.1. Check resistance across red and purple wires with multimeter. Using Table 1, determine the temperature which would be output by the probe based on this resistance. Compare to the temperature reported on a NIST-traceable thermometer.
    9.1.1.1. If the two values correspond to within +/- 2°C, the sensor is functioning properly.
    9.1.1.2. If the two values differ by more than +/- 2^{o}C, the sensor is defective and must be returned to the manufacturer.
9.2. Installation (Section 8.6.5 of SOP U5)
  9.2.1. Locate position on lagoon corner (away from inlet) where sensor should be placed.

9.2.2. Position the probe so that the sensor will be 0.3 m below the waterline of the float.
9.2.3. Once all sensors are installed, connect center of nylon guide rope to sensor float and lift the float using the rope. Rope has markers to indicate position to ensure the float will be 3 m from the edges of the lagoon in the corner.
9.2.4. Connect probe cable from NEMA box to indicated hole in the lagoon probe float.
9.2.5. Verify the waterline of the float matches to the 0.3 m mark on the Styrofoam float.
9.2.6. Attach wires to datalogger in NEMA box using the ⅛″ slotted screwdriver. Connect black wire to the switched excitation channel, purple to ground, clear to ground, and red to the single-ended analog channel.
9.3. Calibration
9.3.1. Perform a single-point calibration every six months.
9.3.1.1. Single-point calibration determines the temperature offset in the polynomial equation. The value of the offset is chosen so that the probe outputs the temperature calculated by the polynomial, not the actual calibration temperature.
9.3.1.1.1. 9.3.1.1.1. For example, if probe is placed into a 0^{o}C chamber, and reads 0.1^{o}C, the offset is -0.16, because at 0^{o}C the polynomial gives a value of -0.06^{o}C (Table 1).
9.3.1.2. Connect sensor probe to datalogger (Section 9.2.15).
9.3.1.2.1. Set datalogger to real time measurement mode (SOP W6).
9.3.1.3. Insert sensor probe into calibration chamber. Record chamber temperature, indicated probe temperature, and computed offset on the Calibration Form (Appendix A).
9.3.1.3.1. If sensor offset exceeds +/- 0.5oC, replace sensor.
9.4. Preventive maintenance after each measurement period
9.4.1. Clean the sensor and sensor cable with soap and water.
9.4.2. Inspect the sensor cable for damage, and repair or replace as necessary to maintain cable integrity and continuity in the wires.
9.5. Troubleshooting
9.5.1. Repeated constant values indicate sensor failure.
9.5.1.1. Check connection at datalogger, and tighten with a screwdriver if necessary.
9.5.1.2. Check condition of Santoprene® jacket of cable for cracks and tears. Replace sensor if damaged.
9.6. Data acquisition, calculations & data reduction requirements
9.6.1. Temperature output (-35 to 50 mVDC) is monitored by the datalogger.
9.6.2. Measurements are recorded by the datalogger every 5 min.
9.6.3. The datalogger converts measured voltage (equation 1 below) to temperature using a 5th-order polynomial (equation 2 below). Data is reported to three significant digits.

$X = Vs/Vx = 800 * mVDC$……………..…………………………………… Equation 1

Where X is a ratio of voltage measured (Vs) to excitation voltage (Vx)

$\text{Temp} = -53.4601 + 90.807X - 83.257X^2 + 52.283X^3 - 16.723X^4 - 2.211X^5$…Equation 2

9.7. Computer hardware & software
  9.7.1. A Campbell Scientific datalogger will extract data from the sensor, record the data, and store it.
  9.7.2. The datalogger operates using on-board BASIC-like programming.
    9.7.2.1. The general program for measurements is provided on pages 4 & 5 of the Model 107 Temperature Probe Instruction Manual (Probe Manual, 2004).

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for meteorological sensors.
10.2. Manage all data according to SOP D1.
10.3. Appendix A of this SOP contains a form to record the calibration information.
10.4. Document the complete data and information in the field data sheets and in the site logbooks with permanent ink, or in electronic notes.
10.5. Overstrike errors in writing with a single line. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Check to be sure the probe is mounted 0.3 m below the waterline of the float.
11.2. Abrupt changes (>5oC within 5 min.) indicate the probe float has turned over. Check float and correct orientation.

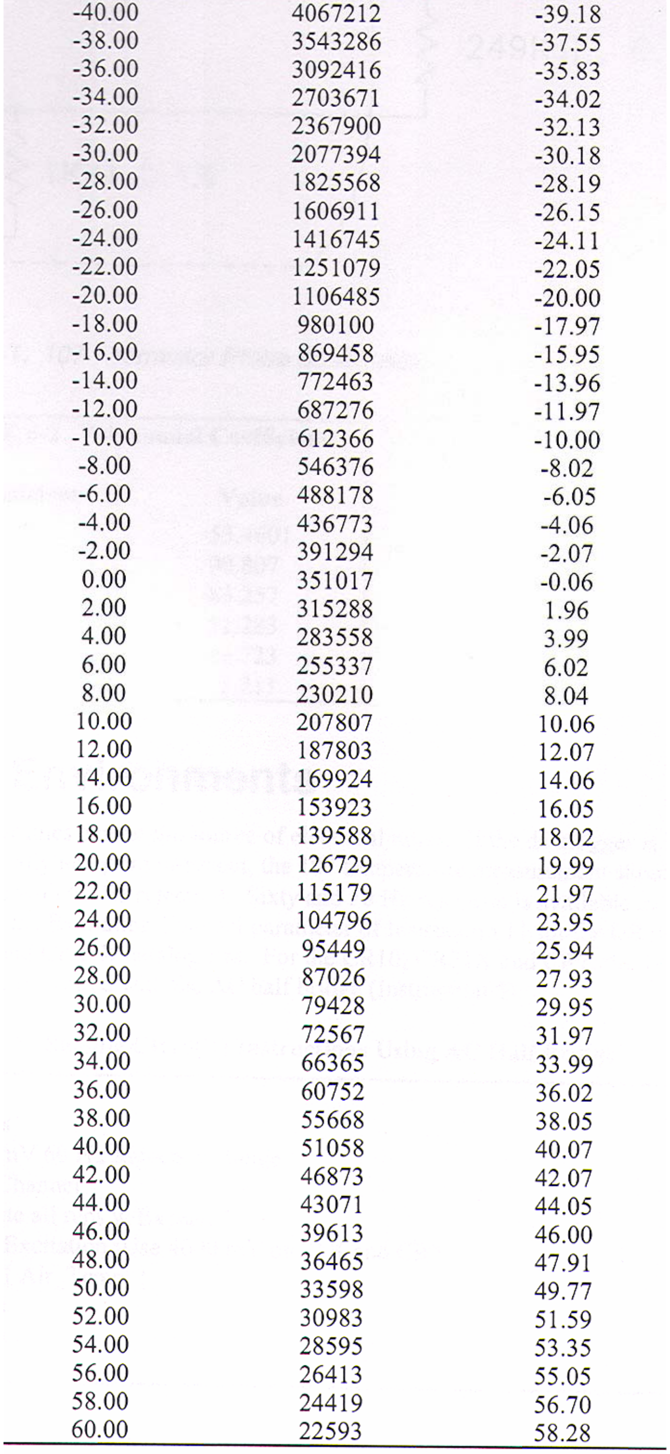
12. References

12.1. Probe Manual. 2004. Model 107 Temperature Probe Instruction Manual. Campbell Scientific, Inc., North Logan, UT.
12.2. Quality Assurance Handbook for Air Pollution Measurement Systems. 1989. Volume IV- Meteorological Measurements, U.S. EPA.
12.3. NCDAQ. 1994. General Guidelines for On-Site Meteorological Data Collection for N.C. Air Quality Analysis. North Carolina Dept. of Air Quality, October.
12.4. De Visscher, A., O. Van Cleemput, L.A. Harper, R.R. Sharpe, P.W. Westerman, Z. Liang, and J. Arogo. 2002, Ammonia emissions from anaerobic swine lagoons: Model development. J. of Applied Meteorology 41:426–433.
12.5. SOP D1. 2006. Management of Open-Source, Weather and Lagoon Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
12.6. SOP W6. 2006. Establishment, Data Acquisition and control of weather and lagoon characterization hardware. Standard Operating Procedure W5. Purdue Applied Meteorology Lab.
12.7. SOP U5. 2006. Installation of Open-Source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.

13. Contact Information

13.1. Distributor: Campbell Scientific, Inc., 815 W 1800 N, Logan, UT, 84321 Technical support: 435-753-2342

Table 1. Correspondence between sensor resistance and measured and reported temperatures.



Temperature °C	Resistance OHMS	Output °C
-40.00	4067212	-39.18
-38.00	3543286	-37.55
-36.00	3092416	-35.83
-34.00	2703671	-34.02
-32.00	2367900	-32.13
-30.00	2077394	-30.18
-28.00	1825568	-28.19
-26.00	1606911	-26.15
-24.00	1416745	-24.11
-22.00	1251079	-22.05
-20.00	1106485	-20.00
-18.00	980100	-17.97
-16.00	869458	-15.95
-14.00	772463	-13.96
-12.00	687276	-11.97
-10.00	612366	-10.00
-8.00	546376	-8.02
-6.00	488178	-6.05
-4.00	436773	-4.06
-2.00	391294	-2.07
0.00	351017	-0.06
2.00	315288	1.96
4.00	283558	3.99
6.00	255337	6.02
8.00	230210	8.04
10.00	207807	10.06
12.00	187803	12.07
14.00	169924	14.06
16.00	153923	16.05
18.00	139588	18.02
20.00	126729	19.99
22.00	115179	21.97
24.00	104796	23.95
26.00	95449	25.94
28.00	87026	27.93
30.00	79428	29.95
32.00	72567	31.97
34.00	66365	33.99
36.00	60752	36.02
38.00	55668	38.05
40.00	51058	40.07
42.00	46873	42.07
44.00	43071	44.05
46.00	39613	46.00
48.00	36465	47.91
50.00	33598	49.77
52.00	30983	51.59
54.00	28595	53.35
56.00	26413	55.05
58.00	24419	56.70
60.00	22593	58.28

Appendix A. Model 107 Thermistor Calibration Form

Serial Number ______________________________

Calibration Date D-M-Y	Calibration Time hh:mm		Temperature (^oC)			Action (Adjust, Replace)	Analyst
	Begin	End	Expected	Measured	Offset (Table 1)		

MEASUREMENT OF LAGOON REDOX STATE WITH THE INNOVATIVE SENSORS CSIM11-ORP SENSOR

Standard Operating Procedure (SOP) L3

MEASUREMENT OF LAGOON REDOX STATE WITH THE INNOVATIVE SENSORS CSIM11-ORP SENSOR

Standard Operating Procedure (SOP) L3

Prepared by

Alfred J. Lawrence and Richard H. Grant

Reviewed by

Wayne P. Robarge (N.C. State Univ.), Bill W. Bogan, and Albert J. Heber

Effective Date: November 6, 2006
Rev. 1.0 Effective Date: February 9, 2008

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
Agronomy, Purdue University
West Lafayette, IN 47907

Table of Contents

Page

1. Scope and Applicability

1.1. The redox state of a livestock waste lagoon is an important parameter that influences emissions from the lagoon.

  1.1.1. For example, redox potentials above ~300 mV will inhibit the growth of methanogenic bacteria in a lagoon, which will lead to a buildup of organic alcohols, aldehydes, and acids (the metabolic products of acetogenic bacteria) (Zhang 2001). Many of these are volatile, and can contribute to odorous volatile organic compound (VOC) emissions from the lagoon.

1.2. Ion-specific probes with built-in preamplifiers are useful for the accurate measurement of Oxidation-Reduction Potential (ORP), a key measure of the redox state of a system.

1.3. This procedure applies only to the Campbell Scientific Model CSIM11 ORP probe.

  1.3.1. The ORP measurement range is from -700 to +1100 mV.

  1.3.2. The ORP range encountered in anaerobic livestock-waste lagoons is -20 to -300 mV.

2. Summary of Method

The density of electrons in solution is measured as the reduction-oxidation potential using an ion-selective probe with a built-in preamplifier (Innovative Sensors, Inc., Model 11 process ORP sensor purchased as Model CSIM11-ORP, Campbell Scientific). A platinum band around the glass electrode provides a surface for reaction and results in the response of the sensor to electron density. The band also reduces sensor drift. A porous Teflon liquid junction resists fouling in the solution and a double junction reference cell increase the service life of the probe. The voltage output of the sensor is proportional to electron density. The probe will be located 0.3 m below the surface of the lagoon and 9 m from where the liquid meets the berm of the lagoon. The probe will be connected to a styrofoam float, which will be held in place with two nylon ropes and a ½” PVC raceway. In addition to keeping the float in place, the PVC raceway will protect the lead cable coming from the probe. The raceway will pivot off of two steel posts which will also support a weatherproof NEMA box, which will house a datalogger that will store data from the probe.

3. Definitions

3.1.	APHA	American Public Health Association
3.2.	Ag/AgCl	Silver: Silver chloride
3.3.	BNC	Bayonet Neill Concelman Connector
3.4.	CSI	Campbell Scientific, Inc
3.5.	emf	Electro-motive force
3.6.	KCl	Potassium chloride
3.7.	NEMA	National Electrical Manufacturers
3.8.	ORP	Oxidation-reduction potential
3.9.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.10.	PAML	Purdue Applied Meteorology Laboratory
3.11.	PVC	Polyvinyl chloride

3.12.	QAPP	Quality Assurance Project Plan
3.13.	SOP	Standard operating procedure
3.14.	VDC	Voltage direct current
3.15.	VOC	Volatile organic compounds

4. Health and Safety

4.1. Be careful when working with solutions. Buffers may cause eye and skin irritations and may be harmful if inhaled or swallowed. Maintain proper lab procedures when filling electrodes or calibrating. Wear proper eye protection, as damage to eyes may result from splash.
4.2. Wear gloves and face shields when handling manure (including lagoon) samples.

5. Cautions

5.1. Be careful when connecting the sensor cable to the datalogger, being sure that connections are tight and are made according to SOP W6.
5.2. Avoid crimping electrical cables and connectors.
5.3. Stray sources of ions such as tap water, soil, dust etc. can contaminate the reference solution. Maintain good laboratory cleanliness when refilling probe.
5.4. Take proper care to avoid damaging the probe’s glass reservoir
  5.4.1. Never wipe the pH bulb, since dust may scratch the delicate gel layer.
  5.4.2. Do not drop the probe.

6. Interferences

6.1. The depth of measurement must be maintained at 0.3 m.
6.2. Algae and other biological organisms will foul the probe, and/or physically block porous liquid junctions.

7. Personnel Qualifications

7.1. Each field staff member must read and understand the sensor instruction manual and this SOP before operating the instrumentation.
7.2. Personnel must be trained in the use of the handheld multimeter before initiating the procedure. Training requires 10 min.

8. Equipment and Supplies

8.1. #2 common head screwdriver
8.2. 1/8″slotted screwdriver
8.3. Bottle with deionized water
8.4. 250-mL and 500-mL beakers
8.5. 2 stands and clamps for sensor and pH reference electrodes
8.6. Protective gloves

8.7. Safety glasses
8.8. Teflon pipe tape
8.9. Multimeter
8.10. ZoBell's Solution Redox Standard (Source: Campbell Scientific, Inc.)
  8.10.1. For Ag/AgCl in 4M KCl, the measured emf is +228 mV at 25˚C
  8.10.2. For Ag/AgCl in saturated KCl, the measured emf is +229 mV at 25˚C
  8.10.3. According to the manufacturer, the shelf life is typically 6 months; however, drifts of >10 mV over 30 d have been reported when solutions are stored at room temperature.
8.11. Standard glass-body reference electrode (Ag/AgCl) with porous liquid junction and BNC connector
8.12. pH preamplifier kit for BNC connector equipped electrodes with leads to connect to multimeter
8.13. 5-VDC power supply for preamplifier
8.14. Analytical balance (200 g range , 0.01 g resolution)
8.15. 1M KCl solution (or reagent grade KCl for preparing solution)
8.16. ORP probe Model CSIM11-ORP, Campbell Scientific, Inc., North Logan, UT
8.17. Stirring rod
8.18. Measure tape
8.19. Reference solution (KCl/AgCl + KNO_3)

9. Procedures

9.1. Probe preparation
  9.1.1. All electrodes are shipped with a wetting cap covering the measuring end. This cap contains a pH 4 buffer saturated with potassium chloride (KCl) for single-junction combination electrodes and potassium nitrate (KNO_3) for double-junction electrodes.
    9.1.1.1. Gently remove wetting cap by unscrewing the bottle from the cap and then sliding the cap and o-ring off the electrode body. Save the cap for storage.
    9.1.1.2. During shipping some air may have moved into the bulb from inside the probe. Grasp the electrode near the cable and gently swing it through an arc to force the internal electrolyte into the pH bulb. Ensure that the internal surface of the porous junctions in the electrodes is in contact with internal reference solutions.
    9.1.1.3. Rinse the electrode with distilled water.
9.2. Acceptance
  9.2.1. Physically inspect ORP probe (Appendix C). Verify that the glass sensing bulb is intact and not scratched, that the porous junction is not visibly leaking, and that the area around the porous junction is not cracked or damaged. Reject the probe if it fails any of these tests. Record assessment on Acceptance Log (Appendix A).
    9.2.1.1. If damaged, return probe to manufacturer for repair or replacement.
  9.2.2. Make 1M KCl solution
    9.2.2.1. Dissolve 74.55 g of reagent grade KCl in 1 L of distilled water.
    9.2.2.2. This solution is stable for 6 months.
  9.2.3. Place 100 mL of KCl solution in a 250-mL beaker.
  9.2.4. Connect green lead from ORP probe to one terminal of the high-input impedance multimeter. Connect the clear-cased reference lead from preamplifier of standard

glass-bodied reference electrode to other terminal of multimeter. Connect ground of ORP probe (black wire) to ground lead from preamplifier of standard glass-bodied reference electrode (Fig. 1).

- 9.2.5. Connect the three-wire preamplifier connector to the 5-VDC power supply. Connector cable has red to positive, black to negative, green to ground. (Fig. 1).
- 9.2.6. Place both ORP and glass-bodied reference electrodes into the 1M KCl solution, and measure the voltage difference between them. The measured voltage should be 0±4 mV for two Ag/AgCl reference electrodes.
  - 9.2.6.1. Use of 1M KCl solution is designed to minimize junction potentials for the two electrodes.
  - 9.2.6.2. Record voltage reading (in mV) on the Sensor Acceptance Log (attached at the end of this SOP) for future reference of ORP probe performance and correction of readings if measured voltage is not zero.
    - 9.2.6.2.1. If the voltage reading is unstable after one min (± 1mV), the porous junction on the probe is likely either leaking or partially obstructed. According to the ORP manual (page 5) change internal (reference) solutions for the ORP probe, and repeat measurements with standard glass-bodied reference electrode.
    - 9.2.6.2.2. If the reading is still unstable, follow the procedure for cleaning clogged liquid junctions outlined in the manual (Section A.1.6 “Electrode Cleaning”), and repeat measurements of the ORP probe with the standard glass-bodied reference electrode.
    - 9.2.6.2.3. If the readings are still unstable, reject ORP probe and replace with a new one.
- 9.2.7. Calibrate the probe (Section 9.4) using redox standard solution to verify acceptability.

9.3. Installation (SOP U5, Section 9.6.7)

- 9.3.1. Locate the position on the lagoon corner (away from inlet) in which the sensor should be placed. Refer to the QAPP and/or Site Monitoring Plan.
- 9.3.2. Position the probe so that the sensor will be 0.3 m below the waterline of the float and install all sensors in tri-sensor float.
- 9.3.3. Connect center of nylon guide rope to tri sensor float and lift using rope. Rope has markers to indicate position to ensure the float will be 3 m from the edges of the lagoon in the corner.
- 9.3.4. Connect the probe cable from NEMA box to the indicated hole in the lagoon probe float.
- 9.3.5. Verify the waterline of the float matches to the 0.3 m mark on the Styrofoam float.
- 9.3.6. Attach wires to datalogger in NEMA box using the ⅛″ slotted screwdriver. Connect brown wire to ground, and red and green wires to the differential-ended analog channel.

9.4. Calibration

- 9.4.1. Based on APHA Standard Method 2580.
- 9.4.2. Connect red wire to positive post and green wire to negative post of multimeter.
  - 9.4.2.1. Set multimeter to mV readings.
- 9.4.3. Rinse probe with de-ionized water three times.

- 9.4.4. Make ZoBell's solution in the field by mixing 1.41 g $K_4Fe(CN)_6{\cdot}3H_2O$ (potassium ferrocyanide), 1.10 g $K_3Fe(CN)_6$ (potassium ferricyanide), and 7.46 g KCl (potassium chloride) in 1 L deionized water.
- 9.4.5. Place ZoBell's Solution in a 500 mL beaker (Section 8.10), and maintain a temperature of 25 ±3 ºC.
- 9.4.6. Measure and record the temperature of the redox standard solution on the calibration form (attached at the end of this SOP as Appendix B).
  - 9.4.6.1. Determine potential of the ZoBell's solution from Table 1 and record on the calibration form under 'expected' (Appendix B).
- 9.4.7. Immerse probe in gently-stirred redox standard solution (ZoBell's Solution).
  - 9.4.7.1. Record measured emf (mV) on the calibration form (Appendix B).
  - 9.4.7.2. If measured emf differs from the expected value by less than ± 30 mV after 10 min, the ORP probe is functioning properly, and may be accepted for use.
  - 9.4.7.3. If measured emf differs from the expected value by more than ± 30 mV after 10 min, replace with unused redox standard solution and repeat Step 9.4.5.
  - 9.4.7.4. If measured emf again differs from the expected value by more than ± 30 mV, replace internal solution and clean platinum-sensing element according to page 5 of the probe manual. Rinse well with distilled water and repeat Step 9.4.4.
  - 9.4.7.5. If measured emf continues to differ from expected value by more than ± 30 mV, the probe performance has degraded beyond acceptable levels. If this occurs at the beginning of a measurement period, replace the probe with a new one. If it occurs at the end of the measurement period, flag or correct the data collected during the period as follows:
    - 9.4.7.5.1. If the measured emf differs from the expected value by ≥50 mV, flag the measurements during the period is invalidated.
    - 9.4.7.5.2. If the measured emf differs from the expected value by less than ± 50 mV, age-correct the measurements made between the beginning and end of measurement period by linear interpolation of the difference in measured values across the time of the measurement period according to:

$$\text{Corrected ORP measurement} = \text{ORP measurement} + T_M * (ORP_F - ORP_I)/(T_P)$$

Where:
T_M = Elapsed time into the measurement period when ORP reading was taken, h
ORP_F = Probe ORP reading at the end of the measurement period
ORP_I = Probe ORP reading at the beginning of the measurement period
T_P = Measurement period duration, h

9.5. Performance assessment and preventive maintenance after each measurement period

- 9.5.1. Remove ORP probe from float and visibly inspect platinum-sensing ring and liquid junction for damage or blockage.
- 9.5.2. Rinse ORP probe with copious amounts of distilled water to remove lagoon effluent.
- 9.5.3. Calibrate according to Section 9.4.4.
- 9.5.4. Clean ORP according to the probe manual.
- 9.5.5. Clean sensor cable with mild soap and water.

9.5.6. Inspect sensor cable for damage and repair or replace as necessary to maintain integrity of the cable and continuity in the wires.
9.5.7. Repeat acceptance protocol (Section 9.2) to assess probe performance at the end of the measurement period and in preparation for future use.
9.5.8. If ORP probe meets acceptance criteria, store according to manufacturers guidelines in preparation for future use.
9.5.9. If the ORP probe fails acceptance criteria, contact PM for replacement probe.

9.6. Troubleshooting
9.6.1. If probe reads 1100 mV or higher:
9.6.1.1. Check for damaged cable. If damaged, replace or repair
9.6.1.2. If the cable is damaged and can't be repaired, contact PM for replacement.
9.6.1.3. Check for loose connections at datalogger; tighten if necessary.
9.6.1.4. Check for coating on platinum sensing element. If needed, clean according to Appendix A, page A3 of the probe manual.
9.6.1.5. Check for clogging of reference probe. If needed, clean according to Appendix A (Page A3) of the probe manual. Repeat acceptance protocol (Section 9.2).
9.6.1.5. Check the probe if filled with reference solution, according to the probe manual.
9.6.2. Noisy or erratic readings
9.6.2.1. Check for coating or clogging of reference electrode. If needed, clean according to Appendix A (Page A3) of the probe manual. Repeat acceptance (Section 9.2).
9.6.3. No response to changes in oxidation reduction potential
9.6.3.1. If electrode reads 0.0 mV when connected to the datalogger, check cable for damage (short circuit). If damaged, replace or repair.

9.7. Data acquisition, calculations & data reduction requirements (SOP D1)
9.7.1. Probe output (-0.413 to 0.413 VDC) is monitored by the datalogger.
9.7.2. Measurements will be recorded by the datalogger every 5 min.
9.7.3. Data is reported to three significant digits.

9.8. Computer hardware & software
9.8.1. Attach the sensors through electrical wires to a Campbell Scientific datalogger.
9.8.2. The Campbell Scientific datalogger will record the data and store it.
9.8.3. The datalogger operates using on-board BASIC-like programming.
9.8.3.1. The general program for measurements is described in the Appendix B of the CSIM11 ORP probe manual
9.8.3.2. Instructions for downloading data from the datalogger are provided in SOP D1.

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for meteorological sensors.
10.2. Manage all data according to SOP D1.
10.3. Acceptance and Calibration Forms are attached at the end of this SOP as Appendix A and Appendix B, respectively.
10.4. Document all data and information on field data sheets and logs and in site logbooks with permanent ink, or in electronic notes.

10.5. Overstrike any errors in writing with a single line. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Verify that the probe is mounted 0.3 m below the waterline of the lagoon probe float.
  11.1.1. Conduct a daily remote check of the data for abrupt changes in measurements (more than ±10 mV in sequential values).
  11.1.2. Verify the correct placement of the probe at the end of each measurement period (up to 20 days).
11.2. Replace redox standard reference solution every 6 months.
11.3. Clean the probe tip according to the probe manual.
  11.3.1. Take note of how far the red refill plug is screwed into the probe body. Remove plug and place it where it will stay clean.
  11.3.2. Rinse reservoir with deionized water repeatedly to remove old solution. Drain out all remaining water.
  11.3.3. Completely fill the reservoir with the new reference solution (Section 13.1). Keep the bottle tip clean and replace bottle cap immediately after using the bottle.
  11.3.4. Replace the red plug and apply new pipe tape. Make sure the plug is screwed back in as far as it was originally.
11.4. Conduct the acceptance protocol (Section 9.2) and calibration (Section 9.4) on all new units, and at the beginning and end of each measurement period.
11.5. Conduct any preventative maintenance during the performance assessment (Section 9.5) at the beginning and end of each measurement period.

12. References

12.1. Probe manual. 2003. Using CSIM11 pH and ORP Probes with Campbell Scientific Dataloggers, Campbell Scientific, Inc., North Logan, UT, 14p
12.2. APHA, Standard Methods for the Examination of Water & Wastewater, 19th Edition, Method 4500-H^+, A.D. Eaton, L.S. Clesceri, A.E. Greenberg (Eds.), AWWA, WEF, 1995.
12.3. Aneja, V.P.; B. Bunton, J.T. Walker, and B.P. Malik. 2001. Measurement and analysis of atmospheric ammonia emissions from anerobic lagoons. Atm. Env. 35:1949-1958.
12.4. Miller, W.J. 2003. Redox Chemistry of Six Anaerobic Swine Lagoons in Eastern North Carolina. MS Thesis, North Carolina State University, Raleigh, NC, 167 p.
12.5. Zhang, R. 2001. Biology and Engineering of Animal Wastewater Lagoons. Online at ucce.ucdavis.edu/files/filelibrary/5049/678.pdf. Acessed 6/08/2006.
12.6. SOP D1. 2006. Management of Open-source, Weather and Lagoon Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
12.7. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.
12.8. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.

13. Contact Information

- 13.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, Utah, 84321
  - 13.1.1. Technical support: 435-753-2342
- 13.2. Manufacturer: Innovative Sensors, Inc, 4123 E. La Palma Ave #200, Anaheim, California 92807
  - 13.2.1. Telephone 714-577-5600, 800-835-5474
  - 13.2.2. Fax: 714-577-5688
- 13.3. Fisher Scientific
  - 13.3.1. www.fishersci.com/Chemical
  - 13.3.2. Rica Chemical No. : 9880-32
  - 13.3.3. Telephone 1-800-766-7000.

Figure 1. Quality Control Check Sample (KCl) connections.

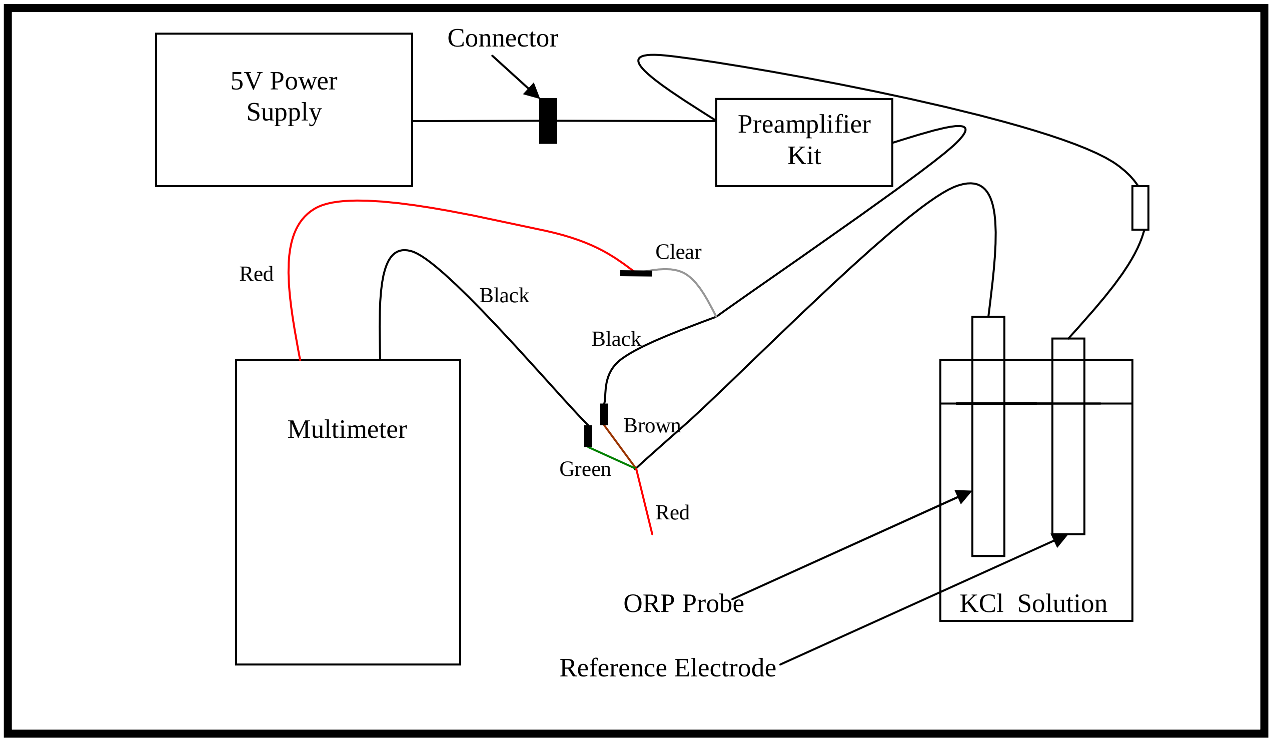


Table 1. Potential of ZoBell's Solution.

Standard Solution	Potentials of Pt Electrode Vs Ag/AgCl reference electrodes at 25 ºC in Standard Solution		
	KCl 1.00M	KCl 4.00M	KCl saturated
ZoBell's solution	+192	+228	+229

Appendix A. Acceptance Log
INNOVATIVE SENSORS MODEL CSIM11 ORP SENSOR

Serial Number ____________

Date & Time	**Condition**		**KCl reference check (mV)**	**Time to stable reading (min)**[1]	**Calibration done?**	**KCl reference check (mV)**	**Action**	**Analyst**
	Pt sensing element	**Porous junction**						

[1] Should be stable within ±1 mV in less than 10 min

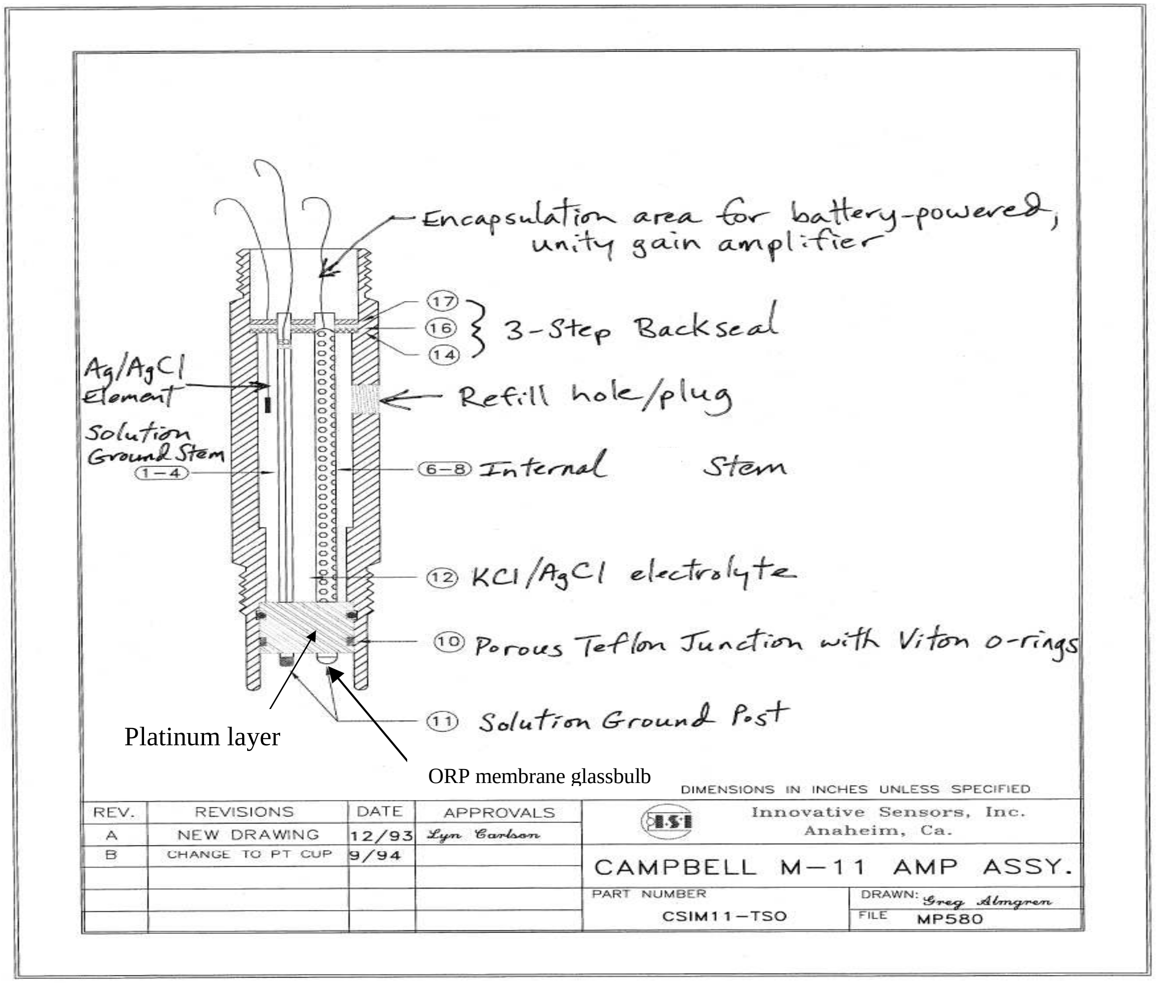
Appendix B. **Calibration Form**
INNOVATIVE SENSORS MODEL CSIM11 ORP SENSOR

Serial Number _____________

Date & Time	T (°C)	Redox standard ORP (mV)			Action (Retest, Replace)	Analyst
		Expected	Measured	Error (Expected-measured)		

Appendix C. ORP Probe Diagram

INNOVATIVE SENSORS MODEL CSIM11 ORP SENSOR



MEASUREMENT OF LAGOON SLUDGE DEPTH
WITH MARKLAND MODEL 10 PORTABLE SLUDGE GUN
Standard Operating Procedure (SOP) L5

MEASUREMENT OF LAGOON SLUDGE DEPTH WITH MARKLAND MODEL 10 PORTABLE SLUDGE GUN

Standard Operating Procedure (SOP) L5

Prepared by

Chris D. Fullerton and Richard H. Grant

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
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Table of Contents

1. Scope and Applicability

1.1. The depth of a livestock waste lagoon can influence emissions from the lagoon.

1.1.1. The ratio of surface area to volume is an important parameter governing emission of NH_3 (Ullman and Mukhtar, 2004).

1.2. Optical transmission of infrared light can be used to detect changes in the solids content of a liquid.

1.3. This SOP document applies only to the Markland Model 10 Sludge Level Detector, or “Sludge Gun”.

1.3.1. The measurement (depth) range of this instrument is 0 to 10 m.

1.3.2. Measurement accuracy (under laboratory conditions) is ±1 cm.

1.3.3. Measurement precision (under laboratory conditions) is ±0.5 cm.

2. Summary of Method

The Markland Model 10 Sludge Level Detector uses high-frequency pulses of a solid-state infrared light-emitting diode, received by a phototransistor, to detect the transmittance of infrared radiation through the liquid enclosed within an open chamber (such as a lagoon or basin). The sensor detects a noise as it receives these pulses; the pitch of this noise increases as the degree of infrared transmittance by the lagoon increases, indicating increasing solid content.

Once during each time the site is set up for a measurement period (one per season), nine individual measurements will be taken from specified areas of the lagoon using the pattern and approximate locations specified on the Depth Plot Worksheet (Appendix D) with the above-described probe. These will be averaged together, and recorded on the “Sludge Depth Field Data Form” (Appendix B). The original Depth Plot Worksheets will then be filed for scanning into the computer and retained according to SOP D1.

3. Definitions

3.1.	SOP	Standard operating procedure

4. Health and Safety

4.1. Wear proper Personal Protective Equipment

4.1.1. Use life jackets at all times when near the edge of the lagoon, and when using the sampling boat.

4.1.2. Always have the life-line rope attached to your waist and in reach of your field partner (on berm) when in the boat.

4.1.3. Do not attempt to take measurements from the lagoon or board the sampling boat if your field partner is not watching from the berm.

  - 4.1.4. Do not attempt to take measurements from the lagoon or board the sampling boat if threatening weather is nearby or imminent.
  - 4.1.5. Use safety glasses when near the lagoon water, and take care not to splash lagoon contents on clothes.
  - 4.1.6. Use rubber gloves when handling the Markland sludge gun, the boat, or any other gear that may have come in contact with the lagoon contents.
- 4.2. Wash and dry gloves before storing
- 4.3. Wash hands after working in the lagoon or on anything that may come in contact with lagoon contents.
- 4.4. Notify your field partner and apply decontamination and correct field dressing if you accidentally cut yourself or puncture the skin in any manner while handling the sludge gun or any material that may have come into contact with the lagoon contents.
- 4.5. It is recommended to wait until close to the end of the day to take measurements from the lagoon if possible so you can proceed to change clothes and shower as soon as possible afterwards.
  - 4.5.1. Have a spare change of clothes at the site with you at all times.
- 4.6. If you fall into the lagoon or become soaked with fluids from the lagoon:
  - 4.6.1. Immediately notify your partner.
  - 4.6.2. Immediately wash your face and hands.
  - 4.6.3. Locate shower facilities and change clothing if your skin has become soaked.
    - 4.6.3.1. If needed, arrange for transport to your hotel to change clothes and shower, return to work site as soon as possible afterwards.
      - 4.6.3.1.1. Place plastic sheeting on the seats and floor of the work vehicle to avoid contaminating them.

5. Cautions

- 5.1. Twisting the cable will damage it.
- 5.2. If the probe is to be lowered in the vicinity of any moving rakes or scrapers, make sure these are stopped first.
- 5.3. Unwind and rewind cable slowly, to avoid stirring up a large ‘cloud’ of sludge, which would result in inaccurate readings.
- 5.4. Never drop the probe into any tank with a hard (e.g. concrete) bottom.

6. Interferences

- 6.1. Battery life is shortened in cold weather. For accurate readings, use new batteries during winter operation (i.e. if temperature is near or below freezing).

7. Personnel Qualifications

- 7.1. Each member of the field staff must read and understand this SOP before operating this instrument.

8. Equipment and Supplies

- 8.1. Model 10 Sludge Level Detector (Markland Specialty Engineering, Toronto, Canada)
- 8.2. Cloth and rubbing alcohol
- 8.3. Dish soap
- 8.4. 500-mL beaker to serve as a calibration chamber
- 8.5. Water softener resin
- 8.6. 50-ft tape measure with metric markings
- 8.7. 1-m ruler with float
- 8.8. 10-cm ruler
- 8.9. Heavy duty rubber gloves (2 pair)
- 8.10. (2) Safety glasses
- 8.11. (2) Life vests
- 8.12. 300-ft Life-line rope capable of lifting 300 lbs.
- 8.13. Roll of plastic sheeting (for protecting vehicle from contamination with fecal matter should transport be necessary with contaminated clothing)
- 8.14. AA alkaline batteries (sensor uses 6)
- 8.15. Heavy duty two man inflatable raft with oars
- 8.16. Portable 12-VDC inflation unit.

9. Procedures

- 9.1. Acceptance
  - 9.1.1. Unpack the unit and check for visible damage or missing parts.
  - 9.1.2. Clean the cable using the following method.
    - 9.1.2.1. Unreel all the cable from the probe and lay it out in a straight line on a clean grassy area. Wipe the entire cable with a clean cloth saturated with rubbing alcohol. Allow a few minutes for the cable to dry.
  - 9.1.3. Make sure the cable is clean and dry. Starting at the sensor end and working towards the “gun” body, string a 50-ft. tape measure beside the cable, with the zero end of the tape at the sensing window in the probe gap (see Fig. 1). Peel off the numbers provided with the instrument from the manufacturer and place the “1” at the 1-m mark on the cable, “2” at the 2-m mark, etc.
  - 9.1.4. Calibrate sensor according to Section 9.2
    - 9.1.4.1. If the sensor cannot be calibrated to within the accuracy and precision limits given in Sections 1.3.2 and 1.3.3, return sensor to manufacturer for repair or replacement.
- 9.2. Calibration
  - 9.2.1. Set sensitivity on the Sludge Level Detector in midpoint of range, using the knob on the back of the handle (Fig. 1). Rotate the control knob so that, with the trigger depressed, there is no sound when the probe is in air or in the liquid at the top of the tank, and the high-pitched note sounds when the probe is in the sludge at the lagoon bottom.
  - 9.2.2. Add 200 mL of resin and 200 mL of tap water to the 500-mL beaker.

9.2.3. Record water depth (in mm) to top of resin on calibration form (Appendix A).
9.2.4. Clamp sensor portion of the instrument to a stand at a height of < 1 m.
9.2.5. Place beaker below sensor.
9.2.6. Lower probe into water until sensor speaker makes a sound.
9.2.7. Holding the cable at the point where it reaches the water line, remove cable and record length of cable from waterline to sensor on calibration form.
9.2.8. Repeat Sections 9.2.4 through 9.2.8 eight additional times.
9.2.9. Calculate precision and accuracy from the 9 sets of measurements as indicated on the calibration form (Appendix A).

9.3. Determining average depth of sludge and water for the lagoon

9.3.1. Refer to Depth Plot Worksheet (Appendix D) for proper sampling locations.
  9.3.1.1. Visualize a grid over the lagoon to be measured consisting of intersecting points by making vertical lines from one edge of the lagoon to the other, at approximately the ¼ point the ½ way point and the ¾ point, and doing the same in the horizontal direction.
9.3.2. Switch gun on. A short ‘beep’ indicates nominal operation.
9.3.3. Proceed to the first sampling point on the grid.
9.3.4. To unwind or rewind cable, point the gun downward and rotate the whole gun in a circular motion. The cable will come off the front of the spool. Unwind only as much cable as needed, then pass it through the pinch-groove at the front of the gun (Fig. 1). To get the cable back up onto the spool, guide the cable with one hand and rotate the whole gun with the other, so the cable comes back onto the spool.
9.3.5. Slowly lower the probe while squeezing the probe trigger.
  9.3.5.1. If the sensitivity control is set correctly, there will be no sound.
  9.3.5.2. If sound is heard, adjust sensitivity according to Section 9.2.1.
  9.3.5.3. As the probe nears the sludge bed, the sensor emits a low-pitched sound.
  9.3.5.4. The volume and pitch will increase as the solids concentration increases.
  9.3.5.5. Keep the trigger depressed and slowly lower the probe to the bottom of the lagoon (the cable will go slack when on the bottom).
    9.3.5.5.1. Record the first cable marker number above the waterline on the Depth Plot Worksheet as value “A” for the current location shown on the worksheet.
    9.3.5.5.2. Using the one-meter ruler, measure the distance from this marker to the waterline, and record as value “B”.
  9.3.5.6. Slowly raise the sensor probe cable until the sound just stops.
    9.3.5.6.1. Record the first cable marker number above the waterline as value “C”
    9.3.5.6.2. Using one-meter ruler, measure distance from marker to waterline, and record as value “D”.
9.3.6. Repeat Section 9.3.5 for each additional required measurement location, filling in all values on the Depth Plot Worksheet.

9.3.7. Determine averages for values A, B, C, and D by adding all the measurements for that value (A, B, C, or D) and dividing by the number of measurement locations (9). Enter the averaged values on the "Sludge Depth Field Data Form" (Appendix B) in the appropriate field. File the completed Depth Plot Worksheet to be scanned and saved according to SOP D1.

9.4. Preventive maintenance after each set of measurements at the field site

9.4.1. Clean sensor and sensor cable with water and soap and dry before storage.

9.4.1.1. Clean sludge depth gun by unwinding cable and lay cable out lengthwise in a clean grassy or snowy area and then wipe sensor and cable with cloth saturated with soap and water and rewind cable using a dry clean cloth.

9.4.2. Inspect sensor cable for damage. Repair or replace as necessary to maintain cable integrity and wire continuity.

9.4.3. Store indoors (i.e. in the open-source instrument trailer) when not in use.

9.5. Troubleshooting

9.5.1. With the probe in air, if a low-pitched ticking occurs as soon as the trigger is depressed, and does not stop upon turning the sensitivity control, this indicates that either the probe is dirty or the batteries need to be replaced.

9.5.1.1. Clean probe and retest.

9.5.1.2. If ticking persists, replace batteries. Remove two screws and take off rear speaker, pull down on the spring-loaded plunger, and slide out the Battery Pack. Insert new batteries, checking orientation against that shown on the pack.

9.5.2. Repeated constant values indicate sensor failure. Replace sensor.

10. Data and Records Management

10.1. Record original calibration and measurement data on appropriate forms (Appendices A and B, respectively), which are kept in a waterproof bound record book designated for lagoon sensors.

10.2. Transfer calibration and measurement data to worksheets of the electronic field notes spreadsheet designated for this method.

10.2.1. Reserve one worksheet for calibration data, and one for measurement data.

10.3. Document all written data and information in site logbooks with permanent ink.

10.4. Overstrike errors in writing with a single line. Initial and date all corrections.

10.5. Manage all data is managed according to SOP D1.

11. Quality Control and Quality Assurance

11.1. Calibrate (Section 9.2) the sensor quarterly.

11.2. Clean sensor and cable (Section 9.4.1) and inspect for damage (Section 9.4.2) after every use.

11.2.1. If damaged, repair wires and encase cable with waterproof heatshrink tubing.

12. References

12.1. Sludge Gun Manual. 2006. Markland Model 10 Sludge Gun Operating Instructions. Markland Specialty Engineering, Ltd., Toronto, Canada

12.2. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.

12.3. Ullman, J.L. and S. Mukhtar. 2004. Implications on ammonia emissions from dairy facilities using dry-lot versus hybrid (free-stall) housing practices. ASAE Paper #044064. American Society of Agricultural Engineers, St. Joseph, MI.

13. Contact Information

13.1. Distributor: Markland Specialty Engineering, Ltd
48 Shaft Road, Toronto, Ontario, Canada M9W 4M2
Tel: 416-244-4980, Fax: 416-244-2287
email: markland@sludgecontrols.com

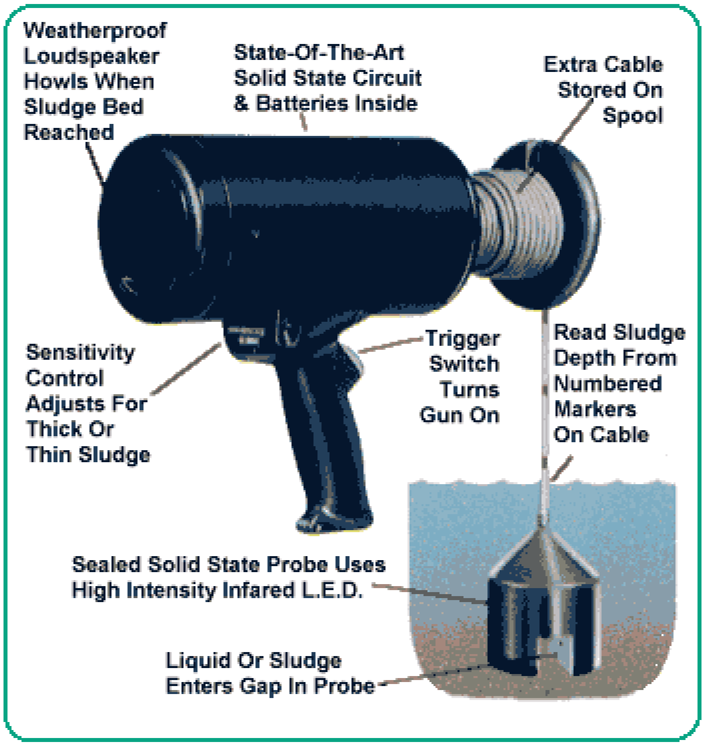

Figure 1. Markland Model 10 Sludge Gun.

Appendix A
Markland Model 10 Sludge Gun Calibration Form

Serial Number ________________ Date (DD-MM-YYYY)

____

Person Calibrating ____ Action:

(Accept / Adjust / Replace)

Actual depth of resin layer (mm)	Measured Depth (mm)
	Trial #1
	Trial #2
	Trial #3
	Trial #4
	Trial #5
	Trial #6
	Trial #7
	Trial #8
	Trial #9
Mean	
Standard Deviation (Precision)	
Accuracy (Measured depth – Actual depth)	

Appendix B
Sludge Depth Field Data Form

Location: __________________________________

Sensor serial number ____________________

Date (D-M-Y)	Time (EST)	**Average Bottom Depth (m)**			**Average Sludge Depth (m)**			Comments	Persons measuring
		Cable Marker # (A)	Distance to Waterline (B)	Bottom Depth (A – B)	Cable Marker # (C)	Distance to Waterline (D)	Depth to Sludge Layer (C – D)		
									/
									/
									/
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Appendix C

MARKLAND SPECIALTY ENGINEERING LTD.
OPERATING INSTRUCTIONS MODEL 10 SLUDGE GUN®

Cable Markers

Make sure the cable is *clean and dry*. String a 50 ft. tape measure beside the cable, with the zero end of the tape at the Probe. Carefully peel off the numbers and place the “1” at the 1ft. mark, “2” at the 2ft. mark, etc… Metric or any other units can also be used. Placing a strip of black vinyl electrical tape midway between the number markers permits more precision in reading the depth.

Sensitivity Adjustment

This thumb adjustable control is at the back of the handle. Rotate the control knob so that with the trigger depressed, there is no sound with the Probe in air or when it is in the liquid at the top of the tank, and the high pitch note sounds when the probe is down in the sludge. This setting is not critical. The sensitivity control is mainly needed for extremely thick or thin slurries, or for boosting the power as the batteries get weak.

Unwinding/Rewinding Cable

Point the Gun downward and rotate the whole Gun in a circular motion and the cable will come off the front of the spool like a fishing line coming off a reel. Unwind only as much cable as needed, then pass it through the pinch- groove at the front of the Gun. To get the cable back up onto the spool, guide the cable with one hand and again rotate the whole Gun with the other hand so the cable comes back onto the spool. ***Do not twist the cable!***

Finding The Sludge Blanket

Lower the Probe while squeezing the trigger. If the Sensitivity control is set right (see Sensitivity Adjustment) no sound will be heard. As the Probe nears the sludge bed a low pitch sound will come from the speaker. This will increase in volume and rise in pitch as the solids concentration increases. Keep the trigger depressed and lower the Probe to the bottom of the tank (the cable will go slack when on the bottom). Read the cable marker opposite the handrail (for example - 25ft.). The speaker will still be howling with the trigger depressed and the Probe in the sludge. *Slowly* raise the cable until the note starts to become lower pitched, and again read the marker opposite the handrail (for example - 20 ft.). Continue *slowly* raising the Probe until the sound stops and read the cable marker (for example - 18 ft.). In this example, you would have 5ft. of dense sludge, covered by 2 ft. of a partially settled sludge ‘cloud’. Remember to move *slowly,* or you will stir up a large ‘cloud’ and confuse the reading.

Battery Replacement

Batteries will last about one year in normal service. With the Probe in air, if a low pitched ticking is heard as soon as the trigger is depressed, and this cannot be removed by turning the sensitivity control, this indicates that either the Probe is dirty and needs washing, or the batteries are weak and will soon need to be replaced. Ignore the short beep that is heard when the Gun is first switched on. This is normal. Battery life is shorter when the Gun is used at low temperatures. To change batteries, remove two screws and take off rear speaker, pull down on spring loaded plunger and slide out the Battery Pack. Replace with AA size Pencell Alkaline batteries, and be careful to install in the direction shown on the Battery Holder.

Appendix C (continued)

MARKLAND SPECIALTY ENGINEERING LTD.
OPERATING INSTRUCTIONS MODEL 10 SLUDGE GUN®

Cold Weather Operation

Battery output is greatly reduced at low temperatures. It is suggested that the Gun be fitted with new batteries if it is going to be used at temperatures near or below freezing. The Gun should be stored indoors when not in use.

Probe

The solid-state Probe is sealed and requires no maintenance other than washing. The cable is sealed at the Probe and can’t be disconnected. If defective, the entire Probe/Cable assembly must be replaced. Care should be taken not to tangle the Probe in Rakes or Scrapers, which should be stopped before lowering the Probe into the tank. Do not drop the Probe onto the concrete tank bottom, lower it slowly. Quickly dropping the Probe stirs up the sludge bed making readings difficult, and could cause Probe damage.

Appendix D

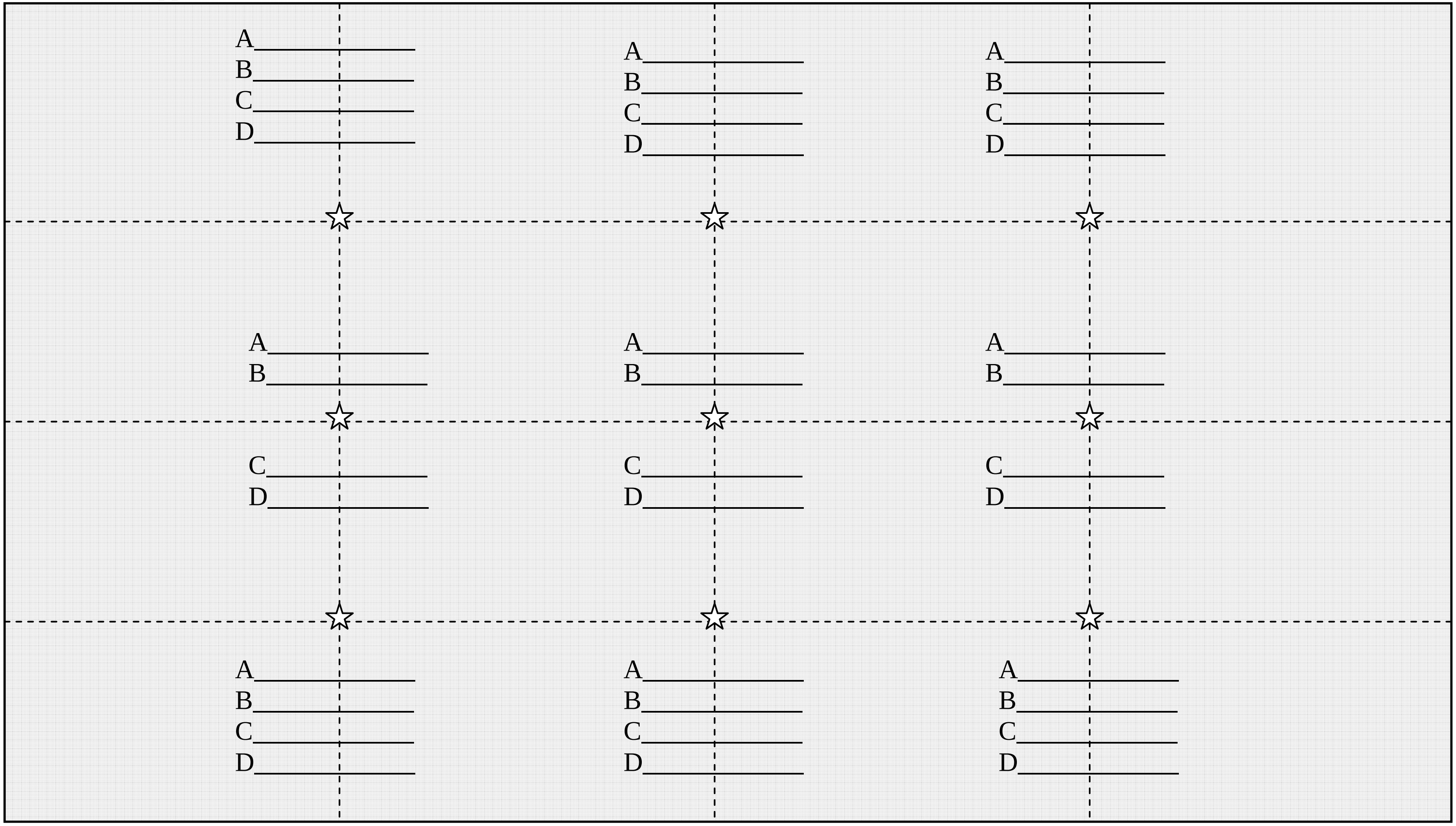
Depth Plot Worksheet

Location: ______________________________

Date:____________________Operator:________________________________

Sensor serial number ____________

☆ Denotes approximate measurement locations



MANURE SAMPLING
Standard Operating Procedure (SOP) M1

MANURE SAMPLING

Standard Operating Procedure (SOP) M1

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PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

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1. Scope and Applicability

1.1. The release of ammonia from manure in livestock barns and storage areas is affected by manure pH, moisture content, and nitrogen and ammonia contents. Manure must be collected to determine these parameters.

1.2. Data on nitrogen content of manure at the end of its storage period is also needed to develop a N balance for the facility (SOP S5).

1.3. This SOP describes the procedures for collecting manure samples from broiler, layer, dairy, and swine barns, and from manure storage areas, during tests of air emissions.

  1.3.1. Manure samples may need to be collected from various types of storage or treatment facilities, including deep pits under buildings, collection channels, drainage pipes, outdoor storage basins, lagoons, and storage piles.

1.4. This SOP provides information on selecting sampling locations and methods to capture representative samples.

1.5. The sampling methods described in this SOP are adapted from References 11.1 – 11.4.

2. Summary of Method

Different sampling equipment and techniques should be selected based on manure characteristics (liquid or dry samples) and the type of facility that is being sampled. Storage basins and lagoons can be sampled with either a conduit-mounted container sampler, a sampling tube with a stopper, or a telescoping ladle. The ladle is also well-suited to sampling collection channels or drainage pipes. Deep pits under slatted floors can be sampled with a sampling tube with a stopper, or with a manure/sludge sampling pump. Manure storage piles that do not contain significant litter, feathers, or other material can be sampled using a tube corer. If, however, litter and feathers are plentiful, sampling can be conducted using a shovel or spade. Several sub-samples can be taken to produce a composite sample. It is important to take a sufficient number of samples to be representative of the manure storage or treatment unit. The times at which manure is sampled will depend on the data required. For example, if the sample is intended to provide data on initial manure parameters (pH, moisture, N and/or NH_3 content) that may affect emissions, it may be desirable to collect fresh manure. If, on the other hand, the final N content of aged manure leaving a storage facility is needed for an N balance, sampling should coincide with the emptying of the storage unit. Exact sampling times for each site will be specified in the Site Monitoring Plans in the QAPP for the specific project.

3. Definitions

3.1.	COC	Chain of custody
3.2.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.3.	QAPP	Quality Assurance Project Plan

4. Health and Safety

4.1. When sampling manure, wear Tyvec suits, shoe covers, and gloves. Wear a respirator to prevent exposure to manure gases.
4.2. Sample, mix, and package all samples in a well-ventilated area.
4.3. Agitation of manure in storage facilities (e.g. deep-pit systems) may release gases that cause very strong odors, and high concentrations that are a severe (i.e. lethal) health hazard (Lorimor et al. 2004).
  4.3.1. If agitation is deemed necessary for accurate sampling, either remove all animals and persons from the facility before beginning agitation, or open or turn on all available ventilation sources (doors, windows, exhaust fans) throughout the agitation and sampling process.
4.4. Be aware of slippery surfaces.
4.5. When collecting samples from confined storage spaces, make sure there is someone watching from a safe area.
4.6. Do not attempt to take samples from manure piles that are too moist and soft to walk on. If the manure becomes too soft, wait to take samples until it is dry enough to walk on, or until it is removed from the building.
4.7. Keep manure sampling equipment and samples away from your mouth and face.
4.8. Be aware of aggressive livestock that may interfere with the sampling process.
4.9. If animals are interfering with the sampling procedure at a particular location, contact the farm manager so that the animals can be temporarily removed from the location.

5. Interferences

5.1. Clean all equipment thoroughly between samplings and sampling locations, using a scrub brush and a bucket of hot, soapy (antibacterial dish soap) water. Check all equipment visually and make sure there is no remaining manure attached.
5.2. Nutrient stratification can occur in non-agitated basins, pits, and lagoons. In these cases, it can be particularly important to collect subsamples from as wide a range of depths as possible, so they can be composited to obtain samples that are representative of the entire vertical profile.

6. Personnel Qualifications

6.1. Personnel should read and understand this SOP and be trained in the use of sampling equipment.
6.2. Personnel should be trained in proper handling and labeling of samples, and in the storage and shipping process.
6.3. The individual taking samples should be able to locate the place from which samples are to be taken.

7. Equipment and Supplies

7.1. Conduit-mounted container sampler (Fig. 1) for sampling basins and lagoons
  7.1.1. The container sampler consists of a long conduit, with a handle at one end and a container with a spring-loaded lid on the other. A cable is attached to the lid of the container, and runs up the conduit and through the handle. When pulled, the cable opens the lid of the container; when released, the spring closes the lid. This type of sampler is particularly useful for collecting samples below the surface of a storage basin or lagoon, if concurrent collection of the surface layer is not required.
7.2. Sampling probe or tube (Fig. 2) for sampling lagoons or deep pits
  7.2.1. Make the sampling tube from a PVC tube.
  7.2.2. For sampling a deep pit, choose a sampler that has an outside diameter smaller than the slot opening of the slats.
  7.2.3. For sampling open pit or lagoon, use a tube with a larger outside diameter (up to 6″) to reduce the sampling time.
  7.2.4. The sampling tube is equipped with a string running the length of the tube. One end of the string is attached to a rubber stopper or ball with a diameter larger than that of the tube; on the opposite end, the string is attached to the sampling tube.
7.3. Telescoping sampler for sampling collection gutters or channels
  7.3.1. CONBAR telescoping dipper, Enviro-Tech Services, Martinez, CA (or equivalent)
  7.3.2. Sampler consists of three nested 4.5′ metal conduit sections, which telescope out to a total length of 12′. The end of the sampler has a screw-tight mounting for a 600-mL polypropylene beaker.
7.4. Manure/sludge sampling pump and collection system for sampling deep pits under slatted floors
  7.4.1. Pump: ¼-horsepower, Gast Model 0523-V191Q-G588DX, or equivalent
  7.4.2. Erlenmeyer filtering flasks (2) 2-L or larger. Polypropylene is preferred over glass for safety reasons.
  7.4.3. Stoppers and tubing to connect the two flasks in series
  7.4.4. Sampling tubing – wall of tubing must be thick enough to withstand collapse under the vacuum from the pump.
  7.4.5. PVC pipe or similar rigid support to guide tubing into the pit and allow sampling at depth
  7.4.6. Cable ties
7.5. Metal tube corer (Fig. 3) for sampling manure piles
  7.5.1. The corer consists of a thin-walled metal tube with a solid metal handle.
7.6. Sledgehammer
7.7. Sharp spade for sampling of feather-containing manure piles
7.8. Five-gallon buckets, hand plungers, and mixing rods for sample homogenization
7.9. Tyvec suits, shoe covers and gloves
7.10. Bucket, scrub brush and antibacterial dish soap
7.11. Freezer for storage of manure samples prior to shipping
7.12. Shipping materials
  7.12.1. Insulated cooler
  7.12.2. One-quart plastic bottles or equivalent

7.12.3. 8″ x 10″ and 9″ x 12″ zip-lock plastic bags
7.12.4. Moisture-proof labels for sampling containers
7.12.5. Shipping peanuts and/or expandable shipping foam
7.12.6. “Blue ice” or other cooling agent
7.12.7. Duct tape or packing tape
7.12.8. Shipping and chain-of-custody (COC) forms

8. Procedures

8.1. Sampling basins or lagoons with container sampler
  8.1.1. Immerse container portion of sampler below the surface of the basin.
  8.1.2. Pull cable to open container lid and allow sample to fill the open container, then release to close. Avoid collecting floating debris, as these may bias the analysis.
  8.1.3. Transfer collected sample into a five-gallon bucket.
  8.1.4. Take multiple samples in order to make a composite sample.
    8.1.4.1. If the composite sample is intended to represent the entire basin or lagoon, take each individual sample at a different location.
    8.1.4.2. If the sample is intended to represent fresh manure, sample from the inlet as manure is entering the basin or lagoon.
    8.1.4.3. The total number of samples needed from each basin or lagoon, and the number of different locations needed, will be specified in the QAPP for a specific project.
  8.1.5. Mix the composite sample with a hand-held plunger (using an up-and-down motion) until it is well-mixed.
  8.1.6. While mixing is maintained, withdraw sample using a cup or similar instrument.
  8.1.7. Fill a one-quart plastic bottle not more than three-quarters full and cap tightly.
  8.1.8. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.

8.2. Sampling deep pits or lagoons with sampling tube
  8.2.1. Insert sampling tube into the lagoon or deep pit until touching the bottom, with the stopper open.
  8.2.2. Pull string attached to stopper to seal the end of the sampling tube. Retract the tube, being careful to avoid tipping the tube and spilling the sample.
  8.2.3. Release the sample into a five-gallon bucket.
  8.2.4. Take multiple samples to make a representative composite sample.
    8.2.4.1. If the composite sample is intended to represent the entire deep pit or lagoon, take each individual sample at a different location.
    8.2.4.2. If the sample is intended to represent fresh manure, sample from the inlet as manure is entering the lagoon, or from the top layer of the deep pit.
    8.2.4.3. The total number of samples needed from each pit or lagoon, and the number of different locations needed, will be specified in the QAPP for the specific project.
  8.2.5. Use a mixing rod to stir the contents of the bucket; this will resuspend settled solids.
  8.2.6. While agitating the liquid, use a ladle to transfer the sample into a plastic bottle until the bottle is three-quarters full.
  8.2.7. Securely fasten the lid onto the container.
  8.2.8. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.

8.3. Sampling lagoons or collection gutters with telescoping sampler
  8.3.1. If not already attached, attach the polypropylene beaker to the end of the sampler. Extend the sampler to the desired length, and immerse container portion of sampler below the surface of the manure.
  8.3.2. Transfer the collected sample into a five-gallon bucket. Follow Steps 8.2.4-8.2.8 above to collect and mix a composite sample and prepare it for shipping.
8.4. Sampling deep pits under slatted floors with a pump
  8.4.1. Connect two filtering flasks in series with the sampling pump, as shown in Fig. 4.
  8.4.2. Attach the sampling tubing to the PVC pipe or other support using cable ties. Slide the tubing/support into the manure in the pit to the desired depth. Unless it is specifically intended to sample the sludge layer that is most likely present at the bottom of the pit, it may be desirable to avoid lowering the sampling tube all the way to the bottom, as the sludge may block the tubing.
  8.4.3. Turn on the sampling pump. Collect approximately 1 L of manure in the first flask (half full). The second flask should serve as a trap to keep manure from entering the pump.
  8.4.4. Turn off the pump and disconnect the sample flask. Mix the contents, either with a stir bar or by swirling, and transfer (pour) the sample into a plastic bottle until the bottle is three-quarters full. Alternatively, if the sample is to be composited with others before taking the final sample, follow Sections 8.1.3–8.1.7.
  8.4.5. Securely fasten the lid onto the container.
  8.4.6. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.
  8.4.7. Discard the remainder of the manure from the sample flask, and rinse the flask before re-connecting to collect the next sample.
8.5. Sampling storage piles with a spade and/or scoop
  8.5.1. With a scoop and a bucket, collect samples throughout the manure storage piles. Take multiple samples in order to make a representative composite sample.
    8.5.1.1. If the composite sample is intended to represent the entire storage pile, take each individual sample at a different location.
    8.5.1.2. If the sample is intended to represent fresh manure, sample from newest portion of the pile (if necessary, consult with farm personnel to identify this portion).
    8.5.1.3. The total number of samples needed from each storage pile, and the number of different locations needed, will be specified in the QAPP for the specific project.
  8.5.2. Once all of the samples have been collected, lay a piece of clean plastic on the ground and place the samples onto the plastic.
  8.5.3. Using a shovel, continuously scoop the outside of the pile to the center of the pile, and mix the center, until the composite pile is thoroughly mixed.
  8.5.4. Use a scoop to collect a composite sample from the mixed pile into a one-quart sealable bag. Fill the bag approximately halfway.
  8.5.5. Squeeze excess air out of the bag, seal the bag, and place the bag into a second bag, which should be larger than one quart (e.g. 9″ x 12″) for ease of handling.
  8.5.6. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.
8.6. Use of core sampler for sampling storage pile
  8.6.1. Insert the corer into the manure storage pile. If needed, use a sledgehammer to pound the tube portion of the corer into the manure, until it touches the bottom of the pile.

- 8.6.2. Remove the core sampler from the manure pile.
- 8.6.3. Using a long rod or broom handle, push sample from corer into a clean bucket.
- 8.6.4. Take multiple samples to make a composite sample. The total number of samples needed from each pile will be specified in the QAPP for the particular project.
  - 8.6.4.1. If the composite sample is intended to represent the entire storage pile, take each individual sample at a different location.
  - 8.6.4.2. If the sample is intended to represent fresh manure, sample from newest portion of the pile (if necessary, consult with farm personnel to identify this portion).
  - 8.6.4.3. The total number of samples needed from each storage pile, and the number of different locations needed, will be specified in the QAPP for the specific project.
- 8.6.5. Once all of the samples have been collected, lay a piece of clean plastic on the ground and place the samples onto the plastic.
- 8.6.6. Using a shovel, break up any chunks that may be present. Continuously scoop the outside of the pile to the center of the pile, and mix the center, until the composite pile is thoroughly mixed.
- 8.6.7. Use a scoop to collect a composite sample from the mixed pile into a one-quart sealable bag. Fill the bag approximately halfway.
- 8.6.8. Squeeze excess air out of the bag, seal the bag, and place the bag into a second bag, which should be larger than one quart (e.g. 9″ x 12″) for ease of handling.
- 8.6.9. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.

8.7. Handling and shipping samples

- 8.7.1. Label all samples with sample location, date and the name of the personnel who collected the samples. Include a completed COC form with each shipment. Enclose COC form in a zip-lock back to keep it dry and clean.
- 8.7.2. Load all samples into the cooler they will be shipped in. Add the desired amount of blue ice, ice-gel packs, or equivalent. If necessary, add sufficient packing material (such as packing peanuts or expandable packing foam) to keep the samples from shifting. Place the packed cooler in the freezer overnight (at minimum).
  - 8.7.2.1. Make sure that the cooler is not contaminated with chemicals or manure, and that it can be properly sealed. Use duct tape or packing tape to keep the cooler closed during shipping.
- 8.7.3. Use overnight shipping. Arrange shipping and receiving to ensure that samples are not held over a weekend or holiday, and that they do not arrive at the lab on a weekend or holiday.

9. Data and Records Management

- 9.1. Maintain all records in a worksheet of the electronic field notes spreadsheet which is designated for this method. Supplement this electronic record with a bound record book designated for the method, which should contain copies of the electronic record.
- 9.2. Manage data according to SOP B5.
- 9.3. Document all data and information on field data sheets, and within site logbooks, with permanent ink, or in electronic field notes.

10. Quality Control and Quality Assurance

10.1. Log all samples. Identify all collected samples, both on their container and in the site logbook, with the sample name and/or number, location, collection method, and date.

10.2. Check all sampling equipment before each sampling trip to make sure they are functioning properly.

11. References

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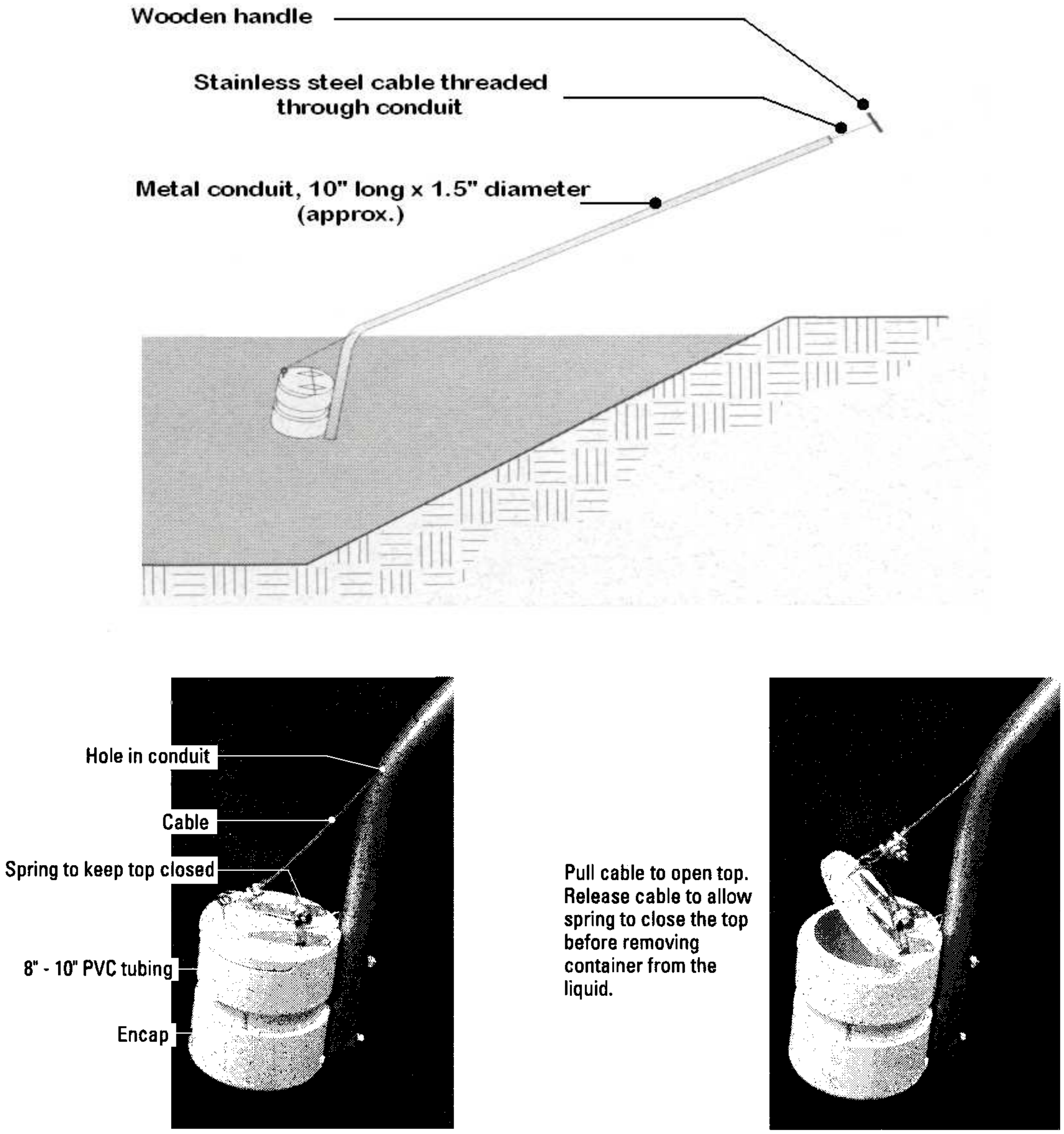

Pull cable to open top. Release cable to allow spring to close the top before removing container from the liquid.

Figure 1. Conduit-mounted container sampler, with close-up showing the operation of the container lid (Lorimor et al, 2004).

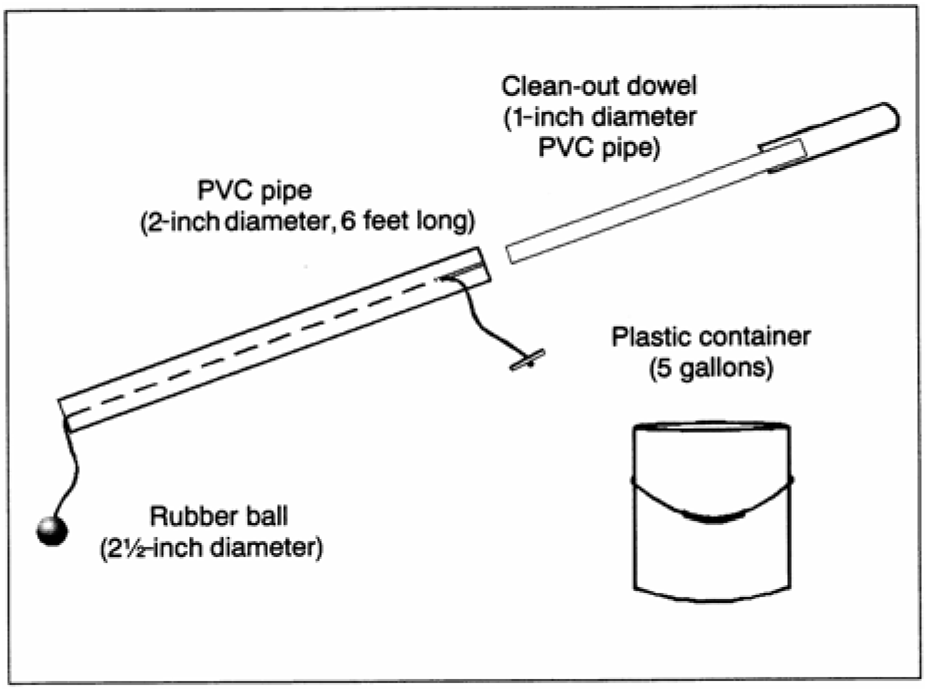

Figure 2. Liquid Slurry Sampling tube (Sonon et al., 2005). Sampler size shown is appropriate for sampling deep pits beneath slatted floors; larger diameters may be used for open lagoons.

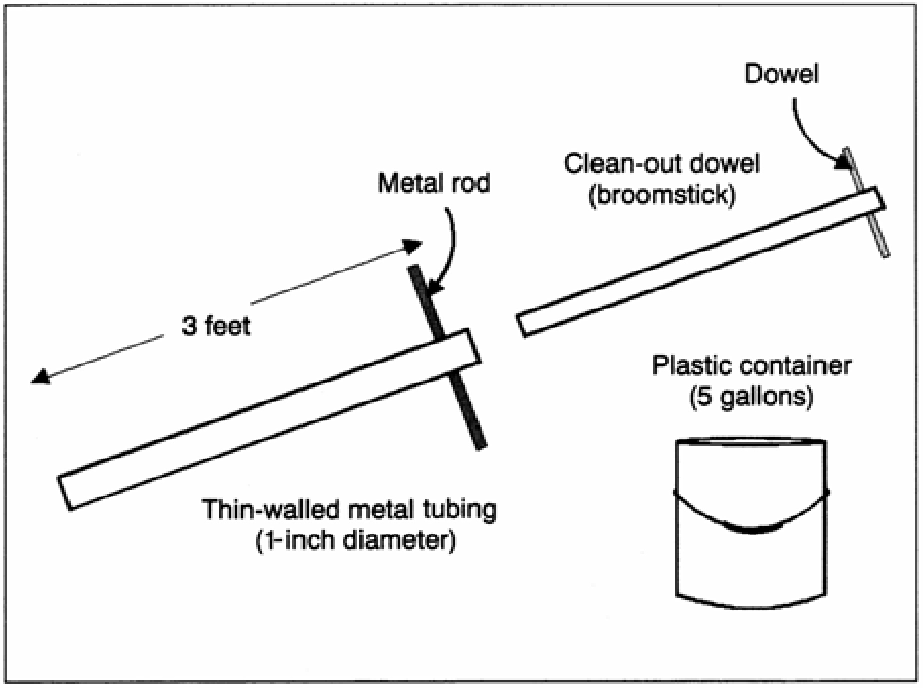

Figure 3. Core Sampler (Sonon et al., 2005).

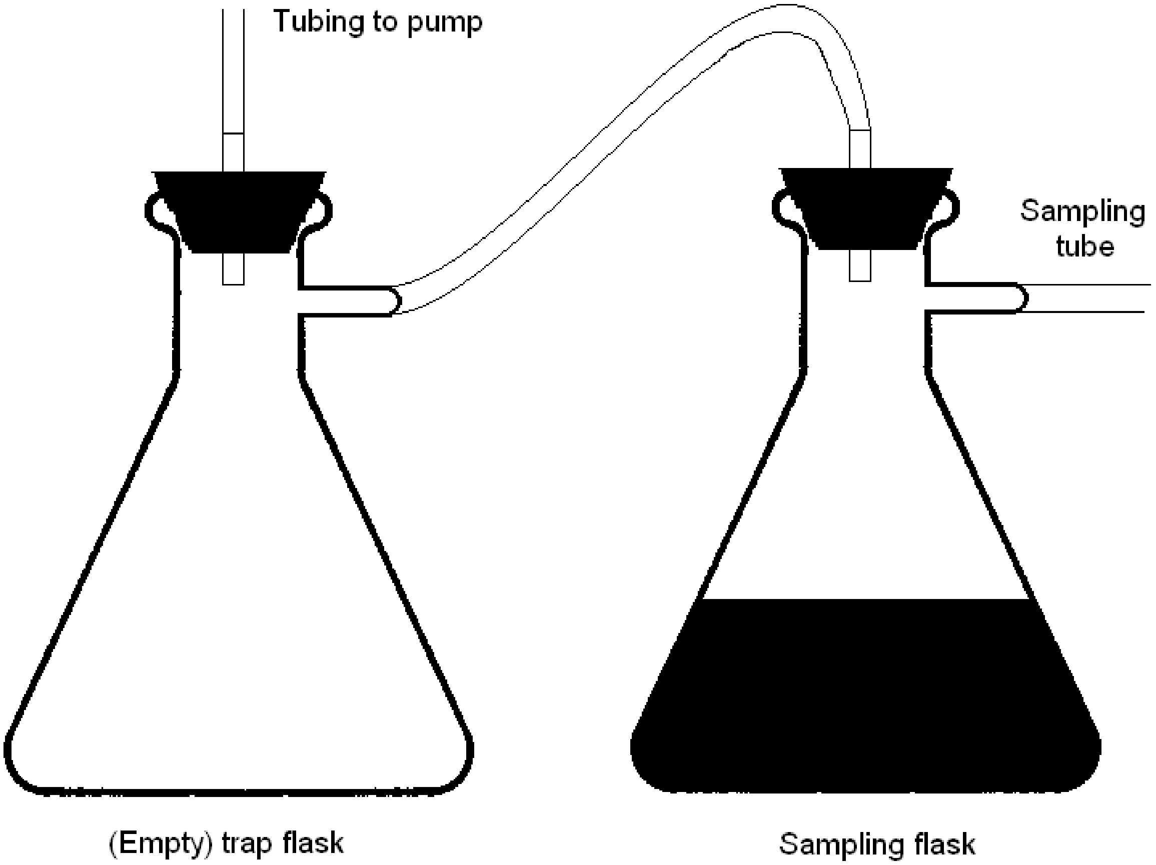

Figure 4. Sampling system for collecting manure from pits under slatted floors (e.g. from deep-pit swine barns).

CONDUCTING pH MEASUREMENTS ON MANURE SAMPLES
Standard Operating Procedure (SOP) M2

CONDUCTING pH MEASUREMENTS ON MANURE SAMPLES

Standard Operating Procedure (SOP) M2

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PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

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1. Scope and Applicability

1.1. A range of pH-dependent chemical and biological interactions and processes can affect the odor and overall composition of the manure.
  1.1.1. Emissions of ammonia (NH_3) increase as pH increases.
  1.1.2. Emissions of hydrogen sulfide (H_2S) decrease as pH increases.
1.2. The method detailed here is adapted from USEPA Method 9040C, “pH Electrometric Measurement”, and is therefore suitable for use on aqueous wastes and those multiphase wastes (including manures) where the aqueous phase constitutes at least 20% of the total volume of the waste. Wastes which originally contain less than 20% water by volume are mixed with water, and allowed to equilibrate, prior to taking the pH measurement.
1.3. This procedure measures the pH of a manure sample using the Orion Model 310 pH meter, equipped with a KCl electrode.
  1.3.1. This instrument covers the pH range from 0.00 to 14.00.
  1.3.2. The instrument has a resolution of 0.01 pH units, and a stated accuracy of ±0.02 pH units.
  1.3.3. The Orion Model 310 can be used within the temperature range of 0°C to 100°C, and includes temperature correction over this range. Its temperature resolution is 0.1°C, with a stated relative accuracy of ±0.5°C.
  1.3.4. This instrument can be used in laboratory or on-site settings.
  1.3.5. This method allows for high-throughput analysis of samples, as analysis of one sample generally requires no more than 5 min.

2. Summary of Method

The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode. The measuring device (pH meter) is calibrated using a series of standard solutions of known pH, which are chosen such that the calibration range brackets the samples whose pH is to be determined. Liquid samples are measured as collected, while solid samples are equilibrated with deionized water in order to obtain a reading. Because measurement of pH is sensitive to temperature, the Orion Model 310 pH meter and 82-02 electrode are equipped with a temperature-correction capability.

3. Health and Safety

3.1. Wear lab coats, gloves and face shields when handling manure samples.
3.2. Buffers may cause eye and skin irritations and may be harmful if inhaled or swallowed.

4. Cautions

4.1. Avoid rubbing or wiping electrode bulb, as this may introduce error due to polarization.
4.2. Do not let stirring bar hit the sensing bulb, as it may crack or break the electrode tip.

5. Interferences

5.1. Glass KCl electrodes, in general, are not subject to solution interferences from any of the following factors:
  5.1.1. Color
  5.1.2. Turbidity
  5.1.3. Colloidal matter
  5.1.4. Oxidizing or reducing agents
  5.1.5. Moderate (<0.1 M) salinity
5.2. Coatings of oily material or particulate matter on the electrode surface can impair its response, and should be removed as they arise. These coatings can usually be removed by gentle wiping with detergent, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.
5.3. Temperature effects on pH measurement using this method arise from two sources:
  5.3.1. Change in electrode output at various temperatures. This interference is controlled by using a pH meter and electrode equipped with temperature compensation.
  5.3.2. Change of pH due to temperature-dependent changes in the sample itself. This error is sample-dependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis.
5.4. Manures have considerable bacterial populations, and metabolic acids produced by these bacteria can cause rapid fluctuations (decreases) in pH. Keep manure samples cool during shipping (SOP M1), and freeze upon arrival in the lab to minimize bacterial growth and activity. Analyze samples as soon after thawing as possible. Observe the general holding times described in Section 10.1.3.

6. Personnel Qualifications

6.1. Personnel should be trained in the use of the analytical instruments, and in laboratory safety, before initiating the procedure.
6.2. Each analyst must read and understand the entire manuals for the pH meter and electrode, and this SOP, before operating the instruments.

7. Equipment and Supplies

7.1. Orion Model 310 pH meter (Thermo Electron Corp., Beverly, MA), or equivalent
7.2. Orion 82-02 PerpHecT ROSS Combination pH Electrode with automatic temperature compensation (Thermo Electron Corp., Beverly, MA)
7.3. Calibration buffers (VWR International, Chester, PA) - These commercially available solutions have been validated by comparison with NIST standards and are recommended by USEPA for routine use.
  7.3.1. pH 4 Buffer , Cat. No. 34170-127
  7.3.2. pH 7 Buffer, Cat. No. 34170-130
  7.3.3. pH 10 Buffer, Cat. No. 34170-133
7.4. Magnetic stirrer and Teflon-coated stirring bars

8. Procedures

8.1. Select the KCl electrode.
8.2. Lower the plastic band covering the hole on the electrode.
8.3. Set pH meter to “standby”.
8.4. Lift electrode from storage solution.
8.5. Using a wash bottle, rinse electrode with redistilled water into a waste beaker.
8.6. Instrument calibration
  8.6.1. Set meter to “pH” mode.
  8.6.2. Press “CAL” key to initiate calibration sequence. “CAL” is displayed for 2 s.
  8.6.3. Press the “YES” key to accept the last calibration range (7-4 or 7-10) or select one of these calibration ranges using the SCROLL keys. Press “YES” to accept.
    8.6.3.1. When measuring samples with acidic pH, use pH 4 buffer for the second calibration step (Choose 7-4 calibration).
    8.6.3.2. When measuring samples with alkaline pH, use pH 10 buffer for the second calibration step (Choose 7-10 calibration).
  8.6.4. “pH 7” will be displayed for 2 s. Place electrode in pH 7 buffer, using a sufficient volume of buffer in a 50-mL beaker to cover the sensing elements of the electrodes, and to give adequate clearance for the magnetic stirring bar. Reading will be displayed and updated as calibration continues. When the “READY” light comes on indicating electrode stability, press the “YES” key to accept.
  8.6.5. “pH 4” (or “pH 10”) will then be displayed for 2 s. Remove electrode from pH 7 buffer. Rinse with deionized water and place electrode in either pH 4 or pH 10 buffer (depending on calibration range that was selected). When the “READY” light comes on, press the “YES” key to accept buffer value. “SLP” (slope) will be displayed for 2 s, after which the calculated slope will be displayed.
  8.6.6. Meter will automatically go into the “MEASURE” mode, at which point the “READY” light will come on to indicate electrode stability.
8.7. Using a wash bottle, rinse electrode with redistilled water into a waste beaker.
8.8. If sample is a liquid, or a slurry with >20% water by volume, place approximately 30-40 mL of sample to be tested into a beaker and gently stir at a constant rate to provide homogeneity and suspension of solids.
  8.8.1. If sample is a solid, or a slurry with <20% water by volume, mix approximately 5 g of sample with water in a 1:1 ratio in a beaker. Gently stir at a constant rate to provide homogeneity and suspension of solids, and allow sample to equilibrate for 30 min before taking the measurement.
8.9. Submerge electrode; when readout is stable, note and record sample pH and temperature.
  8.9.1. If operating in the LogR mode or using an ATC (Automatic Temperature Compensation) probe, the temperature-corrected pH is displayed.
8.10. Rinse electrode between samples with distilled water into the waste beaker.
8.11. When finished, submerge electrode in storage solution and return pH meter to “standby”.

9. Data and Records Management

9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
9.2. Manage all data according to SOP B5.
9.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all corrections.

10. Quality Control and Quality Assurance

10.1. Data integrity
  10.1.1. A second person will verify that the analyses were conducted properly and provide a signature at the end of the data set run and/or at the end of the working day.
  10.1.2. Record the temperature with each set of pH measurements, in accordance with Section 5.3.2.
  10.1.3. In accordance with USEPA Method 9040C, analyze all samples as soon after collection as is feasible.
    10.1.3.1. Hold refrigerated samples for no more than 7 days prior to conducting this analysis. Archived (frozen) samples can be maintained indefinitely.
10.2. Equipment handling and maintenance
  10.2.1. Submerge electrode in storage buffer at all times when not in use.
  10.2.2. Maintain the level of filling solution above the reference junction and at least one inch above the sample level when immersed. Add saturated KCl solution to the electrode if it falls below this level.
  10.2.3. Calibrate the instrument (See Section 8.6) immediately before each batch of samples, and after each batch as a check of how much drift may have occurred.

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DETERMINING SOLIDS CONTENT OF MANURE SAMPLES
Standard Operating Procedure (SOP) M3

DETERMINING SOLIDS CONTENT OF MANURE SAMPLES

Standard Operating Procedure (SOP) M3

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1. Scope and Applicability

1.1. Manure composition significantly affects its emissions of odor and individual chemical components. The solids-to-liquids ratio is an important property.
1.2. This method determines the total solids, volatile solids and fixed solids contents of a manure sample.
1.3. The method described in this SOP is taken from USEPA Method 1684 (2001 Draft version), and is therefore also applicable to soils, sediments, and both raw and processed sludges.
1.4. The stated Method Detection Limits (MDLs) specified for this method are 3 mg/L total solids, 7 mg/L volatile solids, and 7 mg/L fixed solids.

2. Summary of Method

Well-mixed sample aliquots, having a wet weight of between 25 and 50 g, are dried at 103ºC to 105ºC in order to drive off all of the moisture in the sample. This step allows for the determination of total solids. Following cooling, the total-solids portion of the sample is heated to 550ºC, in a step that causes the volatile solids to be released. The sample is again cooled, and the remaining residue represents the fixed-solids portion.

3. Definitions

3.1.	IPR	Initial precision & recovery
3.2.	MDL	Method detection limit
3.3.	OPR	Ongoing precision & recovery
3.4.	QA	Quality Assurance
3.5.	QC	Quality Control
3.6.	Sample batch	All samples which are analyzed at the same time, up to a maximum of twenty (20). If more than 20 samples are analyzed at once, they must be assigned into more than one batch for the purposes of including blanks and OPR standards.

4. Health and Safety

4.1. Use gloves, tongs, and/or suitable sample holders when handling/moving hot sample vessels.

5. Cautions

5.1. When working with wet samples, weigh as quickly as possible after transferring to crucibles (Step 9.2.1), as these can lose water due to evaporation.
5.2. Unless extended drying times are required due to crusting (Section 6.3), do not exceed the drying times specified in this method. Overdrying can result in negative errors due to volatilization of certain salts (Section 6.5), volatilization of organic matter, or heat-

induced chemical decompositions, or to positive errors due to weight gain through sample oxidation.

5.3. Minimize the time that desiccators containing dried samples are open, as samples can adsorb moisture from the air. If strong desiccants are present in the sample (i.e. if weight gain occurs while samples are in the desiccator), use of a vacuum desiccator would be necessary. In all cases, do not remove samples from desiccators until immediately prior to weighing.

6. Interferences

6.1. Keep samples at or below 4°C after collection and during shipping (SOP M1), and prior to analysis, to minimize microbial degradation of sample solids. Analyze all samples within 7 days of collection. Frozen (archived) samples can be maintained indefinitely.

6.2. Samples must be kept homogeneous during the transfer of subsamples to drying vessels, as phase separation at this stage can be a source of considerable error.

6.2.1. Use a magnetic stirrer (See Step 9.2.1.1).

6.2.2. If the sample contains visible suspended solids, do not retrieve subsamples with a narrow-bore pipette. Use wide-bore pipettes, spoons or similar appropriate tools.

6.2.3. If part of the whole sample adheres to the sample container walls despite magnetic stirring, a more intensive homogenization method must be used for mixing. In these cases, sample will be transferred (including scraping of the sample which is adhering to the vessel walls) into a Waring blender and homogenized in this way.

6.2.4. Do not use a magnetic stirrer if magnetic particles are present in the sample.

6.3. If samples form a crust during the drying stage, this can inhibit further evaporation (either of water or volatile solids), and must be dealt with through special handling (either extended drying times or careful physical disruption (breakage) of the crust).

6.4. Following the initial drying step for total solids determination, some samples may retain small amounts of bound water, either as water of crystallization or interstitial water in crystals. Because the loss of this water is generally very slow, these samples may take longer to reach a constant weight.

6.5. Some samples may exhibit loss of CO_2 as bicarbonate is converted to carbonate. Loss of ammonium carbonate may also occur.

6.6. Samples with high levels of oil and grease may require particularly long drying times to reach a constant weight; it may therefore be difficult to obtain incontrovertible results for these samples.

6.7. Some dried samples may be highly hygroscopic, and contain stronger desiccants than are used in the desiccators themselves. Follow Section 5.3.

6.8. Use the same balance for all weighing steps involving a particular batch of samples.

7. Personnel Qualifications

7.1. Personnel should be trained in the use of the analytical instruments.

7.2. Each analyst must read and understand USEPA Method 1684 and this SOP before initiating these procedures, and must demonstrate the ability to generate acceptable data accuracy and precision as outlined in Section 11.2.

8. Equipment and Supplies

8.1. Drying oven
8.2. Muffle furnace
8.3. 15-100-mL crucibles or evaporating dishes made of porcelain or high-silica glass.
8.4. Watch glasses of suitable size to cover the crucibles/evaporating dishes
8.5. Analytical balance: Sartorius Model CP124S, Sartorius AG, Goettingen, Germany (or equivalent) (Capacity = 120 g; Accuracy =0.0001g).
  8.5.1. Reference masses for calibration: 2-mg, 1000-mg, and 50-g class “S” weights
8.6. Magnetic stirrer
8.7. Spatulas
8.8. Desiccator and desiccant (Drierite, Anhydrous Calcium Sulfate)

9. Procedures

9.1. Preparation
  9.1.1. If volatile solids are to be determined, heat crucibles/evaporating dishes and watch glasses to 550ºC for 1 h.
  9.1.2. If only total solids are to be determined, heat crucibles/evaporating dishes and watch glasses to 103ºC to 105ºC for 1 h.
  9.1.3. Cool and store all glassware in a desiccator.
  9.1.4. Prior to use, weigh empty clean crucible and watch glass, and record value (to the nearest 0.01 g) of the two combined.
9.2. Sample handling
  9.2.1. Obtaining subsamples
    9.2.1.1. Fluid (i.e. free-flowing) samples: Stir to homogenize (either by magnetic stirrer or using a spatula). Taking care that the sample remains homogeneous (i.e. that no phase separation occurs), transfer a 25- to 50-g aliquot to a crucible/dish using a pipette, spoon, or other appropriate tool. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.
    9.2.1.2. Wet solid samples: Stir with a spatula to homogenize. Transfer a 25- to 50-g aliquot to a crucible/dish using a spatula, spoon, or other appropriate tool. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.
    9.2.1.3. Dry solid samples: Pulverize the entire sample by hand (using rubber gloves) on a clean surface. Mix, and transfer a 25- to 50-g aliquot to a crucible/dish using a spatula, spoon, or other appropriate tool. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.
  9.2.2. Include one blank (Section 11.8) and one Ongoing Precision and Recovery sample (Section 11.3) with each batch of samples.
  9.2.3. Put in oven to dry for 12 h at 103°C to 105°C.
  9.2.4. Remove from oven with tongs and place in the desiccator to cool to room temperature. Do not remove from desiccator until immediately prior to weighing. See Step 5.3 if working with highly hygroscopic samples.
  9.2.5. Weigh cooled crucible/watch glass/sample, and record value to the nearest 0.01 g.

9.2.6. Place crucible in the (cool) muffle furnace and raise temperature to 550° C and hold for 2 h.
9.2.7. Remove crucible from the muffle furnace with tongs and place in the desiccator to cool to room temperature.
9.2.8. Weigh and record value of crucible/watch glass/ash residue to the nearest 0.01 g.
9.2.9. Return to muffle furnace for 30 minutes, cool and re-weigh. Repeat as necessary until weight change is less than 4% or 50 mg (whichever is less).
9.2.10. Record final value of crucible/watch glass/ash residue to the nearest 0.01 g.

9.3. Calculations
9.3.1. Wet sample weight:
Crucible/watch glass/wet sample (Step 9.2.1) – Crucible/watch glass (Step 9.1.4)
9.3.2. Dry sample weight:
Crucible/watch glass/dry sample (Step 9.2.7) – Crucible/watch glass (Step 9.1.4)
9.3.3. Ash weight:
Crucible/watch glass/ashed sample (Step 9.2.12) – Crucible/watch glass (Step 9.1.4)
9.3.4. % Total Solids = ((Dry sample weight)/(Wet sample weight)) * 100
9.3.5. % Fixed Solids = ((Ash weight)/(Dry sample weight)) * 100
9.3.6. % Volatile Solids = 100% - % Fixed Solids

10. Data and Records Management

10.1. Maintain all laboratory records in an electronic spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
10.2. Document all data and information on data sheets with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance

11.1. Quality Control (QC) Solutions
11.1.1. One of the following solutions will be employed for QC checks of the procedures described in this SOP:
11.1.1.1. NaCl-KHP QC solution
11.1.1.1.1. Dissolve 0.10 g NaCl in reagent-grade (de-ionized) water.
11.1.1.1.2. Add 0.10 g potassium hydrogen phthalate (KHP), and mix to dissolve. Warm and stir if necessary to dissolve the KHP.
11.1.1.1.3. Dilute with de-ionized water to 1.0 L in a volumetric flask.
11.1.1.1.4. Store at 4°C
11.1.1.1.5. This solution contains 200 mg/L of total solids, 81 mg/L of volatile solids, and 119 mg/L fixed solids.
11.1.1.2. Commercially-obtained QC solution: Wibby Environmental Volatile Solids QC Standard (Catalog # QC-VSOL-WP), Wibby Environmental, Golden, CO (or equivalent).

11.2. Analyst Proficiency Demonstration

11.2.1. Initial Precision and Recovery (IPR): Analyst must demonstrate ability to generate acceptable precision and accuracy. Prepare at least four (4) replicate (50-mL) aliquots of a 12-fold dilution of the QC solution. Process all replicates through each step of the analytical method, and perform all calculations. Using the results of the four or more replicates, calculate the average and standard deviation for the concentrations of total, volatile and fixed solids. Compare to the following table:

Analyte	Expected value	Acceptable range	Acceptable S.D.
Total solids (mg/L)	16.7	14.2 – 18.4	10%
Volatile solids (mg/L)	9.92	7.44 – 10.91	30%
Fixed solids (mg/L)	6.75	5.06 – 7.42	20%

11.2.1.1. If averages and standard deviations do not fall within these acceptable ranges, analyst must identify his/her source(s) of error and repeat the IPR procedure.

11.3. Ongoing Precision and Recovery (OPR)

11.3.1. Prepare an OPR solution identical to the IPR solution described in Step 11.2.1 (i.e. a 12-fold dilution of the QC solution).

11.3.2. Include one (1) OPR sample (25-50 mL) with each sample batch, and subject it to all steps of the analytical method.

11.3.3. Concentrations determined from the OPR sample must fall within the following ranges (in mg/L):

- Total solids: 13.4 – 18.4
- Volatile solids: 6.94 – 10.91
- Fixed solids: 4.90 – 7.42

11.3.4. If recovery of any analyte falls outside the acceptable range for a given OPR sample, repeat analysis of that sample batch after appropriate troubleshooting and correction of the problem.

11.4. QC Charts

11.4.1. Track all IPR and OPR results to keep a graphical representation of continued laboratory performance.

11.5. Method Modifications & Re-validation

11.5.1. Each time any modification is made to this method, the analyst will verify that the modified method meets or exceeds the Method Detection Limit and Initial Precision and Recovery achieved by the same analyst using the original (unmodified) method.

11.5.2. Verify MDLs by diluting either the NaCl-KHP QC solution (Section 11.1.1.1) or the commercial standard (Section 11.1.1.2) to the extent(s) necessary to achieve the solids contents listed in Section 1.4. Analyze each of these diluted solutions by the modified method to verify that the analyte can be detected at that level.

11.5.3. The laboratory will maintain records of any modifications made to the method. At minimum, these records must include the following:

11.5.3.1. Names, titles, and contact information for analyst who initiated the modifications and for the QA officer who witnessed and approved the modifications.

11.5.3.2. Narrative stating reason(s) for the modification

11.5.3.3. Results from all QC tests comparing the modified method's performance to that of the original method:

- Initial Precision and Recovery
- Accuracy
- Blanks
- Ongoing Precision and Recovery

11.6. Data Integrity

11.6.1. A second person will be present to verify that analyses were conducted properly and provide a signature at the end of the data set run and/or the end of the working day.

11.7. Replication

11.7.1. Run at least 10% (one in ten) of the samples in each batch in duplicate, with at least one duplicate per batch. Initial masses (i.e. wet weights) of the duplicates will not differ by more than 10%. Replicates must agree to within 10% of their average (% total solids); if not, rerun this batch.

11.8. Blanks

11.8.1. One blank (25-50 mL of deionized water) must be included with each sample batch, and be subjected to all steps of the analytical method.

11.8.2. If material is detected in the blank at or above the MDLs stated in Section 1.4, stop analysis until the source of contamination is identified and eliminated (i.e. a new blank shows no evidence of contamination).

11.9. Instrument Calibration and Standardization

11.9.1. Analytical balance

11.9.1.1. Full calibration (annually at a minimum).

11.9.1.1.1. Calibrate at 2 mg and 1000 mg using class "S" weights.

11.9.1.1.2. Instrument readings must be within ±10% at 2 mg (i.e. ±0.2 mg) and ±0.5% at 1000 mg (i.e. ±5 mg). If these are not achieved, re-calibrate the balance.

11.9.1.2. Calibration check (daily before any samples are weighed)

11.9.1.2.1. Place a 50-g weight and the 2-mg weight on the balance together. Verify that the balance reads 50.002 g (±0.0002 g).

12. References

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12.2. Hoskins, B. 2003. "Laboratory Procedures". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 12-13. Online at http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx. Accessed 9/6/2007.

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DETERMINING TOTAL (KJELDAHL) NITROGEN CONTENT OF MANURE SAMPLES

Standard Operating Procedure (SOP) M4

DETERMINING TOTAL (KJELDAHL) NITROGEN CONTENT OF MANURE SAMPLES

Standard Operating Procedure (SOP) M4

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Effective Date: December 28, 2007

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Table of Contents

1. Scope and Applicability

1.1. Manure composition can significantly affect its emissions, both in terms of general odor and individual chemical components. The total nitrogen content is an important manure property that affects emission of ammonia and other nitrogen-containing compounds. Thus, this parameter is important for both barn and open-source systems.

1.2. This method determines the total Kjeldahl nitrogen (TKN) content of a manure sample, Kjeldahl nitrogen being the sum of ammoniacal nitrogen and organic nitrogen.

1.3. The digestion method described in this SOP is taken from USEPA Method 1687 (2001 draft version), and is therefore also applicable to wastewaters, drinking water or groundwater, and industrial and other wastes.

1.4. The distillation and titrimetric procedures are described in USEPA Method 351.3.

1.4.1. This method has a working range of ca. 0.5-10 mg N/g manure (wet weight basis).

1.4.2. The stated detection limit for the method (Reference 12.4) is 0.01% (0.1 mg/g) N.

2. Summary of Method

Organic nitrogen in a manure sample is first converted to ammonia by metal-catalyzed acid digestion (with the exceptions listed in Section 6.7). The ammonia in the digested sample (the sum of nitrogen that was originally present in the ammoniacal form, plus that which is liberated from organic compounds) is then distilled away from the rest of the sample, at which point it is captured in a dilute sulfuric acid/boric acid solution containing bromocresol green/methyl red indicator. The ammonia concentration of the distillate is then determined by titration with sulfuric acid. Cupric sulfate/titanium dioxide ($CuSO_4/TiO_2$) is used as the sole catalyst in the digestion process. This avoids the toxicity and waste-disposal problems posed with mercury- and/or selenium-containing compounds. A QA/QC section is provided to discuss the calibration and periodic testing of the analytical method to ensure reliable data.

3. Definitions

3.1.	COD	Chemical oxygen demand
3.2.	CV	Calibration verification
3.3.	IPR	Initial precision and recovery
3.4.	MDL	Method detection limit
3.5.	MgO	Magnesium oxide
3.6.	MS	Matrix spike
3.7.	NaOH	Sodium hydroxide
3.8.	OPR	Ongoing precision and recovery
3.9.	QA/QC	Quality assurance/quality control
3.10.	Sample batch	A group of samples analyzed at the same time, up to a maximum of twenty (20). If more than 20 samples are analyzed at a time, divide them into multiple batches for the purpose of including blanks and CV and PR standards.
3.11.	SOP	Standard operating procedure
3.12.	TKN	Total Kjeldahl nitrogen

4. Health and Safety

4.1. Use gloves, tongs, and/or suitable sample holders when handling hot sample tubes.

4.2. Treat each chemical compound as a potential health hazard, and reduce exposure to chemicals to the lowest possible level.

4.2.1. Wear protective clothing (gloves, lab coats, protective eyewear) when working with chemicals.

4.2.2. Conduct all sample digestions in properly-ventilated fume hoods. Use a manifold to remove acid fumes.

4.2.3. Keep analytical balances and other work areas clean; deal promptly with spills.

4.2.4. When using concentrated acids and bases, keep a fully-stocked spill kit (including suitable neutralizers and absorbents) in the work area.

4.3. Maintain a current awareness file of Occupational Safety and Health Administration (OSHA) regulations pertaining to the chemicals specified in this method.

4.4. Maintain a reference file of material safety data sheets, which will be available to all personnel involved in these analyses.

4.5. Sulfuric acid digest is classified as a hazardous waste. Dispose of accordingly.

5. Cautions

5.1. Considerable heat is generated when dissolving sodium hydroxide (NaOH) in water (Step 8.7.2), or by mixing acids with water. Cool the container in an ice bath when preparing NaOH solutions or when mixing acid and water.

5.2. Excessive foaming during distillation can cause foam to pass into the condenser, which can contaminate the receiving flask and condenser with alkaline salts. Treat heavily-foaming samples with an anti-foaming agent.

6. Interferences

6.1. Keep samples at or below 4°C after collection and during shipping (SOP M1), and prior to analysis, to minimize microbial activity. Analyze all samples within 7 days of collection. Frozen (archived) samples can be maintained indefinitely.

6.2. Use only ammonia-free water, which can be prepared by passing reagent water through an ion-exchange column that is packed with a mixture of both strongly acidic cation- and strongly basic anion-exchange resins. Regenerate this column periodically according to the manufacturer's instructions.

6.3. Ammonia gas in the lab can deposit into the sample. Minimize sample exposure to air.

6.4. Nitrate in large quantities (>10 mg/L) will oxidize ammonia produced by the digestion and can introduce a substantial negative error. Although this interference cannot be prevented, its effect can be predicted and accounted for on the basis of preliminary nitrate determination of the sample. Whenever high nitrate levels are a potential issue, nitrate analyses of the sample should be conducted first.

Inorganic salts or solids will increase digestion temperature; however, conducting digestion at temperatures in excess of 400°C will cause pyrolitic loss of nitrogen. If high levels of salts and/or solids are suspected, adding extra H_2SO_4 can partially circumvent this problem. Approximately 1 mL H_2SO_4 per g of salt/solid will preserve the proper ratio, and allow digestion to proceed at the target temperature. Monitoring of digestion temperature will indicate if problems are occurring.

6.5. Large amounts of organic matter can consume the acid in the digestion reagent, causing the digestion temperature to rise above 400°C. Adding 10 mL H_2SO_4 per 3000 mg COD will prevent this. Monitor digestion temperature and pH if this correction is used.

6.6. Nitrogen occurring in certain organic compounds will not be measured by this method. These compounds include those in which the nitrogen occurs in the following forms:

- azide
- azine
- azo
- hydrazone
- nitrate
- nitrite
- nitrile
- nitro
- nitroso
- oxime
- semicarbazone

6.6.1. Levels of these compounds in manure are expected to be negligible.

7. Personnel Qualifications

7.1. Personnel should be trained in the use of the equipment before initiating the procedure.
7.2. Each analyst must read and understand this SOP, as well as USEPA Methods 1687 and 351.3, before initiating this procedure, and must show the ability to generate acceptable data accuracy and precision (Section 11.3).

8. Equipment and Supplies

8.1. Copies of USEPA Methods 1687 and 351.3
8.2. Tecator Instrument Model 2020 digester block and aspirator for sample digestion
8.3. Foss Kjeltec Model 2300 automated distillation and titration analyzer (FOSS Analytical, Hilleroed, Denmark), or equivalent
8.4. Flasks: 100-mL Kjeldahl and 50-mL Erlenmeyer flasks
8.5. Magnetic stirrer and Teflon-coated stir bars
8.6. Spatulas
8.7. Analytical balance (Capacity = 410 g; Accuracy = 0.001 g)
8.7.1. Reference weights for calibration: 2-mg, 1000-mg, and 50-g class “S” weights
8.8. Reagents
8.8.1. Sulfuric acid: Concentrated, 0.020N & 0.25N H_2SO_4, certified (Midland Scientific, Omaha, NE)
8.8.2. Sodium hydroxide: 40% NaOH solution, certified (Midland Scientific, Omaha, NE)

8.8.3. Digestion reagent: TT-43 Pro Pac Kjeldahl digestion tablets (Alfie packers, Inc, Omaha, NE)

8.8.4. Boric acid indicator

8.8.4.1. Add 0.10 g bromocresol green, 0.10 g methyl red, 100 g boric acid, and 20 mL of 0.10N NaOH to a 2-L flask that is half-full of NH_3-free water and dissolve all reagents

8.8.4.2. Add NH_3-free water to the 2-L mark on the flask.

8.8.4.3. Quantitatively transfer contents of the flask to the receiving solution reservoir below the Foss Kjeltech Model 2300.

8.8.4.4. Add 8 moreL of NH_3-free water to the reservoir, for a total of 10 L of solution.

8.9. Standards

8.9.1. Ammonia standards

8.9.1.1. Ammonia stock solution (1000 mg/L NH_3-N): Dissolve 0.382 g of anhydrous ammonium chloride (NH_4Cl), which should be dried at 105°C immediately prior to use, in NH_3-free water and dilute to 100 mL in a volumetric flask with NH_3-free water.

8.9.1.2. Ammonia working standard (10 mg/L NH_3-N): Dilute 10 mL of ammonia stock solution to 1 L in a volumetric flask with NH_3-free water.

8.9.2. Nicotinic acid standard (Contains 100 mg/L organic N): Dissolve 21.637 g nicotinic acid in ~ 150 mL NH_3-free water. Dilute to 200 mL with NH_3-free water.

8.9.3. Calibration standards

8.9.3.1. Prepare calibration standards by spiking 5-g aliquots of blank sand or diatomaceous earth (Section 8.10) directly with nicotinic acid standard solution (Section 8.9.2).

8.9.3.2. Prepare a minimum of five calibration standards, at five different concentrations to produce a calibration curve with at least five points.

8.9.3.3. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The QAPP for a particular project will state the specific concentration needed for this standard.

8.9.3.4. The remaining standards should correspond to the concentration range found in actual samples, without exceeding the titration-based system’s working range.

8.9.4. Calibration Verification (CV) standard

8.9.4.1. Prepare the CV standard such that its N concentration approximates the midpoint of the calibration curve.

8.9.4.2. The source used to prepare the CV standard should be different from that used to prepare the calibration standards. Use either a different bottle of nicotinic acid, or a different compound altogether.

8.9.4.3. If a different ammonium compound is used for the CV stock, adjust the amount weighed according to the ratio of N weight to the molecular weight. Nicotinic acid has one atom of N per molecule and a formula weight of 123.11; it is, therefore 11.37% N by weight (14/123.11). If, for example, nicotinamide (2 N per molecule, formula weight 122.12, 22.93% N) is used as the CV standard, and X mg of nicotinic acid was needed to yield the desired N concentration, then (11.37/22.93) X, or 0.496 X g of nicotinamide would be needed.

8.9.5. Quality control sample (QCS): Standard Reference Material 1570a (Spinach Leaf). National Institute of Standards & Technology, Gaithersburg, MD.

8.10. Blank sand or diatomaceous earth

8.10.1. Bake 500 g diatomaceous earth or clean sand at 400°C for 8 h. Cool and store in a glass container with a sealing lid.

9. Procedures

9.1. Sample Preparation

9.1.1. In a fume hood, preheat the digestion block of the Model 2020 to 380ºC.

9.1.2. Obtain percent total solids of each sample to be analyzed, according to SOP M3.

9.1.3. Label digestion tubes as necessary to accommodate all samples, plus the various QA standards (Duplicates, Precision and Recovery Samples, Calibration Verification Standards, Quality Control Standards, Matrix Spikes, and/or Blanks) as required (See Sections 11.4 through 11.9 for the frequency that these must be included).

9.1.4. Bring samples to room temperature. Thoroughly homogenize the sample. Transfer 50 mL of sample into a digestion tube, and obtain and record the sample weight.

9.1.5. Add Kjeldahl digestion tablets (Section 8.8.3) and 20 mL of concentrated H_2SO_4, and place tube in the preheated digestion block.

9.2. Digestion

9.2.1. Immediately set up the exhaust system, and place the exhaust manifold so that it covers the mouth of each digestion tube. Turn water to vacuum on low.

9.2.2. Digest for 2 h.

9.2.2.1. Be sure that the manifold and hood fan are operating properly to remove the fumes released in the digestion process.

9.2.3. Remove tubes from digestion block with tongs and transfer to cooling tray to cool, leaving the exhaust manifold in place for a minimum of 30 min after the end of digestion. After 30 min, turn off water to vacuum, and remove exhaust manifold.

9.2.4. Add NH_3-free water to each tube to the level of the top horizontal support on the cooling rack (~50 mL). Place samples in the cooler and cool to <20 ºC.

9.3. Distillation

9.3.1. Place samples on the Model 2300 and select “Kjeldahl 5”. This selection ensures that the instrument will dispense a sufficient amount of NaOH to the sample.

9.3.1.1. Use 0.020 N H_2SO_4 for low-N samples, and 0.25 N for all others. Most samples associated with livestock manure, feed, and bedding are assumed to require 0.25 N H_2SO_4.

9.4. Calculations

9.4.1. Calculate TKN (in units of mg/g) in the sample as follows, correcting the amount of titrant (0.25N H_2SO_4) added to each sample by subtracting that needed to titrate a blank to endpoint:

$$\mathrm{TKN} = \frac{\mathrm{Titrant_{sample}\,(mL) - Titrant_{blank}\,(mL)}}{\mathrm{Sample\ wt\ (g)}} \times 3{,}500^{*}$$

*If 0.25N H2SO4 is used in the distillation/titration step. If 0.02 N acid is used, replace 3,500 with 280.

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
10.2. Manage all data according to SOP B5.
10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstirke errors in writing with single lines.
10.4. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Reagent Purity
  11.1.1. Use only NH_3-free water in this method. See Step 6.1 for the methodology used to remove ammonia from water for use in these procedures.
  11.1.2. All other reagents will be ACS Reagent Grade or better.
11.2. Instrument Calibration and Standardization
  11.2.1. Calibrate analytical balance annually (at a minimum).
    11.2.1.1. Calibrate at 2 mg and 1000 mg using class “S” weights.
    11.2.1.2. Instrument calibration must be within ±10% at 2 mg (i.e. ±0.2 mg) and ±0.5% at 1000 mg (i.e. ±5 mg). If these limits are not achieved, the balance must be re-calibrated.
    11.2.1.3. Place a 50-g weight and the 2-mg weight on the balance together. Verify that the balance reads 50.002 g (±0.0002 g).
11.3. Initial Demonstration of Analyst & Laboratory Proficiency
  11.3.1. The following steps are to be followed by every new analyst performing this method, in order to assure the quality of his/her technique. No analyst may perform analyses of actual samples until he/she has passed all of the following:
    11.3.1.1. Determine Method Detection Limit (MDL)
      11.3.1.1.1. Prepare a group of at least seven (7) MDL determination samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) (Section 8.10) with 10 mL NH_3-free water, and spiking each with nicotinic acid standard (Section 8.9.2) such that the organic N concentration of each MDL sample is one to five times the estimated detection limit (Section 1.4.2).
      11.3.1.1.2. Using the seven replicates; process each through every step of the method. Calculate the N concentrations (Section 9.4.1) for each aliquot, and compare with the expected concentrations.
      11.3.1.1.3. If, on average, the calculated concentrations are within 20% of the expected concentrations, analyst and system performance are acceptable .
    11.3.1.2. Initial Precision and Recovery (IPR)
      11.3.1.2.1. Prepare a group of at least four (4) IPR samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) with 10 mL NH_3-free water, and spiking the slurries with nicotinic acid standard so that the organic N concentration of each PR sample is on the order of 1%. Process each sample through every step of the method. Perform the TKN calculations in Section 9.4.1, and determine

the percent recovery by comparing these to the known input amount.

11.3.1.2.2. Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation (s) of the percent recovery for TKN.

11.3.1.2.3. The required performance specifications are 110% ≥ X ≥ 90%, and s ≤ 5%.

11.3.1.2.4. If X and s meet the acceptance criteria, analyst and system performance are acceptable..

11.3.1.2.5. If s exceeds the precision limit (5%), and/or X falls outside the range for recovery, analyst and/or system performance is unacceptable. In this event, correct the problem, and repeat the test.

11.3.1.3. Blank

11.3.1.3.1. Include a minimum of one blank (Section 11.9) to verify that nitrogen is not detected in the blank at a concentration greater than the aqueous or solid MDL (Section 11.4.1.1).

11.3.1.4. Linearity of calibration curve

11.3.1.4.1. Analyst will prepare a five-point calibration curve (Section 8.9.3) to verify his/her ability to generate linear results over the concentration range of interest. If the linear correlation coefficient (r^2) of the calibration curve is ≥0.96, analyst and system performance are acceptable.

11.3.1.5. CV Standard

11.3.1.5.1. Include a minimum of one CV Standard (Section 8.9.4) to verify that the analyst can meet the acceptance criteria outlined in Section 11.6.1.1.

11.4. Ongoing Precision and Recovery (OPR)

11.4.1. MDL

11.4.1.1. Re-determine/verify MDLs annually.

11.4.1.2. Re-determine/verify MDLs whenever a modification is made to the method or analytical system.

11.4.2. Precision and Recovery (PR) samples

11.4.2.1. Each batch of samples (Section 3.10) must contain a minimum of one PR sample. Run solid samples with solid PR samples, and slurry samples with slurry PR samples.

11.4.2.1.1. Prepare a solid PR sample by spiking 5 g of blank sand (or diatomaceous earth) with nicotinic acid standard such that the organic N concentration of the PR sample is approximately five times the MDL (Section 1.4.2).

11.4.2.1.2. Prepare a slurry PR sample by mixing 5 g of blank sand (or diatomaceous earth) with 10 mL NH_3-free water, and spiking the slurry with nicotinic acid standard such that the organic N concentration of the PR sample is approximately five times the MDL (Section 1.4.2).

11.4.2.2. Carry the spiked PR sample through the entire analytical process, and calculate percent recovery.

11.4.2.2.1. If recovery falls within 90 to 110%**,** the system performance is acceptable.

11.4.2.2.2. If recovery falls outside 90% to 110%, system performance is unacceptable, and the problem must be identified and resolved prior to further analyses.

11.4.2.3. All samples must be associated with an acceptable PR standard before their results may be reported.

11.5. Calibration curves

11.5.1. Generate a new calibration curve (containing a minimum of five points as described in Section 8.9.3) whenever a new standard is obtained.

11.6. Calibration Verification (CV) standards

11.6.1. A Calibration Verification Standard (solid or slurry, as appropriate) must be run once per sample batch (every twenty analyses – Section 3.10).

11.6.1.1. Acceptance criteria for the CV Standard is 100% ± 5%.

11.6.1.2. If the CV does not meet acceptance criteria, the problem must be identified and corrected, including possible recalibration of the system if the problem cannot be corrected by other means.

11.6.1.3. All samples must be associated with an acceptable CV Standard before their results may be reported.

11.7. Quality Control standards

11.7.1. In accordance with Method 1687, analyze a QC standard with each day's distillations, or every 12 h, whichever is more frequent.

11.8. Matrix Spikes

11.8.1. To assess the performance of the method on a given sample matrix, spike (in duplicate) a minimum of 5% (1 sample in 20) of the samples from a given site.

11.8.2. Ensure that the concentration of nitrogen spiked into each MS sample is 1-5 times the background (i.e. existing) concentration expected for that particular sample.

11.8.2.1. The spiking level may be established *a priori* based on existing information (either analyst's previous experience or existing literature values) for N content of manures from the particular species.

11.8.3. The concentration added should be expressed as mg/kg, and should be calculated for a 1-g aliquot by multiplying the N concentration (mg/L) added (as nicotinic acid) by the conversion factor 100 (mg/L x 0.1L/0.001kg = 100).

11.8.4. Do not use blanks for MS analysis.

11.8.5. Assessing spike recovery

11.8.5.1. Calculate the percent recovery of TKN in the duplicate matrix spike samples (corrected for the background concentration measured in the corresponding unspiked sample which was used as the basis for the MS samples), as well as the relative percent difference (RPD) between the two values.

11.8.5.1.1. Percent recovery is calculated as follows:

$$\text{Percent Recovery} = \frac{(C_s - C_b)}{S} \times 100$$

Where:
C_s = Sample N concentration determined after spiking
C_b = Sample N concentration determined before spiking
S = Known concentration of the spike

11.8.5.1.2. RPD is calculated as follows:

$$\mathrm{RPD} = \frac{(|D_1 - D_2|)}{D_1 + D_2} \times 100$$

Where:
D_1 = TKN concentration of MS sample #1
D_2 = TKN concentration of MS sample #2

11.8.5.2. Percent recoveries for the matrix spikes should be within three (3) standard deviations above or below the mean, with RPD ≤ 20%.

11.8.5.3. If either the percent recovery or the RPD of TKN in the MS samples falls outside the designated range, while the percent recovery in the PR sample is within the acceptable range, the problem encountered with the MS sample is judged to be matrix-related instead of method-related.

11.9. Blanks

11.9.1. Run a minimum of one blank with each batch of samples (Section 3.10).

11.9.2. Run aqueous (slurry) samples with an aqueous blank (10 mL NH_3-free water), and solid samples with a solid blank (a 5-g aliquot of blank sand).

11.9.3. If material is detected in the aqueous or solid blank at a concentration greater than the MDL (Section 1.4.2), halt analysis of samples until the source of contamination is eliminated and a new blank shows no contamination.

11.9.4. All samples must be associated with an uncontaminated laboratory blank before their results may be reported.

11.10. QC Charts

11.10.1. Track all IPR, OPR, Calibration Verification, QC Standard, MS and Blank results to provide graphical representations of continued laboratory performance.

11.11. Method modifications & re-validation

11.11.1. If any modification is made to this method, verify that the modified method meets or exceeds the Method Detection Limit (11.3.1.1) and Initial Precision and Recovery (11.3.1.2) achieved by the same analyst using the original (i.e. unmodified) method.

11.11.2. Maintain records of any modifications made to the method. At minimum, these records must include the following:

11.11.2.1. Names, titles, and contact information for analyst who initiated the modifications and for the QA Officer who witnessed and approved them

11.11.2.2. Narrative stating reason(s) for the modification

11.11.2.3. Results from all QC tests comparing the modified method's performance to that of the original method:

- Initial Precision and Recovery
- Ongoing Precision and Recovery
- Results with CV, QC Standard, MS samples and Blanks

11.12. Duplication

11.12.1. Run a minimum of 10% of samples (1 in 10) in duplicate, with a minimum of one duplicate per batch. Replicates must agree to within 10% of their average TKN content; if not, the batch of samples must be re-run.

12. References

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DETERMINING AMMONIA CONTENT OF MANURE SAMPLES
Standard Operating Procedure (SOP) M5

DETERMINING AMMONIA CONTENT OF MANURE SAMPLES

Standard Operating Procedure (SOP) M5

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Effective Date: December 28, 2007

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1. Scope and Applicability

1.1. The composition of manure can significantly affect its emissions, both in terms of general odor and individual chemical components. The ammonia content of a manure is generally a good indicator of readily available nitrogen, and therefore affects emission of both ammonia and other nitrogen-containing compounds.

1.2. This method determines a manure sample's total ammoniacal nitrogen (NH_3-N) content.

1.3. The distillation and titrimetric analyses are described in USEPA Method 350.2 and elsewhere (Bremner and Keeney 1965).

1.3.1. This method has a working range of approximately 0.5-10 mg NH_3 per g of manure (wet weight basis), and a minimum detection limit of approximately 0.1 mg/g.

1.3.2. The digestion and distillation method described in this SOP has been scaled down to "micro-Kjeldahl" scale in accordance with Reference 12.2.

2. Summary of Method

The ammonia in a manure sample is distilled away from the rest of the sample, at which point it is captured in a dilute boric acid solution which contains a bromocresol green methyl red indicator. The ammonia concentration of the distillate is then determined by titration with a known concentration of sulfuric acid. A quality assurance/quality control (QA/QC) section is provided to discuss the calibration and periodic testing of the analytical method to ensure that reliable data are collected.

3. Definitions

3.1.	CV	Calibration verification
3.2.	IPR	Initial precision and recovery
3.3.	MDL	Method detection limit
3.4.	MgO	Magnesium oxide
3.5.	MS	Matrix spike
3.6.	NaOH	Sodium hydroxide
3.7.	OPR	Ongoing precision and recovery
3.8.	PR	Precision and recovery
3.9.	QA/QC	Quality assurance/quality control
3.10.	RPD	Relative percent difference
3.11.	Sample batch	A group of sample analyzed at the same time, up to a maximum of twenty (20). If more than 20 samples are analyzed at a time, they must be divided into multiple batches for including blanks and QA standards.
3.12.	SOP	Standard operating procedure

4. Health and Safety

4.1. Treat each chemical compound as a potential health hazard, and reduce exposure to chemicals to the lowest possible level.

4.1.1. Wear protective clothing (gloves, lab coats, protective eyewear) when working with chemicals.
4.1.2. Conduct all operations involving solvents in properly-ventilated fume hoods.
4.1.3. Keep analytical balances and other work areas clean; deal with spills promptly.
4.1.4. When using concentrated acids and bases, keep a fully-stocked spill kit (including suitable neutralizers and absorbents) in the work area at all times.

4.2. Maintain a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method.

4.3. Maintain a reference file of material safety data sheets (MSDS), which will be available to all personnel involved in these analyses.

5. Cautions

5.1. Considerable heat is generated when dissolving sodium hydroxide (NaOH) in water, or when mixing acid with water. Cool the container in an ice bath when preparing NaOH solutions, or when mixing acid with water.

5.2. Excessive foaming during distillation can cause foam to pass into the condenser, which can contaminate the receiving flask and condenser with alkaline salts. Treat heavily-foaming samples with an anti-foaming agent.

6. Interferences

6.1. Keep samples at or below 4°C after collection and during shipping (SOP M1), and prior to analysis, to minimize microbial activity. Analyze all samples within 7 days of collection. Frozen (archived) samples can be maintained indefinitely.

6.2. Use only ammonia-free water, prepared by passing reagent water through an ion-exchange column packed with strongly acidic cation- and strongly basic anion-exchange resins. Regenerate this column periodically according to the manufacturer’s instructions.

6.3. Ammonia gas in the lab can deposit into the sample. Minimize sample exposure to air.

6.4. Nitrate in large quantities (>10 mg/L) can oxidize ammonia in the sample and introduce negative error. Although this interference cannot be prevented, its effect can be predicted and accounted for on the basis of preliminary nitrate determination of the sample. Whenever high nitrate levels are a potential issue, conduct nitrate analyses of the sample before attempting an ammonia analysis.

6.5. Volatile nitrogen-containing compounds such as hydrazine and some amines can cause overestimates of ammonia content. The levels of these in manure should be negligible.

6.6. Any residual Cl^- in the sample must be removed using sodium thiosulfate. This is most often associated with aqueous samples, or samples containing free water, and generally does not affect manure/biosolid samples.

7. Personnel Qualifications

7.1. Personnel should be trained in conducting these analyses before initiating the procedure.

7.2. Each analyst must read and understand this SOP, SOP M3, and USEPA Methods 351.3

and 1687 before initiating this procedure, and must show the ability to generate acceptable data accuracy and precision (Section 11.3).

8. Equipment and Supplies

8.1. Copies of PAAQL SOP M3 and USEPA Methods 351.3 and 1687
8.2. Foss Kjeltec Model 2300 automated distillation and titration analyzer (FOSS Analytical, Hilleroed, Denmark), or equivalent
8.3. Flasks: 100-ml Kjeldahl and 50-ml Erlenmeyer flasks
8.4. Analytical balance (Capacity = 410 g; Accuracy = 0.001 g)
  8.4.1. Reference weights for calibration: 2-mg, 1000-mg, and 50-g class “S” weights
8.5. Reagents and buffers
  8.5.1. Sulfuric acid: Concentrated, 0.020N & 0.25N H_2SO_4, certified (Midland Scientific, Omaha, NE)
  8.5.2. Sodium hydroxide: 40% NaOH solution, certified (Midland Scientific, Omaha, NE)
  8.5.3. Boric acid indicator:
    8.5.3.1. Add 0.10 g bromocresol green, 0.10 g methyl red, 100 g boric acid, and 20 mL of 0.10N NaOH to a 2-L flask that is half-full of NH_3-free water. Dissolve all reagents
    8.5.3.2. Add NH_3-free water to the 2-L mark on the flask.
    8.5.3.3. Quantitatively transfer flask contents to the receiving solution reservoir below the Foss Kjeltech Model 2300.
    8.5.3.4. Add 8 more liters of NH_3-free water to the reservoir, for a total of 10 L of solution.
8.6. Standards
  8.6.1. Ammonia standards
    8.6.1.1. Ammonia stock solution (1000 mg/L NH_3-N):
      8.6.1.1.1. Dry anhydrous ammonium chloride (NH_4Cl) at 105°C immediately before use.
      8.6.1.1.2. Dissolve 0.382 g of this dried NH_4Cl in NH_3-free water and dilute to 100 mL with NH_3-free water in a volumetric flask.
    8.6.1.2. Ammonia working standard (10 mg/L NH_3-N): Dilute 10 mL of ammonia stock solution to 1 L in a volumetric flask with NH_3-free water.
  8.6.2. Calibration standards
    8.6.2.1. Prepare calibration standards by spiking 5-g aliquots of blank sand or diatomaceous earth (Section 8.7) directly with ammonia standard solution (Section 8.6.1).
    8.6.2.2. Prepare a minimum of five calibration standards, at five different concentrations to produce a calibration curve with at least five points.
    8.6.2.3. At least one of the calibration standards must correspond to a sample concentration at or below that necessary to meet the data quality objectives (DQOs) of the project. The QAPP for the particular project will state what concentration meets the DQOs.

8.6.2.4. The remaining standards should correspond to the concentration range found in actual samples, without exceeding the titration-based system's working range.

8.6.3. Calibration Verification (CV) standard

8.6.3.1. Prepare the CV standard so its NH_3 concentration approximates the midpoint of the calibration curve.

8.6.3.2. Prepare solid CV standards for use with solid samples, and slurry CV samples for use with slurry samples.

8.6.3.3. The source used to prepare the CV standard should be different from that used to prepare the calibration standards. Use either a different bottle of the same compound, or a different NH_3-containing compound altogether.

8.6.3.4. If a different ammonium compound is used for the CV stock, adjust the amount weighed according to the ratio of NH_3-N atomic weight to molecular weight. Ammonium chloride has one NH_4 group per molecule and a formula weight of 53.49; it is, therefore 31.78% NH_3 by weight (17/53.49). If, for example, dibasic ammonium phosphate (2 NH_4 per molecule, formula weight 132.06, 25.75% NH_3) is used as the CV standard, and X mg of ammonium chloride was needed to yield the NH_3 concentration at the midpoint of the calibration curve, then (31.78/25.75) X, or 1.23 X g of dibasic ammonium phosphate would be needed to yield the same NH_3 concentration.

8.6.4. Quality Control Sample (QCS): A prepared quality control sample from a standards vendor. Environmental Resource Associates (Arvada, CO) Catalog #545, or equivalent.

8.7. Blank sand or diatomaceous earth

8.7.1. Bake 500 g diatomaceous earth or clean sand at 400 °C for 8 h. Cool and store in a glass container with a sealing lid.

9. Procedures

9.1. Sample Preparation

9.1.1. Obtain percent total solids of each sample, according to SOP M3.

9.1.2. Label Kjeldahl distillation flasks as necessary to accommodate samples, in duplicate, as well as all necessary duplicates, Precision and Recovery Samples, Calibration Verification Standards, Quality Control Standards, Matrix Spikes, and/or Blanks (See Sections 11.4 - 11.9 for the frequency that these must be included).

9.1.3. Add sample.

9.1.3.1. Solid samples: Bring to room temperature. Thoroughly homogenize the sample. Weigh 1-2 g of sample into the Kjeldahl flask.

9.1.3.2. Slurry samples: Bring to room temperature. Mix the sample thoroughly. Transfer (by pipette or spatula) 1-2 mL (or 1-2 g) of sample to the distillation flask, and record the weight of the added sample.

9.2. Distillation

9.2.1. Place samples on the Model 2300 and select "Kjeldahl 5". This selection ensures that the instrument will dispense a sufficient amount of NaOH to the sample.

9.2.1.1. Use 0.020 N H_2SO_4 for low-N samples, and 0.25 N for all others. Most samples associated with livestock manure will require 0.25 N H_2SO_4.

9.3. Calculations

9.3.1. Calculate NH_3-N (in units of mg/g) in the sample as follows, correcting the amount of titrant (0.25N H_2SO_4) added to each sample by subtracting that needed to titrate a blank to endpoint:

$$NH_3\text{-}N = \frac{\text{Titrant}_{\text{sample}}\,(\text{mL}) - \text{Titrant}_{\text{blank}}\,(\text{mL})}{\text{Sample wt (g)}} \times 3{,}500^*$$

*If 0.25N H2SO4 is used in the distillation/titration step. If 0.02 N acid is used, replace 3,500 with 280.

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.

10.2. Manage all data according to SOP B5.

10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line.

10.4. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Reagent Purity

11.1.1. All water used in this method must be NH_3-free. See Step 6.1 for the methodology used to remove NH_3 from water for use in these procedures.

11.1.2. All other reagents will be ACS Reagent Grade or better.

11.2. Instrument calibration and standardization

11.2.1. Calibrate the analytical balance annually (at a minimum).

11.2.1.1. Calibrate at 2 mg and 1000 mg using class “S” weights.

11.2.1.2. Instrument calibration must be within ±10% at 2 mg (i.e. ±0.2 mg) and ±0.5% at 1000 mg (i.e. ±5 mg). If these limits are not achieved, the balance must be re-calibrated.

11.2.1.3. Place a 50-g weight and the 2-mg weight on the balance together. Verify that the balance reads 50.002 g (±0.0002 g).

11.3. Initial demonstration of analyst & laboratory proficiency

11.3.1. The following steps are to be followed by every new analyst performing this method, in order to provide an assurance of the quality of his/her technique. No analyst may perform analyses of actual samples until he/she has passed all of the following:

11.3.1.1. Determine Method Detection Limit (MDL)

11.3.1.1.1. Prepare a group of at least seven (7) MDL determination samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) (Section 8.7) with 10 mL NH_3-free water, and spiking each with ammonia standard (Section 8.6.1) such that the organic NH_3 concentration of each MDL sample is one to five times the estimated detection limit (Section 1.3.1).

11.3.1.1.2. Using the seven replicates; process each through every step of the method. Calculate the NH_3 concentrations (Section 9.3) for each aliquot, and compare with the expected concentrations.

11.3.1.1.3. If, on average, the calculated concentrations are within 20% of the expected concentrations, analyst and system performance are acceptable.

11.3.1.2. Initial Precision and Recovery (IPR)

11.3.1.2.1. Prepare a group of at least four (4) IPR samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) with 10 mL NH_3-free water, and spiking the slurries with ammonia standard such that the NH_3 concentration of each PR sample is on the order of 1%. Process each sample through every step of the method. Perform the % NH_3 calculations in Section 9.3, and determine the percent recovery by comparing these to the known input amount.

11.3.1.2.2. Using the results of the four analyses, compute the average percent recovery (X) and the standard deviation (s) of the percent recovery for ammonia.

11.3.1.2.3. The required performance specifications for PR samples are a recovery of $110\% \geq X \geq 90\%$, and precision $(s) \leq 5\%$.

11.3.1.2.4. If s and X meet the acceptance criteria, analyst and system performance are acceptable.

11.3.1.2.5. If s exceeds the precision limit (5%), and/or X falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem, and repeat the test.

11.3.1.3. Blank

11.3.1.3.1. Analyst will include a minimum of one blank (Section 11.9) to verify that he/she does not detect NH_3 in the blank at a concentration greater than the stated MDL (Section 1.3.1).

11.3.1.4. Linearity of calibration curve

11.3.1.4.1. Analyst will prepare a five-point calibration curve (Section 8.6.2) to verify his/her ability to generate linear results over the concentration range of interest. If the linear correlation coefficient (r^2) of the calibration curve is ≥ 0.96, analyst and system performance are acceptable.

11.3.1.5. CV Standard

11.3.1.5.1. Analyst will include a minimum of one CV Standard (Section 8.6.3) to verify that he/she can meet acceptance criteria (Section 11.6.1.1).

11.4. Ongoing Precision and Recovery (OPR)

11.4.1. MDL

11.4.1.1. Re-determine/verify method detection limits annually.

11.4.1.2. Re-determine/verify method detection limits whenever a modification is made to the method or analytical system.

11.4.2. Precision and Recovery (PR) Samples

11.4.2.1. Each batch (up to 20 samples) (Section 3.11) must contain a minimum of one PR sample. Run solid samples with solid PR samples, and slurry samples with slurry PR samples.

11.4.2.1.1. Prepare a solid PR sample by spiking 5 g of blank sand (or diatomaceous earth) with ammonia working standard such that the NH_3 concentration of the PR sample is approximately five times the MDL (Section 1.3.1).

11.4.2.1.2. Prepare a slurry PR sample by mixing 5 g of blank sand (or diatomaceous earth) with 10 mL NH_3-free water, and spiking the slurry with amoonia working standard such that the NH_3 concentration of the PR sample is approximately five times the MDL (Section 1.3.1).

11.4.2.2. Carry the PR sample through the entire analytical process, and calculate percent recovery.

11.4.2.2.1. If recovery falls within the range of 90% to 110%**,** the system performance is acceptable.

11.4.2.2.2. If recovery falls outside the range of 90% to 110%, the system performance is unacceptable, and the problem must be identified and resolved before performing any further analyses.

11.4.2.3. All samples must be associated with an acceptable PR standard before their results may be reported.

11.5. Calibration curves

11.5.1. Generate a new calibration curve (containing a minimum of five points as described in Section 8.6.2) whenever a new standard is obtained.

11.6. Calibration Verification Standards

11.6.1. Run a Calibration Verification Standard, either solid or slurry as appropriate based on the sample matrix, once per sample batch (every 20 analyses – Section 3.11).

11.6.1.1. Acceptance criteria for the CV Standard is 100 % ± 10%.

11.6.1.2. If the CV does not meet the acceptance criteria, the problem must be identified and corrected, including possible recalibration of the system if the problem cannot be corrected by other means.

11.6.1.3. All samples must be associated with an acceptable CV Standard before their results may be reported.

11.7. Quality Control (QC) Standards

11.7.1. In accordance with Method 1687, analyze a QC standard with each day's distillations, or every 12 h, whichever is more frequent.

11.8. Matrix Spikes (MS)

11.8.1. To assess the performance of the method on a given sample matrix, spike, in duplicate, a minimum of 5% (one sample in 20) of the samples from a given sampling site.

11.8.2. The concentration of NH_3-N spiked into each MS sample shall be 1-5 times the background concentration for the corresponding batch of samples.

11.8.2.1. The spiking level may be established *a priori* based on existing information (either analyst's previous experience or existing literature values) for NH_3 content of manures from the particular species.

11.8.3. The concentration added should be expressed as mg/kg, and should be calculated for a 1-g aliquot by multiplying the N concentration (mg/L) added (as ammonium chloride) by the conversion factor 100 (mg/L x 0.1L/0.001kg = 100).

11.8.4. Do not use blanks for MS analyses.

11.8.5. Assessing spike recovery

11.8.5.1. Calculate the percent recoveries of NH_3-N and relative percent difference (RPD) of the duplicate matrix spike samples (corrected for the background concentration measured in the corresponding unspiked sample which was used as the basis for the MS samples).

11.8.5.1.1. Calculate percent recovery as follows:

$$\text{Percent Recovery} = \frac{(C_s - C_b)}{S} \times 100$$

Where:
C_s = Sample NH_3-N concentration determined after spiking
C_b = Sample NH_3-N concentration determined before spiking
S = Known concentration of the spike

11.8.5.1.2. Calculate RPD as follows:

$$\text{RPD} = \frac{(|D_1 - D_2|)}{D_1 + D_2} \times 100$$

Where:
D_1 = NH_3-N concentration of MS sample #1
D_2 = NH_3-N concentration of MS sample #2

11.8.5.2. Percent recoveries for the matrix spikes should be within three standard deviations of the mean value, with RPD $\leq$ 20%.

11.8.5.3. If either the percent recovery or the RPD of NH_3-N in the MS samples falls outside the designated range, while the percent recovery in the PR sample is within the acceptable range, the problem encountered with the MS sample is judged to be matrix-related instead of method-related.

11.9. Blanks

11.9.1. Run a minimum of one blank with each batch of 20 samples (Section 3.11).

11.9.2. Suitable blanks are 10 mL of NH_3-free water, or 5 g of blank sand.

11.9.3. If NH_3-N is detected in the blank at a concentration greater than the MDL stated in Section 1.3.1, halt analysis of samples until the source of contamination is eliminated and a new blank shows no contamination.

11.9.4. All samples must be associated with an uncontaminated laboratory blank before their results may be reported.

11.10. QC Charts

11.10.1. Track all IPR, OPR, Calibration Verification, QC Standard, MS and Blank results to provide graphical representations of continued laboratory performance.

11.11. Method Modifications & Re-validation

11.11.1. Each time any modification is made to this method, the analyst will verify that the modified method meets or exceeds the Method Detection Limit and Initial Precision and Recovery achieved by the same analyst using the original (unmodified) method.

11.11.2. The laboratory will maintain records of any modifications made to the method. At minimum, these records must include the following:

11.11.2.1. Names, titles, and contact information for analyst who initiated the modifications and for the QA Officer who witnessed and approved them

11.11.2.2. Narrative stating reason(s) for the modification

11.11.2.3. Results from all QC tests comparing the modified method's performance to that of the original method:

- Initial Precision and Recovery
- Ongoing Precision and Recovery
- Results with Calibration Verification, QC Standard, MS samples and Blanks

11.12. Replication

11.12.1. Run duplicates of 10% of the samples in each batch, with a minimum of one duplicate per batch. Initial masses (i.e. wet weights) of the duplicates will not differ by more than 10%. Replicates must agree to within 10% of their average NH_3 content; if not, the batch of samples must be re-run.

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EMISSIONS ESTIMATION USING THE THUNDER BEACH SCIENTIFIC WINDTRAX® BACKWARD LAGRANGIAN STOCHASTIC MODEL
Standard Operating Procedure (SOP) O1

EMISSIONS ESTIMATION USING THE THUNDER BEACH SCIENTIFIC WINDTRAX® BACKWARD LAGRANGIAN STOCHASTIC MODEL

Standard Operating Procedure (SOP) O1

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1. Scope and Applicability

1.1. This protocol describes backward Lagrangian Stochastic (bLS) methods designed to estimate mass flux of air pollutants from an open source, based on concentrations measured along a horizontal line within a vertical plane downwind from the source

1.2. The bLS method has been evaluated, demonstrated, and peer-reviewed for measuring emissions of air pollutants from open sources (See Appendix B).

1.3. The bLS methodology is appropriate for characterizing ground-level area sources and non-point fugitive emission sources of NH_3 and H_2S, such as lagoons and manure storages.

1.4. The bLS method requires both downwind and upwind path-integrated concentrations (PICs) of the contaminants in question, turbulence, and other meteorological data to calculate emission rates.

1.4.1. The bLS methodology described here uses two open-path methods: Ultraviolet Differential Optical Absorption Spectroscopy (UV-DOAS) in a single beam configuration (SOP C1), and Tunable Laser Diode Absorption Spectroscopy (TLDAS) in multiple beam configurations (SOP C2), to obtain the PIC data.

1.4.2. Background PIC (PIC_{bg}) is defined as the lowest PIC measured among upwind beamlines.

1.5. The limitations of this methodology can be instrument- or geometry-based.

1.5.1. Instrument-Specific Limitations: The UV-DOAS and TDLAS systems' maximum pathlength, compounds which can be detected, and detection limits are described in SOP C1 (UV-DOAS) and SOP C2 (TDLAS).

1.5.2. Geometry Limitations: Plume emissions are assumed to originate near ground level (within 3 m from the ground).

1.6. The measurement data consists of 5-min averages, and the bLS models will be uniquely configured for each measurement location prior to sampling.

1.7. The level of acceptable uncertainty in emission rates is +/- 30%.

2. Summary of Method

The commercially-available program WindTrax® will apply the bLS method to determine emissions of H_2S and NH_3 from area sources (e.g. lagoons, corrals). WindTrax® simulates short-range atmospheric dispersion (for horizontal distances within about 1 km of the source) based on measured turbulence statistics. The model has an easy-to-use graphical interface for assessment of turbulence transport on the micro-meteorological scale using Lagrangian stochastic particle models. The simulation elements are represented by icons on a graphical interface showing a plan-view map with the location of sources and sensors at each location. Data files and the underlying numerical models are also represented by icons on the map. The turbulence statistics are based on 16-Hz measurements from the 2.0-m height sonic anemometer described in SOP W2 and SOP U5.

In applying the bLS method, the WindTrax® software makes three significant assumptions: 1) The ground surface around the source is assumed to be horizontally homogeneous; 2) Turbulence is assumed to be homogeneous and stationary; and 3) The PIC measurements for upwind and downwind components are assumed to be contemporaneous.

3. Definitions

3.1. Beamline	Path along which a PIC is determined
3.2. bLS	backward Lagrangian Stochastic. A Lagrangian modeling approach in which the position of a parcel of air is based on turbulence statistics backward in time.
3.3. c	Downwind volume concentration of a contaminant of interest (ppm)
3.4. c_{bg}	Upwind (background) volume concentration of a contaminant of interest (ppm)
3.5. C	Downwind mass concentration of a contaminant of interest (mg/m^3)
3.6. C_{bg}	Upwind (background) concentration of a contaminant of interest (mg/m^3)
3.7. DQI	Data quality indicator
3.8. L	Monin Obukov length, m
3.9. |L|	Absolute value of Monin Obukov length, m
3.10. PAML	Purdue Applied Meteorology Laboratory
3.11. PC	Personal computer
3.12. PIC	Path-Integrated Concentration. The integrated concentration of a gaseous pollutant measured along the beam path length, given in units of parts per million-meter (ppm-m).
3.13. PIC_{bg}	Upwind (background) PIC (ppm-m)
3.14. PI-ORS	Path-Integrated Optical Remote Sensing. Instrument system used to acquire gaseous PIC along open optical beam path.
3.15. ppm	Parts per million concentration by volume
3.16. Q	Emission rate, kg/m^2s
3.17. SMP	Site Monitoring Plan
3.18. SOP	Standard operating procedure
3.19. TDLAS	Tunable Diode Laser Absorption Spectroscopy
3.20. Tolerance parameter	A threshold value used to terminate iterative search algorithms of the bLS methodology.
3.21. u_*	Friction velocity, m/s
3.22. UV-DOAS	Ultraviolet Differential Optical Absorption Spectroscopy

4. Cautions

4.1. Take care when collecting turbulence and survey data, as the quality of this data will have a direct impact on the quality of the bLS method’s calculations. Carefully follow the protocols given in SOP W2.

5. Interferences

5.1. Interferences for collecting UV-DOAS and TDLAS data, which can, in turn, affect the quality of bLS modeling, are given in SOPs C1 and C2, respectively.

6. Personnel Qualifications

6.1. Each analyst must read and understand the pertinent sections of the WindTrax® manual and this SOP before operating the software.

6.2. Basic knowledge of the UV-DOAS and TDLAS systems, and the ability to obtain quality PIC data from them, is assumed.

7. Equipment and Supplies

7.1. Computer hardware and software

7.1.1. The PC used for bLS modeling must have, at minimum, a 3-GHz CPU, 1 GB RAM, and a 40-GB hard drive.

7.1.2. Commercial bLS methodology software (WindTrax®, Thunder Beach Scientific, Nanaimo, BC) is used to analyze the acquired PIC and wind data.

8. Procedures

8.1. Setup

8.1.1. Physical configuration of each source location

8.1.1.1. The location of the UV-DOAS and TDLAS systems that provide PIC values are given in the QAPP and individual SMPs.

8.1.1.2. The location of the wind sensor used to define the turbulence and the elevation of the site is documented in SOP U5.

8.1.2. Meteorological measurements

8.1.2.1. Measurements of barometric pressure, air temperature, and turbulence are described in SOP W2 and SOP W6.

8.2. Data acquisition, calculations & data reduction requirements

8.2.1. The program calculates emission rates and downwind concentrations from the arbitrarily shaped lagoon or waste storage area sources using Lagrangian stochastic models for dispersion calculations in backward mode from PI-ORS PIC measurements.

8.2.1.1. The path-integrated concentration measured downwind of the source, *PIC,* and the background value measured upwind of the source, PIC_{bg}, are converted to beamline-averaged mass concentrations by dividing each *PIC* by the corresponding instrument pathlength to produce the volumetric concentrations, c_{obs} and $c_{obs,bg}$, respectively, and then substituting these values into Equation A.1 to obtain C_{obs} and $C_{obs,bg}$.

8.2.1.2. The model calculates the emission rate (Q_{obs}) corresponding to the measured background and downwind beamline-averaged concentrations based on the assumption that $(C_{obs} – C_{obs,bg}) / Q_{obs}$ is only a function of atmospheric conditions. For the measured atmospheric conditions, this ratio is determined by using the bLS model to simulate the flight path of air parcels backwards from the beamline until each parcel intersects the gound (a ‘touchdown’). The modeled ratio is given as C_{sim} / Q_{sim} , where C_{sim} is the simulated averaged beamline concentration, and Q_{sim} is the simulated source strength corresponding to this simulated concentration. The actual emission rate (Q_{obs}) is calculated using:

$$Q_{obs} = \frac{(C_{obs} - C_{obs,bg})}{\left[C_{sim} / Q_{sim}\right]} \qquad \text{(Equation 1)}$$

8.2.2. The velocity of each bLS-simulated air parcel is varied during each model timestep Δt according to

$$\Delta u_k = \alpha_k \Delta t + \beta_k P_k \qquad \text{(Equation 2)}$$

Where:

k is a directional indicator

$\beta_k = (4.41\varepsilon)^{0.5}$ (implying a Schmidt number of 0.64 at neutral stability)

P_k is a Gaussian random number from population with mean 0 and variance Δt

α_k is a function of turbulence statistics, which is given by

$$\alpha_u = -\frac{1}{2(\sigma_u^2\sigma_w^2 - u_*^4)}\beta_u^2[\sigma_w^2(u-U)+u_*^2 w]+w\frac{\partial U}{\partial z},$$

$$\alpha_v = -\frac{1}{2}\beta_v^2\frac{v}{\sigma_v^2},$$

$$\alpha_w = -\frac{1}{2(\sigma_u^2\sigma_w^2 - u_*^4)}\beta_w^2[u_*^2(u-U)+\sigma_u^2 w]+\frac{1}{2}\frac{\partial \sigma_w^2}{\partial z}+\frac{1}{2(\sigma_u^2\sigma_w^2 - u_*^4)}\left[u_*^2\frac{\partial \sigma_w^2}{\partial z}(u-U)w+\sigma_u^2\frac{\partial \sigma_w^2}{\partial z}w^2\right]$$

(Equation 3)

Where:

u_* is the friction velocity

U is the mean velocity in the streamwise direction over the measurement period

u, v and w are the instantaneous components of the wind in the streamwise, cross-stream, and vertical directions respectiuvely.

8.2.3. The location of each parcel ($x_k(u_1, u_2, u_3$ = x, y, z directions) is calculated as

$$\Delta x_k = u_k \Delta t \qquad \text{(Equation 4)}$$

iteratively for a large number of cycles. At some time, all parcels will impact the surface. The location of the intersection of the parcel with the surface is determined, and the vertical velocity at impact with the surface (w_0) is recorded. Parcels rebound from the surface. If the parcel impacts the surface within the source domain area, then it contributes to the flux. If it does not impact the surface within the source domain, the parcel does not contribute to the flux.

8.2.4. The model calculates "touchdown catalogs" of x, y, w_0 for each concentration measurement point *j* along the beamline. The number of impacts within the source domain, weighted by $w_{0,}$ divided by the number of parcels (*n*) released from *P* equidistant points along each beamline, each with concentration C_{sim}, provides the ratio C_{sim} / Q_{sim} for each concentration measurement height:

$$a_{sim} = \frac{\mathrm{C_{sim}}}{\mathrm{Q_{sim}}} = \frac{1}{P}\sum_{j=1}^{P}\left(\frac{1}{\mathrm{n}}\sum_{\text{within source area}}\frac{2}{|w_0|}\right) \qquad \text{(Equation 5)}$$

.

8.2.4.1. The bLS model is run on-location and at PAML.

8.2.4.1.1. Automated on-site modeling will utilize generalized touchdown catalogs.

8.2.4.1.2. Model runs at PAML will be automated and utilize measurement period-specific touchdown catalogs.

8.2.5. Depending on stability and source/sensor geometry, all sensors might measure some part of the source emission, and none are sure to provide the correct background C_{bg}. WindTrax® calculates the actual background concentration, since the number of given concentrations (PIC values) is at least one greater than the number of unknown emission rates (lagoon).

8.2.6. The PIC values (units of ppm-m) are converted to beamline-averaged mass concentrations by dividing by the corresponding instrument pathlength to produce the volumentric concentrations, c_{obs} and $c_{obs,bg}$, respectively, and then substituting these values into Equation A.1 to obtain C_{obs} and $C_{obs,bg}$ (units of mg/m^2), taking into consideration the molecular weight of the target gas, atmospheric pressure and the ambient temperature.

8.2.7. The emission rate (Q_{obs}) , in units of kg/m^{2}s, is calculated using

$$Q_{obs} = \frac{C_{obs} - C_{obs,bg}}{a_{sim}}\left(\frac{1\,kg}{1{,}000{,}000\,mg}\right) \qquad \text{(Equation 6)}$$

8.3. Calibration and standardization

8.3.1. The bLS methodology relies on accurate PIC data provided by the UV-DOAS instruments (SOP C1).

8.3.2. The bLS methodology requires accurate turbulence measurements (SOP W2).

8.4. Computer hardware & software

8.4.1. This method uses the WindTrax® program.

8.4.2. Location-specific configurations, 5-min turbulence and meteorological data, and 5-min average PICs are stored on a desktop computer.

8.4.3. Software is run on a desktop computer according to SOP D1.

9. Data records Management

9.1. The individual 5-min emission rates determined from each beamline and corresponding QC flags (Section 11.3.2) will be written to a file. Manage all data according to SOP D1.

9.2. Document all data and information checked in the lab (PAML) on lab data sheets and in modeling logbooks with permanent ink, or in electronic notes.

9.3. Overstrike errors in writing with a single line. Initial and date all corrections.

10. Quality Assurance and Quality Control

10.1. Data quality indicator (DQI) goals are established to assess the quality of critical measurements. For PI-ORS systems, they depend on the wind measurements and the model specifics. These are summarized in Table 1.

10.1.1. DQIs for UV-DOAS are given in SOP C1.

10.1.2. No fully-documented DQI has been developed for the bLS method. Based on literature evaluations, values of u_* that are >0.15 m/s and values of $|L|$ that are >2 m appear to provide the threshold of method accuracy needed to obtain emission rate measurements at the 30% uncertainty level.

Table 1. DQIs for the bLS Methodology.

Measurement Parameter	**Analysis Method**	**DQI**
U,V,W wind components	Side-by-side comparison of two wind monitors	Within 10%
UVDOAS [NH_3]	Comparison with standard gas	Within 10%
UVDOAS [H_2S]	Comparison with standard gas	Within 10%
bLS	u_*	>0.15 m/s
bLS	$\|L\|$	> 2 m

10.2. Meteorological measurements

10.2.1. The quality assurance of the wind component measurements from the 2-m height anemometer is described in SOP W2.

10.3. Reasonableness of the calculated flux

10.3.1. Verify that the generated result is reasonable based on the raw PIC data.

10.3.2. Assess plume emission measurements for quality using range limits for input variables.

10.3.2.1. Flag measurements with a “1” when $u_* < 0.15$ m/s (calculated from SOP W2). Flagged values are invalidated.

10.3.2.2. Flag measurements with a “2” when $|L| < 2$ m (calculated from SOP W2). Flagged values are invalidated.

10.4. Plume emission measurement accuracy

10.4.1. Plume emission measurement accuracy is expected to be ± 30%.

10.4.2. Accuracy is based in part on least-squares fit and the uncertainty inherent in the coefficients a_{ii} generated by the statistical LS model. Stochastic errors, related to the number of particles released in the model, are estimated at 7%.

10.4.2.1. Corral emission rates are modeled using 1 downwind PIC and 1 source.

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12. Contacts

12.1. Software manufacturer: Thunder Beach Scientific
  12.1.1. Phone: (250) 753-5955
  12.1.2. e-mail: Brian@thunderbeachscientific.com
12.2. Software adaptation: CEREX Environmental Services
  12.2.1. e-mail: David Yu [dhy1332@yahoo.com]

Appendix A
Conversion of Volumetric Gas Concentrations to Gas Density Concentrations

Validated air pollutant concentration measurements are reported in units of parts per billion (ppb). Standard dry concentrations of all gas measurements will be reported but emissions will be calculated using the non-dry (including water vapor partial pressure) air concentrations. As emissions calculations require units of $\mu g/m^3$, all measurements will be converted from ppb to $\mu g/m^3$ at 20^oC.

The ideal gas law (*$PV = nRT$)* is used to convert volumetric gas concentrations to actual and standardized mass concentrations in non-dry air according to:

$$C = \frac{PcM}{R(273.15 + T)} \qquad \text{(Equation A.1)}$$

Where:

c Volumetric concentration, ppb for mass concentration of $\mu g/m^3$
C Actual mass concentration, mg/m^3 or $\mu g/m^3$
M Molecular weight, M=17.03 and 34.08 for NH_3 and H_2S respectively
P Pressure at sampling location, atm
T Temperature at sampling location, °C
R Universal Gas Constant: 0.08206 L-atm/mol-°K

The equation to standardize the volumetric gas concentrations of any pollutant to the standard mass concentration C' is as follows:

$$C' = \frac{P'C^* M}{R(273.15 + T')} \qquad \text{(Equation A.2)}$$

Where:

C' Standard mass concentration moist basis, $\mu g/sm^3$
P' Standard pressure, 1 atm
T' Standard temperature, 20°C

The dry standard concentration is calculated as follows:

$$C'' = \frac{C'}{(1 - W)} \qquad \text{(Equation A.3)}$$

Where:

C'' Dry standard mass concentration dry basis, $\mu g/dsm^3$
W Humidity mixing ratio

The humidity mixing ratio (W) is calculated as:

$$W = 0.62198\frac{e^{f(T)}\varphi}{P_a - e^{f(T)}\varphi} \qquad \text{(Equation A.4)}$$

Where:

W Humidity mixing ratio, kg/kg

P_a Pressure at the sampling location

φ Relative humidity, decimal

$$f(T) = \frac{C_1}{T} + C_2 + C_3T + C_4T^2 + C_5T^3 + C_6T^4 + C_7\ln T \qquad \text{for } T < 273.15°\text{K}$$

$$f(T) = \frac{C_8}{T} + C_9 + C_{10}T + C_{11}T^2 + C_{12}T^3 + C_{13}\ln T \qquad \text{for } T > 273.16°\text{K}$$

$C_1 = -5.675\text{x}10^3$

$C_2 = 6.392$

$C_3 = -9.678\text{x}10^{-3}$

$C_4 = 6.222\text{x}10^{-7}$

$C_5 = 2.075\text{x}10^{-9}$

$C_6 = -9.484\text{x}10^{-13}$

$C_7 = 4.163$

$C_8 = -5.800\text{x}10^3$

$C_9 = 1.391$

$C_{10} = -4.864\text{x}10^{-2}$

$C_{11} = 4.176\text{x}10^{-5}$

$C_{12} = -1.445\text{x}10^{-8}$

$C_{13} = 6.545.$

Appendix B
Evaluations of the Backward Lagrangian Stochastic Emissions Model

1. Accuracy Evaluations: Theoretical

a. Theoretical analysis of the bLS method versus the Integrated Horizontal Flux (IHF) method for estimating emissions from a lagoon under variable surrounding terrain and atmospheric conditions suggests that the bLS method can be expected to estimate the emissions between -14% to +44% of the IHF method under terrain situations of nearby buildings or woods (Wilson et al., 2001). Stability varied from L of -27 to 103 m.

2. Accuracy Evaluations: Field Trials

a. A comparison of calculated NH_3 emissions (passive gas samplers) from small field plots using the bLS method and the IHF method showed the bLS underestimated emissions by 16-24% compared to the IHF estimate, but were not statistically different (Sommer et al., 2005).

b. A comparison of calculated NH_3 emissions from a 3.1-m diameter (1.1-m high) manure pile using bLS (gas measurements by TDLAS) and IHF (passive gas sampler) methods over 24 h showed agreement to within 15% (after assumptions concerning differences in sampling times) (Sommer et al., 2004). Stability varied from z/L of 2.7 to -0.5 and L of 0.37 to -2 m. Sample averaging of 30 min was used.

c. An evaluation of CH_4 emissions from a defined 6 m x 6 m area source using bLS (gas measurements by TDLAS) method was made over 11 roughly two-hour periods over 6 d. Results indicated that bLS method error for 15-min periods ranged from 300% to 20%, while stability varied from L of -407 to 419 m. Two measurement periods during light winds and sunrise resulted in estimation errors of more than 100%. These measurement periods had the lightest winds of all periods, and appeared to indicate a breakdown of model assumptions. Conditions of strong instability ($-2 \text{ m} \leq L \leq 2 \text{ m}$) and low winds ($u^* < 0.15$ m/s) resulted in model failure. Errors in emission estimates during measurement periods with the allowed stability range also varied with stability: A mean 12% overestimation under near-neutral conditions, 13% underestimation under unstable conditions, and 38% overestimation under stable conditions were observed (Flesch et al., 2004). The single best diagnostic for inaccurate Q estimates was found to be $u_* < 0.15$ m/s. When such cases were eliminated, the average value of the ratio $Q_{modeled}/Q_{actual}$ was 0.97, with a standard deviation over the 15-min averaging periods of 0.28. These results show that the bLS method is capable of calculating individual 15-min period values for Q within about 30%. This study also describes placement of the measurement platforms. They must be placed at least several instrument heights away from the upwind edge of the lagoon and other obstructions, but also near enough that the concentration rise over the background can be measured accurately. A problem with this study was the low CH_4 signal/noise ratio of only 2 to 4, which probably resulted in overestimations.

d. An evaluation of CH_4 emissions from a defined 6m x 6m area source surrounded by a 20 m x 20 m fence (flow disturbance) using bLS (gas measurements by TDLAS) method was made over 10 roughly 2-h periods over 4 d. Results indicated an overestimate of emissions by the bLS model of an average of 10%, ranging from 80% overestimation to 49% underestimation. Errors in the near-field measurements (in front of the fence row) ranged from 40% underestimation to 30% overestimation. When the near-fence measurements were eliminated, the results improved so that on average the modeled *Q* values were within 2% of the actual values, with a standard deviation of about 20% for individual 15-min periods. Stability varied from L of -235 to -17 m (Flesch et al., 2005b). The results from this study show that the bLS technique provides reliable estimates for *Q*, even in the presence of some flow obstruction. It is recommended that ambient wind statistics, rather than disturbed, should be used to obtain the best results. A problem with this study is the low CH_4 signal/noise ratio of only 2 to 4, which probably resulted in overestimations.

e. An evaluation of CH_4 emissions (gas measurement by TDLAS) from grazing dairy cows for 3-week periods in winter and summer indicated that the bLS method overestimated the emission (relative to the IHF method) by 5% to 17% when averaging emissions estimates from five TDL scans. They approximated the sources of the error: 1) stochastic model errors were estimated at 1 to 7%, 2) measurement errors of the atmospheric conditions were estimated at 5%, and 3) the error associated with estimating the grazing animal ‘surface roughness’ was considered to be 15%. The model error was on average 19-23% greater for measurements made at high heights with the error of up to 35% when the measurement height was 5m. There was no tendency for the model error (bias) to vary with stability after filtering for $-2\text{m} \leq L \leq 2\text{m}$ or $u^* < 0.15$ m/s (Laubach and Kelliher, 2005).

f. An evaluation of NH_3 emissions (gas measurements by TDLAS) was made from a lagoon over 6 d. The 15-min measurement periods were filtered using the stability range of $-2 \text{ m} \leq L \leq 2 \text{ m}$ or $u^* < 0.15$ m/s (low winds) resulting in a mean emission rate of 1.69 g/s (SD 1.23 g/s). In this study, estimation problems due to trajectories rising above the measurement height resulted in an increase in the stability exclusion range to $-10\text{m} \leq L \leq 10\text{m}$ (extreme instability with measurements made far from the source) and a mean emission rate of 1.44 g/s (SD 0.57 g/s). Use of actual turbulence (as in this SOP) reduced the mean emission estimate 13% to 1.27 g/s (SD 0.49 g/s). Since there was no measure of actual emissions, the reduced emissions estimate may or may not have contributed to greater accuracy of the estimate. Considering only a 72-hr period in March in which no measurement intervals were removed by filtering resulted in a mean emission of 0.9 g/s, corresponding to 6.5 g/animal/day. Similar estimation during July (extrapolated from 15.5 hrs of measurement) resulted in a mean emission of 2.2 g/s, corresponding to 16 g/animal/day (Flesch et al., 2005a).

g. An evaluation of bLS modeling was made using CH_4 emissions from a 3 m x 3 m grid measured by an open-path laser with turbulence data from a three-dimensional sonic anemometer (McBain and Desjardins 2005). Under conditions when Monin Obukhov similarity theory is valid, the modeled Q value was, on average, within 6% of the actual value, with a standard deviation of 0.16. In addition, obstructions upwind from the sensors were determined to have little impact on the Q estimates, so long as the distance between the sensors and the obstructions was at least 25 times the height of the obstructions.

EMISSIONS ESTIMATION USING
THE ARCADIS RADIAL PLUME MAPPING MODEL
Standard Operating Procedure (SOP) O2

EMISSIONS ESTIMATION USING THE ARCADIS RADIAL PLUME MAPPING MODEL

Standard Operating Procedure (SOP) O2

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1. Scope and Applicability

1.1. This SOP describes a method for characterizing gaseous emissions from non-point pollutant sources. The Radial Plume Mapping (RPM) methodology described here uses scanning Path-Integrated Open-Path Tunable Laser Diode Absorption Spectroscopy (TLDAS) in multiple beam configurations to measure emission fluxes. The RPM methodology has been well developed, evaluated, demonstrated, and peer-reviewed.

1.2. The Vertical Radial Plume Mapping (RPM) configuration described here is designed to measure mass flux of pollutants through a vertical plane, downwind from an emission source.

1.3. The PIC data will be generated by measurement with a scanning TDLAS (See SOP C2). The wind profile will be generated by measurements with sonic anemometers (See SOP W2).

1.4. The TDLAS system's maximum pathlength, compounds which can be detected, and detection limits are described in SOP C2.

  1.4.1. TDLAS measurement and RPM mapping are appropriate for NH3 emissions.

  1.4.2. The sensitivity of the TDLAS system is described in SOP C2.

1.5. The RPM methodology described here was developed from studies that concentrated on developing, evaluating, and demonstrating the various configurations of the RPM methodology. The RPM methodology (configurations, procedures, and algorithms) was patented as a technology for mapping air contaminants using a PI-ORS system with a non-overlapping, variable pathlength, radial beam geometry.

1.6. RPM methodology is appropriate for characterizing ground-level area sources and non-point fugitive emission sources, such as lagoons and manure storage areas. Its limitations can be instrument- or geometry-based.

  1.6.1. The primary geometric limitation of this version of the RPM software is that plume emissions are assumed to originate within 3 m from the ground.

1.7. The level of acceptable uncertainty in emission fluxes determined using this method is ± 10%.

2. Summary of Method

This protocol describes application of an RPM method which is designed specifically for use with the TDLAS system. RPM utilizes multiple non-intersecting beam paths in a vertical plane downwind from the emission source to obtain a mass-equivalent plume map. This map, in conjunction with wind speed and direction, is used to obtain the flux of pollutants through the vertical plane. The measured flux is then used to estimate the emission rate of the upwind source being characterized. The TDLAS system collects spectra from airborne contaminants. There are no true "samples" that require preservation, storage, transport, extraction, digestion, or concentration. A vertical scanning plane, downwind of the source, is used to directly measure the gaseous flux (Section 8.3). The total length of the measurement area required depends on the size of the emission source, and is typically between 100 m and 300 m, depending on location (SOP U5). The height of the scanning area is dependent on the 16 m tower and the lateral distance along the side of the manure storage area (SOP U5). It will nominally represent a rectangular area of approximately 200 m by 15 m. The chemical concentration of each gas species of interest, along each beam path, is obtained following the measurement and analysis procedures for the instruments being used. The PIC measurements along the elevated beam paths provide vertical concentration gradient information of the emitted plume. The beam paths on the ground indicate the approximate ground level concentration profile along the length of the RPM setup. Ground-level refers to the area as close to horizontal as possible, relative to the surface of the measurement area. A bivariate Gaussian function is assumed for the plume mass across the RPM plane, and the parameters of the mass-equivalent bivariate Gaussian function(s) are reconstructed from the measured PIC. These reconstructed parameters are then used to calculate the concentration values across the RPM plane at high resolution. The concentration values (in ppm) are converted to mass concentrations using the molecular weight of the monitored gas species, air temperature and atmospheric pressure. The products of the mass concentration and the wind speed normal to the measurement plane are integrated across the plane to calculate the mass flux through the plane. The physical range and sensitivity of the RPM methodology is determined by the limitations of the TDLAS to acquire the PIC data. The accuracy of results from the RPM method depends on the instrument accuracy as well as: 1) the quality control (QC) criteria of the algorithms, and 2) the accuracy and representativeness of the wind data. Because the estimation of the emission rate is dependent on the wind data, stable and measurable wind conditions are desired so that the source remains upwind of the RPM plane. Wind speed and direction measurements will be recorded at 2 m, 4 m, and 16 m (SOP W2).

3. Definitions

3.1.	CCF	Concordance Correlation Factor. CCF is used to represent the quality of fit for the reconstruction in the path-integrated domain. CCF is similar to the Pearson correlation coefficient, but is adjusted to account for shifts in location and scale.
3.2.	Cycle	One complete sequential data collection of the TDLAS system, through all PDCs in the setup.
3.3.	Gaussian function	A normal distribution curve with a specified mean and standard deviation.
3.4.	PDC	Pathlength-defining components. PDC is used to denote the component on the other end of the optical path from the PI-ORS instrument. Depending on the instrument selected, this could be a source, detector, mirror, or other reflecting object.
3.5.	PIC	Path Integrated Concentration. PIC is the integrated concentration (ppm-m) of an analyte measured along an open optical beam path..

3.6.	PI-ORS	Path-Integrated Optical Remote Sensing. An instrument system used to acquire PIC data.
3.7.	ppm	Parts per million. Typical units of gas concentration for TDLAS, ratioed by volume.
3.8.	Retro-reflector	A reflector that returns a beam of light in the same direction from which it came, regardless of the orientation of the reflector relative to the light source. This is the PDC for the TDLAS-based measurements.
3.9.	RPM	Radial Plume Mapping. A methodology involving the acquisition of PIC data over multiple beam paths originating from a single, stationary PI-ORS instrument. Optimization algorithms are applied to give a time-averaged, mass-equivalent plume map (field of concentration) of air pollutants from an area source.
3.10.	SBFM	Smooth Basis Functions Minimization. An algorithm used in the RPM methodology to fit the parameters of the Gaussian basis function(s) to the measured PIC data.
3.11.	SMP	Site monitoring plan.
3.12.	SSE	Sum of Squared Errors. The SSE is the sum of the squared differences between the measured and predicted PIC at each step of the iterative algorithm used in the RPM methodology.
3.13.	TDLAS	Tunable diode laser absorption spectroscopy
3.14.	TDLAS system	The TDLAS, and all associated components, e.g. mirrors, scanners, software, etc.
3.15.	Tolerance parameter	A threshold value used to terminate iterative search algorithms of the RPM methodology.

4. Health and Safety

4.1. The hazards of performing the RPM methodology are those associated with any field event. Safety procedures should be established and implemented before using this protocol.

4.2. TDLAS system-specific safety protocols should be followed (SOP C2).

5. Cautions

5.1. The quality of the results generated with this method depends on the quality of the wind profile measurements (SOP W2), the survey measurements (SOP U5), and the TDLAS system PIC measurements (SOP C2).

6. Interferences

6.1. General Interferences
  6.1.1. Do not allow vehicle or pedestrian traffic to temporarily obstruct the beam path for any PIC measurement.
  6.1.2. Do not allow any physical obstruction of the beam path (e.g., by buildings, trees, and/or complex topography).
6.2. Weather Interferences
  6.2.1. Fog, rain, or snow obscures the light beam and affects the ability of the TDLAS to continuously measure gas concentrations (SOP C2).
  6.2.2. Wind conditions can greatly affect the results of field measurements.
    6.2.2.1. Optimal results are obtained in wind speeds between 1 and 8 m/s.
    6.2.2.2. In very low wind speeds (<0.5 m/s), the source plume may not be carried through the vertical plane.
    6.2.2.3. Very high wind speeds (>11 m/s) may displace or vibrate the optical alignment of various components of the TDLAS retro-reflectors, and affect the quality of the PIC acquired in multiple beam paths.
    6.2.2.4. Data collected in wind speeds between 8 and 11 m/s should be carefully checked for reliability.

7. Personnel Qualifications

7.1. Each member of the field staff must read and understand the pertinent sections of the RPM program instruction manual, SOP D1, and this SOP.
7.2. Specific training in the RPM software, by the software supplier, is required.
7.3. Basic knowledge of the TDLAS system and the ability to obtain quality path-integrated concentration (PIC) data is assumed.

8. Equipment and Supplies

8.1. Concentration Instrumentation. A TDLAS system (SOP C2) will provide the PIC data used in the RPM emissions rate characterization methodology.
8.2. Vertical Structure. Stable towers (SOP U5) are used to ensure reliable PIC data.
8.3. Computer Software
  8.3.1. Use specific software to control the TDLAS and obtain PIC data.
  8.3.2. Use commercial RPM software for post-analysis of PIC and wind data.
  8.3.3. Use custom software for the pre-processing of PIC data and post-processing of RPM data.
8.4. Meteorological Measurements. Wind measurements used in the RPM methodology (SOP W2). See QC checks described in SOP W2.
8.5. Survey Measurements. A GPS unit is used to measure latitude and longitude coordinates of each component. A laser rangefinder is used to measure each pathlength. Azimuth, and elevation angle of each PDC (SOP U5) is computed by the RPM program.

9. Procedures

9.1. RPM configuration setup. Four RPM planes will surround the lagoon or waste pile according to SOP U5 (Fig. 1).
  9.1.1. The primary purpose of the RPM configuration is to calculate the fluxes through the vertical planes of each of the four sides of the lagoon.
  9.1.2. Five retro-reflectors are used to create the vertical plane:
    9.1.2.1. One at 1/3 the distance of the length of the RPM setup (1-m height)
    9.1.2.2. One at 2/3 the distance of the length of the RPM setup (1-m height)
    9.1.2.3. One the full length of the RPM setup (1 m height)
    9.1.2.4. One positioned on the vertical structure at the full length of the RPM setup and at 8 m height
    9.1.2.5. One positioned at the full length of the RPM setup at the top of the vertical structure (15 m)
    9.1.2.6. Ground-level retro-reflectors refer to those defining optical beams as close to horizontal as possible given the terrain of the measurement site.
  9.1.3. Background PIC will be measured from a measurement plane that is upwind during any given measurement period (one of the four RPM planes).

9.2. Data acquisition, calculations & data reduction requirements
  9.2.1. Wind measurements
    9.2.1.1. The orthogonal components to horizontal wind speed are measured at three heights (2 m, 4 m, 16 m) according to SOP W2 and SOP U5.
    9.2.1.2. Wind component measurements are averaged to 2-s values.
    9.2.1.3. The wind profile is interpolated linearly with height from these measurements by the RPM software for each complete cycle.
  9.2.2. TDLAS data acquisition parameters
    9.2.2.1. TDLAS dwell time per retro-reflector is 15 s.
    9.2.2.2. TDLAS time to reposition scanner for next beam path is roughly 5 s.
    9.2.2.3. TDLAS time per complete scan cycle: approximately100 s.
    9.2.2.4. Data collection should proceed regardless of the wind conditions (if safety permits) until a minimum of 1/2 hour of total data is collected during times when the wind meets the criteria defined in Section 6.2.3.
  9.2.3. RPM theory and algorithms: Once the PIC for all beam paths for each cycle of the gas species of interest are determined valid, the RPM model determines the corresponding wind measurements, reconstructs a plume map in the vertical plane, and determines the flux through the reconstructed plane. The RPM plume reconstruction technique utilizes a two-dimensional smooth basis functions minimization (SBFM) to reconstruct the smoothed mass equivalent concentration map (bivariate Gaussian).
    9.2.3.1. A smooth basis function is assumed to describe the distribution of concentrations, and the search is for the unknown parameters of the basis function. Because the goal here is to determine the plane-integrated concentration, and subsequently the flux through the plane (and not the exact map of concentrations in the plane), only one smoothed basis function (one bivariate Gaussian function) is fitted to reconstruct the

smoothed map. For the beam geometry, it is convenient to express the smooth basis function G in polar coordinates r and θ:

9.2.3.2. Six independent beam paths are needed to determine one bivariate Gaussian function with six independent unknown parameters. However, some reasonable assumptions are made to reduce the number of unknown parameters to four.

$$G(r,\theta) = \frac{A}{2\pi\sigma_y\sigma_z\sqrt{1-\rho_{12}^2}} \exp\left\{-\frac{1}{2\left(1-\rho_{12}^2\right)}\left[\frac{(r\cdot\cos\theta - m_y)^2}{\sigma_y^2} - \frac{2\rho_{12}\left(r\cdot\cos\theta - m_y\right)\left(r\cdot\sin\theta - m_z\right)}{\sigma_y\sigma_z} + \frac{\left(r\cdot\sin\theta - m_z\right)^2}{\sigma_z^2}\right]\right\} \qquad (1)$$

Where:

A = Normalizing coefficient, which adjusts for the peak value of the bivariate surface

ρ_{12} = Correlation coefficient, which defines the direction of the distribution-independent variations in relation to the Cartesian directions *y* and *z* (ρ_{12}=0 means that the distribution variations overlap the Cartesian coordinates)

m_y and m_z = Peak locations in Cartesian coordinates

σ_y and σ_z = Standard deviations in Cartesian coordinates.

9.2.3.2.1. To fit the unknown parameters of the smooth basis function to the PIC data, one has to define an error function for minimization.

9.2.3.2.2. Assume that the reconstructed bivariate Gaussian function is limited only to changes in the vertical and crosswind directions. This sets the correlation parameter (ρ_{12}) equal to zero.

9.2.3.2.3. Assume that the ground-level PIC is the largest of the vertical beams. The peak location in the vertical direction is then fixed to the ground level, setting m_z to zero.

9.2.3.3. A two-phase SBFM approach is used to solve the bivariate Gaussian function. First, a one-dimensional SBFM reconstruction procedure is applied to reconstruct the smoothed ground-level and crosswind concentration profiles. Then, the reconstructed parameters are substituted into the bivariate Gaussian function before applying the two-dimensional SBFM procedure.

9.2.3.4. The one dimensional SBFM reconstruction is applied to the ground-level segmented beam paths (Fig. 1) of the same beam geometry to find the crosswind concentration profile (Hashmonay and Yost 1999). A Gaussian function is fitted to measured PIC ground-level values as a univariate function. The error function for the Simplex minimization procedure is the Sum of Squared Errors (SSE) function:

$$SSE(B,m_y,\sigma_y)=\sum_i\left(PIC_i-\frac{B}{\sqrt{2\pi}\sigma_y}\int_0^{r_i}\exp\left[-\frac{1}{2}\left(\frac{m_y-r}{\sigma_y}\right)^2\right]dr\right)^2 \qquad (2)$$

Where:
B = Area under 1-D Gaussian distribution (integrated concentration)
r_i = Pathlength of the i^{th} beam
m_y = Mean peak location
σ_y = Standard deviation of the Gaussian function
PIC_i = Measured path-integrated concentration value of the i^{th} path

9.2.3.5. Substituting the solved values of the standard deviation (σ_y) and peak location (m_y) of the above one-dimensional SBFM procedure into the bivariate SBFM simplified by the assumptions in Section 9.2.4.2 yields:

$$G(A,\sigma_z)=\frac{A}{2\pi\sigma_y\sigma_z}\exp\left\{-\frac{1}{2}\left[\frac{(r\cdot\cos\theta-m_y)^2}{\sigma_y^2}+\frac{(r\cdot\sin\theta)^2}{\sigma_z^2}\right]\right\} \qquad (3)$$

which is solved by minimizing the SSE using the simplex procedure with PIC values for the one long beam along the ground level and the two elevated beams that sample vertical concentration gradients according to:

$$SSE(A,\sigma_z)=\sum_i\left(PIC_i-\int_0^{r_i}G(A,\sigma_z)dr\right)^2 \qquad (4)$$

9.2.3.6. Once the parameters of the function are found for a specific run, the RPM procedure calculates the concentration values for every 2x2 m square elementary unit in the vertical (y,z) domain.

9.2.3.7. The concentration values are converted from ppm by volume to g/m^3, taking into consideration the molecular weight of the target gas, the atmospheric pressure and the ambient air temperature (Appendix A).

9.2.3.8. The wind speed component perpendicular to the vertical plane at each height z ($\hat{u}_z$) is computed by linear interpolations between the three wind sensors (SOP W2 and SOP U5) and setting a constant wind speed above the top wind sensor to 20 m.

9.2.3.9. If the upwind concentration C_{bg} derived from the upwind PIC (PIC_{bg}) is constant for all heights, the the upwind concentration is at ambient levels and the emission flux Q in g/s is determined by:

$$Q=\sum_{y=0}^{pathlength}\sum_{z=0}^{20m}(C_{y,z}-C_{bg})\hat{u}_z \qquad (5)$$

Where:
$C_{y,z}$ = Interpolated concentration in the RPM y,z plane
C_{bg} = Background concentration determined from the minimum PIC in an upwind RPM plane

9.2.3.10. If the upwind concentration C_{bg} is not constant with height, then the upwind concentration is not at ambient levels, and the emission flux (in g/s) across the *d* RPM plane of measurement (Q_d) is determined by:

$$Q_d = \sum_{y=0}^{pathlength} \sum_{z=0}^{20m} (C_{y,z}) \hat{u}_z \qquad (5)$$

Where:

$C_{y,z}$ = Interpolated concentration in the *d* RPM y,z plane

The flux across each measurement plane is calculated separately with fluxes away from the source (downwind plane) as positive values and towards the source (upwind plane) as negative values. The net flux is computed as the sum of the fluxes. Note that the upwind and downwind RPM planes may consist of two planes each when winds are not perpendicular to a face of the area source.

9.2.3.11. The emission flux across each plane is written to a file.

9.3. Calibration and standardization

- 9.3.1. The RPM methodology relies on accurate PIC data provided by the TDLAS. Calibration of the TDLAS is described in SOP C2.
- 9.3.2. The RPM methodology relies on accurate wind speed measurements. Calibration of the wind sensors is described in SOP W2.

9.4. Computer hardware & software

- 9.4.1. This method utilizes a commercially-available, proprietary RPM emissions program in conjunction with custom software to pre-process the concentration data and post-process the emissions data.
- 9.4.2. This method uses location-specific configurations, 2-s mean wind speeds, and PIC values.
- 9.4.3. This method requires the use of proprietary dongles on the USB port of the processing computer.

10. Data and Records Management

10.1. Write the individual emission rates and DQIs (See Section 11.1) to files, as indicated in SOP D1.

10.2. Manage all data according to SOP D1.

10.3. Conduct all processing at PAML, and ensure that information is documented on lab data sheets and in modeling logbooks with permanent ink, or in electronic notes.

10.4. Overstrike any errors in writing with a single line in ink and initial.

11. Quality Control and Quality Assurance

Data Quality Indicator (DQI) goals for critical measurements: DQI goals are established to assess the quality of critical measurements. The DQI goals for this methodology are listed in Table 1.

Table 1. DQIs for the RPM Methodology

Measurement Parameter	Analysis Method	DQI
TDLAS [NH_3]	Comparison with standard gas	Within 10%
Wind speed	Side-by-side comparison of two wind monitors	Within 20%
Plume reconstruction	CCF	≥0.8[a]
Total mass positioning	A[c]	≥0.9[b]
Plume capture	Fractional change in vertical gradient of PIC	Topmost PIC <=0.9 mid-height PIC

[a] If CCF ≥ 0.8, then the reconstruction is considered valid.

b If CCF < 0.8, then check the A parameter. If A ≥0.9, the reconstruction is not a good fit to the Gaussian function, but the total mass prediction is valid.

[C] A is defined in Equation 6 below.

11.1. Specific QC checks on TDLAS measurements are discussed in SOP C2.
11.2. Specific QC checks on wind measurements are described in SOP W2.
11.3. Specific RPM QC checks:
- 11.3.1. RPM total mass positioning and reconstruction. The RPM methodology plume estimates are assessed for quality using the following:
  - 11.3.1.1. Concordance Correlation Factor (CCF) (Lin, 1989), indicates goodness of fit between measured and predicted PIC. CCF is equal to rA, where r is the Pearson correlation coefficient, and A is a correction factor for the shift in population and location. This shift in total mass positioning is a function of the relationship between the averages and standard deviations of the measured and predicted PIC vectors:

$$A = \left[\frac{1}{2} \left(\frac{\sigma_{PIC_P}}{\sigma_{PIC_M}} + \frac{\sigma_{PIC_M}}{\sigma_{PIC_P}} + \left(\frac{\overline{PIC_P} - \overline{PIC_M}}{\sqrt{\sigma_{PIC_P}\sigma_{PIC_M}}} \right)^2 \right) \right]^{-1} \qquad (6)$$

Where:

σ_{PIC_P} = Standard deviation of the predicted PIC vector
σ_{PIC_M} = Standard deviation of the measured PIC vector
$\overline{PIC_P}$ = Mean of the predicted PIC vector
$\overline{PIC_M}$ = Mean of the measured PIC vector

where the measured PIC vector is of length five (number of scans) and consists of PIC values for each scan beamline. The predicted PIC vector of same length represents the computed values along each beamline based on the modeled concentrations in the y,z plane plume.

11.3.1.2. A CCF ≥0.8 indicates a valid reconstruction.

11.3.1.3. An A≥ 0.9 verifies that the total mass positioning was a reasonable fit of the raw data.

11.3.2. Plume capture: For emission rate estimations using the RPM configuration, maximal plume capture is desired. Calculated emission rates are considered valid when the topmost PIC is less than or equal to 90% of the midlevel PIC. Under these cases, the measurement plane contains ≥ 70% of the plume.

11.3.3. Check for reasonableness of the calculated flux: Verify that the generated result is reasonable based on the raw PIC data and a visual plot of the concentration plane (a product of the model).

12. References

12.1. Hashmonay, R.A.; Yost, M.G. 1999. Innovative approach for estimating gaseous fugitive fluxes using computed tomography and remote optical sensing techniques. J. Air & Waste Manage. Assoc. 49, 966.

12.2. Lin, L. I-K. 1989. A Concordance Correlation Coefficient to evaluate reproducibility. Biometrics. 45, 255-68.

12.3. SOP C2. 2006. Open Measurement of Ammonia with the Boreal Laser GasFinder Tunable Diode Laser Absorption Spectrometer (TDLAS). Standard Operating Procedure C2. Purdue Applied Meteorology Lab.

12.4. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.

12.5. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.

12.6. SOP W2. 2006. Measurement of Wind with the RM Young Model 81000 3-Dimensional Sonic Anemometer. Standard Operating Procedure W2. Purdue Applied Meteorology Lab.

13. Contacts

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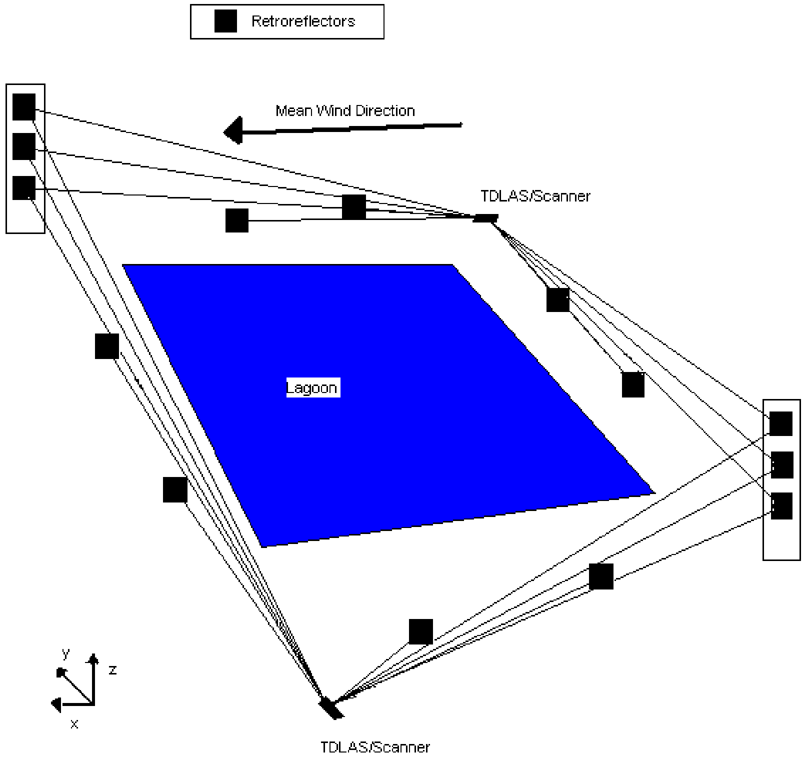

Figure 1. Example of RPM Configuration Setup.

Appendix A
Conversion of volumetric gas concentrations to gas density concentrations

Validated air pollutant concentration measurements are reported in units of parts per billion (ppb). Standard dry concentrations of all gas measurements will be reported, but emissions will be calculated on the non-dry (including water vapor partial pressure) air concentrations. As emissions calculations require units of $\mu g/m^3$ all measurements will be converted from ppm to $\mu g/m^3$ at 20^oC.

The ideal gas law (*PV = nRT)* is used to convert volumetric gas concentrations to actual and standardized mass concentrations in non-dry air according to:

$$C = \frac{PcM}{R(273.15 + T)x10^3} \qquad \text{(Equation A.1)}$$

Where:

c	Volumetric concentration, ppm
C	Actual mass concentration, $\mu g/m^3$
M	Molecular weight, M=17.03 and 34.08 for NH_3 and H_2S respectively
P	Pressure at sampling location, atm
T	Temperature at sampling location, °C
R	Universal Gas Constant: 0.08206 L-atm/mol-°K

Table A.1. Physical constants.

	Ammonia	**Hydrogen sulfide**
Molecular weight	17.03	34.08
Density	0.771 g/L @ 760 mm	1.539 g/L
Solubility in cold water	89.9 g/100 mL	437 cm^3/100 mL

Source: Lide, D.R. ed. 1993. *CRC Handbook of Chemistry and Physics*. Boca Raton: CRC Press.

The equation to standardize the volumetric gas concentrations of any pollutant to the standard mass concentration C' is as follows:

$$C' = \frac{P'cM}{R(273.15 + T')x10^3} \qquad \text{(Equation A.2)}$$

Where:

C′	Standard mass concentration moist basis, $\mu g/sm^3$
P′	Standard pressure, 1 atm
T ′	Standard temperature, 20°C

The dry standard concentration is calculated as follows:

$$C'' = \frac{C'}{(1-W)} \qquad \text{(Equation A.3)}$$

Where:

C'' Dry standard mass concentration dry basis, µg/dsm^3

W Humidity mixing ratio

The humidity mixing ratio (W) is calculated as:

$$W = 0.62198 \frac{e^{f(T)}\varphi}{P_a - e^{f(T)}\varphi} \qquad \text{(Equation A.4)}$$

Where:

W = humidity mixing ratio, kg/kg

P_a = pressure at the sampling location

φ = relative humidity, decimal

$$f(T) = \frac{C_1}{T} + C_2 + C_3 T + C_4 T^2 + C_5 T^3 + C_6 T^4 + C_7 \ln T : \quad \text{(for T < 273.15°K)} \qquad \text{(Equation A.5)}$$

$$f(T) = \frac{C_8}{T} + C_9 + C_{10} T + C_{11} T^2 + C_{12} T^3 + C_{13} \ln T : \quad \text{(for T > 273.16°K)}$$

C_1= -5.675x10^3
C_2= 6.392
C_3= -9.678x10^{-3}
C_4= 6.222x10^{-7}
C_5= 2.075x10^{-9},
C_6= -9.484x10^{-13}
C_7= 4.163
C_8= -5.800x10^3
C_9= 1.391
C_{10}= -4.864x10^{-2}
C_{11}= 4.176x10^{-5}
C_{12}= -1.445x10^{-8}
C_{13}= 6.545.

Appendix B
Example application of methodologies described in this protocol using OP-FTIR

The methodologies described in this protocol are independent of the particular PI-ORS instrument. This appendix has been included to illustrate the application of the methodologies described in this protocol using a scanning open-path FTIR (OP-FTIR).[B1-4] The methodologies described in this protocol have been utilized for several other environmental monitoring programs.[B5-7]

B1.0 Examples of DQI goals and internal QC.

The Data Quality Indicator (DQI) goals for the OP-FTIR instrument are shown in Table B1. The DQI goals were developed based on analysis of the historical data obtained in similar studies and available guidance documents.

Table B1. Data Quality Indicators (DQIs) for OP-FTIR

Measurement Parameter	Analysis Method	Accuracy	Precision	% Complete
PIC determination	OP-FTIR: nitrous oxide conc.	±25%/15%/10%[a]	± 10%	90
Mid-IR absorbance	OP-FTIR: spectra quality	Acceptable[b]	NA	100

a The accuracy acceptance criterion is ±25 percent for pathlengths of less than 50 m, ±15 percent for pathlengths between 50 and 100 m, and ±10 percent for pathlengths greater than 100 m.

b Spectral quality must meet minimal quality (refer to instrumentation manuals and EPA methods for instrument-specific spectral quality criteria).

B1.1 *Procedures for Evaluating OP-FTIR DQIs*. The development and determination of DQIs for open-path measurements is an ongoing process. The following section details the procedure for assessing the OP-FTIR DQI goals presented in Table B1.

B1.1.1 *PIC Determination*. The accuracy and precision of the absorbance data may be verified by analyzing measured atmospheric nitrous oxide concentrations over time. This test is valid only if the background spectrum is void of nitrous oxide, and nitrous oxide is not used in the verification gas release. For the duration of the study, the calculated concentration results for nitrous oxide should be comparable to the natural ambient atmosphere level of 315 ppb ±25 percent for path lengths less than 50 meters, 315 ppb ±15 percent for path lengths between 50 and 100 meters, and 315 ppb ±10 percent for path lengths greater than 100.[B8] Refer to Section 11.3.1.1 of *ASTM Standard Practice E 1982-98* for more information on this procedure.[B2]

B1.1.2 *Mid-IR Absorbance*. The quality of the mid-IR absorbance data can be assessed by performing several qualitative and quantitative checks. These checks include assessing instrument noise, detector nonlinearity, detector saturation, and instrument response. These checks were developed based on *ASTM Standard Practice E 1982-98*. Additional details on how to perform these checks can be found in the EPA document entitled, *ECPB Optical Remote Sensing Facility Manual*.[B9]

B1.2 *Internal Quality Control*. Traditional quality control (QC) samples are not applicable to OP-FTIR experiments. Instead, the analyses of IR-active ambient gases having relatively constant values are included in the quantification process. Examples include nitrous oxide and methane, which are found at relatively constant concentrations of 315 ppb and 1.7 ppm as global backgrounds, respectively. These must be used with caution, because a particular measurement area may prove to be a source of these gases. Methane, for example, is often emitted from hog waste lagoons and landfills.

B1.2.1 *Generation of a Background Spectrum*. As described in EPA Compendium Method TO-16 (1999),[B2, B3] the generation of a background spectrum, more precisely the I_0 spectrum, is best accomplished synthetically. The best approach to this depends upon the software being used for processing the data. Many commercially available software systems are compatible with the data format used by OP-FTIR instruments. However, all of the available software options require analyst input; therefore, there is some subjectivity involved in the process.

B1.2.2 *Data Integrity*. Spectra are analyzed for peaks of common atmospheric constituents, such as water vapor, carbon dioxide, and methane. If these peaks are located outside of their expected spectral region or exhibit any other abnormal characteristics, a spectroscopist should manually examine a select portion of the original data to determine the reason for these deviations and/or the usability of the collected data.

B2.0 RPM example

B2.1 *Introduction*. During the Fall of 2002 and Spring of 2003, the methodologies described in this protocol was evaluated using the controlled release of verification gases and a scanning monostatic OP-FTIR instrument. The experiment was performed at the Duke Forest Facility of Duke University, located in Chapel Hill, North Carolina. A nearly flat and square area of 14,400 m^2 was selected for the study. The verification gases released during the experiments included ethylene and acetylene. This example briefly describes the preliminary results obtained from this controlled demonstration study using the RPM methodology described in SOP O2.

B2.2 Experimental Setup. *This study was supported by the Environmental Security Technology Certification Program (ESTCP) of the Department of Defense (DoD) and audited by the Emissions Monitoring Center (EMC) of the EPA.*

In this study, an OP-FTIR mounted on a scanner and an array of mirrors (PDC) formed multiple, non-intercepting beam paths. Five mirrors were used. The OP-FTIR was scanned sequentially from mirror to mirror, acquiring spectra from each beam path. The OP-FTIR dwelling time on each mirror was 60 s. Ethylene was released and data were collected for 12 cycles. A total of 1.0 lb of ethylene was released over a period of 68 min, which resulted in an average release rate of 0.11 g/s.

B2.3 *Results and Discussion*. PIC data were derived from the collected spectra, and wind data were time-synchronized with the spectral data and averaged for the corresponding measurement cycle. Following this, the PIC data were sorted according to mirror number, and the expanded data matrix was then used as input to the software developed by ARCADIS in Research Triangle Park, NC. The software provides spatial information on the homogeneity of the plume, and calculates the emission flux through the configuration.

B2.3.2 *Results*. Several verification gases were released in an "H" pattern upwind of the RPM configuration to simulate a small area source in Duke Forest (see Fig. B1).[B9] The verification gas cylinders were weighed before and after each release to calculate the actual release rate (in g/s). Spectral data were collected by sequentially scanning the OP-FTIR from mirror to mirror. This data were used to derive PIC data along each beam path, which were used as input to the RPM algorithm to determine a plane-integrated concentration. Wind data were simultaneously collected at two heights (2 and 10 m), and were incorporated with the plane-integrated concentration data to calculate the emission flux through the RPM configuration.

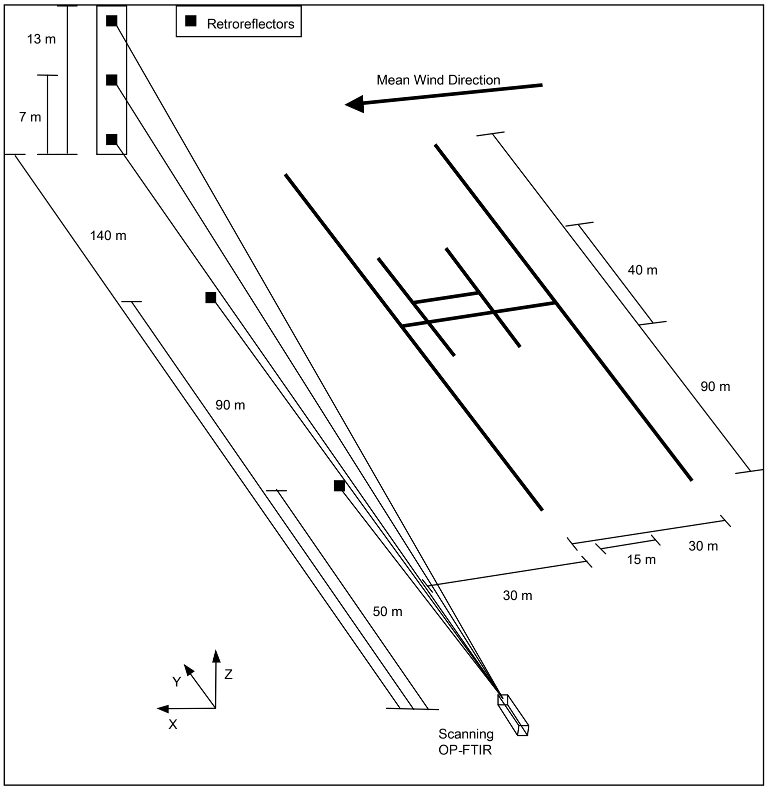

Figure B1. Schematic of the RPM configuration used for the ethylene release.

Table B2 presents the data matrix used to construct the mass-equivalent plume map. Fig. B2 shows the reconstructed mass-equivalent plume map constructed by averaging PIC data from all 12 cycles. The average calculated emission flux was 0.10 g/s.

B2.3.2.1 Average wind speed and direction data for each measurement cycle is shown in Table B2. Wind measurements were interpolated every 2 m between the 2- and 10-m heights. The average wind data for each cycle at 4 m above the ground are shown. The wind direction is measured clockwise from an axis perpendicular to the RPM configuration. In this convention, 0º is perpendicular to the vertical plane.

Table B2 Data matrix used to construct Figure B2.

Beam Path no.	**1**	**2**	**3**	**4**	**5**	**WS (m/s)**	**WD from Normal (deg)**
Physical Beam Path Length (m)	52.1	90.1	137	141.5	141.9		
Cycle 1 (ppm-m)	**2.7**	13.4	17.8	**3.8**	**5.1**	2.7	5
Cycle 2 (ppm-m)	6.4	28.0	19.4	9.0	**5.4**	2.6	1
Cycle 3 (ppm-m)	4.5	23.4	23.1	8.8	**7.9**	2.1	0
Cycle 4 (ppm-m)	**2.8**	28.3	20.4	10.0	**7.3**	2.1	14
Cycle 5 (ppm-m)	5.0	19.8	34.7	**4.2**	**5.3**	2.9	13
Cycle 6 (ppm-m)	**2.9**	27.9	14.4	11.1	**7.8**	2.9	3
Cycle 7 (ppm-m)	10.8	14.1	28.5	**4.7**	**7.9**	3.0	-11
Cycle 8 (ppm-m)	**2.7**	10.3	49.1	4.9	**5.8**	2.3	18
Cycle 9 (ppm-m)	**2.7**	23.1	25.8	23.3	**7.1**	1.8	-2
Cycle 10 (ppm-m)	6.7	11.6	18.7	**4.5**	11.1	1.7	-15
Cycle 11 (ppm-m)	14.4	20.2	36.0	15.7	**7.0**	2.0	-8
Cycle 12 (ppm-m)	**2.6**	**3.3**	30.9	8.3	**6.7**	2.2	14
12-Cycle Average (ppm-m)	5.4	18.6	26.6	9.0	7.0	2.35	3

BOLD values represent one-half the minimum detection limit.

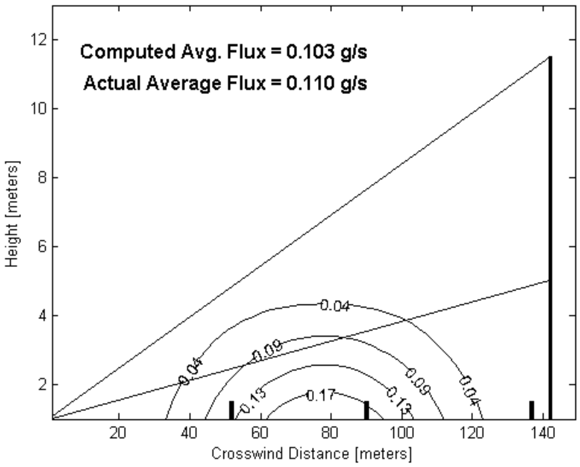

Figure B2. Flux measurement for ethylene.

B2.3.2.2 A moving average is used in the calculation of average values to show temporal variability in the measurements. A moving average involves averaging values from several different consecutive cycles. For example, a data set may be reported using a moving average with a group size of 4, where values from cycles 1 to 4, and 2 to 5 are averaged together to show any variability in the values.

The example in this appendix explores the dependence of the results on the group size of the moving average, and is illustrated in Fig. B3. To assess the accuracy of the reconstruction for each moving average group, the CCF is computed. It is apparent in Fig. B3 that as the group size of the moving averages increase, the standard deviation decreases (smaller error bar). The CCF value increases for larger group sizes of moving averages (indicating a better fit for the measured data), but levels off at a group size of three. A moving average group of three is recommended for the RPM methodology. In some conditions, it may not be advisable to use a larger group size for the moving average.

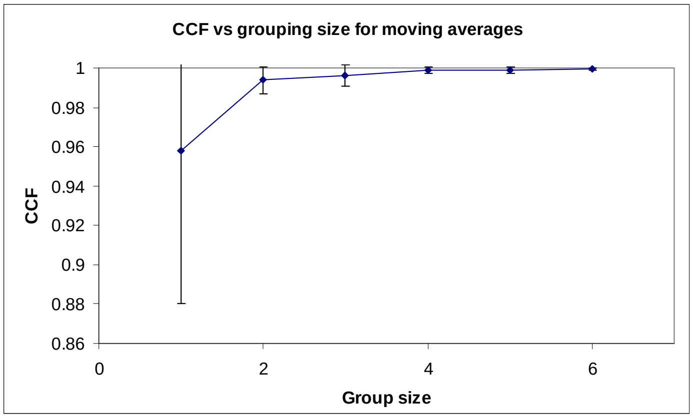

Figure B3. Dependence of CCF on moving average group size.

B2.3.2.3 A time plot of the flux calculation for the ethylene verification release, using a moving average with a group size of three, is shown in Fig. B4. The dashed horizontal line corresponds to the actual mass released; the dashed plot indicates the measured wind direction from perpendicular to the RPM configuration; and the solid plot indicates the calculated emission flux values. The corresponding data are listed in Table B3.

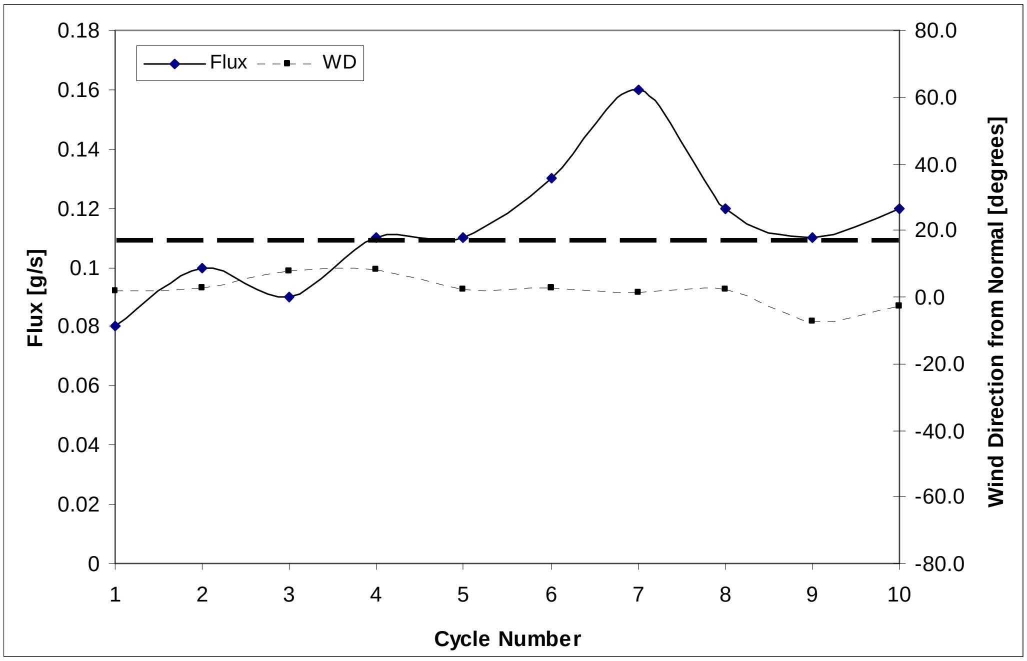

Figure B4. Time series of ethylene emission flux estimation.
(——— Indicates the actual release rate)

Table B3. Moving average of three ethylene emission data points for Figure B4.

Group	CCF	Flux(g/s)	WS(m/s)	WD(deg.)
1	0.95	0.08	2.2	2
2	0.96	0.1	2.1	3
3	0.96	0.09	2.2	8
4	0.96	0.11	2.4	8
5	0.96	0.11	2.7	2
6	0.97	0.13	2.5	3
7	0.98	0.16	2.2	2
8	0.96	0.12	1.8	2
9	0.98	0.11	1.7	-7
10	0.94	0.12	1.8	-3
Average	0.96	0.11		
Std. Dev.	0.012	0.022		

B2.3.2.4 The example below of flux data from the Duke Forest study provides guidance for determining the range of wind directions that will ensure complete capture of the plume (in the horizontal direction) by the RPM configuration. The RPM flux results are representative of the source emission rate only during periods that the prevailing wind directions meet the established criterion.

The determination of the acceptable wind criterion is done by analyzing a data set of calculated flux values, with the corresponding wind direction, at the time of the flux calculation. The data set should consist of data collected over a wide range of wind directions on both sides of the perpendicular direction to the RPM configuration. A plot is create of the calculated flux values as a function of the angle of the prevailing wind direction (from perpendicular to the RPM configuration) of all runs made when the wind direction angles were close to the perpendicular direction of the RPM configuration. Because these runs were collected over a period of six months, the flux values were normalized to the maximum flux value in each run.

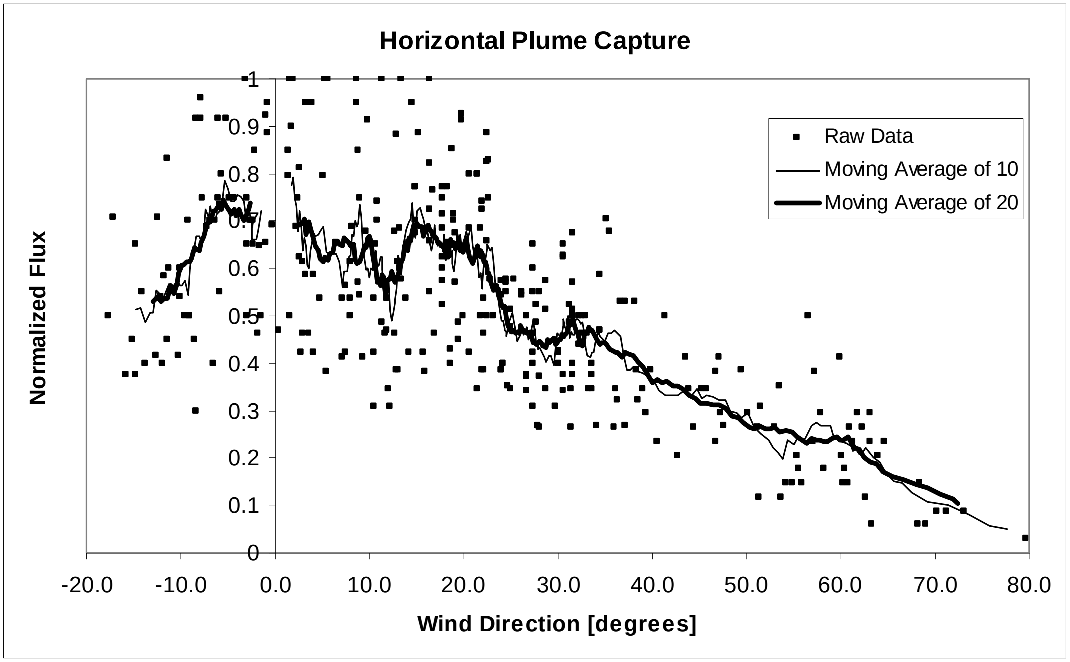

Figure B5. Plot of calculated flux values and corresponding wind direction during the time of measurements.

Fig. B5 presents a plot of this data set. The figure shows the calculated normalized flux values and the corresponding wind direction (from the normal to the RPM configuration during the time of the measurements). The figure shows that the raw flux values steadily increase, up to a certain point, as the prevailing wind direction becomes closer to perpendicular to the configuration. However, over a certain range of wind directions, there appears to be no relationship between calculated flux values and prevailing wind direction. This range is approximately between -10° and +25° for the Duke Forest validation study. This is the approximate range of acceptable wind directions, as the variations in flux values in this range are due to variations in the source strength, and not due to changes in wind direction. It is recommended that these flux plots be smoothed out with a moving average approach to retrieve a more definite and accurate cutoff of the wind criterion. The lined curves in the figure show examples of such moving averages, one with a grouping of 10 data values (thin line) and the other with a grouping of 20 data values (thicker line). These examples of moving averages clearly define the range of wind directions where flux values are independent from the wind direction values.

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USE OF CONTROL CHARTS FOR PERFORMANCE MONITORING OF GAS ANALYZERS AND ANALYTICAL INSTRUMENTS

Standard Operating Procedure (SOP) Q1

USE OF CONTROL CHARTS FOR PERFORMANCE MONITORING OF GAS ANALYZERS AND ANALYTICAL INSTRUMENTS

Standard Operating Procedure (SOP) Q1

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1. Scope and Applicability

1.1. Control charts provide a real-time graphical representation of the performance of a gas analyzer or other analytical instrument, which can be used to assess data quality and to spot long-term trends in the instrument's performance. The purposes of control charts are as follows:
   1.1.1. Visual presentation of the quality control of the analyzers and other instruments
   1.1.2. Real-time assessment of the data quality of the analyzers or measurement devices
   1.1.3. To monitor the long-term performance of the instrument
   1.1.4. To determine when a corrective action must be done

1.2. The control charts described in this SOP can be applied to gas analyzers, and to any other type of measurement device (e.g. temperature, relative humidity, and pressure sensors).

1.3. A control chart plotting the standard deviation is beyond the scope of this SOP.

2. Summary of Method

Control Charts are generated by plotting data from the zero-precision checks (SOP G9) with known reference standards (control sample). After the zero-precision check is conducted, the control chart must immediately update to give real-time assessment of the instrument's performance. A control sample is used to check the instrument's performance. The instrument's response to a control sample is an indication of the instrument's deviation from the true value that is a result of intrinsic instrumental measurement error. Control limits are established to maintain statistical control of the data quality. If the reference standard results in a measurement outside the control limits, the control chart is a tool used to alert the analyst / researcher to increased uncertainty in the instrument's measurements. Other Control Chart parameters include the mean and standard deviation of the last *n* samples, where *n* may be from 5 to 20.

3. Definitions

AirDAC	Air data acquisition and control program
Control Limits	The bounds of the range (above and below the true value) that denote acceptable performance of the analyzer or sensor.
Control Sample	A known reference standard
PAAQL	Purdue Agricultural Air Quality Laboratory
QAPP	Quality Assurance Project Plan

4. Cautions

4.1. Use the correct range for the analyzer or sensor, and for AirDAC. A range that is too small will cause the analyzer or sensor's analog output to be pegged in response to the reference standard.

4.2. The control sample applied to check the analyzer or sensor must be consistent over time. In the case of gas analyzers, for example, use the same concentration for the reference gas for the zero-precision check (SOP G9).

4.3. The control sample should be representative of the range of measurements which the analyzer or sensor is expected to measure. In the case of gas analyzers, verify that the concentration of the reference gas is similar to that in the gas samples being analyzed.

5. Personnel Qualifications

5.1. Personnel must be trained in the use of control charts before working with any analyzer or sensor for which a control chart is used.
5.2. Each analyst must also read and understand this SOP before conducting a multipoint calibration (SOP G8) and zero-precision check (SOP G9).

6. Equipment and Supplies

6.1. Computer and AirDAC software program (SOP B2)
6.2. Microsoft Excel software

7. Procedures

7.1. Setup of the Control Chart
  7.1.1. A template of the Control Chart will be supplied by PAAQL to each site, and will be located in the Instrument Performance Record spreadsheet of each individual instrument. Each instrument (analyzer or sensor) will have its own Control Chart.
  7.1.2. General formatting and style (Fig. 1)
    7.1.2.1. Use Microsoft Excel's x-y plot function for all Control Charts.
    7.1.2.2. Use SI units.
    7.1.2.3. The ratio of the width to height of the chart should be 1.62. This ratio is known as the Golden Ratio, and is related to the Golden Rectangle. A template is in the Instrument Performance Record File.
    7.1.2.4. Use Arial font in graphs and do not bold.
    7.1.2.5. Use 14-point font for the title.
    7.1.2.6. Use 12-point font for all other notations (axes, labels, comments, and numbering).
    7.1.2.7. Place the tic marks on the outside of the graph.
    7.1.2.8. Format each tic label with fixed decimal places.
    7.1.2.9. The x-axis is time (Date). Include one tic mark per week (every 7 d), and label every so many weeks with mm/dd such that the labels fit on the axis.
    7.1.2.10. Don't use vertical labeling on the x-axis.
    7.1.2.11. Put the legend of the graph on the inside of the chart box, not outside.
    7.1.2.12. Place captions outside the chart.
    7.1.2.13. Avoid redundancies in graph labeling. For example, there is no need for a title, since a caption covers it.
    7.1.2.14. Label the control limits.
    7.1.2.15. Multiple baselines can be used for one instrument.
      7.1.2.15.1. Simultaneous baselines for the precision check gas standard and the zero reference gas can be plotted on the same chart.

7.1.2.16. Ancillary data can be plotted on the control chart (e.g. reaction chamber pressure for a chemiluminescence ammonia analyzer).

7.1.2.17. Include the serial number of the instrument in the control chart.

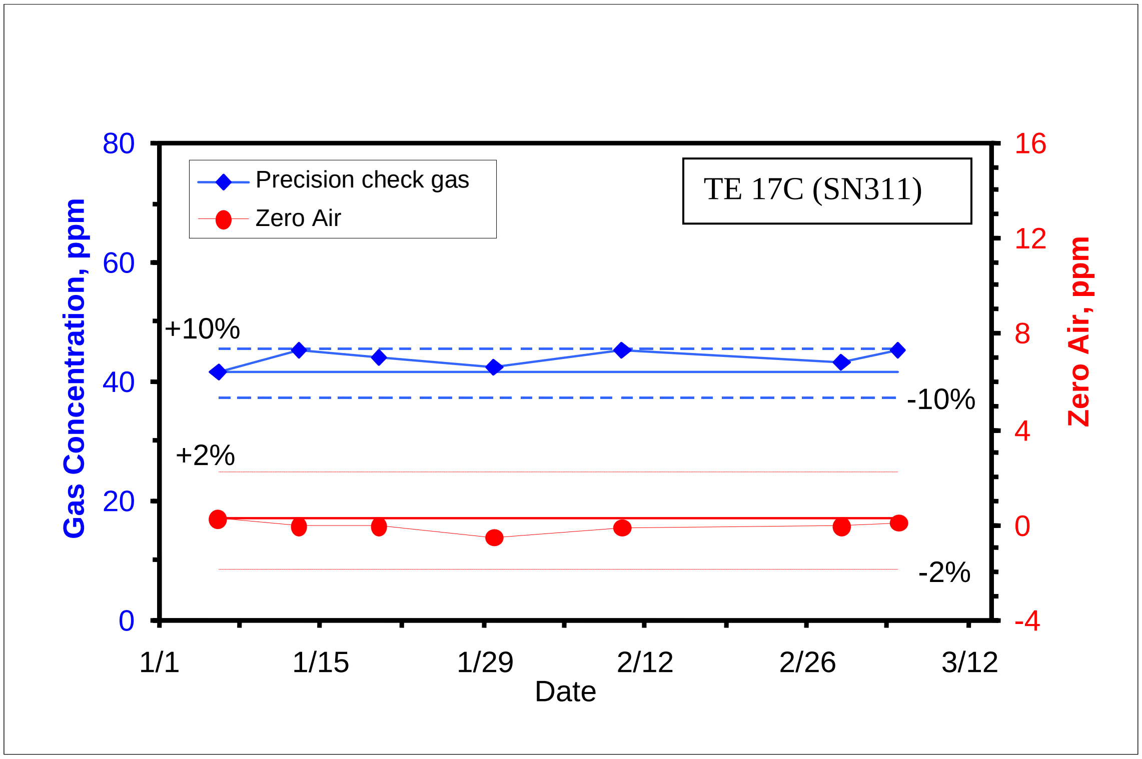

Figure 1. An example of a Control Chart, in this case for a TE 17C Ammonia Analyzer. Two baselines (both components of the zero/precision check) are included, along with their control limits (± 2% for the zero gas and ± 10% for the precision check gas).

7.2. Updating the control chart

7.2.1. Before updating, save the current control chart in the Instrument Performance Record File's "Zero – Precision Record" worksheet to keep a record of past control charts.

7.2.2. The control chart is updated from the zero-precision check data that is tabulated in the Instrument Performance Record File's "Zero – Precision Check" worksheet. This data is entered as described in SOP G9.

7.2.3. The baseline measurement is the first zero-precision check after a multipoint calibration (SOP G8) that is the target value.

7.2.3.1. The baseline measurement is established using a standard reference, the value of which is chosen to be consistent with values that are expected to be measured.

7.2.4. Save the updated control chart in the Instrument Performance Record File's "Zero – Precision Check" worksheet, as described in SOP G9.

7.2.5. Control limits

- 7.2.5.1. Control limits are used to determine whether the analyzer or sensor has drifted out of calibration.
  - 7.2.5.1.1. Do the following when data points fall outside the control limits:
    - 7.2.5.1.1.1. Access the analyzer's or sensor's operation to determine if the equipment is operating properly or is in need of repair.
    - 7.2.5.1.1.2. Recalibrate the analyzer or sensor, and establish a calibration curve and new control chart
- 7.2.5.2. The controls limits are set to a ± percentage of the baseline measurement. This percentage is dependent upon the Data Quality Objectives (DQO's) in the QAPP for the gas analyzer or sensor that the Control Chart is monitoring.
- 7.2.5.3. The control limits can be set in the Instrument Performance Record File's "Zero – Precision Check" worksheet. The percentage is entered into the boxes labeled "Control Limit %" for both the precision control limit and the zero control limit.

8. Data and Records Management

- 8.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 8.2. Manage all data according to SOP B5.
- 8.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all corrections.

9. Quality Control and Quality Assurance

- 9.1. Maintain a control chart for all gas analyzers or sensors specified in the QAPP for the project in question.
- 9.2. Update the Control Chart immediately after performing a zero/precision check of a gas analyzer (SOP G9) or other sensor.
- 9.3. Review the Control Chart to assess data quality and instrument performance every time it is updated.
- 9.4. E-mail the Control Charts to the research leaders (PI, QC Manager, Gas Analyzer Manager, etc.) in a timely manner. The exact list of persons who should receive Control Charts, and the frequency, will be specified in the QAPP for the particular project.
- 9.5. Keep a printout of the most recent Control Chart for each analyzer or sensor in the on-farm instrument shelter (OFIS). Keep these copies available for site audits, either by PAAQL personnel or outside QA auditors.

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PRODUCER COLLABORATIONS AT OPEN-SOURCE MONITORING SITES

Standard Operating Procedure (SOP) S7

PRODUCER COLLABORATIONS AT OPEN-SOURCE MONITORING SITES

Standard Operating Procedure (SOP) S7

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1. Scope and Applicability

1.1. The collection of high-quality air emissions data from a livestock facility requires communication and collaboration between the field operations staff (FOS) and the livestock producer and his/her staff.

  1.1.1. The livestock producer's staff may or may not have a specific interest in the data collected at the open-source site.

1.2. This SOP describes the following:

  1.2.1. Procedures for collaboration between the FOS and livestock producer and his/her staff responsible for open-source sites (lagoons and corrals)

  1.2.2. Information related to the open-source sites that must be collected from livestock producers or his/her staff

  1.2.3. Expectations placed upon collaborating livestock producers or his/her staff, and on the FOS

  1.2.4. Possible modifications to farm operations that may be needed to accommodate the study.

1.3. This SOP applies specifically to the National Air Emissions Monitoring Study.

2. Summary of Method

The research staff must establish and maintain effective communication with any livestock-production that is participating in a research study. The facility design and operational information of the farm must be provided by the producer or his/her staff to the FOS. In addition, the FOS must communicate to the livestock producer and his/her staff those requirements that he/she must meet in order to conduct a successful study. The collaboration and communication will include email messages, phone calls, face-to-face meetings, reports, data forms, presentations, data monitoring, and project documentation such as the Quality Assurance Project Plan (QAPP).

3. Definitions

3.1.	Biosecurity	Measures that are taken to prevent spread of disease to or from livestock or poultry.
3.2.	DM	Data Manager
3.3.	FOS	Field operations staff
3.4.	PM/PI	Project Manager or Pincipal Investigator
3.5.	PPR	Pull plug with recharge
3.6.	QAPP	Quality Assurance Project Plan
3.7.	SOP	Standard Operating Procedure

4. Health and Safety

4.1. Beware of, and do not interrupt, worker traffic. FOS may request a slight delay or interruption in normal farm activities during set up and disassembly of instrumentation, but must always defer to the decisions and activities of the producer and his/her staff.

4.2. Watch out for vehicular traffic such as litter, feed, animal, milk, and egg trucks, loaders, cars, and other vehicles.
4.3. The farm's licensed electrician should conduct or approve all electrical work associated with the study. An example is connecting power to the instrument shelter.
4.4. Be careful around animals and birds to prevent personal injury.
4.5. Post the phone number for medical emergency personnel and directions (map) to the nearest hospital in the instrument trailer close to the entrance.
4.6. Emergency evacuation procedures at each measurement location will be those established by the livestock producer.
4.7. Maintain a first aid kit in the instrument trailer.

5. Cautions

5.1. Take steps to overcome language barriers (e.g. Spanish vs. English). For example, if any worker has Spanish as his/her primary language, distribute a pamphlet in Spanish indicating the objectives of the study and the importance of taking care of the instrumentation. The pamphlet can be complemented with a video and/or PowerPoint presentation in Spanish that can be distributed to all research sites, and can be included in a periodic (quarterly) refresher session (as rotation of workers in the farms can be high).
5.2. Post signs or labels by sensitive test equipment and in measurement paths, indicating that they should not be touched or worked around unless authorized by study personnel. These should also be written in the primary language(s) of the farm workers.
5.3. Fence the region around any light emitters that may cause eye damage.
5.4. FOS must follow all biosecurity protocols and guidelines requested by individual livestock producer or his/her staff. At minimum, these can be expected to include:
  5.4.1. Cleaning research vehicles and equipment entering the farm according to the producer's specific protocol
  5.4.2. Accessing only areas that are directly involved in the study
  5.4.3. Ensuring that FOS have not exceeded producer-specified limits on foreign travel and/or visits to other poultry or livestock farms.
  5.4.4. Notifying the livestock producer prior to visiting the site
5.5. The FOS should, to the greatest extent possible, solicit input and suggestions from the livestock producer and his/her staff as to safe locations for equipment, possible hazards that might occur, and appropriate precautions that might best protect equipment and staff.
5.6. Remember that the producer's involvement and time commitment to the project is less than that of the FOS. The amount of time required for the livestock producer or his/her staff to execute their commitments to the study is a major factor influencing current and future involvement. Take care not to use more of the producer's and/or staff's time than is absolutely required for the study.

6. Interferences

6.1. The participating farms should operate with typical management practices, particularly during active monitoring periods, to minimize the potential for bias in the emission data. If changes to the farm operations occur, they must be documented, and these records retained according to SOP D1.
  6.1.1. Livestock producer or his/her staff should notify FOS if research equipment is

interfering with farm operations and needs to be moved or modified.

- 6.1.2. Livestock producer and his/her staff must agree not to move or modify study equipment themselves.

6.2. FOS must not give tours of the study site without livestock producer's permission. Establish a policy about tours prior to setting up instrumentation on the farm.

6.3. Any add-on research projects at the farm must be approved by the livestock producer.

7. Personnel Qualifications and Training

7.1. All livestock producer and research staff involved in the study should read this SOP.

7.2. A pre-study training and orientation session for the livestock producer and his/her staff will be conducted by the FOS or Project manager, and will include time for the livestock producer or his/her staff to explain the farm operation. The training session will also be conducted in the primary language of the farm workers, with the help of translated video and/or a PowerPoint presentation.

8. Utilities, Equipment and Supplies Provided by Farm Staff

8.1. Personal protection equipment
- 8.1.1. Levels of protective equipment will likely vary at a given facility, depending upon the season, activity, and other factors.
- 8.1.2. Protective clothing may be provided by the FOS, or by the livestock producer or his/her staff, depending, for example, on the preferences of the livestock producer. Reach an agreement on this point with each producer at the beginning of the study.
  - 8.1.2.1. For example, if livestock producer wishes to supply their own boots as part of their biosecurity program, FOS will abide by this preference.

8.2. A suitable area for placement of the open-source instrument trailer, including all-weather (e.g. gravel) access as necessary

8.3. Electric power hookup post (SOPs U4 and U5)

8.4. Utility located to ensure the FOS does not accidentally damage underground cables, gas lines, tiles, drains, or other underground hazards while sinking anchors.

8.5. Phone line (required) and internet service (preferred) (SOP U4)

8.6. A garden hose with a nozzle near the site of the open-source instrument trailer

8.7. Restroom and shower facilities, if available

9. Procedures

9.1. Livestock producer and his/her staff will allow free access to farm by project staff.

9.2. Livestock producer or his/her staff will be apprised of all necessary structural modifications (SOPs U4 and U5).
- 9.2.1. Provide a project schedule and QAPP to the livestock producer at least 90 days prior to initiation of setup.
- 9.2.2. Set up a monthly phone call with farm management to solve problems, provide updates and facilitate collaboration.

- 9.2.3. Detailed information about the farm will be collected for each participating site as part of the site-selection process. Site-selection criteria and parameters that should be collected before initiating a study are included as appendices.
  - 9.2.3.1. Lagoon and basin site forms are included in Appendix A.
  - 9.2.3.2. Corral site forms are included in Appendix B.
- 9.2.4. At the end of each measurement period, each collaborating livestock producer at a lagoon or basin site is requested to provide the following information (Appendix A: Table A2) for all barn(s) whose waste goes to the lagoon or basin:
  - 9.2.4.1. Animal inventory, average weight and average age
  - 9.2.4.2. Record of animal movements in and out of the barn
  - 9.2.4.3. Animal diet (feed composition, including N and S levels, and feed consumption rates).
  - 9.2.4.4. Production (quantity and weight of marketed animals, eggs, milk, etc.)
  - 9.2.4.5. Record of sludge removals
  - 9.2.4.6. Record of pump outs (contractor or livestock producer)
  - 9.2.4.7. Record of water consumption
  - 9.2.4.8. Advance notice of any change in production schedules and methods
- 9.2.5. At the end of each measurement period, each collaborating livestock producer at a corral site is requested to provide the following information (Appendix B: Table B2):
  - 9.2.5.1. Animal inventory, average weight and average age
  - 9.2.5.2. Animal diet (feed composition, including N and S levels, and feed consumption rates)
  - 9.2.5.3. Production (quantity and weight of marketed animals, milk, etc)
  - 9.2.5.4. Record of cleaning operations
  - 9.2.5.5. Record of animal movements in and out of the corral
  - 9.2.5.6. Record of water consumption
  - 9.2.5.7. Advance notice of any change in production schedules and methods

- 9.3. Livestock producer and his/her staff should be made aware that placement of equipment or sensors may in some cases require some site modification. FOS and livestock producer staff should work together to ensure that both the equipment and animals are well-protected.
  - 9.3.1. Trailers placed in lots accessible to animals will likely need to be enclosed with protective fencing.
  - 9.3.2. Equipment placed in areas that may occasionally have vehicular traffic may need additional flagging or warning bumpers.
- 9.4. Livestock producer or his/her representative should contact the PM/PI whenever there are any questions or concerns relating to measurement operations, or if there is catastrophic damage to farm operations.

10. Data and Records Management

- 10.1. Site Identification. Each site will be labeled in a standard format to denote site type (in this case, area component), location and number. For example, the finisher swine basin in Iowa is “IA3A”. This format is described in more detail in SOP B5. Producers shall not be identified in each site plan.
- 10.2. The FOS will maintain a log of farm visits in the lab notebook.

10.3. The livestock producer will maintain a log sheet of non-monitored farm activities that affect emissions (See Appendix C).

10.4. Data collected using Producer Event Forms (Appendix C) will be entered into an Excel spreadsheet and included in the site field notes, as described in SOP D1. Management of this data is also described in SOP D1.

  10.4.1. Scan all producer event forms, and keep them in electronic form and in paper form at the site. Electronic copies will then be ftp transferred to the PM and DM, as described in SOP D1.

10.5. Initial and date any corrections made on hard copies of forms. Use only indelible ink.

11. Quality Control and Quality Assurance

11.1. Each FOS member must maintain a log of animal exposures at other farms.

11.2. Monthly conference calls ensure communication between FOS and livestock producer.

11.3. Livestock producer must check daily to make sure his/her staff are filling out the Producer Event Form, because they do not normally record such data during daily or weekly activities.

11.4. Document all decisions and action items on paper in ink during all planning and status meetings so that it can be confirmed that all parties are in agreement.

11.5. Be sure livestock producer has a copy of the rationale for the study (found in the QAPP) and a copy of the schedule.

  11.5.1. If the livestock producer wishes, provide a copy of any relevant SOPs.

11.6. Training/ information session will include a verification of understanding of the basic aspects of the project, importance of the instrumentation and effects of farm operation on the study. Verification of understanding will be through the use of a question and answer session.

11.7. Livestock producer should designate a responsible staff member (liaison between the farm and the research team) for collecting and transmitting operational information, and also provide the name of his or her substitute when he or she is not available. This person should attend all informational meetings.

12. References

12.1. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.

12.2. SOP U4. 2006. Open-source Instrument Trailer. Standard Operating Procedure U4. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.

12.3. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.

Appendix A Site Information Form for Lagoons and Basins (SOP S7)

Table A1. Lagoon and basin site-selection criteria

Category	Information
Farm name	
Address, phone	Contact Person:
Livestock Producer	Biosecurity: time since farm visit, days:
	Willing to provide production information?
	Cooperative and enthusiastic participant?
	How long has the farm been in operation under current owner?
	Able to provide feed and water consumption data? Sample analysis records?
	Willing to possibly test abatement technologies after the NAEMS?
	Describe management style (tools used, etc.):
Convenience	Distance from university, miles:
	Phone, internet, and electric power available? 240 VAC?
	Lodging nearby for research team?
Facilities	Generally describe rations:
	Animal breed:
	Describe feed delivery system
	Describe waste collection, handling, treatment and storage systems
	Rate lagoon maintenance from 1 to 5 (1 is cleanest):
	Any emission control measures at facility?
Surroundings	Describe surrounding landscape and topography (hills, flat, etc.)
	Describe other pollution sources within one mile:
	Distance to nearest land application sites
	Distance from public road. Type of road (gravel or paved).

Table A2. Characteristics of test site lagoons or basins.

Descriptive parameters	Unit 1	Unit 2	Unit 3
Livestock type			
Year of facility construction			
Separation distance from barn fans, ft			
Type of storage (basin, lagoon or tank)			
Stage of lagoon (1st, 2nd, 3rd)			
Manure contributors to unit			
Animal 1 type (sows, cows, etc.)			
Animal 1 average weight (lb)			
Animal 1 inventory (# head)			
Animal 2 type (sows, cows, etc.)			
Animal 2 average weight (lb)			
Animal 2 inventory (# head)			
Animal 3 type (sows, cows, etc.)			
Animal 3 average weight (lb)			
Animal 3 inventory (# head)			
Manure collection (flush, scrape, PPR)			
Source flush or recharge water (if any)			
Lagoon loading frequency, hours			
Minimum space surrounding unit, ft.			
Volumetric loading rate, lb/d-ft^3			
Surface loading rate, lb/d-ft^2			
Obstructions within 3X height of unit?			
If yes, what kind? (e.g. trees, barns)			
Height of highest obstruction, ft			
Distance from worst obstruction, ft			
Type of cover (crust, straw, none, etc.)			
Are solids separated from influent?			
Odor control: (digester, additives)			
Sludge removal cycle, years			
Last time sludge removed (e.g., 1999)			
Agitation prior to pumpout?			
Manure removal frequency, days			
Pump out (contractor or producer)			
Type of liner (clay, plastic, etc.)			
Volume, cubic ft			
Surface area, square feet			
Berm slope (e.g. 3:1, 3.5:1, 4 :1, etc.)			
Maximum side length, ft			
Minimum side length, ft.			
Actual freeboard, ft.			
Inner berm height, ft.			
Outer berm height, ft.			
Liquid depth, ft.			
Sludge depth, ft.			
Number of inlets (show on drawings)			
Shape (rectangular, oval, etc.)			

Appendix B Dairy Corral Site Information Form (SOP S7)
(Email to Al Heber, Purdue University: heber@purdue.edu)

Table B1. Dairy Corral Site Selection Criteria, SOP S7.

Category	Information
Farm name	
Address, phone	
Livestock Producer	Provide production and herd management information (DC305)?
	Cooperative and enthusiastic about study?
	Willing to make some nonconsequential changes for the test?
	Willing to train herdspersons about importance of test?
	Able to provide feed and water consumption data per cow group?
	Willing to possibly test abatement technologies after the NAEMS?
	Describe management style (tools used, etc.):
Convenience	Distance to site from university, miles:
	Cell phone signal?
	Phone, internet, electric power available?
	Lodging nearby for research team?
Corrals	Generally describe rations (including forages and purchased feeds)
	Describe feed delivery system.
	Describe waste collection, handling, treatment and storage systems
	Rate corral hygiene from 1 to 5 (1 is cleanest).
Surroundings	Describe surrounding landscape and topography.
	Describe other pollution sources within one mile of site;
Farm	Describe all facilities on the site (# hd, size, age);
	Distance to nearest land application sites.
	On-site heifer growing program?

Table B2. Characteristics of test site: corrals, SOP S7.

Descriptive parameters.	Type 1	Type 2	Type 3
Breed			
Inventory (cow capacity)			
Average mass, lb			
Year(s) of construction			
Overall site width (E-W dist), ft			
Overall site length (N-S dist), ft			
Heifer growing program?			
Corrals			
Type (yearlings, lactation, hosp, etc.)			
Number			
Corral orientation			
Corral width, ft			
Corral length, ft			
Corral area, ft^2			
Apron width, ft.			
Apron length, ft.			
Apron cleaning (flush, scrape, vac)			
Maintenance technique (scr, harrow)			
Maintenance frequency (days)			
Shade height, ft.			
Shade length, ft.			
Shade width, deg.			
Side slope, %			
Cross slope, %			
Cow lane width, ft			
Cow lane length, ft			
Windbreak			
Height, ft			
Length, ft			
Lagoon			
Length, ft			
Width, ft			
Depth, ft			
Berm height, ft			
Number of stockpiles			
Weeping pad area, ft^2			
Number of separators			
Number of silage bunkers			
Number of hay barns			
Parlor			
Width, ft			
Length, ft			
Sidewall height, ft			
Roof ridge height, ft.			
Type of ventilation			
Number of air outlets in ridge			
Type of sidewall inlets			
Air circulation fans in ceiling			
Misting?			

Exit lane drenching?			
Holding time, min			
Wash cows?			
Milking frequency, times daily			
Parlor occupancy, # cows			
Holding pen			
Manure removal (flush, hand wash)			
Manure removal frequency			
Width, ft			
Length, ft			
Sidewall height, ft			
Roof ridge height, ft.			
Type of ventilation (NV, MV)			
Number of air outlets in ridge			
Type of sidewall inlets			
How is feed weighed?			
How is manure loads weighed?			
How are animals weighed?			
Number of pumps to monitor			
Access around property for sensors?			

Appendix C Producer Event/Change Form (SOP S7)

In order for the Purdue Air Quality group to do its job, they need to know about events/changes around the lagoon, basin, or corral that affect airflow or air emissions. A few jotted notes here will help them not make mistakes when analyzing the data. Thank you for your cooperation! [Examples of changes include (but are not limited to): major water leaks, power interruptions, sludge removal, pump outs, and diet changes.]

Description of event/change	Start time/date	End time/date	Contact person

Project contact information:
Name(s):______________________________________
Office phone: _____________________________
Cell phone: _____________________________

THE OPEN-SOURCE INSTRUMENT TRAILER

Standard Operating Procedure (SOP) U4

THE OPEN-SOURCE INSTRUMENT TRAILER

Standard Operating Procedure (SOP) U4

Prepared by

Chris D. Fullerton and Richard H. Grant

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006
Rev. 1.0 Effective Date: February 6, 2008

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)

Agronomy, Purdue University

West Lafayette, IN 47907

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1. Scope and Applicability

1.1. This standard operating procedure defines the characteristics of the instrument trailer to be used by each mobile measurement team in the measurement of gaseous emissions from open area sources using open-path and synthetic open path techniques.

1.2. An instrumentation trailer provides weather protection and a central communication point for all measurements made at the source as well as a means to transport the equipment from location to location.

1.3. The trailer is also used to provide a stable platform for the cellular communications antennas and all instrument communications antennas.

2. Definitions

2.1.	Al	Aluminum
2.2.	bLS	Backward Lagrangian Stochastic model
2.3.	CD	Compact disk
2.4.	cfm	Cubic feet per minute
2.5.	COM	Serial communications port
2.6.	DVD	Digital video disk
2.7.	GFCI	Ground fault circuit interrupter
2.8.	GSS	Gas sampling system
2.9.	HVAC	Heating, ventilation, and air conditioning
2.10.	kW	Kilowatt
2.11.	LAN	Local area network
2.12.	PAML	Purdue Applied Meteorology Lab
2.13.	PC	Personal computer
2.14.	PIC	Path-integrated concentration
2.15.	PM	Project manager
2.16.	RF	Radio frequency
2.17.	RPM	Radial Plume model
2.18.	SMP	Site Monitoring Plan
2.19.	S-OPS	Synthetic Open Path Sampling System
2.20.	TDLAS	Tunable Diode Laser Absorption Spectrometer
2.21.	UPS	Un-interruptible power supply
2.22.	USB	Universal serial bus
2.23.	VAC	Volts alternating current
2.24.	VDC	Volts direct current
2.25.		

3. Health and Safety

3.1. Power connections

3.1.1. Exercise care when working with the electrical system of the trailer. Wear proper personal protective equipment, such as leather gloves and safety glasses, when working around electrical circuits.

3.2. There is danger of falling with significant injury or death when working on the roof of the trailer, or on a ladder leading to the roof.
  3.2.1. Roof work safety:
    3.2.1.1. Do not attempt to climb onto slippery roofs. Inspect for and remove ice and snow before getting onto roof surfaces. If the roof surface is wet, wait until the surface is dry.
    3.2.1.2. Do not access the roof during storms, high winds or other adverse weather conditions.
    3.2.1.3. Remove or properly guard any impalement hazards.
    3.2.1.4. Wear shoes with slip-resistant soles.
    3.2.1.5. If you drop something while working on a roof, never try to catch or stop it. Let it fall and keep your own balance secure.
  3.2.2. Ladder safety:
    3.2.2.1. Keep manufactured and job-made ladders in good condition and free of defects.
    3.2.2.2. Inspect ladders before use for broken rungs or other defects so falls don't happen.
    3.2.2.3. Secure ladders near the top or at the bottom to prevent them from slipping.
    3.2.2.4. When you can't tie the ladder off, be sure the ladder is on a stable and level surface so it cannot be knocked over or have its bottom kicked out.
    3.2.2.5. Place ladders at the proper angle (4:1 slope = rise:run).
    3.2.2.6. Extend ladders at least 3 ft above the eave of the roof to provide a handhold, or for balance when getting on and off the ladder from the roof.
    3.2.2.7. Always face the ladder and maintain 3 points of contact when climbing it.

3.3. Be careful climbing the outside steps to and from the exterior door, particularly in inclement weather. Be mindful of wet or iced steps.

3.4. During lightning storms, avoid contact with electrical instruments, or with the exterior shell of the trailer.

3.5. Make yourself aware of the layout of the area upon arrival at a new location, and ask the property owner about where to find shelter in the event of a severe storm or tornado.

3.6. All field personnel must know the physical address of the current field location, so that this may be communicated in an emergency; if you’re not sure ask the property owner or the PM.

3.7. Do not operate the trailer tongue lift jack or attempt to hitch trailer without wearing suitable personnel protection equipment.

3.8. Trailer towing connections
  3.8.1. Be careful not to pinch hands when inserting the trailer socket onto the truck ball. Wear leather gloves.
  3.8.2. The ball and socket hitch connection must be firm.
  3.8.3. Chain the trailer to the truck at the trailer hitch, and be sure the chains do not drag on the ground.
    3.8.3.1. Chain lengths can be shortened to keep them from dragging on the road by twisting the chain prior to hooking it up to the truck.

3.9. Exercise proper care when setting up the trailer, including when setting up the blocks on which the trailer rests. Be mindful of crushing hazards.

3.10. Trailer tires
  3.10.1. Periodically inspect and maintain tow vehicle and trailer tires (including spare tires) and wheels.
  3.10.2. Keep appropriate pressure in the tires at all times. Proper tire pressure affects vehicle handling and the safety of tires. Find the correct tire pressure for a tow vehicle in the owner's manual, or on the tire information placard.
    3.10.2.1. Under inflation reduces the load-carrying capacity of a tow vehicle or trailer, may cause sway and control problems, and may result in overheating, causing blowouts or other tire failure.
    3.10.2.2. Over-inflation causes premature tire wear and affects the handling characteristics of the tow vehicle or trailer.
3.11. Improper loading/weight distribution in the trailer causes towing instability and may cause sudden and unexpected loss of control.
3.12. On a regular basis, have the brakes on both vehicles inspected. Be sure that necessary adjustments are made and any damaged or worn parts are replaced.
3.13. When transporting the trailer to and from the site, make sure that proper spare tires are available, and securely lock all exterior doors. Tow only with vehicles and hitches that are rated for the trailer weight. Follow any applicable state codes, and make sure that the trailer is properly licensed for the state.
3.14. Secure equipment prior to moving: Equipment may shift in transit when the equipment is not securely held to the trailer walls or floor.
  3.14.1. Load and secure instruments and equipment according to Appendix B.
3.15. Make sure gas cylinders are secured as described in SOP G2. Use a strap, chain or other fastener near the bottom of each cylinder, in addition to the top restraint, to prevent the cylinder from moving during transport. As per SOP G2, never transport cylinders with regulators attached.
  3.15.1. Pressurized gases can explode if cylinders are allowed to fall over.
  3.15.2. Leaking calibration gases can cause suffocation. Close cylinders tightly.
3.16. Be aware of traffic patterns on the farm. If possible, do not position the trailer in an area which is frequented by trucks or other vehicles.
3.17. Ensure that ventilation systems work properly, taking into account the following:
  3.17.1. If possible, do not position the trailer in a location where it will be directly in the exhaust stream of barn fans. Locate the trailer air inlet away from the barn fan exhausts.
  3.17.2. Ensure that the filters on the air inlet are changed often enough to mitigate the odors and exhausts which may be encountered on the site.

4. Cautions

4.1. Always check to see that trailer marker lights and both brake lights are functioning before towing trailer on any public roadway.
4.2. Power connections in the trailer
  4.2.1. Line voltage surges may damage computers and communications equipment.
  4.2.2. Improper connection of the trailer supply wiring may cause damage to computers and communications equipment, as well as personal injury.

- 4.3. Take care when installing, maintaining, or securing the communications antennas, and towers. The antenna and associated cables are delicate and must not be bent or broken. Do not drop.
- 4.4. Improper maintenance of the HVAC system can impair performance, leading to uncomfortable conditions.
  - 4.4.1. High levels of dust, feathers, and other airborne debris can readily clog the air inlets for the HVAC systems. This can impair performance, and may result in damage to the motor, compressor, and/or heating element.
  - 4.4.2. Clean or replace filters on the HVAC unit once a month (at minimum).
  - 4.4.3. Check the outdoor coils once a month (at minimum), and clean as necessary.
  - 4.4.4. Do not stack or prop anything against the outside portion of the HVAC unit, or block the outdoor coils in any way.
  - 4.4.5. Do not allow snow to pile up or drift around the outdoor HVAC unit.
  - 4.4.6. Periodically check the permanent drain connection near the inside coil (indoor HVAC unit) to make sure it is unobstructed, and that water is draining properly.
- 4.5. Calibration gases can become depleted if cylinders are not closed tightly.
  - 4.5.1. Replace cylinder when pressure drops below 200 psi.

5. Interferences

- 5.1. The trailer communications systems may not function properly if the trailer is located in the vicinity of a television station or radio station transmitting tower.
- 5.2. Power failures will adversely affect all communications and computational equipment.
- 5.3. Heating and cooling failures may result in environmental conditions which are outside the specifications of the equipment.

6. Personnel Qualifications

- 6.1. All field personnel must wear proper protective equipment while working in the field. This includes, but is not limited to, steel-toed shoes, leather gloves, and safety glasses.
- 6.2. All field personnel must have practiced driving and maneuvering with the trailer before leaving for a site. In addition to the cautions listed in Section 4, and the procedures listed in Section 8.3, guidelines for driving with a trailer are found in Appendix A.
- 6.3. All field personnel must read and understand this SOP prior to using the trailer.
- 6.4. All field personnel must be able to use the severe weather alert radio.
- 6.5. All personnel must be familiar with the general survival skills for severe storms and tornados in the field.
- 6.6. All personnel must be familiar with the dangers of lightning while working in open fields and on electrical/electronic equipment in general (see SOP U5 Appendix C).
- 6.7. All field personnel should know how to administer first aid in the event of an accident or emergency, and know the basics of CPR.
- 6.8. All field personnel should be aware of the location of your field partner(s) at all times. (Field units consist of a scientist and technician that function as a team).
- 6.9. All personnel should tell their team partner and the PM of any special needs or health concerns they might have in the event of an emergency.

6.10. All field personnel should know how to use the provided handheld two-way radios and cell phone to contact their team partner or emergency services personnel in the event of an accident/emergency.

6.11. Personnel must be able to use a multimeter and other necessary diagnostic tools required by the job.

7. Equipment and Supplies

7.1. Tools
  7.1.1. Tire iron
  7.1.2. Multimeter
  7.1.3. Chemical protective gloves
  7.1.4. Wrenches
    7.1.4.1. To fit gas regulators
    7.1.4.2. To fit tower bolts
  7.1.5. Screwdrivers
7.2. Spare bolts and nuts
7.3. 5 ft. ladder
7.4. Work light
7.5. Clock
7.6. Compressed air can for cleaning parts
7.7. 0.5 m TDLAS calibration cell
7.8. 1.0-ft^3 240-V 800-W electric convection oven
7.9. 1.5 cu ft. refrigerator
7.10. Hitch lock to prevent unauthorized towing.
7.11. (5) Lightweight wooden landing blocks for the purpose of leveling trailer, and landing hitch jack.
7.12. Data vault
7.13. Batteries for UPS unit installed inside
7.14. Activated charcoal (if charcoal filters are used)
7.15. Cleaning supplies: hand soap, paper towels, cloths, bucket, broom, trash bags and trash can
7.16. Fly traps or strips, mouse/rat poison
7.17. Trailer: Order or obtain a trailer that meets or exceeds the following minimum specifications, or their equivalents:
  7.17.1. Spare tires (minimum of two, stored in bed of tow vehicle)
  7.17.2. Frame & structural support
    7.17.2.1. 7′ wide x 14′ long
    7.17.2.2. Tandem 3500-lb (2 brake) axles
    7.17.2.3. Standard towing hitch
  7.17.3. Floor
    7.17.3.1. 3/4″ plywood deck
    7.17.3.2. R-11 insulation
    7.17.3.3. Tile floor covering
    7.17.3.4. Recessed D-ring tie downs every 2′ on centerline (starting 1′ from end)

7.17.4. Roof
  7.17.4.1. Minimum 8′ height inside
  7.17.4.2. R-10 insulation w/vapor barrier
  7.17.4.3. 3/8″ plywood sheathing inside
  7.17.4.4. 30-gage galvanized steel
7.17.5. Walls
  7.17.5.1. Heavy duty studs w/top and bottom plates
  7.17.5.2. R-10 insulation
  7.17.5.3. 3/8″ plywood interior walls
  7.17.5.4. D-ring tie downs every 2′ at 2′ and 4′ height (starting 1′ from end)
7.17.6. Exterior doors
  7.17.6.1. (1) Steel/wood door in side of trailer with bar lock
  7.17.6.2. (1) Large single hinge down steel/wood solid door at trailer back for loading/unloading with cam locks and lift assist system
7.17.7. Electrical system
  7.17.7.1. 100-Amp 240 VAC load center
    7.17.7.1.1. Two 50-A circuit branches (Phases) at 120 VAC (this is considered a standard 240VAC single-phase service)
    7.17.7.1.2. Standard 3-wire single phase (includes neutral).
  7.17.7.2. 100-Amp circuit breaker panel
  7.17.7.3. Receptacles
    7.17.7.3.1. (1) 240 VAC 15-Amp interior
    7.17.7.3.2. (2) 110-V 20-A Interior (for master UPS connection)
    7.17.7.3.3. (8) 110 VAC duplex 15-Amp interior
    7.17.7.3.4. (1) 110 VAC 15-Amp exterior with water seal protection
  7.17.7.4. Interior Lighting
    7.17.7.4.1. (3) 48″ 120VAC standard dual tube fluorescent lights with diffusers (installed lengthwise one in front, one in middle, one in rear).
    7.17.7.4.2. (2) 12 VDC “dome lights”.
  7.17.7.5. Lightning arrestor (built into UPS unit)
    7.17.7.5.1. Surge suppressor with minimum of 120,000 Amp/phase capacity (built into UPS unit)
7.17.8. HVAC system
  7.17.8.1. Sidewall closeable pass-through tube (for signal cable pass-through)
  7.17.8.2. 24,000 BTU/hr cooling or that adequate to cool to 70°F if outside is 120°F
  7.17.8.3. 5 kW heater or that adequate to heat to 70°F if outside is -30°F
  7.17.8.4. Return air at unit with remotely located thermostatic control.
7.17.9. General exterior
  7.17.9.1. 24″ Stoneguard on front
  7.17.9.2. Siding: 0.024 Al
  7.17.9.3. (4) Tie down points
  7.17.9.4. 4 Stabilizer jacks
  7.17.9.5. Running lights and tail lights
7.17.10. Furniture
  7.17.10.1. (1) 8′x2′ counter top permanently affixed to the front of the trailer walls

    - 7.17.10.2. (1) stool/chair
  - 7.17.11. Storage
    - 7.17.11.1. (1) wall-mounted file holder
    - 7.17.11.2. (2) 18″ deep lockable cabinets on sidewall
    - 7.17.11.3. (1) Portable file cabinet
    - 7.17.11.4. (1) Three-drawer cabinet affixed to the sidewall
- 7.18. Cable pass-through should accommodate the following
  - 7.18.1. Coaxial cables for RF modems:
    - 7.18.1.1. (4) for communications with TDLAS/scanner system
    - 7.18.1.2. (1) for communications with data logger
    - 7.18.1.3. (1) for communications with sonic anemometers
    - 7.18.1.4. (1) for cellular internet
- 7.19. Tubing pass-through should accommodate the following
  - 7.19.1. (4) 3/8″ OD Teflon tubing:
  - 7.19.2. (1) 1/4″ OD Teflon tubing:
  - 7.19.3. (2) 5/8″ ID PVC tubing
  - 7.19.4. (1) 3/16″ ID PVC tubing:
- 7.20. Install the following in the trailer:
  - 7.20.1. Instrument rack: Constructed of steel and able to accept sliding trays or rails. Open racks help to maintain low instrument temperature and good air circulation firmly secured to the sidewall.
    - 7.20.1.1. (1) INNOVA 1412 multi-gas analyzer (SOP G7)
    - 7.20.1.2. (1) Thermo Fisher Scientific 450i Pulsed Florescence H2S Analyzer (SOP G5)
    - 7.20.1.3. (1) Thermo Fisher Scientific 146i Diluter (SOP C3)
    - 7.20.1.4. (1) LAN server computer (SOP D1), which includes the following:
      - 7.20.1.4.1. Serial port expansion bus
      - 7.20.1.4.2. UPS web adapter card and Network Protection Module
    - 7.20.1.5. (1) Web power switch for internet access of power in trailer
    - 7.20.1.6. (1) LAN server external storage drive
    - 7.20.1.7. (1) Power distribution and surge protection device
    - 7.20.1.8. (1) 120-VAC In/Out double conversion Uninterruptible Power Supply.
    - 7.20.1.9. (1) Read//write CD/DVD drive
    - 7.20.1.10. (1) Flash memory drive reader
  - 7.20.2. Install on wall near instrument rack
    - 7.20.2.1. (1) Cellular RF internet modem for communication with PAML and internet
    - 7.20.2.2. (1) 24VDC power supply for GSS solenoid power
    - 7.20.2.3. Multiple Ethernet ports using an Ethernet switch and router
    - 7.20.2.4. (6) RF modem transceivers (in trailer)
      - 7.20.2.4.1. (1) for meteorological data logger
      - 7.20.2.4.2. (1) for sonic anemometers
      - 7.20.2.4.3. (4) for TDLAS
    - 7.20.2.5. (8) RF modem transceivers (at instruments)
      - 7.20.2.5.1. (1) for meteorological data logger
      - 7.20.2.5.2. (3) Built into power unit of sonic anemometers
      - 7.20.2.5.3. (4) Built into power unit of TDLAS

- 7.20.2.6. (1) Scanner (for copying paper documents)
- 7.20.2.7. USB, RF modems
- 7.20.2.8. Gas sampling system: attached with shelf brackets near or mounted on top of instrumentation rack..
- 7.20.2.9. CR800 Data logger and A21 REL-12 relay controller.

7.20.3. Compressed gases

- 7.20.3.1. Total non-flammable gases and total flammable gases must weigh less than 220 lbs each. If not, Department of Transportation placarding and special inspections and approvals are required.
- 7.20.3.2. One four-cylinder rack on the wheel well and two auxiliary positions in front of wheel well to hold calibration gas cylinders. See SOP G2 for the proper means of securing cylinders to the rack(s), and for the proper regulators to use with each gas. Cylinders should be A3 size or equivalent, except for CEM zero air (size AQ).
  - 7.20.3.2.1. NH_3
  - 7.20.3.2.2. H_2S
  - 7.20.3.2.3. Methane/propane
  - 7.20.3.2.4. Methanol/ethanol
  - 7.20.3.2.5. CEM zero air
  - 7.20.3.2.6. SO_2 calibration
- 7.20.3.3. One cylinder rack on cabinet to hold zero (N_2) gas cylinders.

7.20.4. Miscellaneous radio equipment

- 7.20.4.1. NOAA severe weather alert receiver with internal battery backup
- 7.20.4.2. Two way radios for communications between FOS.

7.20.5. Appliances under counter

- 7.20.5.1. Small refrigerator to store chemicals only
- 7.20.5.2. Convection oven to dry chemicals and desiccants.

7.20.6. Internal configuration of storage areas (Fig. 1)

- 7.20.6.1. Store the following in place on meteorological mast secured using straps attached to D-rings:
  - 7.20.6.1.1. (1) NEMA box with data logger, barometer, and RF modem, and antenna for modem
  - 7.20.6.1.2. (1) Solar radiation sensor and mounting fixture and bracket
  - 7.20.6.1.3. (1) RH/T sensor in passive radiation shield
  - 7.20.6.1.4. (3) Sonic anemometers and their modems
  - 7.20.6.1.5. (2) TDLAS/scanner systems
  - 7.20.6.1.6. (20) Retro-reflectors with mounting brackets (10″ x 9″ x 7″ boxes)
    - 7.20.6.1.6.1. (8) Mounting on tripods
    - 7.20.6.1.6.2. (12) Mounting on fixed towers
- 7.20.6.2. Store the following in the rear of tow vehicle inside plastic pail
  - 7.20.6.2.1. (1-2) 107-L thermistor
  - 7.20.6.2.2. (1-2) ORP redox state sensor
  - 7.20.6.2.3. (1-2) pH sensor
  - 7.20.6.2.4. Lagoon probe float(s) and associated tie down ropes
- 7.20.6.3. Tripods (stored in plastic bin)

  - 7.20.6.3.1. (8) Tripods for retro-reflectors
  - 7.20.6.3.2. (2) Tripods for TDLAS/scanner systems
  - 7.20.6.3.3. (1) Tripod for calibration cells
- 7.20.6.4. (1) Fold-down 3-m mast
- 7.20.6.5. Ground rods (SOP U5)
- 7.20.6.6. 3/8″ ID Teflon tubing for SOPS sampling
  - 7.20.6.6.1. (1) 500′ section of tubing for S-OPS extension
  - 7.20.6.6.2. (1) 1000′ section of tubing for S-OPS extension
  - 7.20.6.6.3. (2) 50′ S-OPS sampling manifold with filtered inlets
- 7.20.6.7. 1/4″ ID tubing for connections between cylinders, analyzers, rotameters, diluter, and calibration chamber.
- 7.20.6.8. Cables
  - 7.20.6.8.1. (2) Power cables for TDLAS/scanner systems
  - 7.20.6.8.2. (2) Power/Signal cables for TDLAS to scanner and scanner to power supply hookup
  - 7.20.6.8.3. (4) Serial communications cables for TDLAS/scanner
  - 7.20.6.8.4. Coaxial cables for RF modems:
  - 7.20.6.8.5. (4) for trailer for communications with TDLAS/scanner system
  - 7.20.6.8.6. (1) for trailer for communications with CR1000 data logger
  - 7.20.6.8.7. (1) for trailer for communications with CR800 data logger
  - 7.20.6.8.8. (1) for trailer for communications with sonic anemometers
  - 7.20.6.8.9. (1) for trailer for communications with Cellular Modem
  - 7.20.6.8.10. (1) spare cable
- 7.20.6.9. Additional computers (stored in protective cases under front counter)
  - 7.20.6.9.1. (2) TDLAS/ laptop computers (SOP C2, SOP O2).
- 7.20.6.10. Communications equipment
  - 7.20.6.10.1. Antenna for RF communications (stored in cabinet drawer):
    - 7.20.6.10.1.1. (4) for trailer for TDLAS/scanner systems
    - 7.20.6.10.1.2. (1) for trailer for communications with data logger
    - 7.20.6.10.1.3. (1) for trailer for communications with sonic anemometers
    - 7.20.6.10.1.4. (1) for trailer for communications with Cellular Modem
  - 7.20.6.10.2. RF modems (internal means in trailer mounted on wall):
    - 7.20.6.10.2.1. (4) internal for TDLAS/scanner systems
    - 7.20.6.10.2.2. (1) internal for data logger communications
    - 7.20.6.10.2.3. (1) internal for polled anemometer communications
    - 7.20.6.10.2.4. (1) Airlink “Raven X” Cellular Internet Modem
    - 7.20.6.10.2.5. (4) external for TDLAS/scanner systems (built into TDLAS power supply boxes)
    - 7.20.6.10.2.6. (1) external secured inside NEMA box for data logger
    - 7.20.6.10.2.7. (3) external built into anemometer power boxes
- 7.20.6.11. Miscellaneous hardware supplies (to be stored in cardboard or plastic boxes):
    - 7.20.6.11.1. Stakes
    - 7.20.6.11.2. Clamps
    - 7.20.6.11.3. Tie-down straps
    - 7.20.6.11.4. Spare bolts for towers and sensor mounting brackets

7.20.6.12. Eye wash station (attached to wall)
7.20.6.13. Two-gallon container for hand/emergency wash water
7.20.6.14. First Aid kit (attached to wall)
7.20.6.15. Fire extinguisher (stored in truck during transit)
7.20.6.16. (2) 1400VA APC brand UPS units for TDLAS system field backups (stored on floor under front counter during transport)
7.20.6.17. TDLAS back-up battery protection box stored on floor during transport (used for containing straps/cords during transport).

8. Procedures

8.1. Acceptance
  8.1.1. Conduct a thorough check of the wiring system before turning power on to the trailer. Make sure that all instruments are disconnected before supplying power to the trailer for the first time.
  8.1.2. Environment monitoring
    8.1.2.1. Heating and cooling
      8.1.2.1.1. .Set thermostat to ambient temperature + 20°F to test heating, or ambient temperature - 20°F to test cooling.
      8.1.2.1.2. If air temperature in trailer stabilizes at set temperature ± 3°F, system passes.
      8.1.2.1.3. If air temperature in trailer does not stabilize at set temperature ± 3°F, system fails; contact manufacturer for repair.
  8.1.3. Power distribution
    8.1.3.1. Test each outlet for line voltage.
      8.1.3.1.1. Identify which outlets go with each circuit breaker and mark in breaker box and in trailer log.
        8.1.3.1.1.1. If an outlet does not have appropriate voltage, check circuit breaker or upper GFCI receptacle (if one is above it).
        8.1.3.1.1.2. Reset if needed and retest.
        8.1.3.1.1.3. If there is still no power, contact manufacturer for rectification.
    8.1.3.2. Test each outlet for correct polarity.
      8.1.3.2.1. If polarity is incorrect, notify manufacturer for resolution.
  8.1.4. Lighting
    8.1.4.1. Turn lights on and check for all lamps to light.
      8.1.4.1.1. If lamp does not light, replace lamp and retry. If problems continue, contact manufacturer for rectification.
  8.1.5. Storage (according to Fig. 1)
    8.1.5.1. Fire-proof data vault for storage of data backups between measurement periods
    8.1.5.2. Drawers in plastic file cabinet
      8.1.5.2.1. Logbooks, calibration tables, and checklists
        8.1.5.2.1.1. For individual sensors
        8.1.5.2.1.2. Data logger installation
      8.1.5.2.2. Site installation forms
        8.1.5.2.2.1. Towers
        8.1.5.2.2.2. Open source gas measurement systems

- 8.1.5.2.3. Material Safety Data sheets for calibration gases, pH calibration solutions, redox calibration solutions and other chemicals
- 8.1.5.2.4. All relevant SOPs
- 8.1.5.2.5. Copy of QAPP
- 8.1.5.2.6. Copy of SMPs
- 8.1.5.2.7. Manuals for all instruments

8.1.5.3. Cabinets according to Fig. 1

- 8.1.5.3.1. Electronic storage media: Writeable DVDs and CDs
- 8.1.5.3.2. pH calibration solutions
- 8.1.5.3.3. Redox calibration solutions
- 8.1.5.3.4. Relative humidity salts
  - 8.1.5.3.4.1. Individual sensors attached to each SOP
- 8.1.5.3.5. Trailer walls
  - 8.1.5.3.5.1. HVAC unit
  - 8.1.5.3.5.2. RF modems

8.1.5.4. Cable storage tie downs according to Fig. 1

8.1.5.5. Tubing storage on walls and on reels as needed.

- 8.1.5.5.1. 1/4″ ID tubing for connections between cylinders, analyzers, rotameters, diluter, and calibration chamber.
- 8.1.5.5.2. 3/8″ ID tubing for S-OPS and connection of S-OPS with GSS
  - 8.1.5.5.2.1. (1) 500′ section of tubing for S-OPS extension
  - 8.1.5.5.2.2. (1) 1000′ section of tubing for S-OPS extension
  - 8.1.5.5.2.3. (2) 50′ S-OPS sampling manifold with filtered inlets

8.1.5.6. Rack mount according to Fig. 1 (SOP D1)

- 8.1.5.6.1. Network LAN server computer
- 8.1.5.6.2. Thermo Fisher Scientific 146i diluter
- 8.1.5.6.3. Thermo Fisher Scientific Pulsed Florescence H_2S analyzer
- 8.1.5.6.4. UPS
- 8.1.5.6.5. Power distribution center for computers
- 8.1.5.6.6. External storage drives
- 8.1.5.6.7. Ethernet communications switch

8.1.5.7. Gas cylinders affixed to wall storage unit/tie downs (SOP G2)

- 8.1.5.7.1. Seven cylinder mounting brackets for compressed gases
- 8.1.5.7.2. NH_3 calibration gas cylinder and appropriate regulator (CGA 705)
- 8.1.5.7.3. H_2S calibration gas cylinder and appropriate regulator (CGA 330)
- 8.1.5.7.4. CEM Zero air (N_2) gas cylinder and appropriate regulator (CGA 580)
- 8.1.5.7.5. CEM Zero air gas cylinder and appropriate regulator (CGA 580)
- 8.1.5.7.6. SO_2 calibration gas cylinder and appropriate regulator (CGA 660)
- 8.1.5.7.7. Methane/propane calibration gas cylinder and appropriate regulator (CGA 350)
- 8.1.5.7.8. Methanol/ethanol calibration gas cylinder and appropriate regulator (CGA 350)

8.1.5.8. First aid kit (attached to wall near side entrance door)

- 8.1.5.8.1. A first aid kit is located in the field trailer. It can be used for minor injuries, including abrasions, contusions, punctures, and burns

8.1.5.9. Eye Wash Stations

8.1.5.9.1. An eye wash station is located in the field trailer. It is to be used when chemicals spill on skin, or to flush a foreign object from the eye. Do not use if the tamper seal is broken, or if the solution is cloudy. Do not drink the solution.

8.1.5.10. A two-gallon water container is located in the trailer for the purpose of washing hands, or for quick access to water in the event of a small emergency (stored on floor).

8.1.5.11. Fire extinguisher

8.1.5.11.1. A dry chemical (ABC) type fire extinguisher is located in the field trailer. ABC-type fire extinguishers can be used for most types of fires, including the following:

Class A - Ordinary Combustibles; examples: trash, wood, and paper.
Class B - Flammable & Combustible Liquids: e.g. gasoline, diesel, grease.
Class C - Electrical Equipment; examples: computers, outlets and cords.

8.1.5.11.2. Fire extinguisher to be carried in truck during transport of trailer

8.2. Troubleshooting

8.2.1. Heating/cooling

8.2.1.1. Check thermostat settings.

8.2.1.2. Check for the presence of 240 VAC at supply.

8.2.1.3. Check breakers in circuit breaker box and GFCI outlet on circuit, and reset as needed.

8.2.2. If power fails for an instrument in field:

8.2.2.1. Check connection to power outlet on post.

8.2.2.2. Check breakers in circuit breaker box, and reset as needed.

8.2.3. If signal fails for an instrument:

8.2.3.1. Follow procedures indicated in the SOP for the instrument.

8.2.4. If RF Communications fails for an instrument:

8.2.4.1. Follow procedures in appropriate instrument SOP (SOPs W6, W2, C1, C2).

8.2.5. If Cellular Communications fails:

8.2.5.1. Check operational lights on RF modem.

8.2.5.1.1. If proper lights are lit, reset router and wait three minutes for it to reboot

8.2.5.1.2. If still not working, turn modem off then back on (reset).

8.2.5.1.3. Wait four minutes then reset router again.

8.2.5.1.4. If problems continue, contact PM at PAML for remediation.

8.3. Transportation

8.3.1. Ensure that all exterior doors are securely locked, all instruments and all gas cylinders are securely fastened.

8.3.2. Ensure vehicle and trailer maintenance is current. This is very important because towing puts additional stress on the tow vehicle.

8.3.3. Check and correct tire pressure on the tow vehicle and trailer.

8.3.4. Check for proper trailer loading to ensure balance is correct. Use common sense when determining trailer balance.

8.3.4.1. The trailer should be weighted so that the tongue drops the rear of the tow vehicle somewhat when dropping onto the hitch ball.

    - 8.3.4.2. If the weight is excessive (tow vehicle squats significantly), check trailer loading and re-orient the contents slightly to provide less tongue weight.
    - 8.3.4.3. Conversely, if the tongue is very light (tow vehicle doesn't respond to dropping ball onto hitch) check trailer loading and re-orient the contents slightly to provide more tongue weight.
      - 8.3.4.3.1. Use caution when entering the trailer after the stabilizers have been raised, as the trailer might suddenly tip back if not securely hitched to tow vehicle, causing damage to trailer, contents, and the possibility of severe personal injury!
  - 8.3.5. Be sure the hitch, coupler, and other equipment that connect the trailer and the tow vehicle are properly secured and adjusted.
  - 8.3.6. Check that the wiring is properly connected—not touching the road, but loose enough to make turns without disconnecting or damaging the wires.
  - 8.3.7. Make sure all running lights, brake lights, turn signals, and hazard lights work.
  - 8.3.8. Verify that the brakes on the tow vehicle and trailer are operating correctly.
  - 8.3.9. Check that all items are securely fastened on and in the trailer.
  - 8.3.10. Be sure the trailer jack, tongue support, and any attached stabilizers are raised and locked in place.
  - 8.3.11. Check to see that the wooden landing blocks have been picked up and loaded into trailer or tow vehicle.
  - 8.3.12. Fully extend side mirrors and check all mirrors to make sure they are properly adjusted and you have good visibility.
  - 8.3.13. Check routes and restrictions on bridges and tunnels.
- 8.4. Site establishment and trailer unloading
  - 8.4.1. Position trailer to match correct location for ground driven anchors .
  - 8.4.2. Prop the hitch and disconnect it from the truck.
  - 8.4.3. Disconnect trailer wiring cable from the tow vehicle.
  - 8.4.4. Level trailer according to Appendix C.
  - 8.4.5. Anchor trailer between ground-driven anchors and trailer anchor points with new strap set and special purpose bolts.
  - 8.4.6. Attach hitch lock on the hitch for security purposes.
  - 8.4.7. Connect trailer power:
    - 8.4.7.1. Attach the bare #6 Grounding wire from the trailer supply box to the ground rod.
    - 8.4.7.2. Using a multimeter set to measure AC voltage, test the provided power access point for the correct supply voltage. Acceptable limits are 210-240 volts phase to phase, and 105-120 volts phase to neutral. Be absolutely certain to turn off the supply source (where possible) before connecting the power cable.
    - 8.4.7.3. Use a long 80-A power cord to connect to an appropriate circuit breaker or switch box at the farm-provided power access point and to the internal electrical panel
      - 8.4.7.3.1. Be sure to hook the correct wire to neutral and each of the of the phases to the respective 120 volt phase lugs of the circuit breaker or fuse block to achieve 240 volts phase to phase and 120 volts phase to neutral.
      - 8.4.7.3.2. Test the neutral to earth ground voltage with a multimeter to determine whether it meets the requirements of the electric code.

8.4.7.3.2.1. Use extreme caution when making this measurement, as potentially hazardous voltages could be present between the earth ground point and the neutral connection to the trailer.

8.4.7.3.3. If the voltage exceeds 2 volts, or the specified value in the local electric code (whichever is lower), take corrective action.

8.4.7.3.3.1. Check the condition of the supply cable and connectors at both ends, paying particular attention to the neutral conductor.

8.4.7.3.3.2. Check the neutral to earth ground voltage at the supply from the farm-provided power access point, to determine if the problem is coming from there. If so, turn off the source and contact the electrician contracted with the respective farm for remediation (SOP S7).

8.4.7.3.3.2.1. Use caution near the suspected faulty power access point.

8.4.7.4. Ensure that the shelter is sufficiently grounded for proper operation of all equipment

8.4.7.5. Check polarity of all wiring before connecting instruments.

8.4.7.6. Check for power in trailer.

8.4.7.6.1. Check to see that the voltage is within acceptable limits (210-240 volts phase to phase and 105-120 volts phase to neutral). If not, check voltages at the farm-provided power access point.

8.4.7.6.1.1. If the voltage is not acceptable at the provided access point, turn off the source and contact the electrician contracted with the respective farm for remediation (SOP S7).

8.4.7.6.1.2. If the voltage is acceptable at the provided access point, but is not correct at the trailer, check the power cable and connections at each end.

8.4.8. Turn on the rack-mounted UPS in accordance with manufacturer’s instructions, wait two minutes for the unit to self test and power up.

8.4.8.1. Verify that the UPS is operating properly.

8.4.9. Unpack radio antenna from trailer.

8.4.10. Mount all communications antennas to the roof mount points on the trailer.

8.4.11. Connect the modem receiver cables to the antennas.

8.4.12. Route the RF modem receiver cables from the outside antennas into the trailer through the antenna cable pass-through, and connect to the modem receivers. Attach the 2.4-GHz antennas to the proper radios and the 900-MHz antenna cable to the 900-MHz modem.

8.4.13. Follow SOP U5 and SOP U7 to connect and set up the modems.

8.4.14. Unpack sensors and conduct installation procedures for each.

8.4.14.1. Return packing boxes to trailer.

8.4.15. Set up synthetic open paths according to SOP C4, U5 or U7, and the respective site monitoring plan (SMP).

8.4.15.1. (1) S-OPS extension is 500′ long and belongs at the path location nearest the trailer.

8.4.15.1.1. Connect section of extension with stainless steel connectors.

8.4.15.2. (1) S-OPS extension is 1000′ long and belongs at the path location furthest from the trailer.

8.4.15.2.1. Connect section of extension with stainless steel connectors.

8.4.15.3. Pass S-OPS extension tube through trailer tubing pass-through and connect to GSS.
8.4.15.4. Return tubing reels to trailer.
8.4.15.5. Attach S-OPS extensions to S-OPS sampling manifolds
8.4.15.6. Follow procedures in SOP C4 to prepare for S-OPS operation.
8.4.16. Check communications between appropriate computer and sensor(s) (SOP W6).
8.4.16.1. TDLAS (SOP C2)
8.4.16.2. 3 sonic anemometers (SOP W2)
8.4.16.3. CR1000 data logger (SOP W6)
8.4.16.4. CR800 data logger (SOP C4)
8.4.16.5. Cellular internet modem
8.4.17. Verify all critical internal equipment is functioning normally through the UPS.
8.5. Site disassembly and trailer loading
8.5.1. Remove Retro-reflectors and anemometers from towers (SOP U5).
8.5.1.1. Perform anemometer intercomparison check using meteorological mast and special mount (stored in rear of tow vehicle).
8.5.2. Remove S-OPS from posts, roll up tubing and disconnect tubing from 47-mm filter in trailer on intake line.
8.5.3. Disconnect RF modem cables and pass back outside.
8.5.3.1. Remove cables from RF modem antennas and store in trailer.
8.5.3.2. Remove radio antennas from roof mount points on the trailer and store in cabinet drawer,
8.5.4. Load trailer with equipment according to plan in Appendix B so that weight distribution is optimized.
8.5.4.1. Store TDLAS/scanners, retro-reflectors, tripods, etc, in appropriate shipping boxes.
8.5.4.2. Remove gas regulators from all cylinders and store regulators in appropriate shipping boxes.
8.5.4.3. Install cylinder caps to protect cylinder valves during transport.
8.5.4.4. Install safety frame over A3 gas cylinders and secure with wing nuts.
8.5.5. Switch power feed off at trailer.
8.5.5.1. Switch power feed off at source.
8.5.6. Disconnect trailer power cable from post power socket.
8.5.7. Disconnect #6 ground wire and store in trailer.
8.5.8. Remove anchor strap from trailer to ground-driven anchors.
8.5.8.1. Store anchor strap bolts in trailer.
8.5.9. Raise all corner jacks and lock into upper (travel) position
8.5.10. Hitch trailer to tow vehicle.
8.5.10.1. Visually inspect the nuts, bolts, and other fasteners to ensure that the hitch remains secured to the tow vehicle and the coupler remains secured to the trailer. The connection point may require periodic lubrication to permit free movement of the coupler to the hitch ball.
8.5.11. Secure trailer to tow vehicle with chain and check slack.
8.5.11.1. Correct problems if chain is loose.
8.5.12. Load towers and mast onto trailer according to SOP U5.

8.5.13. Connect backup and brake lights cables.
  8.5.13.1. Test lights.
  8.5.13.2. If a problem exists, make sure connector-plug prongs and receptacles, light sockets, wire splices, and ground connections are clean and shielded from moisture. Lightly coat all electrical terminal connections with non-conducting (dielectric), light waterproof grease.
    8.5.13.2.1. Clean the prongs with very fine sandpaper, being careful not to damage the contact area.
    8.5.13.2.2. Clean the surface deposits in the connector holes (while lights are off to prevent blowing a fuse). Try to clean off only the deposits and lubricate lightly with dielectric, light waterproof grease.
    8.5.13.2.3. If problems continue, correct wiring problems before leaving nearest repair facility.

8.6. Operations
  8.6.1. Communications (Fig. 2)
    8.6.1.1. Between trailer laptop PCs and TDLAS is via RF modems
    8.6.1.2. Between trailer PC and weather CR1000 data logger is via RF modems
    8.6.1.3. Between trailer PC and PAML is via a Cellular RF modem
    8.6.1.4. Between trailer PC and GSS CR800 data logger is via hard-wired RS-232
    8.6.1.5. The outside weather conditions will be monitored.

8.7. Computer Hardware & Software
  8.7.1. PCs for collecting data and quality control of measurements (SOP D1) (Fig. 2)
  8.7.2. Computers will be on a local-area network.
    8.7.2.1. Each computer will have surge protection via the UPS.
    8.7.2.2. Each computer will have a 20-min capacity backup power supply.
  8.7.3. (1) Rack-mounted network LAN server computer
    8.7.3.1. The computer will run the LoggerNet software for the control of the GSS and meteorological/ lagoon measurement data loggers and the storage of measurements from the dataloggers.
      8.7.3.1.1. All weather conditions (temperature, barometric pressure, solar radiation, relative humidity, surface wetness), and lagoon conditions (pH, temperature, and redox state) will be recorded by the CR1000 data logger and periodically transferred to the LAN server via RF modem link (SOP W6)
      8.7.3.1.2. GSS conditions (sample manifold pressure, flow direction, and mass flow) will be recorded by the CR800 Data logger and continuously transferred to the LAN server (SOP C4).
      8.7.3.1.3. GSS solenoid control for sampling synthetic open paths will be through a relay controller and the CR800 Data logger (SOP C4).
    8.7.3.2. The computer will run “Visual Basic” software to collect and record wind component, turbulence (SOP W2), and NH_3 and VOC measurements (SOP G7).
    8.7.3.3. The computer will run “iPort” software to collect and record H_2S measurements (SOP G5).

9. Data and Records Management

9.1. Document all data and information from the field on field data sheets and in site logbooks with permanent ink, or in electronic notes.
9.2. Overstrike all errors in writing with a single line. Initial and date all such corrections.

10. Quality Control and Quality Assurance

10.1. At the beginning and end of each measurement period on site
  10.1.1. Follow and document a checklist of setup procedures.
  10.1.2. Follow and document a checklist of disassembly procedures.
10.2. Document all problems encountered during setup, and their resolutions, on the checklists.
10.3. If environmental conditions in the trailer exceed equipment specifications, log the event in the daily log and take remedial action. Document the action taken, and the resolution.
10.4. Lock trailer when unoccupied.

11. References

11.1. iPort Instruction Manual. 2007. *i*Port Instruction Manual. Communications software for iSeries and C Series Instruments. Part#102606-00. Thermo Fisher Scientific, Franklin, MA.
11.2. SOP C2. 2006. Measurement of Ammonia with the Boreal Laser GasFinder Tunable Diode Laser Absorption Spectrometer (TDLAS). Standard Operating Procedure C2. Purdue Applied Meteorology Lab.
11.3. SOP C3. 2006. Operation of the Thermo Electron Model 146C Multi-gas Diluter. Standard Operating Procedure C3. Purdue Ag Air Quality Lab
11.4. SOP C4. 2006. Synthetic Open Path Sampling System. Standard Operating Procedure C4. Purdue Applied Meteorology Lab.
11.5. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
11.6. SOP G2. 2006. Compressed Gas Cylinders Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
11.7. SOP G5. 2007. Measurement of Hydrogen Sulfide (H_2S) with the Thermo Electron Corporation Model 450I Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab
11.8. SOP G7. 2006. Use of the INNOVA 1412 Photoacoustic Multi-Gas Monitor. Standard Operating Procedure G7. Purdue Ag Air Quality Lab.
11.9. SOP S7. 2006. Producer Collaborations at Area Source Monitoring Sites. Standard Operating Procedure S7. Purdue Applied Meteorology Lab.
11.10. SOP U5. 2006. Installation of Open Source Measurements. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.
11.11. SOP U7. 2007. The Instrument Trailer and Installation of Open-Source Measurement Equipment (Corrals). Standard Operating Procedure U7. Purdue Applied Meteorology Lab

11.12. SOP W2. 2006. Ultrasonic Anemometer for Wind Velocity. Standard Operating Procedure W2. Purdue Applied Meteorology Lab.
11.13. SOP W6. 2006. Establishment, Data Acquisition and control of weather and lagoon characterization hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.

Figure 1. Trailer storage configuration (14' x 7').

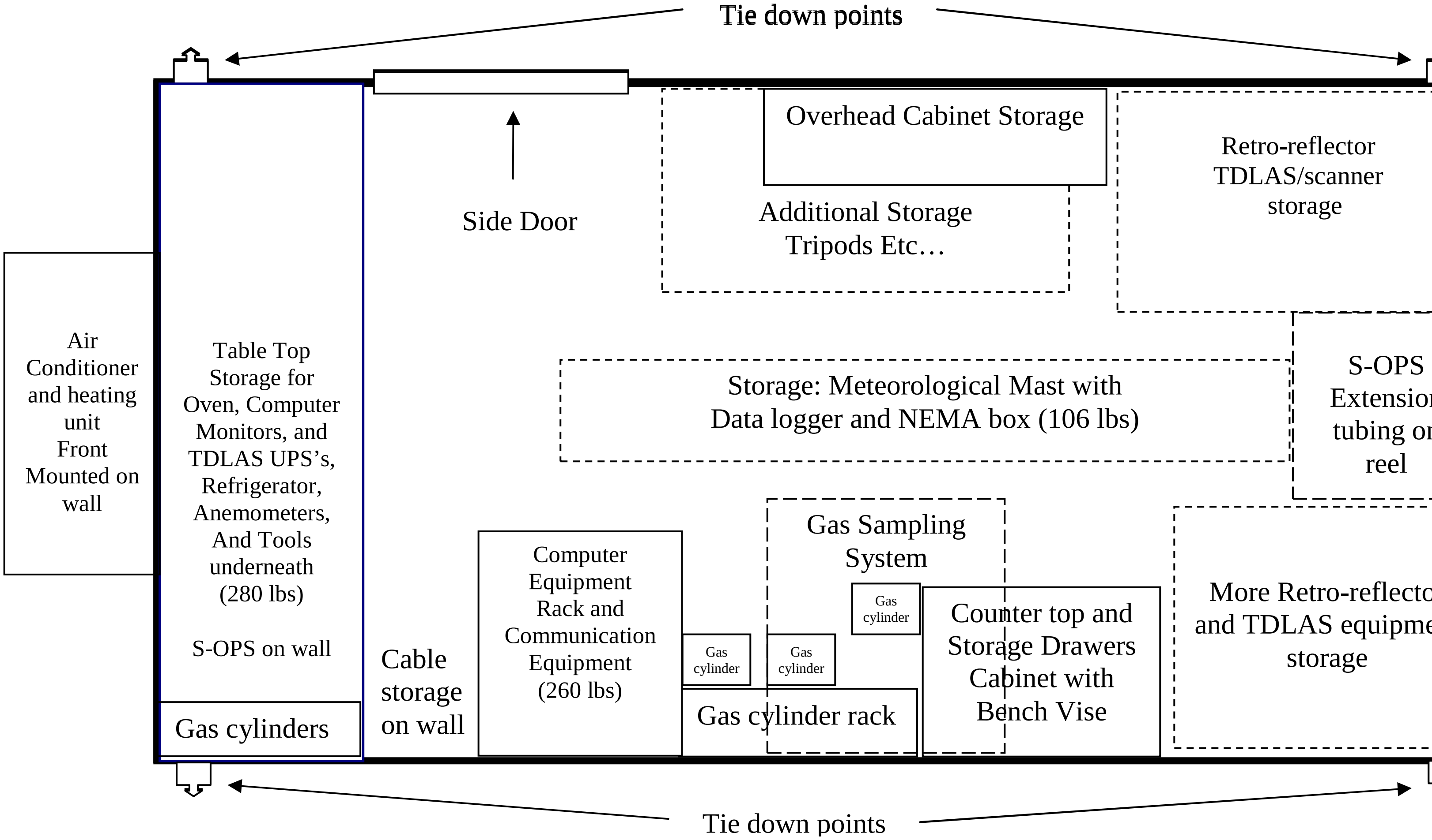
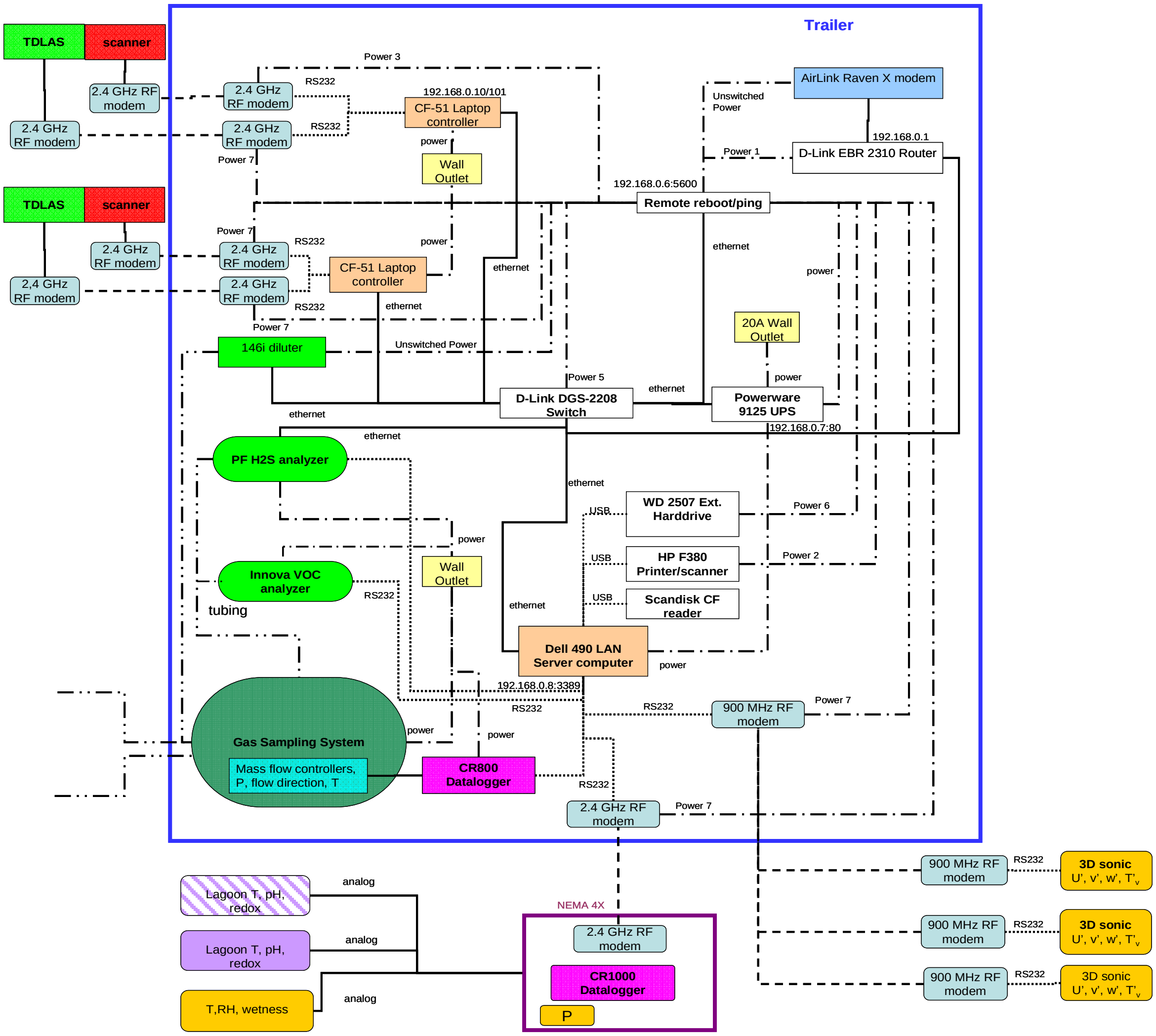


Figure 2. Communications schematic.



Appendix A. Safety Tips for Driving with a Trailer

Take time to practice before driving on main roads and never allow anyone to ride in or on the trailer. Before you leave, remember to check routes and restrictions on bridges and tunnels. Consider the following safety tips each time you drive with a trailer.

General Handling

- Use the driving gear that the manufacturer recommends for towing.
- Drive at moderate speeds. This will place less strain on your tow vehicle and trailer. Trailer instability (sway) is more likely to occur as speed increases.
- Avoid sudden stops and starts that can cause skidding, sliding, or jackknifing.
- Avoid sudden steering maneuvers that might create sway or undue side force on the trailer.
- Slow down when traveling over bumpy roads, railroad crossings, and ditches.
- Make wider turns at curves and corners. Because your trailer's wheels are closer to the inside of a turn than the wheels of your tow vehicle, they are more likely to hit or ride up over curbs.
- To control swaying caused by air pressure changes and wind buffeting when larger vehicles pass from either direction, release the accelerator pedal to slow down and keep a firm grip on the steering wheel.

Braking

- Allow considerably more distance for stopping.
- If you have an electric trailer brake controller and excessive sway occurs, activate the trailer brake controller by hand. Do not attempt to control trailer sway by applying the tow vehicle brakes; this will generally make the sway worse.
- Always anticipate the need to slow down. To reduce speed, shift to a lower gear and press the brakes lightly.

Acceleration and Passing

- When passing a slower vehicle or changing lanes, signal well in advance and make sure you allow extra distance to clear the vehicle before you pull back into the lane.
- Pass on level terrain with plenty of clearance. Avoid passing on steep slopes.
- If necessary, downshift for improved acceleration or speed maintenance.
- When passing on narrow roads, be careful not to go onto a soft shoulder. This could cause your trailer to jackknife or go out of control.

Downgrades and Upgrades

- Downshift to assist with braking on downgrades and to add power for climbing hills.
- On long downgrades, apply brakes at intervals to keep speed in check. Never leave brakes on for extended periods of time or they may overheat.
- Some tow vehicles have specifically calibrated transmission tow-modes. Be sure to use the tow-mode recommended by the manufacturer.

Backing Up

- Put your hand at the bottom of the steering wheel. To turn left, move your hand left. To turn right, move your hand right. Back up slowly. Because mirrors cannot provide all of the visibility you may need when backing up, have someone outside at the rear of the trailer to guide you, whenever possible.
- Use slight movements of the steering wheel to adjust direction. Exaggerated movements will cause greater movement of the trailer. If you have difficulty, pull forward and realign the tow vehicle and trailer and start again.

Parking

- Try to avoid parking on grades. If possible, have someone outside to guide you as you park. Once stopped, but before shifting into Park, have someone place blocks on the downhill side of the trailer wheels. Apply the parking brake, shift into Park, and then remove your foot from the brake pedal. Following this parking sequence is important to make sure your vehicle does not become locked in Park because of extra load on the transmission. For manual transmissions, apply the parking brake and then turn the vehicle off in either first or reverse gear.
- When uncoupling a trailer, place blocks at the front and rear of the trailer tires to ensure that the trailer does not roll away when the coupling is released.
- An unbalanced load may cause the tongue to suddenly rotate upward; therefore, before un-coupling, place jack stands under the rear of the trailer to prevent injury.

Appendix B. Trailer Weight Distribution

The ability to handle and control the tow vehicle and trailer is greatly improved when the cargo is properly loaded and distributed. Refer to your tow vehicle and trailer owner’s manual to find out how to

- Balance weight from side to side
- Distribute cargo weight evenly along the length of the trailer
- Secure and brace all items to prevent them from moving during travel
- Adjust the height of the tow vehicle/trailer interface
- Use common sense when balancing trailer, if the tongue appears to weight the rear of the tow vehicle excessively, check trailer loading and rebalance. Conversely if the tongue appears very light and doesn’t lower the rear of the tow vehicle much, check load and rebalance trailer for more weight in the front.
- Use caution when entering the trailer after the stabilizers have been removed as the trailer might suddenly tip back if not securely hitched to tow vehicle causing damage to trailer, contents, and the possibility of severe personal injury!

Fig. 1 shows the storage locations of major equipment and supplies.

Appendix C. Trailer Leveling Procedure

1. Before Beginning;
    a. Carefully center the trailer into position among the anchor points as the trailer cannot be moved laterally with the available tools once the leveling procedure has begun. And then remove the trailer from the truck hitch. (Use extreme caution to avoid striking the tie downs with the tires of the truck or trailer or the tire may be instantly & irreparably damaged!)
    b. *Be sure everyone stays out of the trailer and out from under the trailer during the leveling procedure.*
2. Begin by lowering the nose of the trailer all the way by cranking the hitch jack down until one corner of the trailer touches the ground.
3. Level the ground under the rear jack stand points and place a couple of the wood blocks under each of the rear corners of the trailer.
4. Lower the jack stand of the LOWEST rear corner all the way to the blocks.
    a. Don’t extend the rear jacks more then 12-14” from the holder inside the trailer as they become inadequate to sustain the lateral forces the trailer may impart upon them if they are extended too far.
    b. Instead add more blocks to bring the ground up to meet them. As a rule the shorter the jack stands are extended the more stable the trailer will be. (6-8” inches is ideal)
5. If the trailer almost level;
    a. Lower the other jack stand (higher side) by only the amount needed to level the trailer if the jacks were both sitting on the blocks. *For example, if the trailer sits 2” higher on the left you would lower the right side all the way to the blocks (As instructed in step #4) and lower the left side until it is two inches from the blocks.*
    b. If the trailer is significantly out of level proceed to step #10.
6. Now all that is needed is to carefully raise the hitch jack until the trailer is level front to back and left to right. (The trailer is now supported on three points, front, right rear, and left rear) *The hitch jack will be difficult to crank as it is supporting half the weight of the entire trailer.*
7. Now level the ground under the two front scissors jacks and place two more wood blocks on the ground under each front jack position and lower the scissor jack for each side onto the blocks and crank in enough pressure to slightly lift that side a bit.
8. Now proceed to securing the trailer with the tie downs.
9. To reverse the procedure start by cranking up on the tongue jack enough to relieve stress on the scissors jacks, crank them up and lower the tongue to relieve the forces on the rear jack stands, remove the pins and push them up into the support and pin them at the highest (traveling) position.
10. If the trailer is significantly out of level (left to right) then place enough boards under the side that is HIGHEST so that the jack stand can be extended only about 6-8” to meet the board surface and pin it into place.

11. Now repeat the step above on the other leg (lowest side) and raise the nose of the trailer (with the hitch jack) enough to place the nose a bit higher then the rear of the trailer.
12. Using the hydraulic jack, place the jack under the frame rail on the side that is lowest at rear edge. (The frame rails are two long steel beams that run the length of the trailer front to back) *Don’t use the jack on any point other then the frame rail.*
13. Place enough boards under the hydraulic jack to bring it up to meet the frame rail without pumping it up much.
14. Now slowly close the relief valve on the bottom of the hydraulic jack and begin pumping the hydraulic jack to raise the low side of the trailer just enough to place another board under the foot of the rear jack stand.
15. Gently open the relief valve on the bottom of the hydraulic jack to lower the corner of the trailer onto the new board.
16. Repeat steps 13-15 until the trailer is level left to right.
17. Raise or lower the hitch jack until the front of the trailer is level with the rear.
18. Now level the ground under the two front scissors jacks and place two more wood blocks on the ground under each front jack position and lower the scissor jack for each side onto the blocks and crank in enough pressure to slightly lift that side a bit.
19. Now proceed to securing the trailer with the tie downs.
20. To reverse the procedure start by cranking up on the tongue jack enough to relieve stress on the scissors jacks, crank them up and lower the tongue to relieve the forces on the rear jack stands, use the hydraulic jack to relieve the side still supporting the trailer, then remove the pins and push the jack stands up into the support and pin them at the highest (traveling) position.

THE INSTALLATION OF OPEN-SOURCE MEASUREMENT EQUIPMENT

Standard Operating Procedure (SOP) U5

THE INSTALLATION OF OPEN-SOURCE MEASUREMENT EQUIPMENT

Standard Operating Procedure (SOP) U5

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Reviewed by

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Effective Date: November 6, 2006
Rev. 1.0 Effective Date: February 6, 2008

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Table of Contents

1. Scope and Applicability

1.1. Micrometeorological techniques are useful for measuring emissions from area sources like anaerobic treatment lagoons at livestock facilities, and include open-path concentration measurements and weather measurements.
1.2. The measurement sites are typically at locations remote from headquarters.
1.3. All equipment needed to measure lagoon emissions must be transported to the site.
1.4. The method requires careful positioning of many optical and physical components of the equipment.
1.5. A generalized representation of a typical open-source area is assumed.

2. Summary of Method

2.1. The objective of this SOP is to specify the procedures associated with the installation and tear-down of equipment and sensors associated with the open-path measurement of gas concentrations. Equipment and sensors include:
  2.1.1. Two fixed corner towers
  2.1.2. Meteorology tower/mast
  2.1.3. Standard fence posts for supporting SOPS inlets
  2.1.4. Synthetic Open Path equipment (SOP C4)
  2.1.5. TDLAS equipment (SOP C2)
  2.1.6. Pulsed fluorescence H_2S analyzer SOP G5)
  2.1.7. Photo-acoustic multi-gas analyzer (SOP G7)
  2.1.8. Instrument trailer (SOP U4)
  2.1.9. Meteorological data logger (SOP W6)
  2.1.10. Weather characterization sensors (SOP W1-W5)
  2.1.11. Lagoon characterization sensors (SOP L1-3)

3. Definitions

3.1.	FOS	Field Operations Staff
3.2.	GFCI	Ground fault circuit interrupter
3.3.	GSS	Gas sampling system
3.4.	LCD	Liquid Crystal Display
3.5.	OSHA	Occupational Safety and Health Administration
3.6.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.7.	PAML	Purdue Applied Meteorology Laboratory
3.8.	PDC	Pathlength-defining component. PDC is used to denote the component on the other end of the optical path from the PI-ORS instrument. Depending on the instrument selected, this could be a source, detector, mirror, or other reflecting object.
3.9.	PI	Principal Investigator
3.10.	PIC	Path Integrated Concentration. Given in units of parts per million-meter (ppm-m), PIC is the integrated concentration of a gaseous pollutant measured along the beam pathlength.

3.11.	PI-ORS	Path-Integrated Optical Remote Sensing. An instrument system used to acquire gaseous PIC data along an open optical beam path.
3.12.	PM	Project Manager
3.13.	PPE	Personal protection equipment
3.14.	QAM	Quality Assurance Manager
3.15.	QAPP	Quality Assurance Project Plan
3.16.	Retro-reflector	A PDC in which the reflected radiation is always back to the direction from which it came
3.17.	SMP	Site Monitoring Plan
3.18.	S-OPS	Synthetic Open Path Sampling System
3.19.	TDLAS	Tunable Diode Laser Absorption Spectroscopy
3.20.	USDOT	US Department of Transportation

4. Health and Safety

4.1. 120 VAC line current is distributed around the lagoon area. Take care to prevent shock, injury and possible loss of life. Always de-energize equipment before performing installation, maintenance, and removal. Hot work is not allowed; if an instrument has capacitors, they must be bled off before work is performed.
4.2. All outdoor receptacles, receptacles 6 ft from exterior doors, outlets around the lagoon, and all extensions cords must be protected by GFCIs.
4.3. Never substitute any of the manufacturer-supplied hardware when constructing or servicing the sheet metal towers. Doing so could cause the tower to collapse, resulting in serious personal injury or death.
4.4. Equipment weighs up to 150 lb. Use proper lifting techniques and personal protection.
4.5. Wear a suitable life jacket if working near the lagoon. One researcher nearly drowned.
4.6. On-site spiders, snakes, insects, and scorpions can be dangerous (Appendix A).
4.7. Wild animals may be in the vicinity of the site (Appendix B).
4.8. Weather (lightning, high winds, hail) can be hazardous (Appendix C).
4.9. Significant injury or death can result from falling off roof or ladder up to the roof.
4.10. Be careful climbing outside steps of trailer, particularly if wet or iced.
4.11. Avoid contact with tower, mast, or with the exterior trailer shell during thunderstorms.
4.12. Beware of traffic patterns on the farm and location of the lagoon and lagoon berm. If possible, do not drive over the lagoon berm.
4.13. The weight of the mast must be considered before lifting.
  4.13.1. Mast weighs 30 lb.

5. Cautions

5.1. Take care when working on the tower, as tools and equipment will likely be bent or damaged if dropped.
  5.1.1. Carefully inspect all rivets and bolt joints to look for cracks or loose bolts. If loose bolts or rivets are found, retighten bolts immediately, replace broken or missing rivets with a screw and nut combination and document their location.
  5.1.2. Carefully inspect the tower to verify it is true and structurally sound.

5.2. Be sure to observe correct polarity/voltage or the instruments may be irreparably damaged!

5.3. The instruments and retro-reflectors are delicate and will be damaged if dropped.

5.3.1. If an instrument is dropped, record the event in the instrument logbook and carefully evaluate the instrument for possible damage prior to installation.

5.4. Proper connection of line power is necessary. Proper care from crimping cables and connectors should be used.

5.5. Take care when climbing on roof in vicinity of antennae, and towers. Antennae, mounts, and cables are delicate and must not be bent or broken. Do not drop.

5.6. Tighten all bolts/wire ties on retro-reflectors. Loose bolts or wire ties may result in a retro-reflector coming loose and falling, resulting in significant damage to the retro-reflector and heater.

5.7. Tighten all bolts on NEMA box. Loose bolts will result in NEMA box slipping down or turning on mast and possibly colliding with and misaligning the other instruments.

5.8. Be sure producer has provided information locating all underground utilities and pipes before driving any stake, ground rod, or other piece of equipment into the ground.

5.9. Lightning damage to sensors is less likely if instruments and towers are properly grounded.

6. Interferences

6.1. The trailer communications systems may not function properly if the trailer is located in the vicinity of a television or radio station transmitting tower.

6.2. Power failures may adversely affect all communications and computers.

6.3. Heating or cooling failures may result in environmental conditions that are outside the specifications of the equipment.

7. Personnel Qualifications

7.1. Personnel must be trained in the use of the handheld multimeter before initiating the procedure. Training requires 10 min.

7.2. All personnel must read and understand this SOP and all cited SOPs (See References).

7.3. Field personnel must be able to lift 40 lb.

8. Equipment and Supplies

8.1. Safety Equipment and PPE

8.1.1. Climbing belt and lanyard (when more than 6 ft off the ground)

8.1.2. Hard hats (where required, or for anyone while work is being done overhead)

8.1.3. Leather gloves

8.1.4. Safety goggles

8.1.5. Ear plugs

8.1.6. Flashlight/trouble light

8.1.7. 100-ft 5/8″ Work rope – for sheet metal tower work

- 8.2. Hand tools (kept in trailer tool box)
  - 8.2.1. Shovel (square mouth)
  - 8.2.2. Cement trowel (for concrete work on tower installation)
  - 8.2.3. Hammer
  - 8.2.4. Small sledge hammer – to drive stakes or grounding rod
  - 8.2.5. Open end wrenches (7/16″, 1/2″, 9/16″ 3/4″, 15/16″)
  - 8.2.6. Socket wrench and 3/4″ and 9/16″ deep well sockets
  - 8.2.7. Allen wrench set (0.05″) for LI2003S – one is supplied with LI2003S
  - 8.2.8. Straight-bit screwdrivers (small, medium, and large)
  - 8.2.9. Phillips screwdrivers (small and medium)
  - 8.2.10. Pliers
  - 8.2.11. Needle-nose pliers
  - 8.2.12. Channel locks
  - 8.2.13. (2) Small diagonal side-cutters
  - 8.2.14. Wire strippers
  - 8.2.15. (2) Adjustable (crescent) wrenches
  - 8.2.16. Cable cutters
  - 8.2.17. (2) Files – one flat and one rat tail
  - 8.2.18. Hacksaw
  - 8.2.19. Pocket knife
  - 8.2.20. Sheet metal cutters (tin snips)
  - 8.2.21. Tool aprons
  - 8.2.22. Nylon cable ties
  - 8.2.23. Enclosure supply kits (CSI P/N 7363)
  - 8.2.24. Electrician’s putty
  - 8.2.25. Bailing wire
  - 8.2.26. Common screws to mount enclosure
  - 8.2.27. Wire multi-color
  - 8.2.28. Caulking gun and caulk
  - 8.2.29. Long bolts, washers, and nuts
  - 8.2.30. Self tapping sheet metal screws
  - 8.2.31. Grabber screws
- 8.3. Hardware
  - 8.3.1. Spare nuts, washers, U-bolts, etc.
  - 8.3.2. Spare cable clamps
  - 8.3.3. Ground wire
  - 8.3.4. (4) Ground rods
- 8.4. Soldering tools (kept in trailer tool box)
  - 8.4.1. Soldering iron
  - 8.4.2. Cigarette lighter
  - 8.4.3. Solder
  - 8.4.4. Heat shrink assortment
  - 8.4.5. Electrical tape
- 8.5. Power tools (kept in trailer tool box)
  - 8.5.1. Cordless variable speed drill with spare battery and charger

  - 8.5.2. Drill bits
  - 8.5.3. Screw/nut driver bits
- 8.6. Electrical equipment
  - 8.6.1. Calculator (kept in trailer)
  - 8.6.2. (2) 1400VA UPS units for TDLAS in-field backups
  - 8.6.3. Portable computer (kept with FOS)
  - 8.6.4. Handheld multi-meter (current, resistance, and voltage testing instrument)
  - 8.6.5. Spare cables (kept in trailer)
  - 8.6.6. 100-ft extension cord
- 8.7. Calibration equipment (kept in trailer drawer)
  - 8.7.1. Brunton 8099 compass or equivalent (for radiation and anemometer sensor alignment)
  - 8.7.2. Torpedo level (12” to 24”)
  - 8.7.3. Tape measures, 10-ft and 100-ft
  - 8.7.4. Handheld Garmin 76 GPS unit or equivalent (kept with FOS)
  - 8.7.5. Nikon 8358 Laser rangefinder (1200m) or equivalent (kept with FOS)
- 8.8. Miscellaneous
  - 8.8.1. 5-ft step ladder
  - 8.8.2. Locks and keys
  - 8.8.3. Batteries and battery cables
  - 8.8.4. RF Antennas
    - 8.8.4.1. (1) for meteorological data logger
    - 8.8.4.2. (1) for sonic anemometers
    - 8.8.4.3. (4) for TDLAS/scanners
  - 8.8.5. (3) Stakes for mast
  - 8.8.6. Digital camera with wide angle capability (kept with FOS)
- 8.9. Original equipment set for each sheet metal tower installation (Appendix D) for initial installation
  - 8.9.1. Delhi DMX60 or optional DMX68 stub kit (shown installed in Fig. 1A)
  - 8.9.2. Delhi DMX60 or optional DMX68 tower (6 sections - Fig. 1B)
  - 8.9.3. Delhi DMX60 or optional DMX68 bolt kit
  - 8.9.4. Custom-made 4-m anemometer mount (built by PAML)
  - 8.9.5. (3) Ground point lugs (for attaching ground wire to tower legs)
  - 8.9.6. (3) Ground rods with clamp
  - 8.9.7. Lightning rod with mounting bracket
  - 8.9.8. (2) 10′ 2x4 boards (for concrete form Fig. 1C)
  - 8.9.9. (1/2) lb 2 1/2″ framing nails (build forms with)
  - 8.9.10. (3) Bricks common size, any color or style (to set base stubs on)
- 8.10. Documentation
  - 8.10.1. Sensor manuals
  - 8.10.2. Site monitoring plan
  - 8.10.3. Mounting angle for solar panel
  - 8.10.4. Station log and pen
  - 8.10.5. Felt-tipped marking pen

- 8.11. Communication equipment
  - 8.11.1. Head-held radios
  - 8.11.2. Cellular phone
  - 8.11.3. Appropriate phone numbers
- 8.12. Equipment and materials to be stored in the bed of the tow vehicle
  - 8.12.1. Two man heavy duty Composite Plastic Dingy (SOP L5).
  - 8.12.2. Roll of plastic sheeting (SOP L5).
  - 8.12.3. (3) 2-m holding stakes (corkscrew anchors may be substituted).
  - 8.12.4. 100′ Nylon rope (for tri-sensor float).
- 8.13. Equipment and sensors to be installed (stored in trailer according to SOP U4)
  - 8.13.1. (1) Campbell Scientific CM110 tripod tower
  - 8.13.2. (1) NEMA box with CR1000 datalogger, CS100 Barometer, and XStream® RS-232/485 RF Modem
    - 8.13.2.1. (1) Antenna for modem
  - 8.13.3. (1) LiCOR 200 solar radiation sensor and mounting fixture and bracket
  - 8.13.4. (1) Vaisala HMP45C RH/T sensor in passive radiation shield
  - 8.13.5. (1) Surface wetness grid and mounting bracket
  - 8.13.6. (3) RMYoung 81000 sonic anemometers, each with XStream® RS-232/485 RF Modem
  - 8.13.7. (1 or 2) 107-L Thermistor
  - 8.13.8. (1 or 2) Innovative sensors CSIM11 ORP redox state sensor
  - 8.13.9. (1 or 2) Innovative sensors CSIM11 pH sensor
  - 8.13.10. (2) TDLAS/scanners on tripod
  - 8.13.11. (2) TDLAS/scanner power supply/ communications boxes (with 2 antennas).
  - 8.13.12. (2) TDLAS/scanner UPS systems.
  - 8.13.13. (20) Retro-reflectors

9. Procedures

- 9.1. Acceptance
  - 9.1.1. Criteria for acceptance of all meteorological and lagoon sensors are described in the SOP covering each instrument.
  - 9.1.2. Data logger acceptance criteria are described in SOP W6.
  - 9.1.3. Expected windload
    - 9.1.3.1. Retro-reflectors consist of at least four retro-reflector cubes mounted in a 10″ x 9″ x 7″ box.
      - 9.1.3.1.1. Three reflectors on each of two sides
      - 9.1.3.1.2. Windload on tower: 2.4 ft^2
    - 9.1.3.2. Sonic anemometer cross-sectional area is approx 18″ x 1″.
      - 9.1.3.2.1. Two anemometers
      - 9.1.3.2.2. Windload: 0.2 ft^2
  - 9.1.4. Allowable wind displacement of the retro-reflectors
    - 9.1.4.1. The diameter of the laser beam when it exits the TDLAS is about 2.5 mm, and the divergence is approximately 1.5 mrad (milliradians). This means that at a

distance of 150 m, the beam diameter is 1.5 x 150 = 225 mm (9″). Similarly, for a path of 300 m, the beam diameter would be 450 mm (18″).

- 9.1.4.2. Retro-reflector area is 6″ x 8″
- 9.1.4.3. Allowable movement of tower at top is approximately 18″ at 150 m and 36″ at 300 m (assuming reflection of 3″ width beam needed).

9.2. Acceptance of sheet metal tower and components (Appendix D)

- 9.2.1. 56' Sheet metal towers (DMX60) consist of (6) 8-ft tower sections, one 6′ 1 ¼″ mast pipe with mounting clamps, one stub kit, and one manufacturer-supplied bolt kit. The 64' model (DMX68) is identical except it has one additional bottom section.
  - 9.2.1.1. Lay the tower on its side if not already lying down.
  - 9.2.1.2. Cut the steel banding (Fig. 1B) holding the sections together using sheet metal cutters.
  - 9.2.1.3. Separate all sheet metal tower sections by pulling the smallest section out of the tower bundle from the bottom (largest end).
  - 9.2.1.4. Visually inspect all sides and both ends of each section for damage (deformed or damaged end pieces or broken/missing rivets).
    - 9.2.1.4.1. If damage is found, contact the manufacturer or shipper for remediation.
    - 9.2.1.4.2. Each sheet metal tower must have a hardware kit containing at least thirty-six (36) hardened steel specialty bolt sets of varying sizes (each bolt set contains one bolt, one nut, and one washer).
      - 9.2.1.4.2.1. These are specially-manufactured bolts with a built-in centering taper. Standard hardware parts must never be substituted for any of the hardware supplied with the tower. If any of the bolt set pieces are missing contact the PM and have replacements brought to the site before continuing to construct the tower.
    - 9.2.1.4.3. Each section of the tower gets six bolts of the same size to fasten it to the next section. All the intended bolt holes are predrilled for the tower. The largest bolts go in the bottom section and smaller bolts are used on each consecutive section. The bolts will exactly fit the holes in the tower for the section of which they are intended. Never use a small size bolt in a large predrilled hole and never ream any hole to fit a larger bolt. Three large standard hardware bolt sets will be placed in the bottom of the base stub for each tower leg to be cemented into the concrete and prevent slippage (Fig. 1A). **This is the only place where standard (non-manufacturer-supplied) hardware components are to be used except when replacing missing or damaged rivets.**
    - 9.2.1.4.4. If any parts are missing or damaged, contact the PM to locate or replace the missing component. Again, never substitute any standard hardware for the manufacturer-supplied tower components.

9.3. 3-m meteorology tower/mast (Campbell Scientific CM110)

- 9.3.1. Characteristics
  - 9.3.1.1. Aluminum construction
  - 9.3.1.2. 10-ft (3-m) height
  - 9.3.1.3. Storage in trailer (SOP U4)
  - 9.3.1.4. Withstands sustained winds of 75 mph

9.3.1.5. Withstands wind gusts to 95 mph
9.3.1.6. Vertical load limit is 100 lb
9.3.1.7. Weighs 30 lb
9.3.2. Assembly test (see Manual)
9.3.2.1. Stand tripod up on end and rotate feet to be perpendicular to the legs.
9.3.2.2. Each leg has a slide collar with black plastic T-bolt and a spring-loaded pin that locks in holes located on the underside of the leg.
9.3.2.2.1. Loosen T-bolts and extend each leg until pin engages in the center hole.
9.3.2.3. With legs extended, orient tripod so that the open channel of the tripod faces parallel to the lagoon edge.
9.3.2.4. Attach lower mast section by aligning holes at the bottom of the mast section with the upper set of slanted holes at the tripod base. Insert pin through holes and rotate the wire retainer over the end of the pin. (The pin should be at the bottom of the slanted hole when the mast is upright.)
9.3.2.5. Lift the mast up so that the pin is in the upper end of the slotted hole to allow the mast to be tilted down to a horizontal position.
9.3.2.5.1. Verify functionality.
9.3.2.6. Lift up the mast so that the pin in is the lower end of the slotted hole to align the mast to the vertical.
9.3.2.6.1. Verify functionality.
9.3.2.7. Secure the mast in the vertical position by installing the locking bracket.
9.3.2.7.1. Insert the top of the bracket into the notches in the mast tripod base. Using both thumbs, press the bracket into the body of the base until the lower tabs lock into position.
9.3.2.7.2. Install the locking pin.
9.3.2.8. Evaluate rigidity.
9.4. Retro-reflectors
9.4.1. Reflectors consist of at least five retro-reflector cubes mounted in a 10″ x 9″ x 7″ box. They are heated by a Thermon DSE-70 Heater (Fig. 2) or other equivalent device.
9.4.2. The heater is supplied by 110VAC.
9.4.3. When powered, the 40-W heater is capable of heating base of reflector cubes 50°C above ambient temperature.
9.4.3.1. If the heater does not heat the reflector cubes adequately, inspect wiring and contact Boreal Laser for replacement or repair.
9.5. Document overall layout
9.5.1. Photograph site from the following orientations:
9.5.1.1. Cardinal directions (N, S, E, W) from location of each wind sensor
9.5.1.2. Views perpendicular to each beamline, using lens with wide angle view
9.6. Installation
9.6.1. Always refer to the individual Site Monitoring Plan for each site when determining exact placement of **all** sensors, tripods, masts, towers, etc.
9.6.1.1. Correct locations of all instruments and devices will have been pre-located during initial site setup and will already have anchors driven at the correct location.

9.6.2. At each power access location, check GFCI for ability to trip.

9.6.3. Due to terrain and clearance limitations, it is necessary to use a self-supporting 15-m tower capable of supporting retro-reflectors and anemometers.

9.6.3.1. The Delhi QDMX52 is a 48-ft, self-supporting sheet metal tower model with a 4-ft mast mounted to the top, and is capable of a 3-ft^2 wind load at the top.

9.6.3.2. The Delhi DMX68 is a 64-ft self-supporting sheet metal tower similar to the DMX60 with the same wind load specifications) used to overcome height and position limitations at the lagoon corners

9.6.3.3. The sheet metal tower will have its base (bottom) section bolted to stubs, and the stubs will be cemented into concrete and allowed to cure. The tower will then be erected by bolting on additional sections hauled upward by hand using a rope to bring up each additional section (this is a common practice for this type of tower).

9.6.3.4. The towers are outfitted during initial installation with standard 110V exterior in-use covered outlets for each instrument, UV-resistant outdoor wiring and a drop cord near the bottom to plug into a nearby power outlet to provide power for all necessary instruments during measurement periods.

9.6.3.5. The towers are fitted with grounding hardware during initial construction consisting of a single 8′ ground rod and a short section of #6 bare copper wire.

9.6.3.6. The entire tower will stand for the duration of all the measurement periods at the site where it is installed and only the instruments and retro-reflectors will be removed in between measurement periods.

9.6.3.7. The tower will be disassembled and removed using the same method used to install the tower but in reverse only when all measurements for the project are complete at the site.

9.6.3.8. The sheet metal tower will have the retro-reflector mounting points pre-located and marked with red paint during initial setup of the site.

9.6.3.9. The 16-m anemometer will attach directly to the top of the short mast pipe on the top of one of the towers at each site, and a special Galvanized pipe mount will be constructed at PAML for each site to mount the 4-m anemometer at the correct distance as specified in SOP W2 and bolted to the tower at the required location on the proper tower for each site (4-m above berm height) using common hardware. This mount will remain on the tower at the site for the project duration.

9.6.3.10. Free-standing sheet metal towers will be used at all sites requiring retro-reflectors and anemometers to be mounted above 3 m.

9.6.3.11. Follow the installation procedure in Appendix D (Sheet metal tower installation) in such locations.

9.6.4. 3-m meteorology tower/mast (Fig. 4)

9.6.4.1. Install at location indicated by SMP (anchor and locating stake should already be present at the proper location).

9.6.4.2. Normally data logger, NEMA box, and meteorological characterization sensors, as specified in SOP W6, remain attached to the mast after initial installation for the duration of the project (except for service).

9.6.4.2.1. If unable to find locating stake, determine location with GPS unit.

- 9.6.4.2.2. Record any deviations in notes on open path sensor installation report (Appendix E).
- 9.6.4.2.3. Stand tripod up on end and rotate feet to be perpendicular to the legs.
- 9.6.4.2.4. Each leg has a slide collar with black plastic T-bolt and a spring-loaded pin that locks in holes located on the underside of the leg.
  - 9.6.4.2.4.1. Loosen T-bolts and extend each leg until pin engages in center hole.
- 9.6.4.2.5. With legs extended, orient tripod so that the open channel of the tripod faces parallel to the lagoon edge.
- 9.6.4.2.6. Lift the mast up so that the pin is in the upper end of the slotted hole.
- 9.6.4.2.7. Tilt mast to the horizontal such that the mast rests on the ladder.

9.6.4.3. Initial installation requires the insertion of the anemometer mount for three sonic anemometers for intercomparison

9.6.4.4. Unpack and install (3) sonic anemometers.
- 9.6.4.4.1. Storage location indicated in SOP U4
- 9.6.4.4.2. Install anemometers at the top of the mast for intercomparisons (SOP W2).
- 9.6.4.4.3. Unpack and inspect instrument Power/Radio Units, check cable and repair or replace as needed. Log inspection results on checklist (Appendix F) with comments and remediation.
- 9.6.4.4.4. Connect instrument cables from power units to anemometers.

9.6.4.5. Lift up the mast so that the pin in is the lower end of the slotted hole to align the mast to the vertical position. Secure with locking bracket.
- 9.6.4.5.1. Insert the top of the bracket into the notches in the mast tripod base and using both thumbs press the bracket into the body of the base until the lower tabs lock into position.
- 9.6.4.5.2. Install the locking pin

9.6.4.6. Verify that the mast is vertical using a torpedo level.
- 9.6.4.6.1. If not, adjust the length of the mast legs until the mast is vertical.
- 9.6.4.6.2. Stake all legs of mast using medium hammer.

9.6.4.7. Unpack and drive ground rod in ground near base of mast using large hammer. (Storage location shown in Fig. 1 of SOP U4)
- 9.6.4.7.1. Attach ground wire from mast to ground rod and secure with wire ties or tape.

9.6.4.8. Inspect power cable and repair or replace as needed. Log inspection results on checklist (Appendix F) with comments and remediation.

9.6.4.9. Conduct sonic anemometer intercomparison (SOP W2)

9.6.4.10. Remove sonic anemometers from intercomparison mount and place in shipping containers.

9.6.4.11. Using shipping containers as ancheotic chambers, check anemometer zero values according to SOP W2.

9.6.4.12. Remove intercomparison sonic sensor mount and install single sensor mount on mast top

9.6.4.13. Remount one sonic anemometer on the top of the mast

9.6.4.14. Return shipping container to trailer

9.6.4.15. Disconnect sonic Power/Radio Units from other two sonic anemometers and power.

9.6.5. Fixed tower instrument installation (Fig 3).
  9.6.5.1. Locate correct locations of retro-reflectors and anemometers on tower by using pre-painted cross sections (Red colors represent a retro-reflector location and yellow colors represent the 4-m Anemometer location). Log any deviations of open path sensor installation report (Appendix E).
    9.6.5.1.1. Sonic anemometers
      9.6.5.1.1.1. Install the 16-m sonic anemometer at top of the mast on the correct tower for the site (location shown in the SMP). Orient the instrument to true N.
      9.6.5.1.1.2. Return storage container to trailer.
      9.6.5.1.1.3. Install sonic anemometer on the 4-m high mount arm (already mounted to tower and marked with yellow paint) and check level of instrument to ensure it is vertical and orient instrument to true north.
      9.6.5.1.1.4. Return shipping container to trailer(storage location shown in SOP U4).
        9.6.5.1.1.4.1. Inspect cables and repair or replace as needed. Log inspection results on checklist (Appendix F) with comments and remediation.
    9.6.5.1.2. Connect instrument cables to anemometers (SOP W2) and secure down the tower at three-foot intervals using electrical tape.
    9.6.5.1.3. Connect to power source and verify sensor operation by viewing data in instrument trailer.
    9.6.5.1.4. If anemometer data is not present, troubleshoot system using the following method.
      9.6.5.1.4.1. Open anemometer power boxes and check for power at RF modem.
      9.6.5.1.4.2. Check anemometer for power by listening for “buzz” from sensor.
      9.6.5.1.4.3. Check antenna connection at base radio and data connection into LAN server.
      9.6.5.1.4.4. If still not functioning, contact PAML for further help or remediation.
  9.6.5.2. Retro-reflectors
    9.6.5.2.1. Unpack 6 retro-reflectors (storage location shown in SOP U4) and attach the custom made mounting backboards of tower mounting from trailer.
    9.6.5.2.2. Mount 3 reflectors on the red paint marked sections of the fixed tower at the 1, 7, and 15 m heights so that the open sides of the retro-reflector enclosure are facing an adjacent corner of the lagoon; secure with wire ties.
    9.6.5.2.3. Mount 3 reflectors on the red paint marked sections of the fixed tower at the 1, 7, and 15 m heights so that the open sides of the retro-reflector enclosure are facing the other adjacent corner of the lagoon; secure with wire ties.
    9.6.5.2.4. Inspect cables and repair as needed. Log inspection results with comments and remediation
    9.6.5.2.5. Connect retro-reflector power cables to power boxes on tower and secure with tape.
    9.6.5.2.6. Repeat procedure for mounting retro-reflectors on tower at opposite side of lagoon.

9.6.6. Calibration verification of pH and redox probe must be done prior to installation of probes on tri-sensor float.
  9.6.6.1. Unpack and remove lagoon sensors and cables from storage area in trailer (SOP U4) and uncoil cables only enough to conduct calibration verification.

9.6.6.2. Conduct calibration verifications of redox and pH probes according to SOP L1 and L3..

9.6.7. Installation of tri-sensor float and lagoon characterization sensors

9.6.7.1. Life jacket needs to be worn when working on the inside of the berm top.

9.6.7.2. Float, rope, and 2-m holding stakes are located in bed of tow vehicle. Lagoon sensors are located in trailer (SOP U4).

9.6.7.3. Refer to Fig. 5 for installation of tri-sensor float. Float will be secured using a rope stretched across a corner of the lagoon and secured by two 2-m stakes placed so as to hold the float a minimum of three meters from the waterline of each edge in the corner of the lagoon.

9.6.7.4. Locate the position (at least 100-m from the lagoon inflow pipe) on the lagoon corner where the tri-sensor float should be placed based on appropriate SMP.

9.6.7.5. Take the 100-ft nylon rope from the bed of tow vehicle and stretch it out lengthwise in a dry location, grab one end and walk towards the other in a straight line, lay your end next to the other and walk back to the center. The location where the rope bends 180° will be the center and mounting location for the tri-sensor float. Mark the center with a simple single knot. Now measure 4 m from that knot and tie another knot. Now going the other direction from the first (center) knot, again measure 4 m and tie another knot. These knots will help ensure the float is placed 3 meters from the waterline of each edge in the corner.

9.6.7.5.1. To space the float 3 m from each edge, the distance the rope must cross the water's edge from each corner at is 6 m given the corner is 90°. Using the formula $A^2+B^2=C^2$, the length of the rope diagonally from waterline to waterline must be 8.5 m. The distance to the water's edge from the center each way is 4.25 m. The outside knots will cross each edge at the waterline 6 m from the corner on each side, ensuring the float is approximately 3 m from each bank.

9.6.7.6. Using the rope you just marked, a stake for each of you to mark the desired location, and your partner, stretch the rope diagonally across the corner from just outside the berm to find the location outside the berm to drive the holding stake. Use the marker knots you made in the rope to ensure the correct positioning of the stakes by aligning the two outside knots are at the water's edge so the middle knot is in the center of the water at the corner.

9.6.7.7. Drop your stake at the location you wish to drive it, Have your partner do the same things at his location. Walk the rope back to dry land and lay it down.

9.6.7.8. Now go back and drive the stakes just outside lagoon berm at each previously marked location. (Be sure the producer has provided locates and the area is free of underground wires pipes etc… before driving stakes!).

9.6.7.9. Secure guide rope to tri-sensor float assembly using the center knot in the rope as the center marker for the float.

9.6.7.10. Set the tri-sensor float on a suitable work surface to mount the probes.

9.6.7.10.1. Remove lagoon sensors and cables from trailer and uncoil cables.

9.6.7.10.2. Inspect cable and repair or replace as needed. Log inspection results on checklist (Appendix F) with comments and remediation

9.6.7.10.3. Position and secure all probes in the tri-sensor float so that the respective sensor will be at the correct distance below the waterline of the float (See SOP L1 (PH sensor), SOP L2 (Temperature sensor), and SOP L3 (Redox sensor) for correct positioning information of the probes).

9.6.7.10.4. Connect the probe cable to the rope closest to the meteorological mast and using wire ties attach the cables to the section of rope that will contact the water.

9.6.7.11. Using the rope to lift the tri-sensor float assembly, have your partner take one end and you take the other end and the probe cable, and carefully suspend the probe and carry over the location in the lagoon where the float will rest by pulling each end of the rope against the center. Gently lower the float into the water being careful not to tip it or set it in any debris in the lagoon (Be sure to wear your life jacket while working near the lagoon edge).

9.6.7.12. Secure the ends of the rope to each of the holding stakes to ensure proper location of the tri-sensor float.

9.6.7.13. Visually verify the waterline of the float matches to the 0.3 m mark on the tri-sensor float and the that the rope has enough slack to allow for proper floatation while still maintaining the float's proper location.

9.6.7.14. Run cable into the NEMA box through the proper knockout and secure. Be sure to coil any extra cable at the foot of the mast and secure with nylon wire ties (Do not cut the cable to length).

9.6.7.15. Attach wires to data logger in NEMA box using the ⅛″ slotted screwdriver. Connect black wire to the switched excitation channel, purple to ground, clear to ground, and red to the single-ended analog channel.

9.6.7.16. Connect data logger to RF modem.

9.6.7.17. Verify the instrument cable is floating as expected and ensure that it is secured at the mast base and the NEMA box.

9.6.8. Connect meteorological (NEMA box) power cable and verify that all sensors, data logger, and RF modem have power and are operating as expected.

9.6.8.1. Note any deviations from normal parameters, repair or replace the troubled component if any, and document the repair in field notes.

9.6.8.2. Check sonic anemometer (SOP W2).

9.6.8.3. Check humidity and temperature sensor (SOP W1).

9.6.8.4. Check wetness grid sensor (SOP W3).

9.6.8.5. Check solar radiation sensor (SOP E3)

9.6.8.6. Check barometric pressure sensor (SOP W5).

9.6.8.7. Check lagoon temperature sensor (SOP L1).

9.6.8.8. Check lagoon pH sensor (SOP L2).

9.6.8.9. Check lagoon redox sensor (SOP L3).

9.6.8.10. Check communications (SOP W6).

9.6.8.11. Verify that the solar radiation sensor mount is off the south side of the mast.

9.6.8.11.1. If not, correct position.

9.6.8.12. Check again that the mast is vertical using a torpedo level.

9.6.8.12.1. If not, re-adjust the length of the mast legs until the mast is vertical.

9.6.9. Install S-OPS path
  9.6.9.1. Locate S-OPS paths according to appropriate SMP.
  9.6.9.2. If not in place already, locate and install fence posts with 5-m spacing along path of S-OPS.
  9.6.9.3. Be sure that all inlets of S-OPS have rubber stoppers installed <u>and taped</u> and tubing end is taped.
  9.6.9.4. Roll out S-OPS tubing and attach to fence posts according to SOP C4.
  9.6.9.5. Roll out S-OPS extension tubing and connect to S-OPS according to SOP C4 and Figure 7.
  9.6.9.6. Connect S-OPS extension tubing to GSS in trailer through trailer tubing pass-through.
  9.6.9.7. Check connections of CR800 data logger, AS21-12V relay controller, GSS, and LAN computer, and verify that GSS, data logger, and relay controller have power and are operating as expected using LoggerNet software.
  9.6.9.8. Start GSS and check relay/solenoid, flow direction, mass flow, and pressure sensor operation (SOP C4).
  9.6.9.9. Verify sampling and bypass airflow in GSS (SOP C4).
  9.6.9.10. Check GSS and sampling system for leaks (SOP C4).
  9.6.9.11. Remove rubber stoppers from inlets and store in trailer.
  9.6.9.12. Note any deviations from normal parameters, repair or replace the troubled component if any, and document the repair in field notes.
  9.6.9.13. Conduct sampling system flow balance (SOP C4).
9.6.10. Innova VOC multi-analyzer (SOP G7)
  9.6.10.1. Check connections of Innova and LAN computer and verify that Innova has power and is operating as expected.
  9.6.10.2. Verify calibration of Innova (SOP G7).
9.6.11. Thermo Fisher Corp 450I Pulsed Fluorescence H_2S Analyzer (SOP G5).
  9.6.11.1. Check connections of 450I and LAN computer and verify that 450I has power and is operating as expected.
  9.6.11.2. Start iPort software for control of 450I
  9.6.11.3. Verify calibration of 450I (SOP G5).
9.6.12. TDLAS and scanner
  9.6.12.1. Locate stake and anchor for TDLAS (SOP C2) and scanner tripod at the corner of the lagoon (Fig. 6) as defined in the individual SMP (use GPS if necessary to find anchor location).
    9.6.12.1.1. Platform specifications related to beam alignment are found in Appendix G.
      9.6.12.1.1.1. Specified platform is an original equipment manufacturer selected tripod and meets all platform specifications.
    9.6.12.1.2. Setup and level tripod platform for TDLAS/scanner at 1 m above berm height and secure to anchor with Nylon ratchet strap (if necessary use GPS unit to help locate the anchor).
    9.6.12.1.3. Record deviations (if any) in locations of any TDLAS/scanner components on the site open path sensor installation report (Appendix E).
  9.6.12.2. Remove TDLAS/scanner UPS from storage area in trailer and install next to tripod.

9.6.12.3. Remove TDLAS/scanner power/radio unit and associated cables from storage area in trailer (SOP U4) and uncoil.

9.6.12.3.1. Inspect cable and repair or replace as needed. Log inspection results on checklist (Appendix F) with comments and remediation.

9.6.12.4. Connect power/signal cables at TDLAS/scanner (SOP C2)

9.6.12.5. Connect power cable UPS at power access point.

9.6.12.5.1. Verify power at UPS.

9.6.12.6. Connect power cable from TDLAS/scanner power supply to UPS

9.6.12.6.1. Verify power at TDLAS and scanner with LCD display on TDLAS unit (SOP C2).

9.6.12.7. Unpack and remove all remaining retro-reflectors and tripods from trailer storage location (SOP U4).

9.6.12.8. Position tripods with 'surface' reflectors at the staked locations 1-m height above lagoon berm at 1/3 and 2/3 of the distance between corner towers and TDLAS and scanner, according to Fig. 6 (Retro-reflectors D, E, F, G, N, O, P, Q).

9.6.12.8.1. Visually align reflectors so that the retro-reflector box opens towards TDLAS/scanner system.

9.6.12.8.2. Determine position of retro-reflectors by locating marker stake and anchor, if necessary use GPS unit.

9.6.12.8.3. Secure tripod to anchor using tie down strap.

9.6.12.8.4. Record position deviations (if any) on site open-path sensor installation report (Appendix E).

9.6.12.8.5. Inspect power cable and repair or replace as needed. Inspection results logged on checklist (Appendix F) with comments and remediation.

9.6.12.8.6. Connect power cables from each retro-reflector enclosure to nearest power access point.

9.6.12.8.6.1. Verify power at retro-reflector with visible red light on heater unit

9.7. Lagoon sludge depth measurements

9.7.1. Measurements may be done either at time of site installation or disassembly.

9.7.2. Wear protective clothing when working in lagoon.

9.7.3. Wear life jacket when working inside of the berm top.

9.7.4. Use the boat,(stored in truck bed) to move around lagoon to position for measurements of lagoon depth and sludge depth.

9.7.5. Number and location of measurements are indicated by the SMP for the site.

9.7.6. Procedure for measurements is described in SOP M5.

9.8. Disassembly

9.8.1. Fixed towers

9.8.1.1. Disconnect power cables to retro-reflectors and remove from towers, Remove and store mounting boards, store retro-reflectors in protective boxes and secure in trailer (SOP U4).

9.8.1.2. Disconnect cables from sonic anemometers and remove from tower.

9.8.1.3. Dismount anemometers and perform intercomparison and stationarity checks. Pack anemometers in shipping container and store in trailer in designated storage area (SOP U4).

9.8.1.4. Unplug power cable feeding tower, roll up and secure to tower.

9.8.2. 3-m meteorology tower/mast.
  9.8.2.1. Perform intercomparison tests on anemometers (Sections 9.6.4.4 through 9.6.4.11) before disassembly and storage of meteorological mast.
  9.8.2.2. Disconnect power to system at power access point.
  9.8.2.3. Disconnect all lagoon sensor cables from data logger and pass back outside NEMA box
  9.8.2.4. Remove all sonic anemometers, pack in designated container and store in trailer (SOP U4).
  9.8.2.5. Remove ground rod, ground wire, and pack in designated area in trailer (SOP U4).
  9.8.2.6. Remove stakes at each foot, pack in designated containers and store in trailer (SOP U4).
  9.8.2.7. Loosen clamps and fold mast legs, store in trailer complete with remaining instruments and NEMA box still attached (SOP U4) and secure to center of floor using D-rings and ratchet straps.
9.8.3. S-OPS disassembly
  9.8.3.1. Document sample flows according to SOP C4.
  9.8.3.2. Plug inlets with rubber stoppers and tape stopper to filter holder.
  9.8.3.3. Conduct system leak test according to SOP C4.
  9.8.3.4. Disconnect inlets from fence posts.
  9.8.3.5. Tape any open tubing ends, and roll up S-OPS tubing according to SOP C4.
  9.8.3.6. Disconnect S-OPS extension tubing from GSS and tape ends.
  9.8.3.7. Remove S-OPS extension tubing from trailer tubing pass-through.
  9.8.3.8. Roll up S-OPS extension tubing, taping open ends as necessary according to Figure 7.
9.8.4. Innova 1412 VOC multi-analyzer (SOP G7)
  9.8.4.1. Verify calibration of Innova according to SOP G7.
9.8.5. Thermo Fisher 450I Pulsed Fluorescence H2S Analyzer (SOP G5).
  9.8.5.1. Verify calibration of 450I according to SOP G5.
9.8.6. TDLAS and scanner
  9.8.6.1. Disconnect power to UPS system at power access point.
  9.8.6.2. Disconnect power/signal cables at TDLAS/ Scanner unit and UPS system.
  9.8.6.3. Store UPS with plastic box in trailer.
  9.8.6.4. Coil cable and fasten into storage area in trailer at location (SOP U4).
  9.8.6.5. Dismount TDLAS/Scanner unit from tripod, perform and log calibration check and then pack in shipping container, store TDLAS, Scanner unit, and tripod in trailer in designated protective boxes in storage area (SOP U4).
9.8.7. Retro-reflectors
  9.8.7.1. Disconnect power for all retro-reflector enclosures at power access points.
  9.8.7.2. Dismount retro-reflectors from tripods and store retro-reflector enclosures and tripods in trailer in designated storage area (SOP U4).
9.8.8. Lagoon sensors and tri-sensor float
  9.8.8.1. Disconnect signal cables at NEMA box.
  9.8.8.2. Extract tri-sensor float and sensor package from lagoon using rope attached to tri-sensor float, with two persons and pulling each end against the center to lift

the float from the water and walk the assembly to a dry area for cleaning and removing the sensors.

9.8.8.2.1. Rinse the float assembly, instrument cable, and rope with clean water and remove sensors.

9.8.8.2.2. Store float and rope in bed of tow vehicle.

9.8.8.3. Perform calibration check on redox and pH sensor and log results according to SOP L1 and SOP L3.

9.8.8.4. Store sensors with cable attached in trailer in designated storage area (SOP U4).

9.8.8.5. Remove 2-m holding stakes and store in bed of tow vehicle.

9.9. Look over the entire measurement area for any tools, sensors, or parts that may have been left out, if found, store them in the proper location.

10. Data and Records Management

10.1. Fill out an inspection checklist (Appendix F) at the beginning of each measurement period.

11. Quality Control and Quality Assurance

11.1. Maintain locations of sensors and retro-reflectors within 1 m of those specified in the SMP.

11.2. Perform the following QC and QA measures once at each location each time the location is set up to be measured and again if there are any data failures at the site due to human error, vandalism, theft, lightning/surge, storm, or other damage to the instruments during the measurement period.

11.3. At each power access location, check GFCI for ability to trip.

11.4. Verify that 120VAC supply power is available at each power access point.

11.5. Inspect the grounding rod for firmness in ground. Log inspection results on checklist (Appendix F) with remediation indicated.

11.6. Stability of instrument platforms:

11.6.1. Verify that the mast is staked tightly. Log inspection results on checklist (Appendix F) with remediation indicated.

11.7. Check that all masts, towers, and tripods are vertical.

11.8. Check for any indications of broken, bent or cracked mast or tower structural components. Log inspection results on checklist (Appendix F) with comments and remediation.

11.9. Orientation of sensors: Verify that the sonic anemometers are seated in their positions (SOP W2).

11.10. Verify that the lagoon probe cables have wire ties every 1 m along the rope all the way to the edge of the waterline. Log inspection results on checklist (Appendix F) with comments and remediation.

11.11. Check all retro-reflector heater power cables for damage. Log inspection results on checklist (Appendix F) with comments and remediation.

11.12. Check all sensor wires and cable at transmitter, receivers, scanner, and meteorological data logger NEMA box for tightness and damage. Check all enclosures for water intrusion. Log inspection results on checklist (Appendix F) with comments and remediation.

11.13. Additional QA procedures for sensors are indicated in each respective SOP.

12. References

12.1. LoggerNet Instruction Manual. 2005. LoggerNet Instruction Manual. Campbell Scientific Inc, Logan, UT.

12.2. iPort Instruction Manual. 2007. *i*Port Instruction Manual. Communications software for iSeries and C Series Instruments. Part#102606-00. Thermo Fisher Scientific, Franklin, MA.

12.3. CM110 Tripod Mast Manual. 2005. Tripod installation manual, Models CM110, CM115, CM120, Campbell Scientific Inc., Logan, UT.

12.4. SOP C2. 2006. Open Path Tunable Diode Laser Acoustic Spectroscopy (TDLAS). Standard Operating Procedure C2. Purdue Applied Meteorology Lab.

12.5. SOP C4. 2006. Synthetic Open Path Sampling System. Standard Operating Procedure C4. Purdue Applied Meteorology Lab.

12.6. SOP E3. 2006. The Measurement of Solar Radiation with the Licor Model 200SL or 200SZ Silicon Pyranometer. Standard Operating Procedure E3. Purdue Ag Air Quality Lab.

12.7. SOP G5. 2007. Measurement of Hydrogen Sulfide (H_2S) with the Thermo Fisher Scientific Model 450I Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab

12.8. SOP G7. 2007. Standard Operating Procedure for the Operation of the INNOVA 1412 Photoacoustic multi-gas analyzer. Purdue Ag Air Quality Lab.

12.9. SOP L1. 2006. Measurement of Lagoon pH with Innovative Sensors Model CSIM11 Sensor. Standard Operating Procedure L1. Purdue Applied Meteorology Lab.

12.10. SOP L2. 2006. Measurement of Lagoon Temperature with Campbell Scientific Model 107-L Thermistor. Standard Operating Procedure L2. Purdue Applied Meteorology Lab.

12.11. SOP L3. 2006. Measurement of Lagoon Redox State with Campbell Scientific CSIM11_ORP Sensor. Standard Operating Procedure L3. Purdue Applied Meteorology Lab.

12.12. SOP L5. 2006. Markland Sludge Gun. Standard Operating Procedure L5. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.

12.13. SOP U4. 2006. Open-source Instrument Trailer. Standard Operating Procedure U4. Purdue Applied Meteorology Lab.

12.14. SOP W1. 2006. Humidity and Temperature Probe. Standard Operating Procedure W1. Purdue Applied Meteorology Lab.

12.15. SOP W2. 2006. Ultrasonic Anemometer for Wind Velocity. Standard Operating Procedure W2. Purdue Applied Meteorology Lab.

12.16. SOP W3. 2006. Wetness Grid for Measuring Precipitation. Standard Operating Procedure W3. Purdue Applied Meteorology Lab.

12.17. SOP W5. 2006. The Measurement of Barometric Pressure with the Setra Model 278 (Campbell Scientific CS100) Barometer. Standard Operating Procedure W5. Purdue Applied Meteorology Lab.

12.18. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.

13. Contacts

13.1. Source for Delhi Sheet metal towers:
Lafayette Electronics Supply
405B North Earl Avenue
Lafayette, Indiana 47904 (Shipping)
P.O. Box 4549 Lafayette, Indiana 47903 (Mailing) or near site distributor (if available locally).
Tel: 800-842-1527

13.2. Source for CM 110 Masts:
Campbell Scientific, Inc.
815 West 1800 North,
Logan, UT 84321
Technical support: 435-753-2342

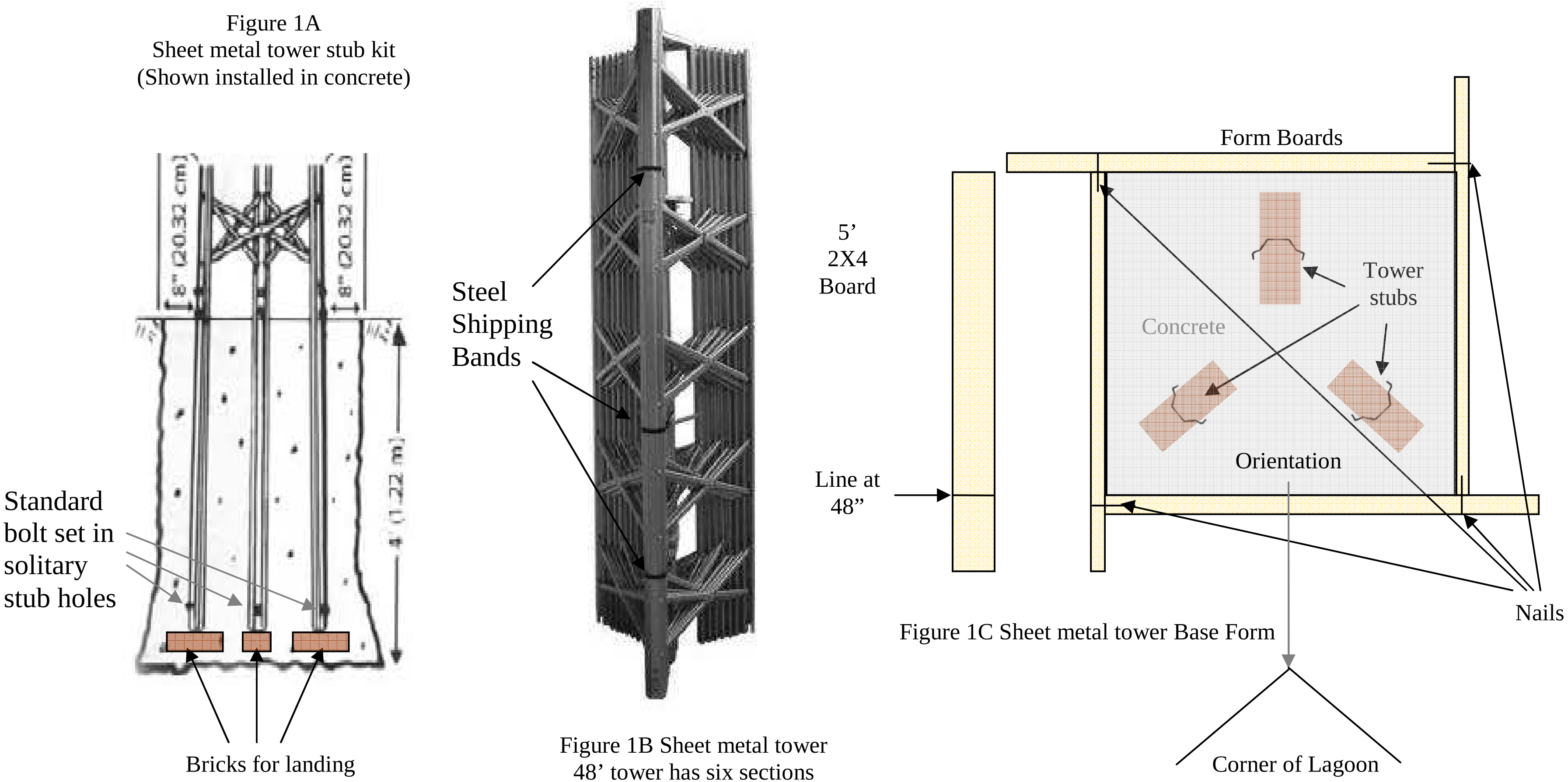


Figure 1A
Sheet metal tower stub kit
(Shown installed in concrete)

Figure 1B Sheet metal tower
48' tower has six sections
(Not eight as shown here but is
otherwise identical to the tower shown)

Figure 1C Sheet metal tower Base Form

Note: These figures are not shown to scale

Figure 1. Schematic diagrams for optional sheet metal tower components and the form for pouring the tower base.

Figure 2. Retro-reflector housing configuration.

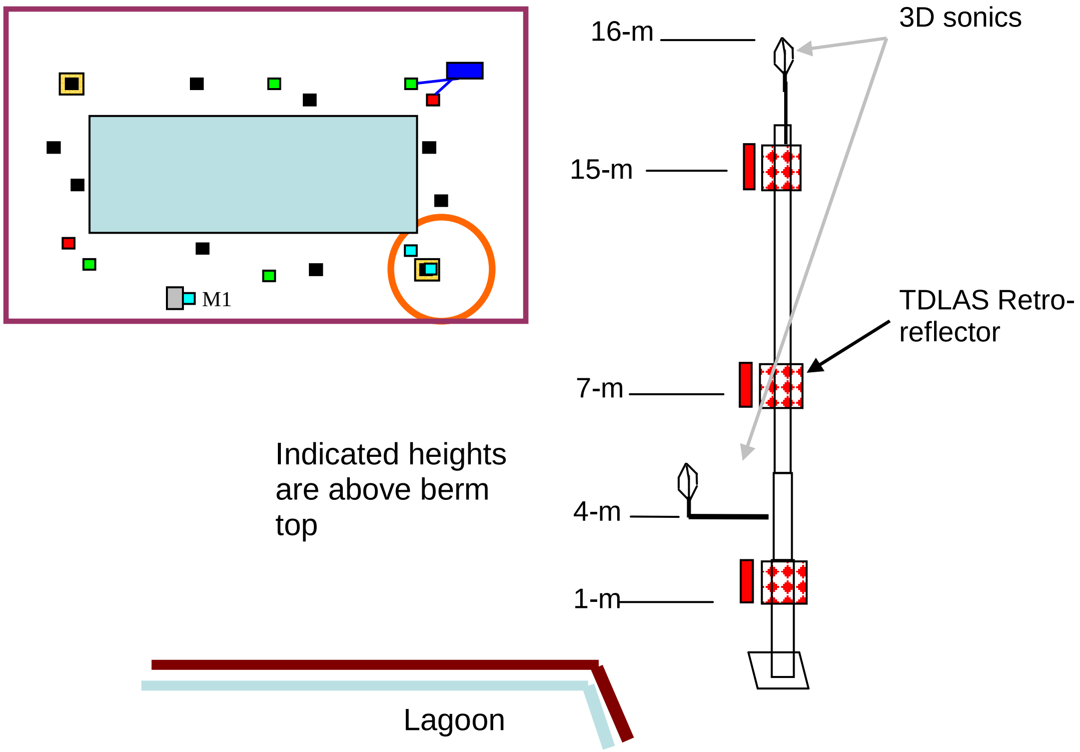


Figure 3. Ideal fixed tower configuration.

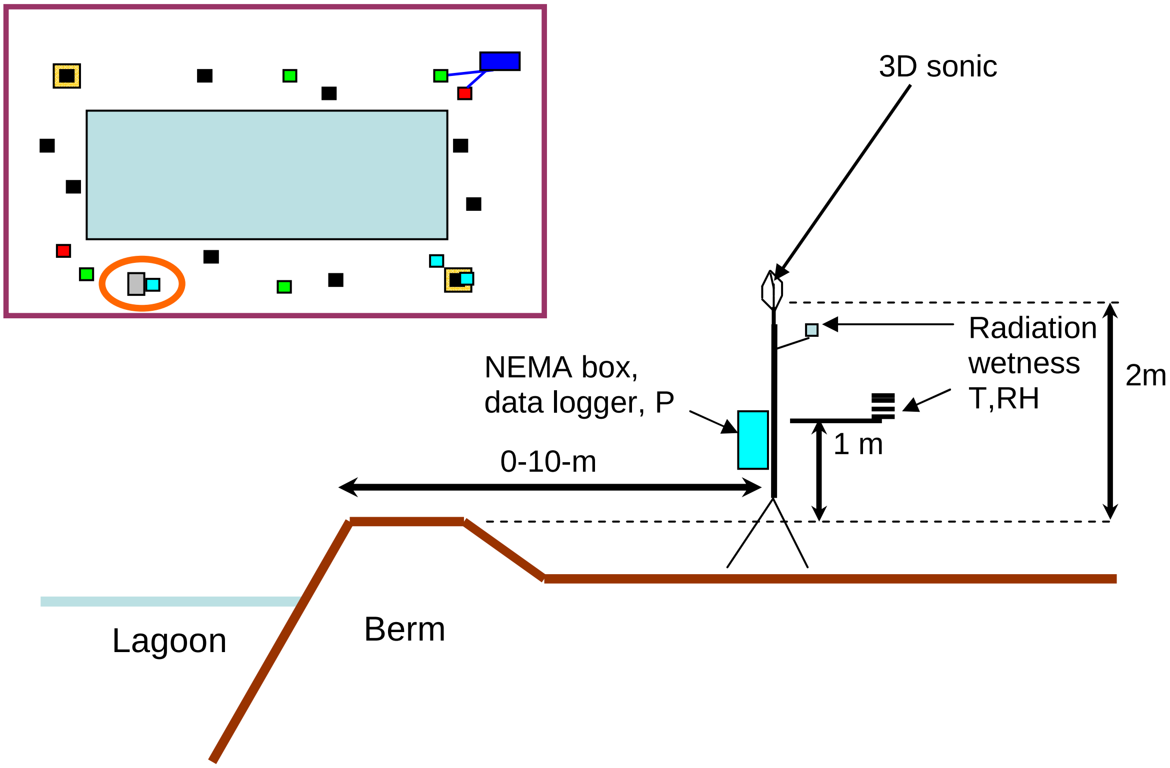


Figure 4. Ideal configuration for the meterological mast.

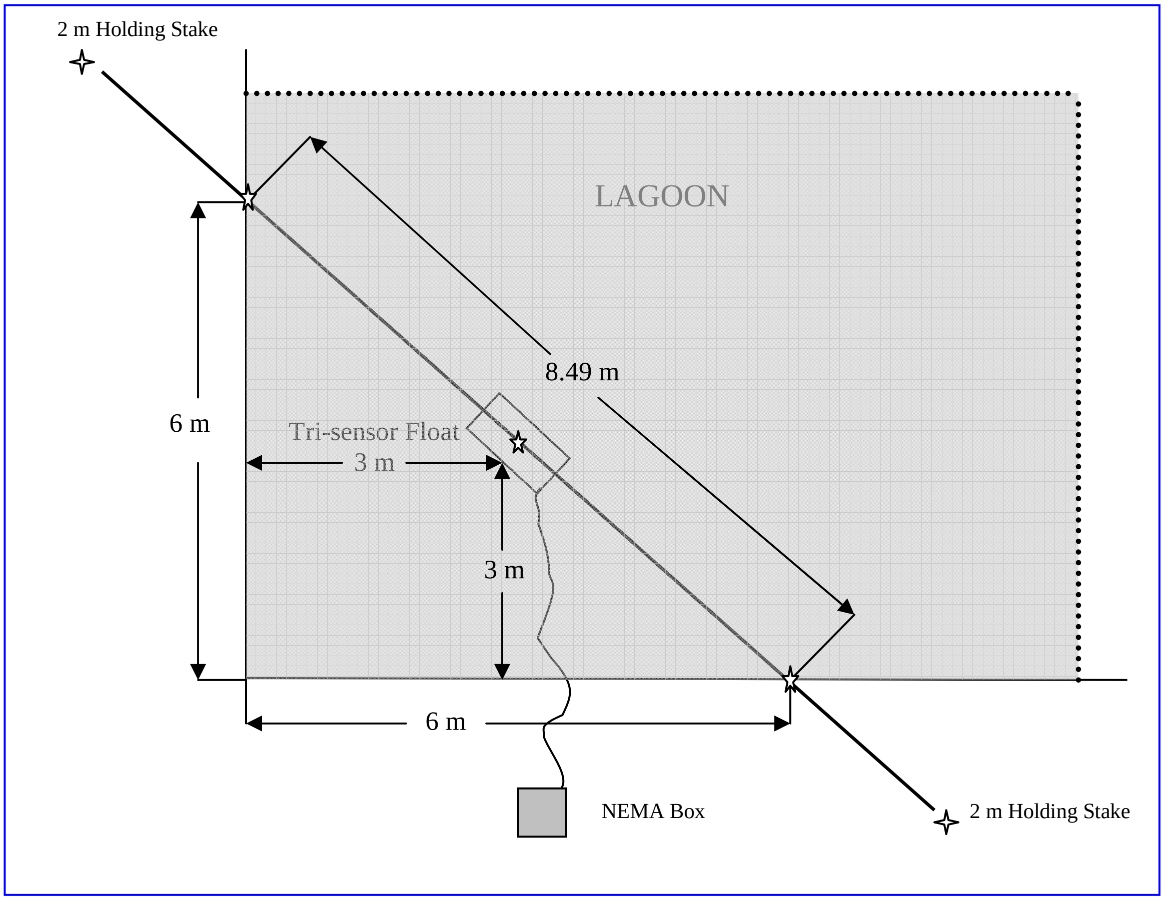


Figure 5. Proper deployment and orientation of the tri-sensor float.

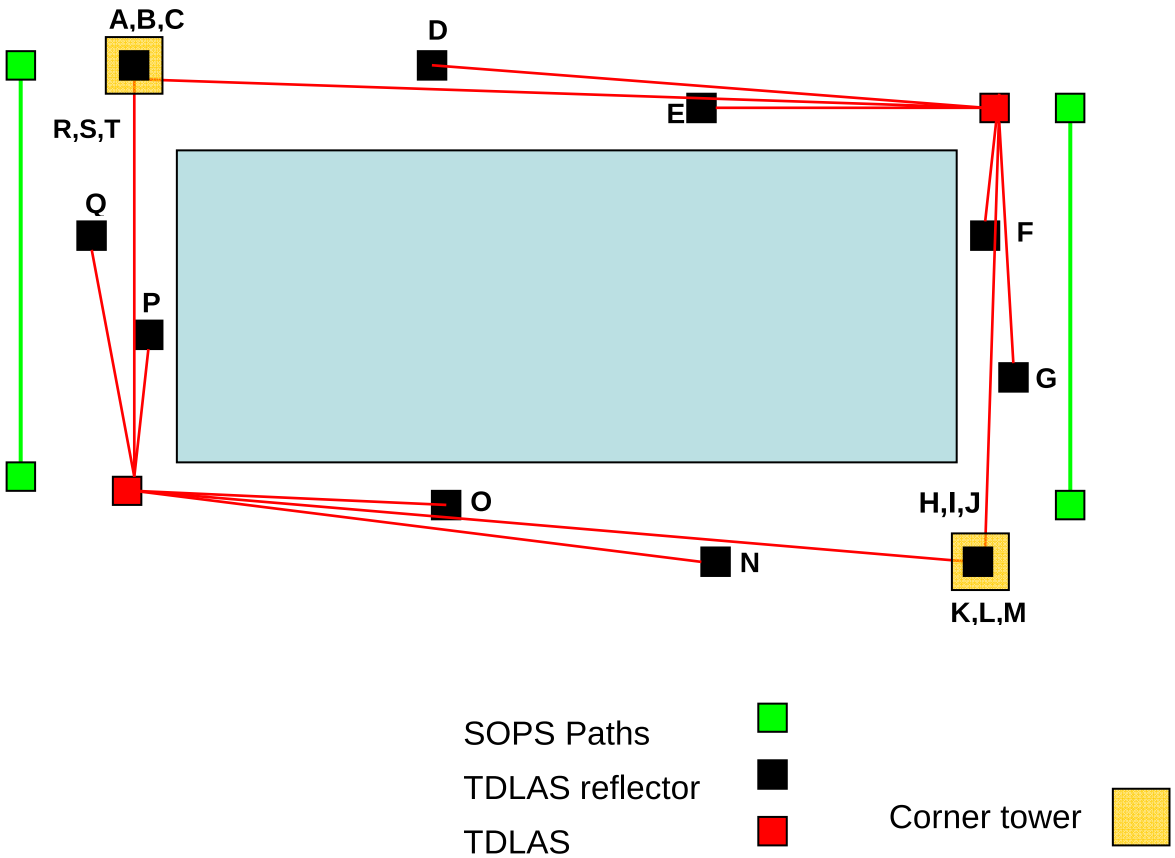


Figure 6. Ideal open-source gas measurement configuration for a lagoon source.

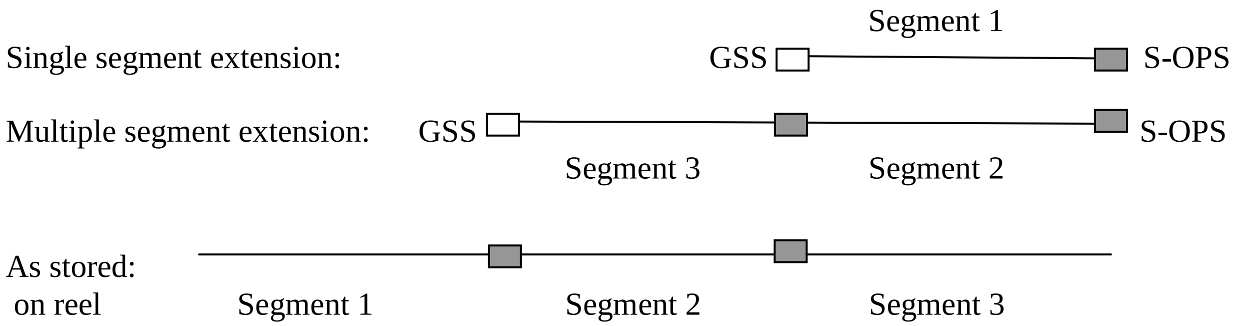


Figure 7 – Extension tubing configuration.
Connector material: Stainless steel (▨) and Teflon (□). Extension tubing is stored on the reel with Stainless Steel unions connecting segments together. An additional union is needed to attach both the single and multiple segment extension lines to the two S-OPS.

APPENDIX A
RESPONSE FOR BITES AND STINGS

- Spiders: Some spiders can cause great pain, and injury.

  - The Black Widow is a small, shiny spider, less than half an inch long but with a leg span of up to two inches. It has a red hourglass shape on its underside. A bite from a Black Widow spider can cause severe pain and muscle spasms, heavy sweating, stomach cramps, nausea, vomiting, tightness in the chest, breathing difficulty and a sharp rise in blood pressure.
  - The Brown Recluse spider is brown or brown-yellow in color and has a dark violin-shaped area on the back. The brown recluse is found under rocks and woodpiles as well as closets and attics. A bite from the brown recluse can cause severe pain, reddening, blistering, and death of the tissue followed by deep ulceration at the site of the wound. The bite may not be noticed at first. Pain at the bite site begins one to four hours later.
  - If you should get bitten by a spider, try to identify the type of spider. If possible, kill the spider without smashing it beyond recognition. Inform the site scientist and seek medical attention for your bite.

- Snakes: Most snakes are nonpoisonous. The bite of a nonpoisonous snake can cause pain and infection, but is rarely serious. There are three types of poisonous snakes in the study area, and their bites can cause serious illness and even death. They are:
  a. Eastern/Western/Pygmy Diamondback Rattlesnakes
  b. Water Moccasin
  c. Copperhead
  - First aid steps to remember if bitten
    a. Stay calm
    b. Look at snake for identification (shape of head, color and markings)
    c. Seek help
    d. Keep the bite at the level of the heart
    e. **Do not incise the bite**
- Insects and other invertebrates:
  - Ticks: The Lone Star Tick, the American Dog Tick, the Brown Dog Tick, and the Black Legged Tick or Deer Tick are some common ticks. Most ticks are capable of transmitting a variety of diseases, including Rocky Mountain Spotted Fever, Colorado Tick Fever, Lyme Disease and even Tularemia (a disease that causes toxins in the blood; high fever is a symptom).
    a. First aid -- remove ticks with tweezers. Gently grasp the tick behind the head and remove with a slow, steady pull. Wash the area thoroughly with soap and water.

RESPONSE FOR BITES AND STINGS
(Continued)

  b. Mark the date of the bite on the calendar and also report it to the program manager. If any flu-like symptoms, such as fever, muscle pain, extreme fatigue, headache, chills, joint pain, swollen glands or a rash appear, see your doctor. Lyme disease symptoms may not develop for up to two months.
  c. To avoid ticks, tuck pant legs into socks and spray an insect repellent on pants.
  d. Perform a self check for ticks at the end of the work day or at the completion of work in areas that contain ticks.

- Fleas/Chiggers: To avoid exposure to chiggers and fleas, do not walk in uncultivated areas and heavy vegetation. Spray an insect repellent on pants. High boots and socks mitigate chigger bites.
- Gnats/Mosquitoes: Although most gnats are not bothersome, Kansas has a gnat (Buffalo Gnat) that bites much like a mosquito. To repel these gnats it is recommended to use Avon *Skin So Soft* or vanilla extract. To avoid contact with mosquitoes, spray an insect repellent on the outside of clothing and on bare skin if repellent is designated for it.
- Blister Beetles: Blister Beetles are small insects that have yellow and black stripes running the entire length of the body. The bite produces immediate pain, redness, itching and swelling that can persist for hours.
- Hornets/Wasps/Bees:
  a. Bees have a stinger attached to a poison sac that is left in the wound when a sting occurs. Wasps and hornets may sting repeatedly.
  b. First aid -- remove the stinger from the wound by scraping a blunt object, such as a dull blade, across the wound. Symptoms usually result within a few hours. If symptoms such as difficulty breathing or rapid, severe swelling occur, seek medical attention immediately.
- Scorpions: All scorpions can produce a painful sting, but serious life threatening symptoms are rare. First aid is similar to that given for a wasp sting.

APPENDIX B.
RESPONSE TO WILD ANMIALS

- The badger is a small (average 20 lb) mammal that feeds on small rodents, fruits and roots. They have very sharp teeth and long, sharp claws and can be quite aggressive. They are a defensive animal and will usually only attack if provoked. The badger, in cooler weather, will make a burrow and remain there for several months. The opening to the burrow is six to eight inches in diameter. Should you spot a badger hole, **keep your distance and do not put your feet or hands in the hole**.

- The skunk is a small mammal that produces a very unpleasant odor when excited or threatened. Please keep your distance from these small mammals. If bitten by a skunk (many carry rabies), you should seek medical attention immediately.

- Coyotes are a small dog-like mammal. They attack small animals and livestock. For the most part they are very shy and afraid of humans. They do carry rabies; so, if bitten, you should seek immediate medical attention.

APPENDIX C
WEATHER RISKS

Severe Weather. Be aware of weather conditions at all times while on site. Do not work outside during lightning storms. In case of severe thunder storm and/or a tornado warnings, cease work and seek shelter. It is your responsibility to locate the nearest available tornado shelter. In case an unexpected tornado occurs, lay flat in the nearest depression until the tornado has passed. The site PI or scientist has the authority to stop work, at his discretion, during severe weather activity.

Cold Weather exposure. There are two main personnel effects cold weather can produce.

a. Hypothermia : Hypothermia results when the body loses heat faster than it can produce it. When this situation first starts to occur the blood vessels start to constrict, and hands and feet are first affected. Next, involuntary shivers begin to occur. The shivering is your body's first warning sign. Further heat loss produces speech difficulty, forgetfulness, loss of manual dexterity, collapse, and finally death.

b. Frost bite: As your blood vessels constrict to keep vital organs warm, hands and feet can experience frost bite. Hands and feet are the first body parts affected. Frost bite begins with a burning sensation in your hands and feet and then enters a numbing phase. The numbing phase is very dangerous and actual frost bite may occur.

Cold Weather Exposure Prevention: Dress warmly, wear layered clothing. Cover all exposed flesh. If clothes become wet, change as soon as possible and take frequent breaks in a heated area. Do not drink alcoholic beverages.

Hot weather exposure. There are three heat-related illnesses that hot weather exposure can produce. They are:

a. Heat stroke is life threatening. The victim's temperature control mechanisms stop working. The body temperature can rise so high that brain damage and death may occur if the body is not cooled quickly.
   (1) Symptoms include dry, red skin, hot, and very high body temperature.
   (2) First aid is to cool the person down as quickly as possible and call for medical help.

b. Heat exhaustion is less dangerous than heat stroke. It typically occurs when people work in a warm, humid place where body fluids are lost through heavy sweating.
   (1) Symptoms include cool, moist, pale, or red skin or heavy sweating, dilated pupils, headache, nausea, and dizziness.
   (2) First aid is to get the person in a cooler place, place him or her on their back with feet elevated. Cool by fanning or using cold packs and give 1/2 cup water every 15 minutes.

c. Heat Cramps are muscular pains and spasms due to heavy exertion. Heat cramps usually involve the abdominal muscles or the legs. It is generally thought that the loss of water and salt from heavy sweating causes cramps..

Hot-weather illness prevention: Drink plenty of liquids such as water, fruit and vegetable juices, and electrolyte solutions (avoid soda pop, alcohol, and coffee). In addition limit strenuous activity during hot periods, wear loose fitting, light colored clothing, preferably made of natural fibers such as cotton (man-made fibers do not absorb water well). Wear a hat.

APPENDIX D.

Sheet Metal Tower Installation and Removal

Sheet metal tower installation (Base form)

1. Use proper protective equipment when working with sheet metal tower.
    a. Wear a pair of boots with heavy soles for climbing the tower during installation.
    b. Use a pair of leather gloves for handling the rope and tower sections.
    c. When climbing, wear the safety climbing harness and lanyard.
    d. Have your partner wear a helmet and leather gloves when working under the tower and assisting you.
    e. Uncured concrete is acetic in nature and can cause mild burns and irritation to unprotected skin, wash concrete from skin as soon as possible if you get uncured concrete on your skin.
    f. When working on the tower, be sure to securely fasten one of the two way radios on your person for communication with your partner and remove any item from your person that might fall and cause injury or be damaged (pocket or belt worn knives, cell phones etc.).
2. Do not start this procedure if below-freezing temperatures, high winds (gusts in excess of 15 mph), storms, or heavy precipitation are expected in less than 24 h, as the tower base may not set correctly in those conditions.
3. Contact the local cement provider and ask them if they can supply you with concrete this day at the time you think you may need it. You will need approximately 1.8 yd^3 for each sheet metal tower base.
    a. Each sheet metal tower base takes approximately 1-4 h to prepare, depending on the difficulty of the excavation of the base.
    b. The base generally must be excavated by hand or done very carefully with a small excavator, as the walls of the excavation need to be relatively vertical on all sides.
    c. Contact the producer or his/her staff before beginning concrete work to determine where you can dispose of any excess concrete, and where the concrete provider can clean out the truck chute.
    d. Inform the producer or his/her staff there will be about 2 yd^3 of dirt excavated from each tower base. Ask if they can dispose of or move the dirt, and ensure that the farm staff does not disturb the tower base during the installation process.
    e. Obtain clearance from the producer and a suggested route to allow the heavy concrete truck to drive over the area to deliver concrete. These trucks are very heavy and can damage some underground pipes and landscaping.
4. Assemble one of the stubs (Fig. 1A) from the stub kit to the outside of the leg on the largest end of the biggest tower section at the two predrilled holes in each base stub and tower section, using two bolt sets from the largest bolt set in the tower bolt kit. Securely tighten the stub mounting bolts using a box end wrench and the ½″drive socket and ratchet. The head of

the bolt should be on the outside of the stub on all sections (use the box end wrench here) and the nut and washer should go on the inside of the tower leg (use the socket and ratchet there).

   a. Install a large standard hardware bolt set (one bolt, one nut, and a washer) in the solitary pre-drilled hole at the bottom of the base stub you just bolted to the tower base. This will be immersed in concrete to prevent the tower stub from pulling out of the concrete after it hardens.
   b. Turn the tower over one step to the right and repeat step #4 until all the base stubs (3) have been installed onto bottom of the base section (largest end of the largest section) and then lay the assembly aside.

5. Using a shovel, dig a square hole 44″x44″x4′ deep at each sheet metal tower location to fill with concrete and form the base for each tower. **The tower location cannot easily be moved more then a few inches once the excavation of the base form has begun!**
   a. Dig only as many holes as you have time to finish this day. Start this in the morning, so that there is time to finish all the tower bases in one day. Sheet metal tower base installation (assembling base section, digging bases, building and filling concrete forms) takes between two to more than five hours depending on the difficulty of the soil excavation of the base form for each tower. Tubular tower base assembly, digging bases, and building and filling concrete forms takes approximately an hour for each tower.
6. Once the excavation of the base form is complete, place the three bricks in the bottom of the form hole at the approximate location where the ends of the base stubs will sit to land the stubs and help keep the tower at the proper level during curing of the concrete (Figs. 1A and 1C show the position of the bricks in the hole).
   a. The tower should be oriented so that one side of the tower is facing the corner of the lagoon (Fig. 1C) and a leg faces each adjacent corner for proper mounting of the retro-reflectors later.
7. Level the bricks at the bottom of the form hole using the carpenters level so that the tower will sit perfectly vertical once the stubs are placed on the landing bricks.
8. Have your partner hand you the tower base assembly (stubs first) and place it on the bricks. Adjust the bricks so that the stubs sit squarely in the center of them (Fig. 1C).
9. Have your partner check each tower leg above the hole with the level in the vertical position and adjust the bricks so that the bubble in the level has exactly the same slight offset for each leg. Note that the bubble in the level should be in the center (indicating the leg is nearly perfectly vertical), but the bubble will rest on one line slightly more then the other on each leg. When the offset is exactly the same for each leg, the tower section is vertically true.
10. Call the local cement provider; tell them you are now ready for concrete delivery.
11. Construct the concrete form for the tower base as follows (Fig. 1C):
    a. Using the tape measure, a pencil, and a “T” square draw a line straight across one 10′ 2x4 in the center.
    b. Using the circular saw cut the board at the line to form two 5′ sections.
    c. Repeat to cut two more 5′ sections from the other 10′ 2x4 (four 5′ sections are required for each tower base).
    d. Using the tape measure, a pencil, and a “T” square draw a line straight across all of the 5′ sections, at 48″ from one end of each board.

    e. Using the 2.5″ framing nails, make a square form for each of the tubular towers by nailing the boards at the lines to form a 48″ square (Fig. 1C).

12. Place the form on the tower base hole by lifting the form over the tower base assembly.
    a. If you disturb the tower, you must recheck the tower (Step 8) to ensure it is still vertical.
13. Have the concrete truck back up to the hole and deliver the concrete into the formed hole while holding the tower base assembly with both hands and a foot to ensure the tower doesn’t move while pouring the concrete.
14. After filling the base form use a level to ensure the tower is still vertically true.
15. Smooth the concrete of each base using a trowel so that it is even with the form.
16. You must re check the tower once again to ensure it is still vertical using the method described in Step #9 now and again before you leave the site in the evening for each sheet metal tower base you construct.
17. Allow the bases to sit for at least 72 h before constructing the towers or climbing on the bases.
    a. The base forms can easily be removed the following day by tapping the extended sections of form boards with a hammer.

Sheet metal tower installation (Tower installation)

18. Separate all the tower sections and locate all necessary bolts for the tower, if any bolts are missing, locate or replace them before proceeding.
19. Lay the tower sections near the base in the order in which they will go up (largest first, smallest last).
20. Place the bolts for the first section in your pocket, tie the work rope onto the climbing belt loop and proceed to the top of the base section. Secure yourself to the top of the section so that the top of the tower is at your waist using the lanyard.
    a. Wrap the lanyard from the loop where it is hooked to the left side of your climbing belt, around the legs of the base section near the top and hook to the loop on the right side of your climbing belt.
    b. Wrap the lanyard around the tower legs to eliminate slack so that you are closely secured to the face of the top section at your waist.
    c. Avoid wrapping the rope **over** the ends of the section you are standing on as it will obstruct your ability to assemble the section, and might slip back over the leg while you are lifting the next section, causing you to lose your balance or fall.
21. Have your partner tie the free end of your work rope to the top of the next section to be installed (this will be the smallest end of the largest uninstalled section).
    a. Tie the rope firmly so it doesn’t become loosened as the section is hoisted up but loosely enough that it can easily be untied once it reaches the top.
22. Have your partner move back out of the way in case you drop the section. Now, hoist the section using the rope. Hook one rung of the section of tower you just lifted over one of the ends of the section you are standing on when it reaches you.
23. Untie the rope from the section and let the free end of the rope fall back to the ground.

24. Standing firm on the tower, grasp the section with both hands and lift it about half way up to where it will sit and again hook it over the end of the section you are standing on so that you can re-orient your hands.
25. Now, grasp the section again and lift the bottom of it the rest of the way above the section you are standing on and lower the bottom of it (largest end) into the top ends of the section you are standing on.
    a. Each section slips into the top of the previous section where it sits on a set of riveted stops designed to allow the parts to rest at the correct position while the sections are bolted together.
26. Insert each of the six bolt sets into the pre-drilled bolt holes to fasten the two sections together and tighten.
    a. Be sure to use the largest bolt set that will fit in the hole from the bolt kit. Each section has the holes drilled to exactly fit the correct bolts.
    b. Place the bolt into the hole from the outside of the tower so that the head of the bolt is on the outside and the nut and washer are on the inside.
    c. Tighten the bolts firmly, but do not over-tighten the bolts and cause them to break, as they are specially-made bolts. Standard hardware must not be substituted for them.
27. Climb the section you just installed and repeat the procedure from Step #21 to install the remaining sections of tower.
    a. Fatigue, height, and other conditions such as light winds do make the task a bit more difficult with each section you install but the sections get smaller and lighter as you go up, and this tends to offset the difficulty level.
28. Proceed to “Optional sheet metal tower installation (Equipment installation)”.

Sheet metal tower installation (Initial Equipment installation)

29. Have your partner unpack and assemble the following:
    a. Lightning rod and mounting bracket (storage location shown in SOP U4)
    b. Connect one end of the green #6 ground cable to the lighting rod mounting bracket
    c. Mount the assembly near the top of the mast pipe.
    d. Unpack and install sonic anemometer at top of the mast pipe (This will become the 16-m location) storage location shown in Fig. 1 of SOP U4.
    e. Unpack the sonic long anemometer power cable from storage area in trailer (Storage location shown in Fig. 1 of SOP U4) and uncoil it.
    f. Inspect cable and repair or replace as needed. Log inspection results on checklist (Appendix F) with comments and remediation.
    g. Connect power cable to anemometer (SOP W2).
    h. Secure the ground and anemometer cables to the mast pipe using electrical tape.
    i. Leave two feet un-taped at the bottom of the mast pipe to slip into the tower mounting location at the top section.
    j. Securely tie the assembly to the work rope with the top facing upward.
30. Carefully hoist the mast assembly to the top of the tower using the work rope, have your partner help control the assembly using the ground cable and the remaining free end of the rope.

31. Slip the bottom of the assembly into the tower and secure using the manufacturer-supplied mounting hardware kit for the mast pipe.
32. Tape the cable at the top of the tower and every two feet along the way as you work your way down (these cables will remain on the tower for the remainder of the measurement period, so leave a small loop of anemometer power cable at top for later repairs if necessary).
33. Have your partner unpack 6 retro-reflectors (storage location shown in SOP U4) and attach mounting board using wire ties.
    a. Inspect cables and repair or replace as needed. Log inspection results with comments and remediation.
    b. Have your partner tie a retro-reflector and mounting board, one at a time, to the work rope.
34. Hoist the parts up to the 15-m retro-reflector mounting location and have your partner help control the ascent with the dangling section of rope.
35. Tie the rope off so it is within reach near the top and attach the retro-reflector, then repeat the procedure for the second retro-reflector.
36. Mount two (2) retro-reflectors on the tower at the 15-m above berm height, one on each leg facing adjacent corners of the lagoon. Align the units so that the open sides of each retro-reflector enclosure are facing the area where the TDLAS spectrometers are to be located.
37. Connect the power cables from the retro-reflectors to the power boxes on the tower and secure them with tape.
38. Proceed to the 7-m mounting location on the tower. Have your partner tie another retro-reflector to the work rope and repeat the procedure used for the 15-m retro-reflector mounting to install the retro-reflectors at the 7-m location.
39. As before, mount two (2) retro-reflectors on the tower at the 7-m above berm height, one on each leg facing adjacent corners of the lagoon. Align the units so that the open sides of each retro-reflector enclosure are facing the area where the TDLAS spectrometers are to be located.
40. Connect the power cables from the retro-reflectors to the power boxes on the towers, and secure them with tape.
41. Proceed to the 4-m anemometer mounting location.
42. Have your partner do the following:
    a. Locate the 4-m custom-made anemometer mount (may have been shipped to the site or transported in the bed of tow vehicle).
    b. Securely tie the assembly to the work rope with the top facing upward.
    c. Carefully hoist the assembly to the 4-m level of the tower using the work rope, have your partner help control the assembly using the free portion of remaining rope.
    d. Attach the custom mount to the tower using the hardware supplied with the custom made mount.
    e. Unpack and install sonic anemometer (storage location shown in Fig. 1 of SOP U4) at the short mounting stub on custom mount.
    f. Unpack sonic anemometer power cable from storage area in trailer (Storage location shown in Fig. 1 of SOP U4) and uncoil. Inspect cable and repair or replace as needed. Log inspection results on checklist (Appendix F) with comments and remediation.
    g. Connect power cable to anemometer (SOP W2).
    h. Secure the anemometer power cable to the 4-m custom mount using electrical tape.

   i. Tape the cable here and every two feet along the way as you work your way down (this cable will also remain on the tower for the remainder of the measurement period) be sure to leave a small loop of wire for later repairs if necessary.

43. Proceed down to the base of the tower.
44. Mount two (2) retro-reflectors on the tower at 1 m above berm height, one on each leg facing adjacent corners of the lagoon. Align the units so that the open sides of each retro-reflector enclosure are facing the area where the TDLAS spectrometers are to be located.
45. Connect the power cables from the retro-reflectors to the power boxes on the tower, and secure them with tape here and at every two feet down to the tower base.
46. Unpack and drive an 8′ ground rod in the ground near the base using large hammer (Storage location shown in Fig. 1 of SOP U4).
    a. Drive the ground rod near the tower base.
    b. Connect to a tower leg using a piece of the bare #6 grounding wire using the provided hardware.
47. Connect the drop cord feeding the tower to the power access point and check for normal operation.

Sheet metal tower installation (Equipment removal)

48. Remove all instrumentation and equipment from the tower and pack into the respective storage boxes and locations in the instrument trailer.
49. If this is not the last measurement period at the site:
    a. Leave the grounding cable on the tower.
    b. Disconnect the drop cord feeding the tower from the power source, coil up and tape it to side of tower.
50. If this is the last measurement period, remove all cables and equipment from tower and proceed to “Optional sheet metal tower installation (Tower removal)” below.

Sheet metal tower installation (Tower removal)

51. Discuss the tower base removal with the producer. He/she may want to leave the base in the ground and cut off the base stubs, or remove the base and fill the hole him/her self. If not, the bases weighs nearly 9,000 lb, and can be removed with the aid of a large backhoe or excavator. Disposal of the concrete will require a tri-axle or other heavy truck to haul the cube to a landfill location. Fill the hole immediately to prevent injury to humans or animals that might fall into the holes. Contact an excavating company to remove the base and take care of those details. Note the following:
    a. If the base is left in the ground the stubs MUST be cut off flush with the concrete to prevent impalement or serious injury from falling over or onto them.
    b. If a torch is used to cut the stubs off flush with the concrete, be careful, as sections of the concrete can fracture under high heat stress with sometimes near-explosive force.
52. Climb tower and firmly secure yourself waist high at the top of the second section down from the top with the lanyard.

   a. Wrap the lanyard from the loop where it is hooked to the left side of your climbing belt, around the legs of the base section near the top and hook to the loop on the right side of your climbing belt.
   b. Wrap the lanyard around tower legs to eliminate slack so that you are closely secured to the top of the section at your waist.
   c. Avoid wrapping the rope **over** the top end of the section you are standing on (the area where the bolts are), as it will obstruct your ability to disassemble the section above and might slip back over the leg while you are lifting off the section above, causing you to lose your balance or fall.

53. Remove tower section above:
    a. Remove the bolt sets attaching the section above you to the tower and secure them in your pocket or a satchel.
    b. Use both hands and wiggle the section out of the section you are standing on and let it down as far as is comfortable then hook it over the exposed leg of the section you are standing on.
    c. Now, re-adjust your hands and lower the section down further to hook the top of it over the section you are standing on and tie the work rope to the section to be lowered.
    d. Lower the section to your partner and have them untie the rope and lay the section aside.
54. Move down to the next lowest section, and repeat Step #58 until all sections of the tower have been removed.
55. Load the largest section into the bed of the tow vehicle (smallest end forward).
56. Load each progressively smaller section into the center of the previous section in the tow vehicle (smallest ends first) until the entire tower is loaded inside itself in the rear of the tow vehicle for transport to PAML.
57. Store the tower bolt sets in a bag or bucket in the rear of tow vehicle.
58. Remove ground rods and store in trailer.
59. Remove tower base or cut off the base stubs as discussed in Step #56.

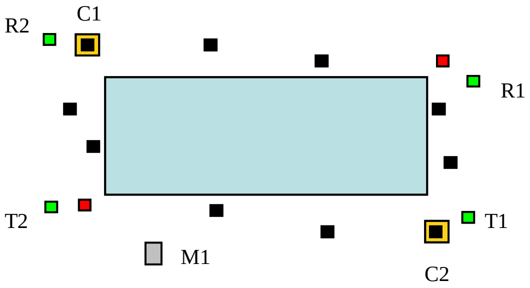
If the base and stubs are not to be removed immediately cover the stubs with a pile of wood, bucket, or some other item to prevent risk of serious injury to humans or animals.

14. APPENDIX E
Site Open-path Sensor Installation Report

Location: ______________________

Date: ______/______/_______

FOS: ________________________________



Mast location deviation? (M1) ______Deg (N) _____Min______Sec

---

SOPS Path #1 start location (T1) ______Deg (N) _____Min______Sec

SOPS Path #1 end location (R1) ______Deg (N) _____Min______Sec

Distance between Path #1 outlet tube and GSS manifold: _____________________ m

Deviation of path#1 height above lagoon berm : _______________________ m

SOPS Path #2 start location (T2) ______Deg (N) _____Min______Sec

SOPS Path #2 end location (R2) ______Deg (N) _____Min______Sec

Distance between Path #2 outlet tube and GSS manifold: _____________________ m

Deviation of path #2 height above lagoon berm : _______________________ m

---

Notes:

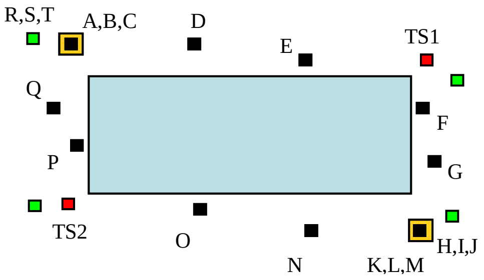
Site Open path sensor Installation Report

(Continued)

Location: ________________________

Date: ______/______/_______

FOS: ___________________________________

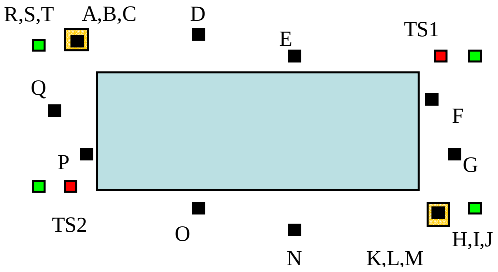


TDLAS/scanner 1 location deviation (TS1) ______Deg (N) _____Min______Sec

	---------------- Location -----------------------				
Retro-reflector	Deg (N)	Min	Sec	Distance from TDLAS (m)	Actual height above berm (m)
A					
B					
C					
D					
E					
F					
G					
H					
I					
J					

Site Open path sensor Installation Report

(Continued)



Location: ______________________

Date: ______/______/_______

FOS: ________________________________

TDLAS/scanner 2 location deviation (TS2) ______Deg (N) _____Min______Sec

---------------- Location -----------------------

Retro-reflector	Deg (N)	Min	Sec	Distance from TDLAS (m)	Actual height above berm (m)
K					
L					
M					
N					
O					
P					
Q					
R					
S					
T					

Notes: (continue on separate page if necessary)

APPENDIX F

Inspection Checklist Form

(Cables)

Date: ___________________

Item	All OK	One Bad	Repaired or Replaced Unit # or Description	Note #
Retro-reflector Cables (15-m/Tower)				
Retro-reflector Cables (7-m/Tower)				
Retro-reflector Cables (1- m/Tower)				
Retro-reflector Cables (1-m 2/3)				
Retro-reflector Cables (1-m 1/3)				
SOPS Tubing Path #1				
SOPS Tubing Path #2				
TD-LAS Power/Signal Cables				
Meteorological Unit Power Cable				
Lagoon Sensor Data Cables				
Sonic Anemometer Cables (16-m)				
Sonic Anemometer Cables (4-m)				
Sonic Anemometer Cables (1-m)				

All notes to be documented on back of form and note # referenced to the appropriate entry!

Name: ____

Inspection Checklist Form SOP U5

(Continued)

(Hardware)

Date: ___________________

Item	**OK**	**Problem**	Note #
GFCI Trip and Power OK?			
Lightning Rod OK?			
Ground Wire OK?			
Mast Stakes			
SOPS paths operating at normal flow rates?			
Indication of flow at all SOPS orifices?			
All Towers, Tripods, & Mast Look Vertical?			
All Towers, Tripods, & Mast Intact?			
Sonic Anemometers Mounted & Oriented Correctly?			
Lagoon Cable Floats Intact And Spaced Correctly?			
All Heater Cables Look OK, Mounted Securely?			
All Other Sensor Cables Look Intact			
Meteorological Boxes Weather Tight?			
Desiccant Fresh In Meteorological Boxes?			

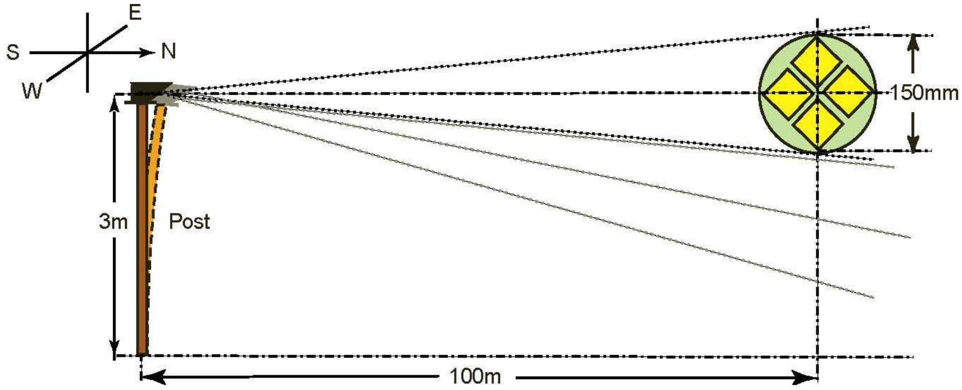
All notes to be documented on back of form and note # referenced to the appropriate entry!

Name: ____

APPENDIX G

Calculating the Effects of Movement

Consider the example of a 150 mm-diameter retro-reflector array located 100 m due N of the remote head:



The TDLAS/scanner can move vertically or horizontally 150 mm (6″) to either side of the centreline before the beam will move completely off the retro array. This ***translational*** movement is not common. However, because the TDLAS/scanner system is fixed at the bottom, ***torsional*** or bending movement of the TDLAS/scanner is much more likely, due to wind loading or thermal distortion. Any bending perpendicular to the centreline is manifested as a lateral motion, and will have a minimal effect. Conversely, any bending along the centerline will have a serious negative effect: The top of a 3-m tall post can only move 4.5 mm (0.18″) before the beam will have moved all the way off the retro. For a 500-m path length, with the retro-reflector array doubled in diameter to 300 mm (12″), the top of 3-m post can only move 1.8 mm (0.07″) before beam will completely miss the retro-reflector.

The diameter of the laser beam when it exits the TDLAS is about 2.5 mm, and the divergence is approximately 1.5 mrad (milliradians). This means that at a distance of 100 m the beam diameter is 1.5 x 100 = 150 mm (6″). Similarly, for a 1,000-m path, the beam diameter would be 1,500 mm (59″).

The remote head can be mounted on a tripod, post, or platform, and directs the laser beam to a reflector, which can be from a few meters to several hundred meters distant.

THE POWERWARE 9125 UNINTERRUPTIBLE POWER SUPPLY

Standard Operating Procedure (SOP) U6

THE POWERWARE 9125 UNINTERRUPTIBLE POWER SUPPLY

Standard Operating Procedure (SOP) U6

Prepared by
Bill W. Bogan and Sam M. Hanni

Reviewed by
Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
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1. Scope and Applicability

1.1. A means is needed to prevent power outages or disruptions to the equipment in the on-farm instrument shelter (OFIS) in order to ensure that data collection is not interrupted.
1.2. A means is also needed to protect the equipment from possible poor power quality. Power surges or spikes, for example, could be damaging to some of the gas analyzers, or to the DAC system.
1.3. This SOP details the use of the Powerware Model 9125 uninterruptible power system (UPS), which provides both of these functions.
1.4. SOP U1 provides a list of which equipment should be connected to the UPS. Sampling pumps and converter ovens are not connected to the UPS.

2. Summary of Method

The Powerware 9125 (Powerware Corporation, Espoo, Finland) UPS filters incoming AC power, and provides consistent, spike-free power with a pure sine wave output. The unit's double-conversion online design constantly conditions and controls AC output, assuring zero delay when transferring to backup power in the event of a power failure. The unit incorporates proprietary "Advanced Battery Management" technology, which allows the unit to operate without draining the battery, extends battery life, reduces battery recharge time, and provides a warning when the batteries are nearing the end of their useful life. The Model 9125 can accept up to four Extended Battery Modules (EBMs), further extending the length of time that the UPS can support equipment in the event of a power disruption or outage. Batteries are hot-swappable, so that power to the equipment does not need to be interrupted.

3. Definitions

3.1.	EBM	Extended battery module
3.2.	OFIS	On-Farm Instrument Shelter
3.3.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.4.	QAPP	Quality Assurance Project Plan
3.5.	UPS	Uninterruptible power system

4. Health and Safety

4.1. The Powerware 9125 contains potentially lethal voltages. There are no user-serviceable parts in the unit. Do not attempt any service to the UPS other than changing of batteries. Under no circumstances should repairs to the UPS or its batteries be attempted by anyone other than authorized service personnel.
4.2. Because the Model 9125 contains batteries as a backup power supply, these voltages may be present even when the unit is not plugged in.
4.3. The batteries themselves can cause electrical shocks if they are handled improperly. Do not contact both poles of the battery simultaneously, and do not attempt to service the batteries. Damaged batteries can cause acid burns.
4.4. Remove watches, rings, or any other metal objects when handling the batteries.

4.5. Use only tools with insulated handles around the batteries.
4.6. Do not lay tools or any other metal parts or equipment on top of the batteries, or allow them to contact the battery poles.
4.7. When connecting the UPS and EBM, some arcing may occur. This is normal, and will not harm personnel. Minimize arcing by quickly inserting the EBM cable into the UPS battery connector, and establishing a firm connection.
4.8. Batteries are lead-acid, and must be properly disposed of or recycled. Do not discard in the trash.
4.9. Never unplug the unit while it is turned on, as this will remove the safety ground from the unit, and therefore from all equipment which is connected to the unit.
4.10. The UPS and EBM weigh approximately 100 lb combined. Do not attempt to lift the UPS and EBM without assistance.
4.11. Operate this UPS only in temperature- and humidity-controlled environments (i.e. inside the OFIS). Do not operate if the temperature exceeds 40ºC (104ºF), or if the humidity exceeds 95%. Operation under these conditions can present a fire or shock hazard.
4.12. Total earth leakage current from all equipment plugged into this UPS must be <1.5 mA.

5. Cautions

5.1. If the UPS is positioned vertically, the air vents must be at the top of the unit.
5.2. Although EBM batteries will have charged to 80% of their capacity after approximately 2 h in a plugged-in unit, it is recommended that batteries be allowed to charge for 24 h after they are initially installed, or after long-term storage. Batteries may not perform optimally if the power is interrupted to the UPS during this period.
5.3. Do not turn the UPS off while it is in Configuration mode (Section 9.2), as this will immediately shut off all power to the equipment as the UPS goes into Standby mode.
5.4. Do not plug laser printers into the UPS, as their power requirements are too high.
5.5. Make sure the receptacle into which the UPS is connected has the correct polarity. Setup of the OFIS will include a polarity check (with a GFCI receptacle tester) of all outlets.
5.6. Disconnect power to the UPS when initially connecting power to the OFIS.

6. Interferences

6.1. Batteries in the EBM can wear out. The 🔋 indicator and alarm signal that batteries may need to be replaced. If this indicator is lit, press and hold the Test/Alarm Reset button (Section 9.2) for 3 s. If the indicator remains lit, check the battery connections and, if necessary, replace the batteries.
6.2. For full battery life, operate the UPS at ambient temperatures as close to 25°C (77°F) as possible. Battery life can be reduced if the unit is operated outside this range.
6.3. Dust entering the unit can affect its performance. Keep the surrounding area as dust-free as possible, and vacuum the exterior of the unit periodically if it becomes dusty.

7. Personnel Qualifications

7.1. Personnel should be trained in the use of the UPS before installing or operating it.
7.2. Each individual must read and understand the Model 9125 manual and this SOP.

8. Equipment and Supplies

8.1. Uninterruptible power system (Model, 9125 700-3000 VA, Powerware Corporation, Espoo, Finland)
8.2. EBM: (Powerware Corporation, Espoo, Finland)
8.3. GFCI receptacle tester (to verify outlet polarity)

9. Procedures

9.1. Initial acceptance and installation
- 9.1.1. Inspect the unit and its box to ensure that no damage has occurred during shipping. Chapter 3 of the UPS Manual describes procedures to file a claim for receipt of a damaged unit.
- 9.1.2. Check the required battery recharge date on the label of the shipping carton. If the date has passed without the batteries being recharged, do not use the UPS until new batteries are obtained. Contact the distributor (Section 13.2).
- 9.1.3. Install the Model 9125, and any EBMs, in an instrument rack, following the “Rack-Mount Setup” procedure in the UPS Manual (Pg. 18).
- 9.1.4. If installing an EBM, plug the EBM cable into the UPS battery connector (Fig. 1). If installing more than one EBM, plug the EBM cable of the second (and subsequent) module into the EBM Battery Connector of the previous module (Fig. 1). See the note in Section 4.1.6 concerning arcing which may occur at this point.

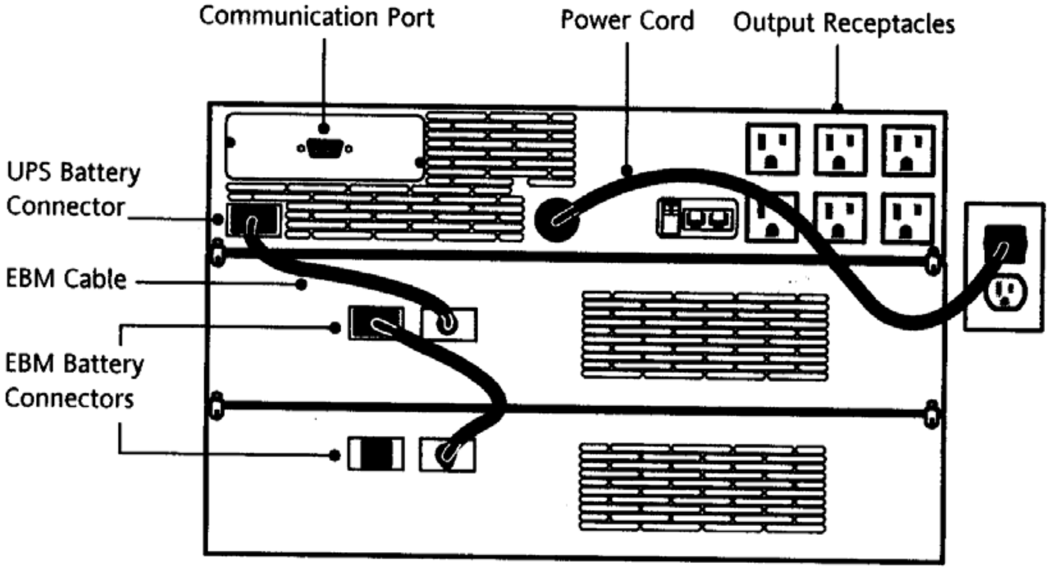

Figure 1. Rear Panel of the Model 9125 and two EBMs, showing connections.

9.2. Operation

9.2.1. Connect UPS to power source. It will conduct a 5-s self-test, and enter Standby mode.

9.2.2. Turn UPS on. The Normal Mode indicator on the front panel (Fig. 2) will come on, and a bar graph indicator will display the connected load (as percentage of capacity).

9.2.3. When the unit is plugged into a working power source (i.e. conditioning power only, not supplying it), it will run in Normal Mode.

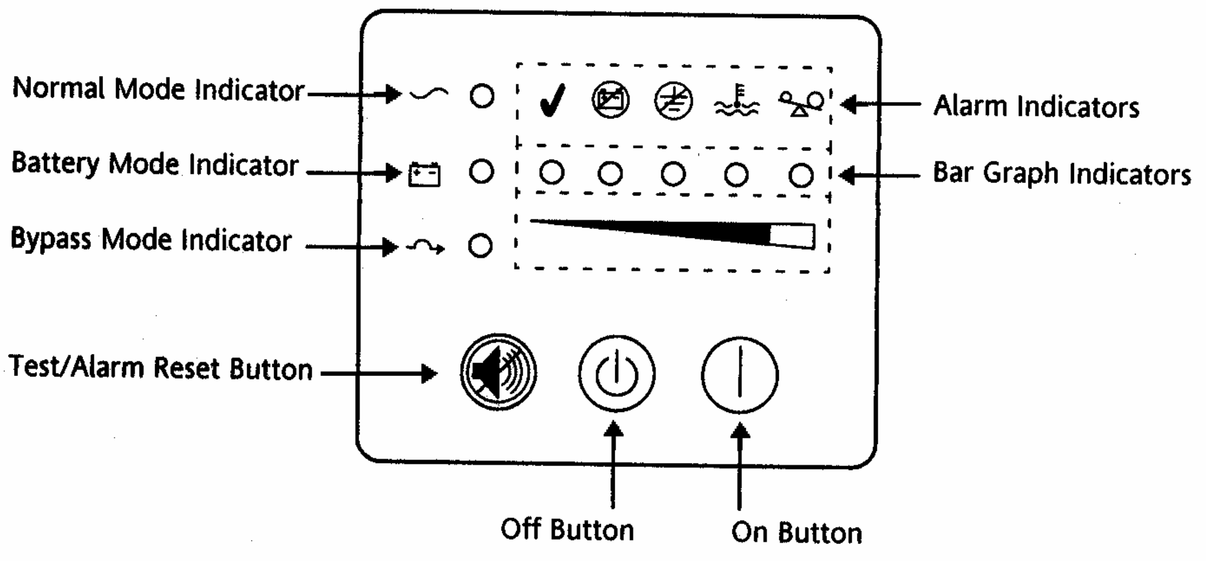

Figure 2. Front panel of the Model 9125, including power buttons and indicator lights.

9.2.4. If power to the Model 9125 is disrupted, it will go into Battery Mode. The Battery Mode indicator will be lit, and an alarm will beep.

9.2.4.1. If power is restored before the batteries become drained, the unit will automatically return to Normal Mode, and begin recharging the batteries.

9.2.4.2. If power is not restored quickly enough, the unit will use up its battery power. The ⊘ indicator will flash, and a continuous alarm will sound, when there is approximately 3 min of battery power remaining. When the check-mark indicator to the left of the low battery indicator begins to flash, a shutdown is imminent. See Section 11.1.1.

9.2.5. The unit will go into Bypass Mode if its temperature is too high, if it detects a fault in its batteries or internal electronics, or if it is overloaded. While in Bypass Mode, the unit continues to condition the utility power source, but the option to switch to Battery Mode in the event of a disruption is not available.

9.3. Configuration

9.3.1. 120 VAC input power is the default setting for the low-voltage version of the Model 9125 (the 700-3000 VA), and should not be changed. If it is changed, reconfigure the unit as follows:

9.3.1.1. Press the On and Test/Alarm Reset buttons simultaneously for one beep. The five Bar Graph indicators will either illuminate (option enabled) or not (option disabled). The proper configuration is as follows:

9.3.1.1.1. First indicator (AC Input Failure Alarm): Illuminated/enabled
9.3.1.1.2. Second indicator (Site Wiring Fault Alarm): Illuminated/enabled
9.3.1.1.3. Third indicator (127 VAC Input): Not illuminated/disabled
9.3.1.1.4. Fourth indicator (110 VAC Input): Not illuminated/disabled
9.3.1.1.5. Fifth indicator (120 VAC Input): Illuminated/enabled

9.3.1.2. If any of these are set incorrectly, use the On button to scroll to the correct indicator. The LEDs immediately above the indicators will light individually to denote which indicator is selected. Once an indicator is selected, if that option is disabled, the indicator will switch from not illuminated to flashing.

9.3.1.2.1. Once the desired indicator is selected, change its status by pressing the Test/Alarm Reset button once.

9.3.2. The unit will automatically exit Configuration Mode if no changes are made for 2 min.

10. Data and Records Management

10.1.1. Record the following in the electronic field notes:
10.1.2. Data pertaining to initial setup (receptacle polarity check, earth leakage current)
10.1.3. Dates and results of all UPS self-tests
10.1.4. Dates and specifics of any changes in configuration
10.1.5. Manage the field notes according to SOP B5.

11. Quality Control and Quality Assurance

11.1. The Model 9125 provides audible alarms for a number of conditions that might affect the unit’s performance. A full list of these alarms is provided in Table 10 (Pgs. 56-57) of the UPS Manual. Included among these are the following:

11.1.1. Warnings indicate when the unit switches to battery power, when the unit has only 3 min of battery power remaining, or when a shutdown is imminent due to low battery power. Under all of these circumstances, switch off all the equipment using the power supplied by UPS. Write down the time and cause of this action in the field notebook, and transfer to the electronic fieldnotes when power is restored.

11.1.1.1. If the total load of all equipment is very close to the maximum of the power circuit, turn off power to most of the equipment, so that there will not be a sudden significant draw when power is restored.

11.1.2. Warnings indicate when the batteries in a plugged-in unit are not charging properly. The last of these can mean that the batteries are not connected properly, or that they need to be replaced. Follow Section 6.1 to run a self-test and address this warning.

11.1.3. Warnings indicate when the unit is overtemperature. Turn off and unplug the unit for at least 5 min to cool, check the airflow around the unit, and verify that there is no dust buildup on the vents. If the alarm persists when the unit is turned back on, contact the distributor.

11.1.4. Warnings indicate when the unit is overloaded. Turn off and unplug the unit for at least 5 min, unplug some of the equipment from the UPS and restart. Determine if a higher-capacity UPS is required. Fig. 3 shows the pattern in which indicator lights will be lit to display the load level of the UPS.

11.2. Even in the absence of alarm messages, perform a self-test (Section 6.1) of the unit on a weekly basis. Record self-tests in the electronic field notes.

11.3. The hot-swapping feature of the batteries allows battery replacement without interrupting power to the equipment that is connected to the UPS, thus preserving the continuity of data.

12. References

12.1. UPS Manual. 2003. Powerware 9125 Two-in-One User’s Guide. Powerware Corporation, Espoo, Finland.

12.2. SOP U1. 2006. On-farm Instrument Shelters for Barn Sources. Standard Operating Procedure U1. Purdue Ag Air Quality Lab.

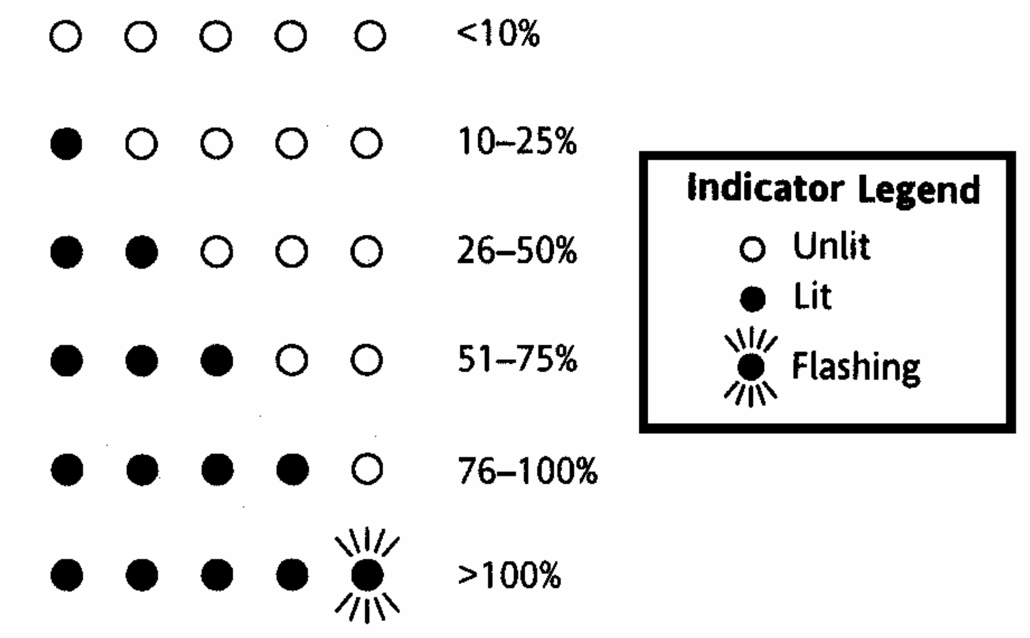

Figure 3. Status of Model 9125 indicator lights in Normal Mode at various loads (% capacity).

13. Contact Information

13.1. Manufacturer:

Powerware Corporation
Koskelontie 13, FIN-02920 Espoo, FINLAND
Phone: +358-9-452 661
Fax: +358-9-452 665 68

13.2. Distributor:

Perfect Power
3405 W. 96th St., Indianapolis, IN 46268
Phone: 317-874-4260
Fax: 219-462-2227

MEASUREMENT OF ATMOSPHERIC TEMPERATURE AND HUMIDITY WITH THE VAISALA MODEL HMP45C SENSOR AND SOLAR SHIELD

Standard Operating Procedure (SOP) W1

MEASUREMENT OF ATMOSPHERIC TEMPERATURE AND HUMIDITY WITH THE VAISALA MODEL HMP45C SENSOR AND SOLAR SHIELD

Standard Operating Procedure (SOP) W1

Prepared by
Richard H. Grant and Jenafer M. Wolf

Reviewed by
Congna Li, Albert J. Heber, and Bill W. Bogan

Effective Date: November 6, 2006
Rev. 1.0 Effective Date: February 23, 2008

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1. Scope and Applicability

1.1. The range of the HMP45C Temperature and Humidity Sensor, in normal atmospheric conditions, is 10 to 100% RH, and -40°C to 60°C temperature.
1.2. The accuracy of the sensor's relative humidity (RH) readings is ±3% RH (for RH from 90% - 100%) or ±2% (for RH from 0% - 90%).
1.3. The accuracy of the sensor's temperature readings is ±0.4°C for temperatures between -20°C and +60°C.
1.4. This procedure applies only to the Vasiala HMP45C temperature and RH probe when it is used, together with a solar radiation shield, to monitor temperature at an outdoor location.
  1.4.1. This SOP is not intended to cover use of the Vaisala Model HMP45C to monitor temperature and RH inside a barn or other building. This is covered in SOP E2.

2. Summary of Method

The HMP45C Temperature and Humidity Sensor contains a platinum resistance temperature detector and a Vasiala HUMICAP® 180 capacitive RH sensor. Capacitance of the HUMICAP® semiconductor increases with increasing humidity. A solar shield is used in conjunction with the sensor to eliminate direct heating of the sensor by sunlight, which could otherwise lead to inaccurately high temperature readings.

3. Definitions

ARM	Atmospheric Radiation Monitoring Network
h_r	Relative humidity ratio
QAPP	Quality Assurance Project Plan
PAAQL	Purdue Agricultural Air Quality Laboratory
PAML	Purdue Applied Meteorology Laboratory
RH	Relative humidity
CS	Campbell Scientific, Inc
NOAA	National Oceanic and Atmospheric Administration
NWS	National Weather Service

4. Cautions

4.1. Do not touch the HUMICAP® RH sensor chip (located within condensate screen) with ungloved fingers.
4.2. The black outer jacket of the cable is Santoprene® rubber that will support combustion in air.
4.3. Be careful when connecting the sensor cable to the datalogger, being sure the connections are tight and made according to the SOP W6.
4.4. The sensors are electrical. Avoid crimping cables and connectors.

5. Interferences

5.1. Hydrogen sulfide levels above 10 ppm interfere with the sensor; however, ambient (outdoor) levels of hydrogen sulfide are not expected to be this high.
5.2. Ammonia concentrations above 100,000 ppm interefere with the sensor; however, ambient (outdoor) concentrations of ammonia are not expected to be this high.
5.3. Buildup of salt on condensate filter or HUMICAP chip
5.4. The sensor must be maintained in the passive radiation shield, as solar radiation will otherwise interfere with the measurement by heating the sensor.

6. Personnel Qualifications

6.1. Each member of the field staff must read and understand the sensor instruction manual and this SOP before operating this equipment.

7. Equipment and Supplies

7.1. #2 common head screwdriver
7.2. 1/8″slotted screwdriver
7.3. Heat shrink tubing
7.4. Heat blower
7.5. Distilled water bottle
7.6. Vacuum glass jar with silica gel dessicant
7.7. Soft toothbrush
7.8. Dish soap (without hand lotion)
7.9. Sling psychrometer
7.10. Data logger for acceptance and calibration
7.11. Still-air temperature calibration chamber
7.12. NIST-traceable certified immersion calibration thermometer
7.13. Ultrasonic cleaner
7.14. Plastic bottle
7.15. Vaisala HMK15 humidity calibrator
  7.15.1. Calibrated Lithium chloride salt packet (traceable to Standards Laboratory of the Centre for Metrology and Accreditation, Finland)
  7.15.2. Calibrated Potassium Sulfate salt packet (traceable to Standards Laboratory of the Centre for Metrology and Accreditation, Finland)
  7.15.3. Calibration thermometer (traceable to Standards Laboratory of the Centre for Metrology and Accreditation, Finland)
7.16. HMP45C probe
7.17. Solar shield
7.18. LI-610 portable dew point generator
7.19. Dew point generator/HMP45C calibration chamber

8. Procedures

8.1. Acceptance
  8.1.1. Attach sensor wires to data logger using the 1/8″slotted screwdriver according to wiring scheme in SOP W6.
    8.1.1.1. Clear wire to ground, Black wire to ground, White wire to signal ground, Yellow wire to single-ended analog input, Blue wire to single-ended analog input, Red wire to switched 12V on data logger.
  8.1.2. Program data logger to read sensor according to program provided in SOP W6, setting sampling time to 5 s and changing averaging time to one minute (from 5 min during normal operation as indicated in SOP W6).
    8.1.2.1. Compile program
  8.1.3. Measure RH and temperature using a sling psychrometer.
    8.1.3.1. The thermometers on the sling psychrometer are NIST-traceable, with an accuracy of $\pm 0.5^{o}$C
    8.1.3.2. The sling psychrometer must have a clean wick to achieve an accuracy of $\pm 0.5^{o}$C, according to NOAA-NWS specifications. The accuracy of the thermometers in the psychrometer is verified by a single point comparison to an immersion thermometer calibrated to a NIST standard.
    8.1.3.3. Shield the psychrometer from direct solar radiation.
    8.1.3.4. Wet the (clean) wick of the sling psychrometer with distilled water and whirl the psychrometer at a rate of about four revolutions per second. Stand clear of obstructions while whirling. Whirl for about one minute, then read the thermometers immediately, the wet bulb first, and record values on acceptance sheet (example attached). Whirl again for another 10 s, read the thermometers again, and record on acceptance sheet. If the readings are unchanged from the first time, this is the measured value. If the readings are different at the second reading, repeat until two successive sets of readings are the same.
    8.1.3.5. Calculate the RH from the dry and wet bulb temperatures according to the chart in Appendix A.
    8.1.3.6. Read average value from data logger and record on acceptance worksheet (Appendix B).
      8.1.3.6.1. If measured RH is greater than 20% different from that derived from standard sling psychrometer measurement, then the sensor is faulty and must be returned to manufacturer.
      8.1.3.6.2. If the measured air temperature differs by more than 1°C from that of the dry bulb temperature of the sling psychrometer, then the sensor is faulty, and must be returned to manufacturer.

8.2. Installation
  8.2.1. The HM45C probe is housed in a radiation shield mounted on the 10 ft. mast according to SOP U5.
  8.2.2. Securely mount sensor into the radiation shield using common head screwdriver.
  8.2.3. Pass cable down tower and through specified weatherproofed NEMA 4X datalogger knockout.

- 8.2.4. Attach wires to datalogger using the1/8″ slotted screwdriver.
  - 8.2.4.1. Clear wire to ground, Black wire to ground, White wire to signal ground, Yellow wire to single-ended analog input, Blue wire to single-ended analog input, Red wire to switched 12V on datalogger, according to SOP W6.

8.3. Preventive maintenance

- 8.3.1. Clean filter yearly.
  - 8.3.1.1. Unscrew the filter cap from the HMP45C. Remove it and place it in a soap solution consisting of dish detergent.
    - 8.3.1.1.1. Put in a plastic bottle. Shake the container gently.
    - 8.3.1.1.2. Once the heavy concentration of dirt has been removed, use a small-diameter blunt punch to press out the inner filter frame from the cap. There is a small hole on top of the filter cap where a punch can be inserted to remove the insert.
    - 8.3.1.1.3. Remove dirt from the outside of the paper filter with a soft toothbrush.
    - 8.3.1.1.4. Remove filter paper from frame and complete cleaning cycle by putting it in ultrasonic cleaner, again using dish soap as the cleaner.
    - 8.3.1.1.5. Rinse thoroughly in clean water.
    - 8.3.1.1.6. Put filter in vacuum glass jar containing silica gel to remove moisture.
    - 8.3.1.1.7. After 1 day, reinstall filter into filter cap and reinstall filter cap on HMP45C.

8.4. Temperature calibration

- 8.4.1. Sensor is single-point calibrated in a temperature-controlled environment.
  - 8.4.1.1. Insert HMP45C and calibration thermometer into a still-air chamber that is in equilibrium with its surroundings.
  - 8.4.1.2. Allow 10 min for equilibration after inserting sensors into still-air chamber
  - 8.4.1.3. Record the mean temperature over the next 5 min from the data logger output of the HMP45C measurement, and the reading from the calibration thermometer on the calibration data sheet (example attached).
  - 8.4.1.4. Calculate difference in temperature reported by calibration thermometer and sensor.
    - 8.4.1.4.1. If the temperature error is >0.5 °C, return sensor to factory for repair.
- 8.4.2. If difference in temperature reported by calibration thermometer (expected) and sensor (measured) is within 2°C, adjust temperature measurements taken since the last calibration to account for the drift, according to Equation 1.

$$T_{corr}(t) = T(t)\left[1-\frac{\overline{T_{measured}}-\overline{T_{expected}}}{\overline{T_{expected}}}\right]\left(\frac{t}{p}\right) \qquad \text{(Equation 1)}$$

Where:

T_{corr} = Back-corrected value at time *t* into the last measurement period
$T(t)$ = Original measured value at time *t*,
$T_{measured}$ = Measured temperature during calibration comparison
$T_{expected}$ = Temperature indicated by the calibration thermometer during calibration comparison
p = Length of time between calibrations.

8.5. RH calibration. Standardized humidities for calibration can be established using either a dew point generator or saturated salt solutions.

8.6. Calibration using LI-610 portable dew point generator:

8.6.1. Construct dew point generator/HMP45C calibration chamber.

8.6.1.1. Cut a piece of 2-inch PVC pipe to a length of 17 cm.

8.6.1.2. Cut a 2-inch diameter disc from a piece of 3/8-inch-thick plastic.

8.6.1.3. Cut a 3/4-inch diameter hole in center of plastic disc to fit sensor probe diameter.

8.6.1.4. Using PVC cement, glue disc onto one end of the 17-cm long PVC pipe and let cement set for at least 15 min.

8.6.1.5. Drill a #27 hole into 2-inch PVC pipe cap and tap hole using a 1/8-27 tapping bit.

8.6.1.6. Using PVC cement, glue and screw cap onto other end of 17-cm long PVC pipe and let cement set for at least 15 min. This hole will be used as an inlet of airflow during calibration.

8.6.1.7. Create air exhaust vents in chamber to be used during calibration. Drill four evenly-spaced holes around PVC piping approximately 1 inch from the end of pipe containing the plastic disc. Be sure holes are smaller than inlet hole.

8.6.1.8. Screw 1/8-27 tubing nipple into tapped hole in PVC piping and connect tubing from the nipple to OUTPUT 1 on LI-610 portable dew point generator.

8.6.2. Calibrate relative humidity probe using LI-610 (Fig. 1).

8.6.2.1. Insert HMP45C probe into the end of the PVC pipe containing the disc.

8.6.2.2. Be sure the distilled water in the LI-610 dew point generator is filled to the proper level (Reference 11.4).

8.6.2.3. Turn the POWER switch ON

8.6.2.4. Attach HMP45C wires to datalogger using 1/8″ slotted screwdriver.

8.6.2.5. Determine the ambient temperature from the datalogger.

8.6.2.6. Set the function switch on the LI-610 to the TEMP SET position.

8.6.2.6.1. Calculate the saturation vapor pressure based on the ambient temperature (T) in degrees Celsius, according to Eq. 2:

$$e_s = 0.61365e^{\frac{17.502T}{240.97+T}} \qquad \text{(Equation 2)}$$

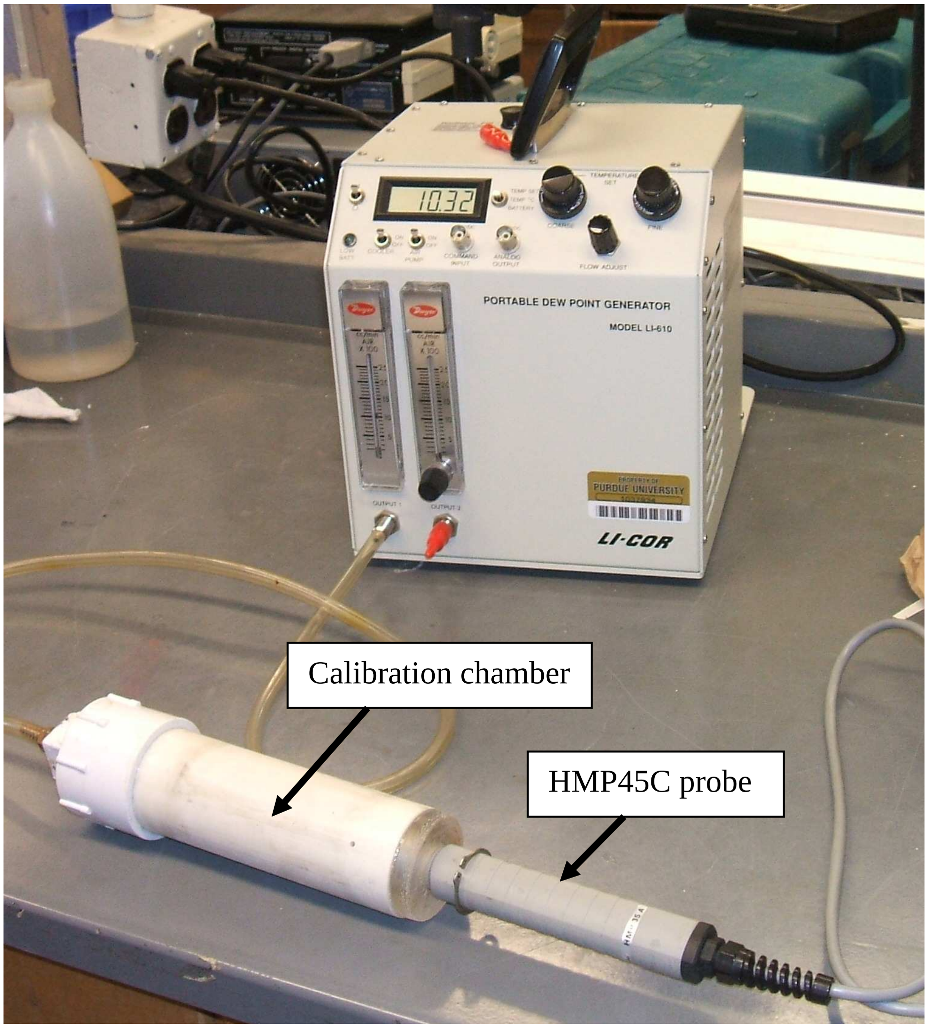

Figure 1. HMP45C Relative Humidity Calibration using LI-610 Dew Point Generator.

8.6.2.6.2. Calculate the set point (dew point) temperature (T_d) using Eq. 3:

$$T_d = h_r \frac{240.97 \ln(e_s / 0.61365)}{17.502 - \ln(e_s / 0.61365)} \qquad \text{(Equation 3)}$$

for humidity h_r of 0.11

8.6.2.6.3. Use the COARSE and FINE TEMP SET knobs to set a desired dew point temperature.

8.6.2.7. Set the function switch to the TEMP ºC position, and turn the COOLER switch ON.

8.6.2.8. Turn the AIR PUMP switch ON

8.6.2.8.1. Turn the knob on the rotameter above OUTPUT 2 clockwise until the flow is shut off and the rotameter shows zero flow.

8.6.2.8.2. Use the FLOW ADJUST knob to control the rate of air flow through the LI-610.

8.6.2.8.2.1. Set flow to 500 cm^3/min. This allows for 5 exchanges through the chamber in 3 min.

8.6.2.8.2.2. Watch for the display to show the desired dew point temperature.

8.6.2.8.2.3. Wait approximately 1 hour for HMP45C to stabilize in the calibration chamber.

8.6.2.8.3. Record temperature, measured humidity and applicable calibration table RH on the calibration data sheet (Appendix C).

8.6.2.9. Repeat sections 8.6.2.6- 8.6.2.7 for h_r= 0.50 and 0.98.

8.7. Saturated salt solution calibration

8.7.1. Conduct calibration in a temperature-controlled environment.

8.7.2. Calibration conducted in HMK 15 calibrator chambers.

8.7.3. Prepare solutions of lithium chloride (LiCl), sodium chloride (NaCl), and potassium sulfate (K_2SO_4) in three chambers of the HMK15 calibrator according to HMK15 manual.

8.7.4. Leave the HMK15 calibrator and the probe at the calibration site for at least 30 min before starting the calibration in order to let the probe temperature stabilize to the room temperature.

8.7.5. Take off the filter protecting the HMP45C sensor. Take care not to damage the sensor.

8.7.6. Low humidity calibration

8.7.6.1. Insert the HMK15 calibration thermometer into the 13.5-mm hole of the LiCl salt chamber (low RH reference). Press it downwards until it passes the O-rings.

8.7.6.2. Insert probe into the 13.5-mm hole of the LiCl salt chamber. Press it downwards until it passes the O-rings. The shorter the time the hole stays open before inserting the probe, the shorter the stabilization time required.

8.7.6.3. Wait 10-30 min for RH stabilization.

8.7.6.4. Read the closest humidity value from the calibration Table 1, given the temperature of the thermometer.

8.7.6.4.1. Record temperature, measured humidity and applicable calibration table RH on the calibration data sheet (example in Appendix C).

8.7.6.4.2. Compare measured RH with Greenspan's value in Table 1.

8.7.6.4.2.1. If measured RH is within expected annual drift (+/-3% RH), sensor is good. Adjust the DRY potentiometer (Fig. 2) to correspond to value given in the calibration table.

8.7.6.4.2.2. If measured RH is outside expected annual drift (+/-3% RH), sensor is defective and must be sent to factory for repair or replacement.

8.7.7. High humidity calibration

8.7.7.1. Insert the HMK15 calibration thermometer into the 13.5-mm hole of the K_2SO_4 salt chamber (high RH reference). Press it downwards until it passes the O-rings.

8.7.7.2. Insert the probe into a suitable hole of the K_2SO_4 salt chamber. Press it downwards until it passes the O-rings. The shorter the time the hole stays open before inserting the probe, the shorter the stabilization time required.

8.7.7.3. Wait 20-40 min for RH stabilization.

8.7.7.4. Read the closest humidity value from the calibration table for the chamber temperature (Table 1).

8.7.7.4.1. Record temperature, measured RH and applicable calibration table RH on the calibration data sheet (Appendix C).

8.7.7.4.2. Compare measured RH with Greenspan’s value in Table 1.

8.7.7.4.2.1. If measured RH is within expected annual drift (+/-10% RH), sensor is good. Adjust the WET potentiometer (Fig. 2) to correspond to the value given in the calibration table.

8.7.7.4.2.2. If measured RH is outside expected annual drift (+/-10% RH), sensor is defective and must be sent to factory for repair or replacement.

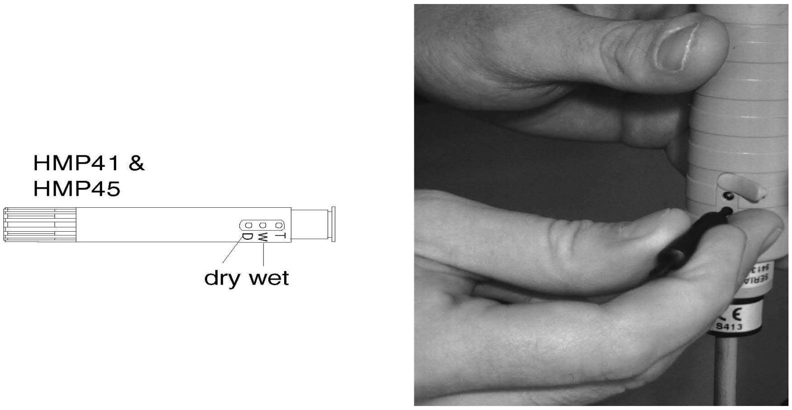

Figure 2. Potentiometer adjustment location on the HMP45C.

8.8. Troubleshooting

8.8.1. Lack of response of either temperature of RH to variation in ambient conditions can be due to the following:

8.8.1.1. Poor connections of the sensor at the datalogger: Check electrical connections (especially excitation voltage wire) according to SOP W6.

8.8.1.2. Damage to plastic casing of the signal cable: If damaged slightly, repair with heat-shrink tubing. If damaged significantly, replace sensor and return sensor to manufacturer for repair.

8.8.2. Lack of at least 95% RH at saturation conditions (fog and light rain) is possibly due to contamination of the condensation filter or HUMICAP. Check filter and HUMICAP and clean or replace as needed.

8.9. Data acquisition, calculations & data reduction.
  8.9.1. RH output (Voltages 0.008 to 1 VDC) is monitored by the datalogger.
  8.9.2. Temperature output (Voltages 0.008 to 1 VDC) is monitored by the datalogger.
  8.9.3. Data are reported to three significant digits.
8.10. Computer hardware & software
  8.10.1. The sensors will be attached through electrical wires to the datalogger that will extract data from the instrument (Section 9.1.4).
  8.10.2. The datalogger will record the data and store it.
  8.10.3. Datalogger operates using on-board BASIC-like programming.
    8.10.3.1. General program for measurements stated in Model HMP45C Temperature and Humidity Probe Instruction Manual.

9. Data and Records Management

9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for meteorological sensors.
9.2. Manage all data according to SOP D1.
9.3. At the end of this SOP is a form to record the calibration information.
9.4. Document all data and information on field data sheets and in site logbooks with permanent ink, or in electronic notes.
9.5. Date, initial, and overstrike errors in writing with a single line.

10. Quality Control and Quality Assurance

10.1. Annual drift of RH measurement increases with increased temperature and RH.
  10.1.1. Annual drift of RH measurement of 6% to 10% for 100% humidity and 35 to 45 °C and up to 4% for 100% humidity and 10 to 25 °C, as reported in personal communication from the NOAA High Plains Climate Center.
  10.1.2. Annual drift of RH measurement of less than 3% at 10% humidity for temperatures from 10 to 45 °C as reported in personal communication from the NOAA High Plains Climate Center.
10.2. Calibration
  10.2.1. RH sensor is calibrated annually using either a Vaisala HMK15 Humidity calibrator with salt solutions of lithium chloride (LiCl) and potassium sulfate (K_2SO_4) or a LiCOR portable dew point generator.
  10.2.2. Temperature sensor is calibrated annually
10.3. Reasonableness of data values (daily check)
  10.3.1. Relative humidity
    10.3.1.1. If a data value is missing, flag.it as invalid.
    10.3.1.2. If a data value is less than -2% or greater than 104% (range of the sensor calibration and uncertainty), flag it as invalid.
    10.3.1.3. If the standard deviation of the data over one hour is greater than 20%, flag the data as invalid.

10.3.1.4. If a data value differs by more than 10% from the temperature-corrected RH from the nearest National Weather Service (NWS) weather station, flag it as invalid.

10.3.1.4.1. Calculate humidity mixing ratio at NWS weather station (W) using station barometric pressure and RH according to Appendix D.

10.3.1.4.2. Calculate saturation humidity mixing ratio (Ws) for measurement location using location barometric pressure and air temperature setting RH at 100% (1.0) according to Appendix D.

10.3.1.4.3. Calculate the RH at measurement location as W/Ws*100.

10.3.1.5. If data is flagged for any of the above reasons, troubleshoot sensor according to Section 8.6.

10.3.2. Temperature

10.3.2.1. If a data value is missing, flag.it as invalid..

10.3.2.2. If a data value is less than -40 °C or greater than +50 °C (bounds of sensor calibration), flag it as invalid.

10.3.2.3. If the standard deviation of the data over an hour is <2 °C, flag the data as invalid.

10.3.2.4. If data values change more than 5 °C (9 °F) from the previous hour, flag as invalid.

10.3.2.5. If data values vary by less than 0.5 °C (0.9 °F) for 12 consecutive hours, flag as invalid.

10.3.2.6. If data value is greater than the local record high or is less than the local record low on a monthly average, flag as invalid.

10.3.2.7. If data value differs by more than 3 °C from the altitude-corrected air temperature from the nearest National Weather Service (NWS) weather station, flag as invalid.

10.3.2.7.1. Calculate altitude-adjusted air temperature at NWS weather station (Tadj) based on the station air temperature according to:

$$T_{adj}=T_{NWS}+(Z_{NWS}-Z_{site})*0.0098$$

Where:
T_{adj} and T_{NWS} are in units of ^{o}C
Z_{NWS} and Z_{site} are the altitudes of the NWS station and site (in m)

10.3.2.8. If data is flagged for any of the above reasons, troubleshoot sensor according to Section 8.6

10.4. Physical inspection

10.4.1. Integrity of the plastic casing of the signal cable will be checked.

10.4.1.1. If damaged slightly, repair with heat-shrink tubing.

10.4.1.2. If damaged significantly, replace sensor and return sensor to manufacturer for repair.

10.4.2. Check for debris in solar radiation shield. Remove if present.

10.4.3. Check screen at sensor end for dirt. If dirty, clean screen according to Section 9.2.

10.4.4. Check HUMICAP® sensor chip for contaminants.

10.4.4.1. If dirt or crystals are present, remove chip with latex-gloved fingers and rinse with distilled water. Do not scratch while cleaning. Let air dry before re-assembling.

10.5. Calibrate sensor in laboratory according to procedures outlined in Sections 8.6 - 8.7.

11. References

11.1. ARM. 2004. Atmospheric Radiation Monitoring Network: Standard Operating Procedures
11.2. Campbell Scientific Inc. 2004. Model HMP45C Temperature and Humidity Probe Instruction Manual.
11.3. K. Hubbard. 2005. Personal communication. High Plains Climate Center, NOAA, December 30.
11.4. LI-COR Inc. 1991. LI-610 Portable Dew Point Generator Instruction Manual, 3-3.
11.5. Vaisala Inc. 2002. Humidity Calibrator HMK15 User's Guide, M210185en-B.
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11.7. NOAA High Plains Climate Center. undated. Manual for the Automated Weather Data Network Maintenance and Calibration. High Plains Climate Center, Lincoln, NE.
11.8. USEPA. 1989. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV- Meteorological Measurements.
11.9. USEPA. 2000. Meteorological Monitoring Guidance for Regulatory Modeling Applications, EPA-454/R-99-005.
11.10. NCDAQ. 1994. General Guidelines for On-Site Meteorological Data Collection for N.C. Air Quality Analysis.
11.11. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.
11.12. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.
11.13. SOP D1. 2006. Management of Open Source, Weather and Lagoon-Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.

12. Contact Information

12.1. HMP45C Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, Utah, 84321
  12.1.1. Technical support: 435-753-2342.
12.2. HMK15 Distributor: Vaisala Sales: 888-824-7252 x1053, email: Barry Eisen, b.eisan@vaisala.com
  12.2.1. Technical support: North American Service Center, Boston, MA, USA
  Phone: 888 VAISALA or 781 933 4500, Fax: 781 933 8029

Table 1. Calibration table[1]

°C	LiCl	MgCl2	NaCl	K_2SO_4
0	11.3 ±0.5	33.7 ±0.3	75 ±0.3	98.8 ±1.1
5	11.3 ±0.5	33.6 ±0.3	75 ±0.3	98.5 ±0.9
10	11.3 ±0.4	33.5 ±0.2	75 ±0.2	98.2 ±0.8
15	11.3 ±0.4	33.3 ±0.2	75 ±0.2	97.9 ±0.6
20	11.3 ±0.3	33.1 ±0.2	75 ±0.1	97.6 ±0.5
25	11.3 ±0.3	32.8 ±0.2	75 ±0.1	97.3 ±0.5
30	11.3 ±0.2	32.4 ±0.1	75 ±0.1	97.0 ±0.4
35	11.3 ±0.2	32.1 ±0.1	74 ±0.1	96.7 ±0.4
40	11.2 ±0.2	31.6 ±0.1	74 ±0.1	96.4 ±0.4
45	11.2 ±0.2	31.1 ±0.1	74 ±0.2	96.1 ±0.4
50	11.1 ±0.2	30.5 ±0.1	74 ±0.2	95.8 ±0.5

[1] Greenspan, L.: Journal of Research of the National Bureau of Standards -A Physics and Chemistry Vol. 81A, No. 1 January-February 1977, pp. 89-95

Appendix A - Dry Bulb/Wet Bulb - Relative Humidity Conversion Chart

This table gives the approximate relative humidity in percent directly from the reading of the air temperature (dry bulb) (t °C) and the wet bulb (T °C). **Condensed from Bulletin of the U.S. Weather Bureau No. 1071**

Dry bulb Temp.- Wet Bulb Temp.

Dry Bulb	2.0	2.2	2.4	2.6	2.8	3.0	3.2	3.4	3.6	3.8	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0	10.5	11.0
5	72	69	67	64	61	58	56	53	51	48	45	39	33	26	20	13	7	..	..	..	..	..	..	..	..
6	73	70	68	65	63	60	58	55	53	50	48	41	35	29	24	17	11	5	..	..	..	..	..	..	..
7	74	72	69	67	64	62	59	57	54	52	50	44	38	32	26	21	15	10	..	..	..	..	..	..	..
8	75	73	70	68	65	63	61	58	56	54	51	46	40	35	29	24	19	14	8	..	..	..	..	..	..
9	76	74	71	69	67	64	62	60	58	55	53	48	42	37	32	27	22	17	12	7	..	..	..	..	..
10	77	74	72	70	68	66	63	61	59	57	55	50	44	39	34	29	24	20	15	10	6	..	..	..	..
11	78	75	73	71	69	67	65	62	60	58	56	51	46	41	36	32	27	22	18	13	9	5	..	..	..
12	78	76	74	72	70	68	66	64	62	60	58	53	48	43	39	34	29	25	21	16	12	8	..	..	..
13	79	77	75	73	71	69	67	65	63	61	59	54	50	45	41	36	32	28	23	19	15	11	7	..	..
14	79	78	76	74	72	70	68	66	64	62	60	56	51	47	42	38	34	30	26	22	18	14	10	6	..
15	80	78	76	74	73	71	69	67	65	63	61	57	53	48	44	40	36	32	27	24	20	16	13	9	6

Dry bulb Temp.- Wet Bulb Temp.

Dry Bulb	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0	10.5	11.0	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	16.0	17.0	18.0
16	81	76	71	67	63	58	54	50	46	42	38	34	30	26	23	19	15	12	8	5	..	..	..	..	..	..	..	..	..	
17	81	76	72	68	64	60	55	51	47	43	40	36	32	28	25	21	18	14	11	8	..	..	..	..	..	..	..	..	..	
18	82	77	73	69	65	61	57	53	49	45	41	38	34	30	27	23	20	17	14	10	7	..	..	..	..	..	..	..	..	
19	82	78	74	70	65	62	58	54	50	46	43	39	36	32	29	26	22	19	16	13	10	7	..	..	..	..	..	..	..	..
20	83	78	74	70	66	63	59	55	51	48	44	41	37	34	31	28	24	21	18	15	12	9	6	..	..	..	..	..	..	..
21	83	79	75	71	67	64	60	56	53	49	46	42	39	36	32	29	26	23	20	17	14	12	9	6	..	..	..	..	..	..
22	83	80	76	72	68	64	61	57	54	50	47	44	40	37	34	31	28	25	22	19	17	14	11	8	6	..	..	..	..	..
23	84	80	76	72	69	65	62	58	55	52	48	45	42	39	36	33	30	27	24	21	19	16	13	11	8	6	.	..	..	..
24	84	80	77	73	69	66	62	59	56	53	49	46	43	40	37	34	31	29	26	23	20	18	15	13	10	8	5	..	..	..
25	84	81	77	74	70	67	63	60	57	54	50	47	44	41	39	36	33	30	28	25	22	20	17	15	12	10	8	..	..	..
26	85	81	78	74	71	67	64	61	58	54	51	49	46	43	40	37	34	32	29	26	24	21	19	17	14	12	10	5	..	..
27	85	82	78	75	71	68	65	62	58	56	52	50	47	44	41	38	36	33	31	28	26	23	21	18	16	14	12	7	..	..
28	85	82	78	75	72	69	65	62	59	56	53	51	48	45	42	40	37	34	32	29	27	25	22	20	18	16	13	9	5	..
29	86	82	79	76	72	69	66	63	60	57	54	52	49	46	43	41	38	36	33	31	28	26	24	22	19	17	15	11	7	..
30	86	83	79	76	73	70	67	64	61	58	55	52	50	47	44	42	39	37	35	32	30	28	25	23	21	19	17	13	9	5

Appendix B
HMP45C Acceptance Worksheet

Serial Number ________________

Evaluator : _____________________________________

Date (DD/MM/YYYY):___________________________

	Psychrometer					**HMP35C**		**Action**
Time (HH:MM:SS)	**Temp. (°C)**				**Calculated RH (%)**	**Measured RH (%)**	**RH difference (%)**	
	Dry Bulb	**Wet bulb #1**	**Wet bulb #2**	**Wet bulb Final**				

Appendix C : Calibration Record (RH): Vaisala HMP45C Sensor

Instrument Serial Number:_________________________________ Location:__________________________________

Date (D-M-Y) Time: Begin/End	Temperature (^{o}C)	% RH		Action Taken (Adjust, Replace)	Analyst
		Expected (Table)	Measured		

Calibration Record (Temperature): Vaisala HMP45C Sensor

Instrument Serial Number:________________________________ Location:_________________________________

Date (D-M-Y) Time: Begin-End	Temperature (^oC)			Action Taken (Adjust, Replace)	Analyst
	Calibration	Measured	Difference		

Appendix D
Conversion between relative humidity and humidity mixing ratio

Conversion of relative humidity (φ) to humidity mixing ratio (W) is calculated as:

$$W = 0.62198\frac{e^{f(T)}\varphi}{P_a - e^{f(T)}\varphi} \qquad \text{(Equation A.4)}$$

Where:

W	Humidity mixing ratio, kg/kg
P_a	Pressure at the sampling location
φ	Relative humidity, decimal

$$f(T) = \frac{C_1}{T} + C_2 + C_3 T + C_4 T^2 + C_5 T^3 + C_6 T^4 + C_7 \ln T \qquad \text{for } T < 273.15 \text{ K}$$

$$f(T) = \frac{C_8}{T} + C_9 + C_{10} T + C_{11} T^2 + C_{12} T^3 + C_{13} \ln T \qquad \text{for } T > 273.16 \text{ K}$$

Where
T= Air temperature (K)
C_1= -5.675x10^{3}
C_2= 6.392
C_3= -9.678x10^{-3}
C_4= 6.222x10^{-7}
C_5= 2.075x10^{-9}
C_6= -9.484x10^{-13}
C_7= 4.163
C_8= -5.800x10^{3}
C_9= 1.391
C_{10}= -4.864x10^{-2}
C_{11}= 4.176x10^{-5}
C_{12}= -1.445x10^{-8}
C_{13}= 6.545.

MEASUREMENT OF WIND WITH THE RM YOUNG MODEL 81000 3-DIMENSIONAL SONIC ANEMOMETER

Standard Operating Procedure (SOP) W2

MEASUREMENT OF WIND WITH THE RM YOUNG MODEL 81000 3-DIMENSIONAL SONIC ANEMOMETER

Standard Operating Procedure (SOP) W2

Prepared by

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Reviewed by

Albert J. Heber and Bill W. Bogan

Effective Date: November 6, 2006
Rev. 1.0 Effective Date: February 23, 2008

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)

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1. Scope and Applicability

1.1. The measurement of the directional components of wind is used in the gaseous emissions measurement methodologies.

1.1.1. Statistical values of instantaneous wind components and the virtual temperature are used in the backward Lagrangian Stochastic method of emission measurement (SOP O1).

1.1.2. Statistical values of instantaneous horizontal wind components are used in the radial plume method of emission measurement (SOP O2).

1.2. The difference in time required for sound to travel through the moving air is a fast response method of measuring each directional component of the wind.

1.3. The detection limit of the method described in this SOP is 0.05 m/s.

1.4. The range of wind speeds which can be measured under this SOP is 0.05 to 40 m/s.

1.5. For wind speed, the manufacturer-reported sensor accuracy is 0.02 m/s, while the manufacturer-reported sensor precision (rms) is 0.05 m/s for speeds of 0 to 5 m/s, 1% of reading for speeds from 5 to 30 m/s and 3% of reading for speeds from 30 to 40 m/s.

1.6. This procedure applies only to the RM Young Model 81000 Ultrasonic Anemometer.

2. Summary of Method

The RM Young Model 81000 measures three-dimensional wind velocity and speed of sound based on the transit time of ultrasonic acoustic signals at 4Hz to 32 Hz. Sonic temperature is derived from speed of sound which is corrected for crosswind effects. Measurement data are available as serial output using RS-232 or RS-485 connections. Operating parameters may be edited via simplified menus using an ordinary serial communication program like HyperTerminal ®. All parameters are stored in non-volatile memory. Superior environmental resistance is achieved by using UV stabilized thermoplastic, stainless steel, and anodized aluminum components.

3. Definitions

COM port	A serial communications port of a computer
HyperTerminal®	A Windows® program that provides communication through a serial communications port of a computer
PAAQL	Purdue Agricultural Air Quality Laboratory
PAML	Purdue Applied Meteorology Laboratory
QAPP	Quality Assurance Project Plan
RS-232	A serial communications protocol
RS-485	A serial communications protocol

4. Health and Safety

4.1. The sensor has electrical components, and proper care should be used as with all electrical equipment.

5. Cautions

5.1. The sensor is acoustical and therefore cannot be dropped without damage.
5.2. Be sure that the sensor is properly grounded.
5.3. Tilt of sensor induces an error in the measurement of approximately 6% in the correlations of wind deviations and 3% in the derived friction velocity with 1^o tilt.

6. Interferences

6.1. Wakes from obstructions such as buildings, storage piles, trees and other vegetation which are closer than ten times the sensor height.
  6.1.1. Incorrect siting of the anemometer will cause greater uncertainties in the data due to failure of assumptions of homogeneity and stationarity.
6.2. Sensor response under high winds can result in extreme values.
6.3. The sensor does not respond under conditions of accumulated freezing rain, heavy frost and snow on the transducers.
6.4. Wake flow around support struts reduces sensor performance.

7. Personnel Qualifications

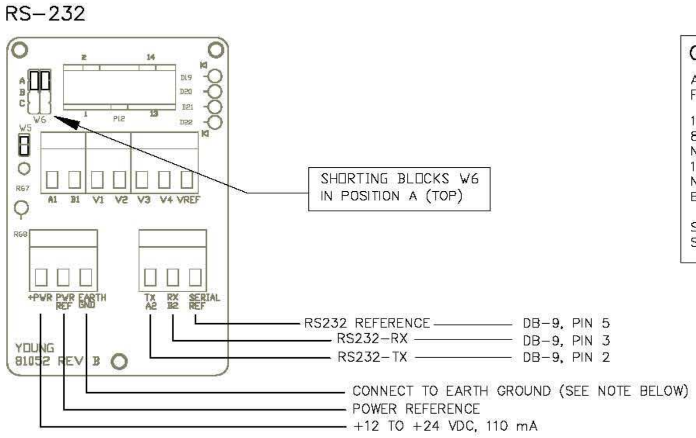
7.1. Read and understand the sensor instruction manual and this SOP before operating this sensor.

8. Equipment and Supplies

8.1. 3-D sonic anemometer
8.2. #2 common head screwdriver
8.3. 1/8″ slotted screwdriver
8.4. Anechoic box
8.5. Laptop computer
8.6. DB9 modem cable
8.7. Labview software
8.8. XStream® RS-232/485 RF Modem
8.9. Sonic anemometer intercomparison stand

9. Procedures

9.1. Instrument acceptance
  9.1.1. Carefully unpack the unit and inspect for physical damage. Report any damage to the shipper. The sensor arrives fully calibrated and ready to use.
  9.1.2. Remove junction box cover. Connect power and signal wires to terminals as in the wiring diagram below. Connect serial cable to computer COM port.
    9.1.2.1. Supply power must be in the range of 12 to 24 VDC **at the sensor junction box terminals** in order for the sensor to operate properly.



GENERAL NOTES

ALL SERIAL COMMUNICATIONS USE THE FOLLOWING SETTINGS:

1 START
8 DATA
NO PARITY
1 STOP
NO FLOW CONTROL
BAUD RATE SET BY USER

SHORTING BLOCKS ON W6 MUST BE IN POSITION SHOWN.

9.1.3. Start HyperTerminal® on the computer with baud rate at 38400 and flow control set to NONE.

9.1.4. Apply power to the 81000 sensor. There will be a 4-s delay for initialization; then, the unit will begin to output data at 4 Hz using the following format: speed (m/s), azimuth (deg), elevation (deg), speed-of-sound (m/s), and sonic-temperature (°C). Verify that all values are present on the display. Typical output is:

```
0.00    0.0    0.0  346.70    25.14
0.00    0.0    0.0  346.68    25.11
0.00    0.0    0.0  346.76    25.25
0.00    0.0    0.0  346.80    25.30
0.00    0.0    0.0  346.82    25.35
```

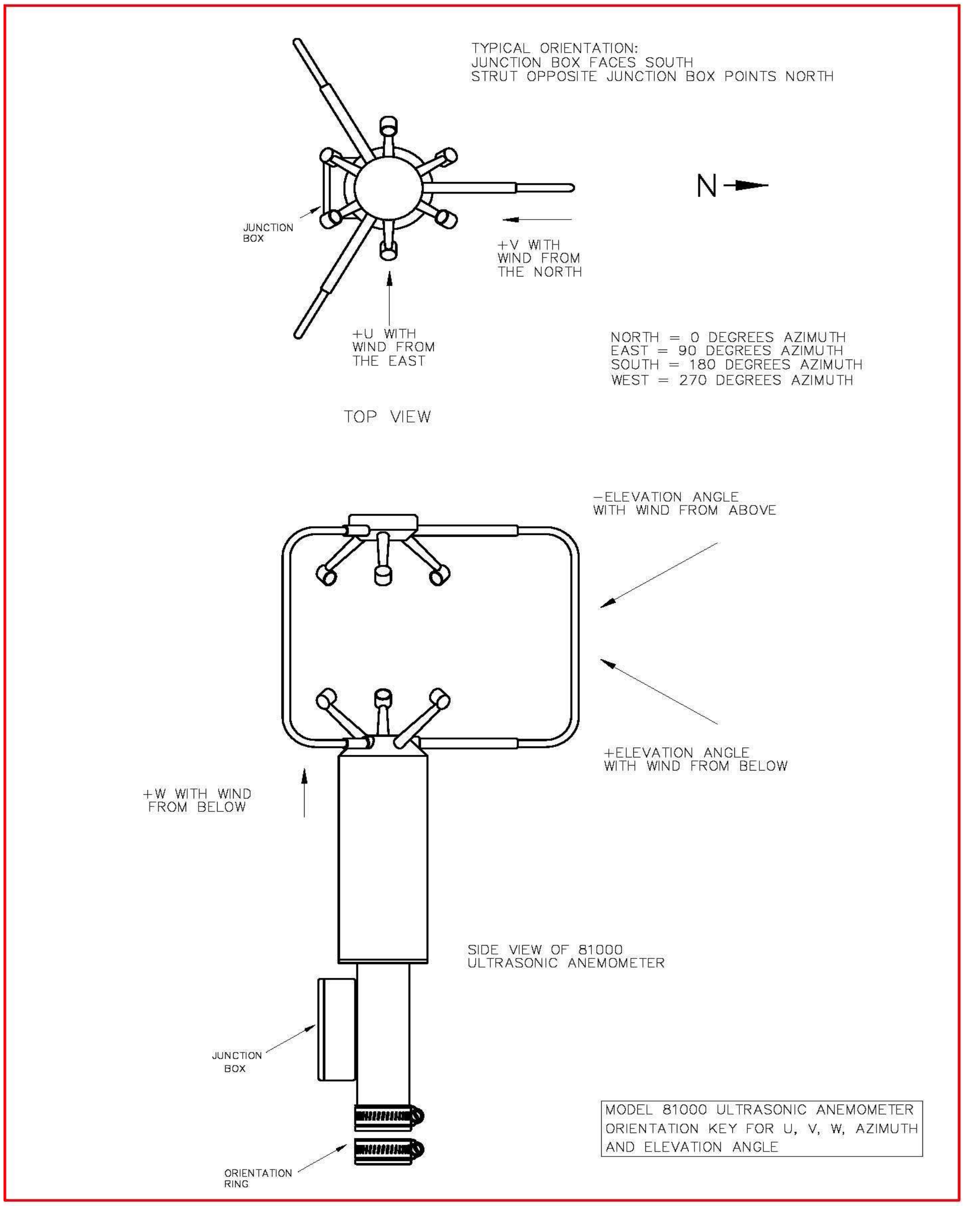
Wind below the 0.05 m/s detection limit is output as 0.00 m/s. Azimuth may be any value from 0.0 to 359.9 degrees. When wind speed is below threshold, azimuth output is maintained at the last value read before wind speed went below threshold. Elevation remains zero until threshold is exceeded. Speed of sound ranges from 300 m/s to 360 m/s, depending on temperature. At 20°C, the value is ~ 344 m/s. Sonic temperature may be compared to a standard Celsius thermometer and should agree within ±2°C. If values appear questionable or any value is not displayed, remove power and check all wiring connections. If the problem cannot be corrected, contact service support (Section 13.1).

9.1.5. Verify sensor response by gently blowing through the measuring section. Wind from the north side (marked “N”), should produce a positive SPEED response and an AZIMUTH display corresponding to North (i.e.: values around 359.9 or 0.0). Wind from the opposite direction should produce AZIMUTH values indicating south, (around 180) and so forth. Downward wind produces negative ELEVATION values, upward wind produces positive values.

9.2. Instrument installation

9.2.1. Mount the sensor securely on top of a mast or tower such that it will not twist, rotate, or sway, according to SOP U5.

9.2.2. Mount the sensor with the following orientation:



9.2.3. Attach earth ground for proper operation.

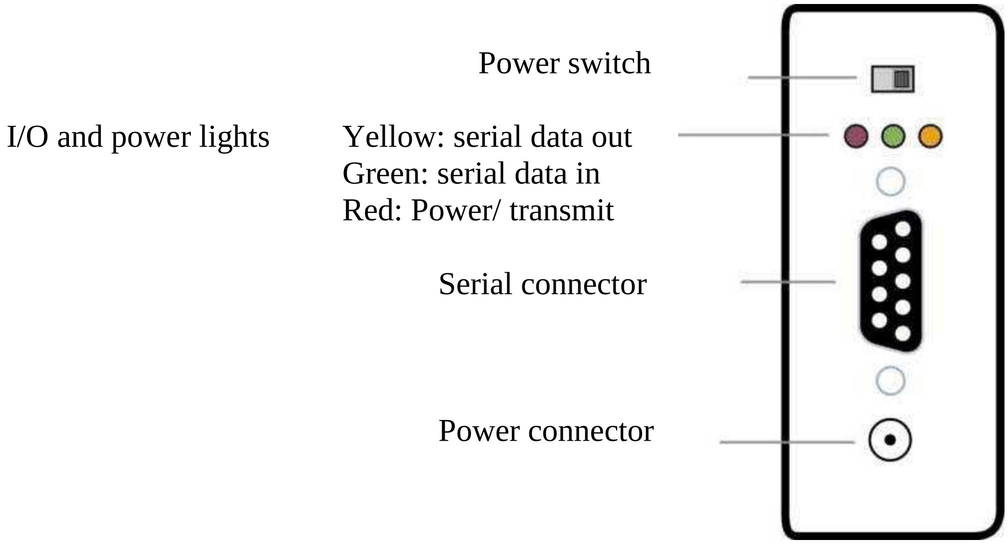
9.3. Instrument operation

9.3.1. Connect a serial cable from the XStream RS-232/485 RF Modem to the COM port of a laptop computer.

9.3.2. Connect antenna to RF Modem.

9.3.3. Provide power to radio modem by connecting power cable to power connector and switching on power switch (See figure below)

9.3.4. Verify power to radio modem by checking red light on modem indicated below:



9.3.4.1. Using X-CTU® program on the laptop computer, verify configuration of Modem (8 data bits, 1 stop bit, no parity, 19200 baud), in accordance with SOP U5.

9.3.5. Connect the serial cable from XStream RS-232/485 RF Modem serial output connector (See figure in Section 9.3.5) to the anemometer, according to figure in Section 9.1.2.

9.3.5.1. Test communication link to the computer in the trailer.

9.3.6. Establish communications with the sensor, according to Sections 9.1.3 and 9.1.4.

9.3.7. Provide power to sensor, and wait 4-s for initialization.

9.3.8. Using the HyperTerminal® window, send the ESC character (ASCII 27) three times in quick succession. This takes the unit out of OPERATE mode and causes the COMMAND MENU to appear:

```
COMMANDS (VERSION 6.6.07)
 -------------------------
R) REPORT
S) SETUP
X) EXIT TO OPERATE MODE
```

9.3.9. Go to SETUP. The SETUP menu appears as follows:

```
SET PARAMETERS
 -------------------------
 A) AVERAGING
 B) BAUD
 E) ERROR HANDLING
 N) SCALING MULT
 O) OUTPUT RATE
 P) POLL CHARACTER (ADDR)
 S) SER OUT FORMAT
 T) THRESHOLD
 U) UNITS
 V) V OUT FORMAT
W) WAKE CORRECTION
 X) EXIT TO MAIN MENU
```

9.3.9.1. Set AVERAGING at 0.
9.3.9.2. Set ERROR HANDLING to “`INCLUDE INVALID DATA`”.
9.3.9.3. Set OUTPUT RATE at 16-Hz
9.3.9.4. Set SER OUT FORMAT as “CUSTOM”. The following appears:

```
CURRENT SERIAL OUTPUT FORMAT:
[       ]
CONSTRUCT AN OUTPUT FORMAT BY SELECTING FROM THE LIST BELOW.
ELEMENTS MAY BE IN ANY ORDER. REFER TO MANUAL FOR DETAILS.
5) UVW
6) 2D SPEED
7) 3D SPEED
8) AZIMUTH
9) ELEVATION
A) SOS
B) Ts
E) ERR CODE
V) INTERNAL VOLTAGE


ENTER CUSTOM STRING (12 CHARACTERS MAX):
```

9.3.9.4.1. Enter Custom string as “5BE”.
9.3.9.5. Set WAKE CORRECTION to “YES”.
9.3.9.6. Exit to main menu and return to OPERATE mode.
9.3.9.7. Locate the appropriate input serial channel on the computer in trailer (SOP U4) and evaluate output for reasonable values.
9.3.9.8. Check output of sensors on the instrument trailer computer.
9.3.9.8.1. If output is not providing three wind vectors, a temperature and an error code, in accordance with the manual, re-enter setup menu and correct problem, starting anew at Section 9.3.5.

9.4. Instrument calibration

- 9.4.1. The sensor response under constant wind is calibrated at the factory.
- 9.4.2. The sensor cannot be calibrated for turbulence components.
- 9.4.3. <u>Standard intercomparison</u>: Compare the output of all three in-use sensors with that of three unused sensors (Standard Triad). This is done after every 6 mos.
  - 9.4.3.1. Locate the sensors to minimize wind interference given prevailing winds.
  - 9.4.3.2. Conduct measurements with the sensors for a period of 24-48 h, so that a variety of atmospheric conditions are included. Then, reverse the locations of the sensors and sample for another 24-48 h.
  - 9.4.3.3. Compare the measurements obtained from the three sensors of the Standard Triad to ensure that the mean wind speed agrees to within 0.2 m/s. If the sensors agree within this amount, then use the average of all 3 sensors of the Triad as the standard. If one of the sensors lies outside this range, then use the average of the two remaining sensors as the standard.
    - 9.4.3.3.1. Troubleshoot any Standard sensor that is out of agreement with other sensors, and resolve problem (Section 9.5).
  - 9.4.3.4. Compare the mean wind speed from each used sensor with the standard set obtained above. If the mean difference is less than 0.2 m/s, then the used sensor being compared is in good working order. If the mean difference is greater than 0.2 m/s, then the used sensor being compared is defective
    - 9.4.3.4.1. Troubleshoot defective sensor and resolve problem (Section 9.5).
- 9.4.4. <u>In-field intercomparison</u>: Compare the sensor with two identical units which are in routine use.
  - 9.4.4.1. Locate all sensors next to each other on the sonic anemometer intercomparison stand. Orient the stand so that it is perpendicular to the wind, to minimize interference among the instruments.
  - 9.4.4.2. Conduct measurements with all sensors for a one-hour period. Record the mean wind speeds for the three sensors over this period on the Field Intercomparison Form (Appendix B).
  - 9.4.4.3. Determine the three paired differences between the sensors' measurements.
    - 9.4.4.3.1. If all paired differences are less than 0.2 m/s, then the sensors are in good working order.
    - 9.4.4.3.2. If one of the sensor's measurement differs by more than 0.2 m/s from the other sensors' measurements, then conduct tests for another one-hour period. If the difference remains greater than 0.2 m/s, then the sensor is defective, and must be returned to the factory for repair or replacement. Otherwise, if the difference in the second hour is less than 0.2 m/s, then the differences in the first hour were likely due to unsteady wind conditions.
      - 9.4.4.3.2.1. If a sensor fails this test, inspect all measurements made since the sensor last passed to determine the time of failure. Invalidate measurements made after that time.
      - 9.4.4.3.2.2. Contact PM for sensor replacement.

9.5. Troubleshooting
- 9.5.1. If no measurements are reported at computer:
  - 9.5.1.1. Check for obstructions in the acoustic path. Invalid measurements occur when the acoustic path of the sonic signal is blocked or internal circuits fail.
    - 9.5.1.1.1. Acoustic blockage may be caused by rain drops, ice, snow, or other debris, and is indicated by the output error code (see Section 11).
  - 9.5.1.2. Check communications.
    - 9.5.1.2.1. Verify communications link between radio modems (Yellow and Green lights intermittently lit during communications) on modems; (See figure in Section 9.3.3).
    - 9.5.1.2.2. Verify firm connections for power, signal, and antenna for serial communications.
      - 9.5.1.2.2.1. Correct as needed.
    - 9.5.1.2.3. Verify power at modems in trailer and at anemometer, as indicated by a lit red light on the modems (See figure in Section 9.3.3).
      - 9.5.1.2.3.1. If not within acceptable range, check continuity of wires and tightness of cable connections and correct as needed.
        - 9.5.1.2.3.1.1. If all connections are tight and of good continuity, send sensor to manufacturer for repair and/or replacement of the appropriate modem.
    - 9.5.1.2.4. Verify Destination Address of radio modems (SOP W6), and correct as needed.
    - 9.5.1.2.5. Check that the correct baud rate is selected (See Section 9.1.3) and correct as needed.
  - 9.5.1.3. Sensor troubleshooting
    - 9.5.1.3.1. Verify power at anemometer by voltage measurement with multimeter: Voltage should be 12 to 24 VDC **at the sensor junction box terminals** (See figure in Section 9.1.2).
      - 9.5.1.3.1.1. If voltage is not within acceptable range, check continuity of wires and tightness of cable connections.
        - 9.5.1.3.1.1.1. Correct as needed.
        - 9.5.1.3.1.1.2. If all connections are tight and of good continuity, send sensor to manufacturer for repair or replacement.

9.6. Data acquisition, calculations & data reduction requirements
- 9.6.1. Data are collected on the LAN server (SOP D1).
- 9.6.2. Corrections for wake effects are internally conducted.
- 9.6.3. Identify spikes in 16-Hz values of *U*, *V*, *W*, *T* using 100-s mean filter and dynamically calculated standard deviation.
  - 9.6.3.1. Calculations are made by a computer program on the LAN server (SOP D1).
  - 9.6.3.2. Spikes are defined as data points with values more than 3 standard deviations away from running mean.
    - 9.6.3.2.1. Spike data are omitted.
  - 9.6.3.3. Count spikes and calculate their mean value.
  - 9.6.3.4. Inspect and flag data falling outside of acceptable limits based on spike counts.
    - 9.6.3.4.1. Write to a data file the spike count, as well as the mean value of spikes, over a 5-min interval.

9.6.3.4.2. If spike count is greater than 160 for any variable, then the 5-min measurement period for that variable is flagged and investigated.

9.6.3.4.2.1. Do not calculate the derived variables in Sections 9.6.4 and 9.6.5 using any invalidated variables as dependencies.

9.6.3.4.3. If spikes count is less than 160 for a variable, then that variable passes the spike test.

9.6.3.4.3.1. Store instantaneous *U*, *V*, *W*, *T* and corresponding qc flags (See table in Section 11.4.1) in temporary data file.

9.6.3.4.3.2. Store 16-Hz sampling of instantaneous values of wind components *u'* and *w'* from 2 m and 16 m anemometers over the period from 12:00 PM to 12:30 PM in a data file (for spectral analysis on LAN server).

[NOTE: There is no correction for tilt of the sensor since inhomogeneities of the surface type (lagoon vs. land surrounding lagoon) and terrain elevation (lagoon berm) create streamlines assumed to be variable with wind direction].

9.6.4. Mean horizontal winds derived from 2-s periods of 16-Hz sampling of instantaneous wind components *u'* and *v'*.

9.6.4.1. Computations are made by computer program on the LAN server (SOP D1).

9.6.4.2. Compute the means of U, V ($\overline{U}, \overline{V}$). Computed values are used in the radial plume method of emission measurement (SOP O2).

9.6.5. Turbulence statistics derived from 5-min periods of 16-Hz sampling of instantaneous wind components *u'*, *v'*, *w'* and *T'*.

9.6.5.1. Computations are made by computer program on the LAN server (SOP D1).

9.6.5.2. Compute the means of U, V, W, and T ($\overline{U}, \overline{V}, \overline{W}, \overline{T}$). Computed values are used in the quality assurance routines and the backward Lagrangian Stochastic emissions measurement (SOP O1).

9.6.5.3. Compute the variance of the three orthogonal wind components σ_u^2, σ_v^2, σ_w^2 and σ_T^2 ($\overline{u'u'}, \overline{v'v'}, \overline{w'w'}, \overline{T'T'}$). Computed values are used in the Quality Control and Assurance checks (Section 11) and the backward Lagrangian Stochastic emissions measurement (SOP O1).

9.6.5.4. Compute averaged correlations of instantaneous deviations in wind components *u'*, *v'*, *w'* and the temperature *T'* derived from the speed of sound: $\overline{u'w'}, \overline{u'v'}, \overline{v'w'}, \overline{u'T'}, \overline{w'T'}, \overline{v'T'}$. Computed values are used in the Quality Control and Assurance checks (Section 11) and the backward Lagrangian Stochastic emissions measurement (SOP O1).

9.6.6. Mean horizontal winds derived from 5 and 30-min periods of 16-Hz sampling of instantaneous wind components *u′* and *v′*.

9.6.6.1. Computations are made by computer program at PAML.

9.6.6.2. Computed values are used to characterize wind conditions.

9.6.6.3. Compute mean vector wind speed ($\overline{S}$) according to the following:

$$\overline{S} = \sqrt{\overline{U}^2 + \overline{V}^2}$$

9.6.6.4. Compute mean wind direction ($\overline{\Theta}$) according to:

$$\overline{\Theta} = \begin{cases} 0^{\circ} & \text{if } \overline{\mathrm{U}} = 0 \text{ and } \overline{V} > 0 \\ 180^{\circ} & \text{if } \overline{\mathrm{U}} = 0 \text{ and } \overline{V} < 0 \\ -\tan^{-1}\left(\overline{V} \big/ \overline{U} \right) + 90^{\circ} & \text{if } \overline{\mathrm{U}} > 0 \\ -\tan^{-1}\left(\overline{V} \big/ \overline{U} \right) + 270^{\circ} & \text{if } \overline{\mathrm{U}} < 0 \end{cases}$$

The convention for the sonic anemometer output is that U is positive for wind from the east, and V is positive for wind from the north. The directions obtained from the above formula are 0° for wind from the north, 90° for wind from the east, and so on.

9.6.7. Turbulence statistics derived from 5 and 30-min periods of 16-Hz sampling of instantaneous wind components *u', v', w'* and virtual temperature *T'*.

9.6.7.1. Computations are made by computer program at PAML.

9.6.7.2. Re-compute LAN-computed variances σ_u^2, σ_v^2, and σ_w^2 and correlations $\overline{u'w'}, \overline{u'v'}, \overline{v'w'}, \overline{u'T'}, \overline{w'T'}, \overline{v'T'}$ with rotated coordinates so that mean wind is in the u direction.

9.6.7.2.1. Rotated values are used in the stationarity and homogeneity quality assurance tests (Sections 11.3.2, 11.3.3)

9.6.7.3. Compute the friction velocity (u_*):

$$u_* = \sqrt[4]{\overline{u'w'}^2 + \overline{v'w'}^2} \qquad \text{(Flesch et al. 2005)}$$

9.6.7.3.1. Computed values are used to characterize wind conditions, and to qualify backward Lagrangian Stochastic emission measurements (SOP O1).

9.6.7.4. Compute the characteristic temperature:

$$T_* = \overline{w'T'} / u_* \qquad \text{(Kaimal and Finnigan 1994)}$$

9.6.7.4.1. Computed values are used to characterize wind conditions.

9.6.7.5. Compute the Obukov length (*L*):

$$z / L = \frac{-kzg\overline{w'T'}}{\overline{T}u_*^{3}} \qquad \text{(Flesch et al. 2005)}$$

Where:
z = Sensor height (m)
k = von Karman's constant (0.4)
g = Acceleration due to gravity (9.8 m/s)

9.6.7.5.1. Computed values are used to characterize wind conditions and qualify backward Lagrangian Stochastic emission measurements (SOP O1).

9.6.7.6. The program estimates the roughness length (z_o) as the average value of the three heights of wind measurement, each calculated as:

$$z_o = \frac{(z-d)}{e^{kU/u_* - \psi_m}} \quad \text{(Flesch et al. 2005)}$$

Where (Flesch et al. 2004, Businger et al. 1971, Paulson 1970):

ψ_M = $4.7\,(z\text{-d})/L$ for $z/L > 0.0001$

ψ_M = 0 for $0.0001 > z/L > 0.0001$

ψ_M = $-2\,ln[(1+x)/2]\ -ln\,(1+x^2)/2\ ]+ 2\tan^{-1} x - \pi/2$ and

X = $(1\text{-}15(z\text{-d})/L\ )^{1/4}$ for $z/L>0.0001$

9.6.7.6.1. Computed values are used to characterize wind conditions.

9.6.7.7. Estimate dissipation rate ε:

$$\varepsilon = \frac{u_*^3}{kz}\varphi_\varepsilon \qquad \text{(Flesch et al. 2004)}$$

Where:

$\varphi_\varepsilon = 1 + 5z/L$ for $L>0$

$\varphi_\varepsilon = \dfrac{b_w^4(1-3z/L)^{1.33}+1}{(b_w^4+1)(1-3z/L)^{0.33}(1-6z/L)^{0.25}}$ for $L<0$

9.6.7.7.1. Computed values are used to characterize wind conditions.

9.6.8. Write values of $\overline{U},\overline{V},\overline{W},\overline{T},\overline{S},\overline{\Theta}$, σ_u^2($\overline{u'u'}$), σ_v^2($\overline{v'v'}$), σ_w^2 ($\overline{w'w'}$) u_*, T_*, z/L, z_o, ε and corresponding qc flags (Table 1) to data file

9.7. Computer hardware & software

9.7.1. Using electrical wires, attach sensor serial port to an XStream® RS-232/485 RF Modem.

9.7.2. A computer in the instrumentation trailer will receive the data using an identically addressed XStream® RS-232/485 RF Modem on a COM port.

9.7.3. Computer will record and process the data, and store it using LABView software.

10. Data and Records Management

10.1. Manage data according to SOP D1.

10.2. Document all data and information checked in the field on field data sheets and in site logbooks with permanent ink, or in electronic notes.

10.3. Overstrike errors in writing with a single line; date and initial all such corrections.

11. Quality Control and Quality Assurance

11.1. Physical inspection
  11.1.1. Inspect sensor support arms for dents or deep scratches in the metal.
    11.1.1.1. If present, conduct an in-field intercomparison (Section 9.4.3).
11.2. Zero check
  11.2.1. Enclose sensor within an anechoic box and check for non-zero values on all wind components (similar to ASTM 2001).
    11.2.1.1. Record sensor biases in the U, V, and W components on an audit form similar to the one in Appendix A of this SOP.
    11.2.1.2. If sensor does not indicate values of less than 0.2 m/s in all three components, then send it back to the factory for repair or replacement.
11.3. Path obstruction check
  11.3.1. The instrument's internal processing determines individual invalid measurements due to a path obstruction (e.g. debris, dew, mist, ice, or snow) preventing or radically altering acoustic transit time measurement. Instrument output includes invalid data and an error code, the sum of values for the following conditions:

```
2   Zero samples on at least one acoustic path
4   Path A speed < -50 m/s
8   Path A speed > +50 m/s
16  Path B speed < -50 m/s
32  Path B speed > +50 m/s
64  Path C speed < -50 m/s
128 Path C speed > +50 m/s
```

    11.3.1.1. If less than 10% of the sampling interval has non-zero error codes, the measurement period is considered valid and computation on the valid data beginning at section 9.6 will be conducted.
    11.3.1.2. If greater than 10% of the sampling interval has a non-zero error code, the measurement period is given an error code (Table 1).
  11.3.2. <u>Stationarity check</u> Determine stationarity in the time series by comparing 5-min covariances of u' and w' and w' and T_v' (Section 9.6.6.2) with the 30-min covariance (Section 9.6.5) where i and j are different variables:

$$\sum \left[\left(\overline{u'_i\,u'_j}\right)_{5\,\mathrm{min}} - \left(\overline{u'_i\,u'_j}\right)_{30\,\mathrm{min}}\right] \Big/ \left(\overline{u'_i\,u'_j}\right)_{30\,\mathrm{min}}$$ (Foken and Wichura 1996)

where the 5-min periods included in the sum are sequential within the 30-min interval. Computations are done by computers at PAML.
    11.3.2.1. If greater than 0.30 with the inclusion of any 5-min period to the sum, the 5-min period of data is given a QA/QC error code (Table 1).
  11.3.3. <u>Homogeneity check</u>: Given values computed according to Section 9.6.6.2, check for homogeneity of turbulence during 30-min sample periods (six sequential 5-min periods) by comparing the theoretically derived and measured integral turbulence

scales of w', u' and T' (u_* and T_*) to the standard deviation of w' and T'. Computations are done by computers at PAML.

11.3.3.1. The theoretically-derived values are computed according to (Foken and Wichura 1996):

z/L	<-1	-1...-0.0625	-0.0625...<0	>=0
$\frac{\sigma_w}{u_*}$	$2.00(-z/L)^{1/6}$	$2.00(-z/L)^{1/8}$	1.41	1.25
$\frac{\sigma_u}{u_*}$	$2.83(-z/L)^{1/6}$	$2.83(-z/L)^{1/8}$	1.99	2.4
$\frac{\sigma_T}{T_*}$	$1.00(-z/L)^{1/3}$	$1.00(-z/L)^{1/4}$	$0.50(-z/L)^{1/2}$	

11.3.3.2. Compute the differences between theoretical and measured values as:

$$\sum\left[\frac{\sigma_w}{u_*}-\left(\frac{\sigma_w}{u_*}\right)_{theory}\right]\Big/\left(\frac{\sigma_w}{u_*}\right)_{theory}$$

$$\sum\left[\frac{\sigma_u}{u_*}-\left(\frac{\sigma_u}{u_*}\right)_{theory}\right]\Big/\left(\frac{\sigma_u}{u_*}\right)_{theory}$$

$$\sum\left[\frac{\sigma_T}{T_*}-\left(\frac{\sigma_T}{T_*}\right)_{theory}\right]\Big/\left(\frac{\sigma_T}{T_*}\right)_{theory}$$

11.3.3.2.1. If the value of any of these is greater than 0.30, the 30-min period is given a QA/QC error code (Table1).

11.4. Turbulence structure check: Conduct spectral turbulence checks of the 16-Hz sampling of instantaneous wind components *u'*and *w'* collected over an approximately 30-min period between 12:00 AM and 12:30 PM. Computations are done by computers at PAML.

11.4.1. Store instantaneous values of wind components *u'*, *v'* and *w'* from all anemometers in a data file for processing at PAML.

11.4.2. Calculate spectral statistics of *u'*and *w'*:

11.4.3. Calculate the spectral density function of the first 32,384 values at the all heights by fast Fourier transform.

11.4.4. Using u_* calculated for the 30-min period, calculate the spectral density function normalized to u_*^2 and plot as

$fS(f)/u_*^2$ by f for both measurement heights.

11.4.5. Compare shape of spectra and evaluate similarity.
  11.4.5.1. If more than one peak in the spectra for either height, flag period values as suspect (Table 1).
  11.4.5.2. If the peak spectral density in the 16-m height spectra is at a higher frequency than that for the 2-m height, flag period values as suspect (Table 1).

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12.20. Wilzak, JM, SP Oncley, SA Stage. 2001. Sonic anemometer tile correction algorithms. Boundary-Layer Meteorol. 99, 127-150.

13. Contact Information

13.1. Anemometer manufacturer:
R. M. Young Company, 2801 Aero Park Drive, Traverse City, MI, 49686, USA,
email: met.sales@youngusa.com
Customer support and repair: 231-946-3980, Fax 231-946-4772

13.2. Modem manufacturer:
Maxstream, Inc., 355 South 520 West, Suite 180, Lindon, UT 84042
Phone: (801) 765-9885
Fax: (801) 765-9895
email: rf-xperts@maxstream.net

Table 1. Data QA/QC conditions.

Individual measurements

Condition
Value not suspect
Value missing (>10% of records in 5 min period)
Value below minimum or above maximum or value is +/- infinity
Spike count >160
Value has variance >10 m^2/s^2 (u, v, w) or 10 C^2 (T)
> 10% of sampling interval has path obstruction

Combinational measurements

$u'w'$ not stationary (30-min period)
$w'T'$ not stationary (30-min period)
σ_u inhomogeneous turbulence in 5-min period
σ_w inhomogeneous turbulence in 5-min period
σ_T inhomogeneous turbulence in 5-min period

APPENDIX A
AUDIT FORM

SOP W2 RM YOUNG MODEL 81000 3-DIMENSIONAL SONIC ANEMOMETER

Date/time Yymm/dd hhmm-hhmm	Bias in U (m/s)	Bias in V (m/s)	Bias in W (m/s)	Auditor

APPENDIX B
FIELD INTERCOMPARISON FORM

SOP W2 RM YOUNG MODEL 81000 3-DIMENSIONAL SONIC ANEMOMETER

Date/time Yymm/dd hhmm-hhmm	**Mean Speed (m/s)**			**Auditor**
	Sensor 1	**Sensor 2**	**Sensor 3**	

MEASUREMENT OF WETNESS WITH THE CAMPBELL SCIENTIFIC MODEL RESISTANCE GRID

Standard Operating Procedure (SOP) W3

MEASUREMENT OF WETNESS WITH THE CAMPBELL SCIENTIFIC MODEL RESISTANCE GRID

Standard Operating Procedure (SOP) W3

Prepared by

Juan Carlos Ramirez and Richard H. Grant

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006
Rev. 1.0 Effective Date: February 6, 2008

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
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1. Scope and Applicability

1.1. Electrical resistance grids are useful for the determination of surface wetness.
1.2. Surface wetness measurements indicate precipitation, an important weather variable.
1.3. This procedure applies only to the Campbell Scientific Model 237 Leaf Wetness Sensor Resistance Grid, to be used coated with white latex ceiling house paint.
1.4. This device is used as a wet/dry sensor in this application. Sensor readings in between wet and dry are simply interpreted as “Conditions other than wet or dry”.
  1.4.1. If measuring resistance with a multimeter, connect the leads to the red and black sensor leads. The sensor lead has a built-in 100,000–Ω, resistor in series with the sensor that must be included in any resistance calculations. See R1 in Fig. 1 of the Model 237 manual. The resistance (including the 100,000-Ω resistor) will be approximately 120,000 Ω – 500,000 Ω when wet, and above 3,100,000 Ω when dry.

2. Summary of Method

2.1. The 237 Leaf Wetness Sensor design emulates the surface area of a leaf. The dimensions of the sensor are 2.75″ W x 3″ L x 0.25″ D, and its weight is 0.5 lb. Surface wetness is detected using a resistance grid (see Fig. 1 at right) of interlaced gold-plated copper etched ‘fingers’ on an electronic circuit board. Condensation connects the etched ‘fingers’ of the board, resulting in a resistance change. The change in resistance is detected by change in voltage of a half bridge of fixed resisters (built into sensor cable) excited by AC current.

Figure 1.
Leaf Wetness Sensor

3. Definitions

3.1.	CS	Campbell Scientific, Inc
3.2.	NEMA	National Electrical Manufacturers Association
3.3.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.4.	PAML	Purdue Applied Meteorology Laboratory
3.5.	QAPP	Quality Assurance Project Plan
3.6.	SOP	Standard operating procedure

4. Cautions

4.1. Be careful when connecting the sensor cable to the data logger, being sure the connections are tight and made according to SOP W6.
4.2. Replace the sensor if the cable should become detached from the sensor (do not attempt to resolder it to the sensor), as the cable contains built-in components (a half bridge of fixed resistors) specifically designed to make the sensor operate correctly.
4.3. As with any electrical device, use proper care when crimping cables and connectors.

5. Interferences

5.1. Dust will reduce the sensitivity of the sensor to drying conditions.
5.2. Fingerprints and grease will reduce the sensitivity of the sensor to wetting conditions.

6. Personnel Qualifications

6.1. Each member of the field staff must read the sensor instruction manual and this SOP before operating this equipment.
6.2. Personnel must be trained in the use of the handheld multimeter before initiating the procedure. Training requires 10 min.

7. Equipment and Supplies

7.1. Leaf Wetness Sensor (Campbell Scientific, Model 237, Logan, Utah)
7.2. #2 common head screwdriver
7.3. 1/8″ slotted screwdriver
7.4. Bottle of distilled water for cleaning and testing sensor
7.5. Multimeter

8. Procedures

8.1. Preparation for use
  8.1.1. Clean the Model 237 Leaf wetness sensor thoroughly using acetone; allow to dry.
  8.1.2. Coat the sensor with a single coat of standard Latex exterior house paint; allow to thoroughly dry.
8.2. Acceptance
  8.2.1. Attach multimeter probes to red and black wires of sensor cable.
  8.2.2. Set multimeter to measure resistance.
    8.2.2.1. Read sensor response on multimeter when sensor is dry.
      8.2.2.1.1. Value should be greater than 3,100,000 Ω.
      8.2.2.1.2. If less than 3,100,000 Ω, allow sensor to dry in a warm environment for a minimum of 24 h. If the sensor still reads less then 3,100,000 Ω, return sensor to manufacturer.
    8.2.2.2. Wet sensor with tap water and read sensor response on multimeter.
      8.2.2.2.1. Value should be in the range of 120,000 – 500,000 Ω (±40,000 Ω).
      8.2.2.2.2. If wet resistance is <80,000 Ω or >540,000 Ω, return sensor to manufacturer.
8.3. Calibration check
  8.3.1. Note that this device is not a precision instrument, and exact resistance readings may vary considerably, depending on a wide variety of conditions including the mineral content of the distilled test or rain water.
  8.3.2. Attach red and black sensor wires to probe wires of multimeter.
  8.3.3. Set multimeter to measure resistance.
    8.3.3.1. Read sensor response on multimeter when sensor is dry.

- 8.3.3.1.1. Record value on calibration check sheet (Appendix A). If resistance is over-range on the multimeter's highest resistance scale, record resistance as infinite.
- 8.3.3.1.2. Value should be greater than 3,100,000 Ω.
- 8.3.3.1.3. If not, return sensor to manufacturer.
- 8.3.3.2. Wet sensor with distilled water and read sensor response on multimeter.
  - 8.3.3.2.1. Record value on calibration check sheet (Appendix A)
  - 8.3.3.2.2. Value should be within 120,000 Ω – 500,000 Ω (wet resistance tolerance is not critical).
  - 8.3.3.2.3. If after one minute, resistance value is less than 80,000 Ω, or over 1,000,000 Ω, return sensor to manufacturer.

8.4. Installation

- 8.4.1. Mount sensor on the 3-m (10-ft) meteorological mast (SOP U5) at 2-m (6.6-ft) height, according to SOP W6.
- 8.4.2. Pass cable along ground to weatherproofed NEMA 4X box (SOP W6) and through specified knockout.
- 8.4.3. Attach sensor wires to data logger according to SOP W6 using the 1/8″ slotted screwdriver.
  - 8.4.3.1. Connect black wire to 5 VAC excitation, purple wire to ground, clear wire to ground, and red wire to analog single ended measurement channel.
  - 8.4.3.2. Data logger will log voltage returned from the wetness sensor's output wire (red wire) as a ratio of the measured voltage divided by the excitation voltage (Vs/Vx). The datalogger applies 5 VAC to the sensor's exciter (black) wire.
    - 8.4.3.2.1. Sensor resistance (Rs) is recorded as a voltage ratio using the following calculation: Vs/Vx=R2/(R2+R1+Rs).
    - 8.4.3.2.2. R1 and R2 are fixed values; R1=100,000 Ω and R2=1,000 Ω (Fig. 1 of Model 237 manual).
    - 8.4.3.2.3. For example, if the sensor resistance is 101,200 Ω, the voltage ratio recorded would be 0.0049455 (1000(R2)/(102,200(R2+R1+Rs)).
  - 8.4.3.3. This logged voltage will indicate the condition the sensor is currently detecting.
  - 8.4.3.4. Values from 4.524 mV to 1.663 mV (120,000 Ω < Rs < 500,000 Ω will indicate "Wet" conditions.
  - 8.4.3.5. Values less than 0.3124 mV (Rs >3,100,000 Ω) will indicate "Dry" conditions.
  - 8.4.3.6. Values between 1.663 mV and 0.3124 mV 500,000 Ω < Rs < 3,099,999 Ω) will indicate transition "Conditions other then wet or dry".
- 8.4.4. While sensor is dry, check to see that data logger is indicating "Dry" conditions. Check wiring connections for error if response is incorrect.
- 8.4.5. Rinse the wetness grid with tap water and verify data logger's proper "Wet" response. Check wiring connections for error if response is incorrect.

8.5. Troubleshooting

- 8.5.1. If readings are noisy or erratic, check for poor connections.
- 8.5.2. If sensor does not respond to wetness, check for broken wires or connections.

8.6. Data acquisition, calculations & data reduction requirements

- 8.6.1. Analog input is monitored by the datalogger.

- 8.6.1.1. Ratio of excitation to sensor resistance is recorded and then calculated at PAML to be “Wet”, “Dry” or “Conditions other then wet or dry”, based on sensor resistance calculated from recorded voltage ratio.
- 8.6.2. Datalogger will record measurements every 5 minutes.
- 8.7. Computer hardware & software.
  - 8.7.1. Attach the sensor to a Campbell Scientific datalogger through electrical wires that will extract data from the instruments.
  - 8.7.2. The Campbell Scientific data logger will record the data and store it.
  - 8.7.3. Datalogger operates using on-board BASIC-like programming.
    - 8.7.3.1. The general program for measurements is stated in the Model 237 Manual.

9. Data and Records Management

- 9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, and in a record book designated for meteorological sensors.
- 9.2. Manage all data according to SOP D1.
- 9.3. Document all data and information on field notes with permanent ink, or in electronic notes. Overstrike errors in ink with a single line, then initial and date the overstrike.

10. Quality Control and Quality Assurance

- 10.1. Daily check from PAML
  - 10.1.1. If value is -6999, flag with “1”.
    - 10.1.1.1. Voltages over the maximum measurable input are indicated by data logger as -6999, and indicate sensor failure (short circuit). Dispatch personnel to check sensor and sensor wiring on site.
  - 10.1.2. Check weather data from an outside source for the area such as http://www.crh.noaa.gov/iwx/index.php?mystation=KLAF (Lafayette, IN). If indicated data does not match conditions shown by outside weather sources on two or more days:
    - 10.1.2.1. Flag recorded value with a “2” as invalid.
    - 10.1.2.2. Dispatch personnel to check sensor and sensor wiring on site.
      - 10.1.2.2.1. If damage is indicated, repair or replace as indicated in Section 8.4.
- 10.2. Perform the following checks at the beginning and end of each measurement period.
  - 10.2.1. Check to be sure probe surface is clean.
    - 10.2.1.1. If dirty, clean with distilled water and cloth.
  - 10.2.2. Check integrity of the plastic casing of the signal cable.
    - 10.2.2.1. If damaged slightly, repair with heat-shrink tubing.
    - 10.2.2.2. If damaged significantly, replace sensor and return sensor to manufacturer for repair.
  - 10.2.3. Wet sensor with tap water.
    - 10.2.3.1. Check datalogger for value corresponding to a wet sensor
    - 10.2.3.2. If data logger does not indicate wet conditions, check wiring (Section 8.2).
  - 10.2.4. Dry sensor.
    - 10.2.4.1. Check datalogger for value corresponding to a dry sensor.

10.2.4.2. If data logger does not indicate dry conditions, check wiring (Section 8.2).

10.2.5. Record results of checks on the site logbook and electronic spreadsheet.

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11.3. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.

11.4. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.

12. Contact Information

12.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, Utah, 84321

12.1.1. Technical support: 435-753-2342

Appendix A

Calibration Check Form: Campbell Scientific Resistance Grid Wetness Sensor

Instrument Serial Number:__________________________________ Location:___________________________________

Date (D-M-Y)	Time		Resistance (Ω)		Action Taken (Adjust, Replace)	Analyst
	Begin	End	Dry	Wet		

THE MEASUREMENT OF BAROMETRIC PRESSURE WITH THE SETRA MODEL 278 (CAMPBELL SCIENTIFIC CS100) BAROMETER

Standard Operating Procedure (SOP) W5

THE MEASUREMENT OF BAROMETRIC PRESSURE WITH THE SETRA MODEL 278 (CAMPBELL SCIENTIFIC CS100) BAROMETER

Standard Operating Procedure (SOP) W5

Prepared by

Richard H. Grant

Reviewed by

Albert. J. Heber and Bill W. Bogan

Effective Date: November 6, 2006

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1. Scope and Applicability

1.1. Barometric pressure measurements are needed to determine the mass concentration of gases from volume concentration measurements.
1.2. The resonant frequency of silicon is a useful means of measuring pressure.
1.3. The lower detection limit for the instrumentation described in this SOP is 600 hPa.
1.4. The range of the instrument, under normal atmospheric conditions, is from 600 to 1100 hPa.
1.5. Sensor response is 5mV / hPa.
1.6. Sensor accuracy is ±3 hPa
1.7. This procedure applies only to the Setra model 278 barometric pressure sensor, purchased as CS100 Barometric Pressure Sensor
1.8. This procedure applies to the open source portion of the National Air Emissions Study.

2. Summary of Method

Barometric pressure is measured using two closely-spaced, parallel, electrically-isolated metallic surfaces, one of which is essentially a diaphragm capable of slight flexing under applied pressure. The surfaces are mounted to allow slight mechanical flexing by small changes in pressure. Changes in the gap between the surfaces change the electrical capacitance of the gap, which is detected and converted to a proportional high level analog signal. The Setra model 278 sensor is used under this SOP because it is well-suited to long-term environmental applications, as its housing and all of its wettable parts are constructed of stainless steel or polyester. The sensor itself is enclosed in a thermally stable, low-hysteresis glass fused ceramic capsule which minimizes temperature effects on pressure readings.

3. Definitions

ARM	Atmospheric Radiation Monitoring Network
CS	Campbell Scientific, Inc
hPa	The S.I. unit of pressure is Pascal. The worldwide meteorological community uses the hectopascal(hPa) i.e. a hundred Pascals, which is the metric equivalent of a millibar. “Standard atmospheric pressure=1013 hPa = 101,325 Pa= 101.3 kPa=760 mm Hg=14.696 psi.”
NEMA	National Electrical Manufacturers Association
PAAQL	Purdue Agricultural Air Quality Laboratory
PAML	Purdue Applied Meteorology Laboratory
QAPP	Quality Assurance Project Plan
VDC	Direct Current voltage

4. Cautions

4.1. Static charge damages the sensor. To avoid damage, take adequate anti-static measures when handling.
  4.1.1. Wear an antistatic wrist strap that is properly connected to earth ground when in contact with the sensor.
  4.1.2. If an antistatic wrist strap is not available, discharge static electricity by touching a grounded metal object before touching the sensor itself.

5. Interferences

5.1. Failure to provide an open vent at the bottom of the NEMA 4X box will interfere with barometric measurements by blocking the sensor's ability to sense atmospheric pressure changes.
5.2. Failure to protect sensor from condensation or liquid water may cause permanent damage. See Section 8.2.2 for proper installation to avoid condensation. Place desiccant inside the NEMA box to absorb any moisture which does occur.

6. Personnel Qualifications

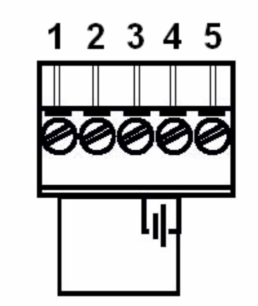
6.1. Personnel must be trained in the use of the handheld frequency multimeter before initiating the procedure. Training requires 10 min.
6.2. Personnel must be trained in the use of the power supply before initiating the procedure. Training requires 10 min.
6.3. Each staff member must read and understand the sensor instruction manual and this SOP before operating this sensor.

7. Equipment and Supplies

7.1. Multimeter
7.2. #2 Phillips head screwdriver
7.3. 1/8" slotted screwdriver
7.4. 2-inch length of jumper wire
7.5. 12 VDC power supply
7.6. 2-in. section of plastic tubing
7.7. Toothbrush
7.8. Electrical cleaner
7.9. Fortin Barometer (kept at PAML)
7.10. CS100 Barometric Pressure Sensor
7.11. NEMA 4X Box
7.12. Campbell Scientific Data Logger

8. Procedures

8.1. Acceptance



8.1.1. Connect terminal 4 to positive terminal of 12 VDC supply and terminal 3 to power supply ground (See diagram at right).

8.1.2. Connect a jumper across terminals 1 and 4.

8.1.3. With multimeter off, connect terminal 2 to negative terminal of multimeter and terminal 5 to positive terminal of multimeter.

8.1.4. Turn multimeter on and set to mV DC.

8.1.5. After 2 s, read measured voltage on multimeter.

8.1.6. Voltage should be approximately 1-2.25 VDC (800-1050 hPa).

8.1.6.1. If values are less than 1V or greater than 2.25 VDC, the sensor is defective, and should be returned to the manufacturer for replacement or repair.

8.2. Setup

8.2.1. Mount the instrument on the inside of the NEMA 4X box on the 10-foot mast according to SOP U5.

8.2.2. Mount the sensor with the pneumatic connector pointing vertically downwards to prevent condensation collecting on the pressure cavity and ensure that water cannot enter the sensor. Place fork lug under the mounting screw for grounding.

8.2.3. Using the 1/8”slotted screwdriver, connect the red wire to 12 VDC supply, black to power ground, yellow to signal ground, clear to shield, green to the external trigger (control terminal), and blue to a single-ended analog terminal of the data logger, according to SOP W6.

8.2.4. On power-up, the sensor requires less than 1 second of warm-up time.

8.2.5. The voltage across the blue and yellow wires (when terminals 1 and 4 are jumper wired as in Step 8.1.2) should be approximately 1-2.25 VDC (800-1050 hPa).

8.2.5.1. If values are less than 1 VDC, the sensor is defective, and should be returned to the manufacturer for replacement or repair.

8.3. Calibration

8.3.1. Calibrate the sensor every 6 mos., or at a different frequency if specified by the QAPP for a particular project.

8.3.2. Calibrate the sensor system (sensor and data logger) against a Fortin Barometer at PAML.

8.3.2.1. Follow the procedures for reading and adjusting the Fortin barometer documented in Appendix A of this SOP.

8.3.2.2. Read instantaneous measurements from datalogger/ sensor system at the time of each Fortin Barometer reading.

8.3.3. Conduct six paired measurements over a two–day period, average mean of their respective readings are taken out and if this matches then it is calibrated.

8.3.4. Record the results of the calibration on the sensor lab notebook form (Appendix B).

8.3.4.1. If the mean difference between Fortin barometer and senor/datalogger measurement is less than or equal to 2 hPa, then the sensor passes calibration and the drift is back-corrected according to Equation 1.

$$P_{corr}(t) = P(t)\left[1 - \frac{\overline{P_{measured}(i) - P_{\exp ected}(i)}}{\overline{P_{\exp ectd}}}\right]\left(\frac{t}{p}\right) \qquad \text{(Equation 1)}$$

Where:
P_{corr} = Back-corrected value at time t into the last measurement period
$P(t)$ = Original measured value at time t
$P_{measured}$ = Measured barometric pressure during calibration comparison
$P_{expected}$ = Barometric pressure indicated by the Fortin barometer during calibration comparison
p = Length of time between calibrations

8.3.4.2. If the mean difference between Fortin barometer and senor/datalogger measurement is >2 hPa, return sensor to manufacturer for repair or replacement.
8.3.4.2.1. If the mean difference is <6 hPa, adjust measurements since the last passed calibration linearly over time to the current calibration to account for the drift according to Equation 1. Correction coefficients determined at this point can be entered into the PAML computers to correct data collected during the next measurement period; the QAPP for a particular project will specify whether or not this is to be done.
8.3.4.2.2. If the mean difference is >6 hPa, invalidate barometric pressure measurements over the preceeding measurement period.

8.4. Troubleshooting
8.4.1. If pressure measurements vary widely over 24 h, check that the NEMA box is not ‘hermetically sealed’. A hermetic seal is evident when air is distinctly forced out at the sides of the opening when the box is closed.
8.4.2. Be sure there is no condensate in the NEMA box.
8.4.3. Visually inspect the casing for damage.
8.4.4. Ensure there is a secure, clean ground connection.
8.4.5. Ensure that the pneumatic connection and tubing are secure and undamaged.
8.4.6. Replace desiccant in the NEMA box as needed.

8.5. Data acquisition, calculations & data reduction requirements
8.5.1. Monitor the analog voltage output with a Campbell Scientific datalogger or equivalent data acquisition system (See SOP W6).
8.5.2. Take measurements every 5 min
8.5.3. Report voltage data to five significant digits.

8.6. Computer hardware & software
8.6.1. Attach the sensors through electrical wires to a Campbell Scientific datalogger (or equivalent data acquisition system) that will extract data from the instruments.
8.6.2. The logger program for reading the sensor is written in a version of BASIC language.
8.6.2.1. The program converts the sensor voltage to hPa using the following formula:

$$\text{hPa} = 600 + 0.2\ \text{mVDC} \qquad \text{(Equation 2)}$$

8.6.2.2. The sensor units are logged as hPa.
8.6.3. Record and store the data with the datalogger.

9. Data and Records Management

9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for meteorological sensors.
9.2. Manage data according to SOP D1.
9.3. Record the calibration information on the form provided in Appendix B.
9.3.1. Maintain a separate record sheet for each individual sensor.
9.4. Document all data and information on field data sheets and in site logbooks with permanent ink, or in electronic notes.
9.5. Overstrike errors in writing with a single line. Initial and date all corrections.

10. Quality Control and Quality Assurance

10.1. Daily
10.1.1. If value is missing, flag with “1”.
10.1.2. If value is less than 600 hPa or greater than 1100 hPa (i.e. outside the range of the sensor), then flag value with “2”.
10.1.3. Calculate the altitude-adjusted barometric pressure from the nearest National Weather Service Weather Station.
10.1.3.1. Correct NWS measured pressure to height of measurement location according to Equation 2 (Holton, 1979):

$$P_{site} = P_{NWS} e^{-(Z_{site} - Z_{NWS})/H} \qquad \text{(Equation 3)}$$

Where:

P_{site} = Height-adjusted NWS pressure for site
P_{NWS} = Pressure at NWS station
Z_{site} = Site elevation
Z_{NWS} = NWS station elevation
H = $287(T_{NWS})/9.80665$
T_{NWS} = Absolute temperature (°K) at NWS station.

10.1.4. If the altitude-adjusted difference between the barometric pressure at the sampling site and the nearest local weather station is > 4 hPa , then flag the day’s data with “3”.
10.1.4.1. Further investigation of the cause for the difference (such as synoptic weather front over the area) will be conducted.
10.2. Repeat the Acceptance Check (Section 8.1) before beginning a measurement period (i.e. during the setup of equipment at a site).
10.3. Do the following at the beginning and end of each measurement period:
10.3.1. Visually inspect the casing for damage.
10.3.1.1. Replace sensor if damaged.

- 10.3.2. Ensure there is a good secure and clean ground connection.
  - 10.3.2.1.Secure ground if needed.
- 10.3.3. Ensure that the pneumatic connection and tubing is secure and undamaged.
  - 10.3.3.1.Replace tubing if needed.
- 10.3.4. Check dessicant in the NEMA box.
  - 10.3.4.1. Replace as needed.
- 10.3.5. Check cleanliness of connectors.
  - 10.3.5.1.If corroded, clean with toothbrush and electrical cleaner.

11. References

11.1. ARM. 2005.: Temperature, Humidity, Wind, and Pressure System Handbook. Atmospheric Radiation Measurement Climate Research Facility, ARM TR-030, 13p.
11.2. Campbell Scientific. 2005. Model CS100 Barometric Pressure Sensor Instruction Manual, 8p.
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11.4. Setra Systems Inc. 2004. Installation guide: Model 278 Barometric Pressure Sensor, 5p.
11.5. National Physical Laboratory. 2006. “Frequently asked questions - pressure and vacuum: How do I use a Fortin or Kew Pattern mercury barometer?”, http://www.npl.co.uk/pressure/faqs/usehgbaro.html#calcpress, accessed 1/11/2006
11.6. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U6. Purdue Applied Meteorology Lab.
11.7. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.
11.8. SOP D1. 2006. Management of Open-Source, Weather, And Lagoon-Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
11.9. USDOC Weather Bureau. 1963. Manual of Barometry (WBAN) Volume 1, 1st edition, 1963
11.10. USEPA. 1989. Quality Assurance Handbook For Air Pollution Measurement Systems, Volume IV- Meteorological Measurements.
11.11. USEPA. 2000. Meteorological Monitoring Guidance for Regulatory Modeling Applications, EPA-454/R-99-005.

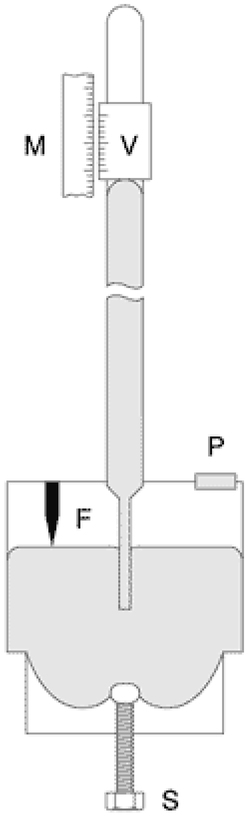
12. Contact Information

- 12.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, UT, 84321
  - 12.1.1. Technical support: 435-753-2342
- 12.2. Manufacturer: Setra Systems, Inc., 159 Swanson Road, Boxborough, MA 01719
  - 12.2.1. Repair Dept. Fax: 978-266-2158/Phone: 978-266-2194

Appendix A. Fortin Barometer Operation

The correct procedure for measuring pressure with a Fortin Barometer is:

- Adjust the cistern mercury surface to within a small distance of the fiducial point.
- Tap the barometer (to achieve a stable meniscus).
- Set the fiducial point.
- Set the vernier.
- Measure the barometer's temperature (and add calibration correction).
- Read the vernier.
- Apply the calibration correction for the barometer.
- Apply the temperature and gravity corrections (from tables or formula).



Fortin barometers should be used with their axis of rotation vertical - such that a setting of the mercury surface to the fiducial point remains correct after axial rotation through any angle.

<u>Setting the Fiducial Point (F in diagram at right)</u>

The lower mercury surface in a Fortin barometer has to be set to a datum level before adjusting its vernier (see schematic diagram) and the accuracy of pressure measurement depends crucially upon proper setting.

To achieve this, the mercury surface should first be lowered until it is clearly *below* the fiducial point (F in diagram at right). After tapping the barometer, the level-adjusting screw (S in diagram at right) should be turned slowly until the thin background of light between the mercury surface and the point just disappears, while viewed along a horizontal line of sight. To confirm proper setting, observe the point of contact at an angle of elevation of about 30° and determine whether or not the fiducial point is making an indentation in the mercury.

When the setting is correct there should be no more than the slightest dimple where the fiducial point makes contact with the mercury surface. If the surface is bright and the setting correct, the tip of the point will appear to coincide with its reflected image in the mercury surface. Correct setting is achieved when the mercury surface is *raised* to the fiducial point but not lowered - if the mercury surface overshoots the point it should be lowered and then raised again.

<u>Setting the vernier</u>: With suitably bright background illumination (using flashlight aimed at the white plastic background for the vernier), and taking care to correctly align the front and back setting edges, the vernier cursor (V in diagram at right) should be lowered until it appears on the

summit of the mercury meniscus. Proper setting can be aided by making small 'pecking' head movements to ensure that both cursor edges just meet the meniscus. Read the vernier.

Measuring temperature: Mercury barometers have a temperature coefficient of nearly 0.2 hPa per °C so it is important to ensure that their thermometers respond to ambient temperature changes in the same fashion as the barometer. Read the temperature off the thermometer attached to the barometer case.

Calculation of pressure from barometer readings: Three corrections to the measured barometric pressure must be made:

- Temperature correction
- Calibration correction
- Gravity correction

Theoretically, the corrections are made according to the following equations:

$$t = (t` - 32) * 5/9$$

$$\text{Pressure} = \frac{g}{9.806\,65}\left\{R + c - R\frac{(b-a)t}{(1+bt)}\right\}$$

where R is the barometer reading (in inches Hg)
c is its calibration correction (+.003 in Hg for barometer at PAML)
g is the acceleration due to gravity at the level of the barometer in m/s^2 (see below)

b is the coefficient of expansion of mercury (taken to be 0.000 181 8 $°C^{-1}$ for meteorological purposes)
a is the coefficient of linear expansion of the scale (taken to be 0.000 018 4 $°C^{-1}$ for brass)
t is the temperature of the barometer in °C
$t`$ is the temperature of the barometer in °F

and $g = 9.780\,318\,4\,(1 + A\sin^2 L - B\sin^2 2L) - 3.086\times10^{-6} H$

where A = 0.005 302 4
B = 0.000 005 9
L = Latitude (40.5)
H = height of barometer meters above sea level (197 m)

At West Lafayette, g = 9.80153, so the gravity correction is 9.80153 / 9.80665 = 0.999.

The correction terms involving a, b, and c were combined in the following table. To correct for the barometer local gravity and temperature, multiply the measured value by the value from the following tables and subtract from the measured value, then multiply by 0.999.

deg. F	correction	deg. F	correction	deg. F	correction
50	0.001631	70	0.003436	90	0.005234
50.5	0.001676	70.5	0.003481	90.5	0.005279
51	0.001721	71	0.003526	91	0.005324
51.5	0.001767	71.5	0.003571	91.5	0.005369
52	0.001812	72	0.003617	92	0.005414
52.5	0.001857	72.5	0.003662	92.5	0.005459
53	0.001902	73	0.003707	93	0.005504
53.5	0.001947	73.5	0.003752	93.5	0.005548
54	0.001993	74	0.003797	94	0.005593
54.5	0.002038	74.5	0.003842	94.5	0.005638
55	0.002083	75	0.003887	95	0.005683
55.5	0.002128	75.5	0.003932	95.5	0.005728
56	0.002173	76	0.003977	96	0.005772
56.5	0.002219	76.5	0.004022	96.5	0.005817
57	0.002264	77	0.004067	97	0.005862
57.5	0.002309	77.5	0.004111	97.5	0.005907
58	0.002354	78	0.004156	98	0.005952
58.5	0.002399	78.5	0.004201		
59	0.002444	79	0.004246		
59.5	0.002489	79.5	0.004291		
60	0.002535	80	0.004336		
60.5	0.00258	80.5	0.004381		
61	0.002625	81	0.004426		
61.5	0.00267	81.5	0.004471		
62	0.002715	82	0.004516		
62.5	0.00276	82.5	0.004561		
63	0.002805	83	0.004606		
63.5	0.00285	83.5	0.004651		
64	0.002896	84	0.004696		
64.5	0.002941	84.5	0.004741		
65	0.002986	85	0.004786		
65.5	0.003031	85.5	0.004831		
66	0.003076	86	0.004875		
66.5	0.003121	86.5	0.00492		
67	0.003166	87	0.004965		
67.5	0.003211	87.5	0.00501		
68	0.003256	88	0.005055		
68.5	0.003301	88.5	0.0051		
69	0.003346	89	0.005145		
69.5	0.003391	89.5	0.00519		

Appendix B.

Calibration Record: Setra 278 Barometric Pressure Sensor

Instrument Serial Number:_________________________________ Location:__________________________________

Date (D-M-Y) Time: Begin-End (HHMM-HHMM)	Temp. (°C)	Manometer pressure (in. Hg)	Hg Temp. correction (in. Hg)	Pressure			Difference hPa	Action*	Analyst
				Corrected		Measured			
				in. Hg	hPa**	hPa			

* Sensor adjustment or replacement made after the measurement, if necessary. If action is taken, next entry should show result of re-calibration following that action.

**1hPa = 0.02953 in Hg

ESTABLISHMENT, DATA ACQUISITION AND CONTROL OF WEATHER AND LAGOON CHARACTERIZATION HARDWARE

Standard Operating Procedure (SOP) W6

ESTABLISHMENT, DATA ACQUISITION AND CONTROL OF WEATHER AND LAGOON CHARACTERIZATION HARDWARE

Standard Operating Procedure (SOP) W6

Prepared by

Richard H. Grant

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
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Table of Contents

1. Scope and Applicability

1.1. Weather and lagoon characteristics data are needed when measuring lagoon emissions at livestock facilities.
1.2. The measurement sites are typically at locations remote from headquarters.
1.3. The instrumentation on the tower must be in proper communication with the datalogger, which, in turn, must communicate properly with the PC in the trailer.
1.4. All equipment needed for these measurements must be transported to the site.

2. Summary of Method

2.1. Set up meteorological towers in an unobstructed, level area at the corner of the lagoon, referring to SOP U5, and to the site-specific site monitoring plan. Mount weather related sensors on the towers, as per instructions in their individual SOPs; these include barometric pressure (SOP W5), surface wetness (SOP W3), solar radiation (SOP W4), T/RH (SOP W1), and sonic anemometers (SOP W2). Another group of sensors, including T (SOP L1), pH (SOP L2), and redox state (SOP L3), is deployed, via the use of a float, into the lagoon. All these sensors are connected to a datalogger, which communicates with a ‘LAN server’ at the site.

3. Definitions

3.1.	DM	Data Manager
3.2.	FIFO	First in first out
3.3.	FOS	Field operations staff
3.4.	LAN server	Local Area Network server
3.5.	OSHA	Occupational Safety and Health Administration
3.6.	PC	Personal computer (laptop)
3.7.	PDC	Pathlength-defining components. PDC is used to denote the component on the other end of the optical path from the PI-ORS instrument. Depending on the instrument selected, this could be a source, detector, mirror, or other reflecting object.
3.8.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.9.	PAML	Purdue Applied Meteorology Laboratory
3.10.	PI-ORS	Path-Integrated Optical Remote Sensing
3.11.	PM	Project Manager
3.12.	PPE	Personal protection equipment
3.13.	QAPP	Quality Assurance Project Plan
3.14.	QAM	Quality Assurance Manager
3.15.	RF	Radio frequency
3.16.	RH	Relative humidity
3.17.	SE	Single-ended
3.18.	Site PI	Site Principal Investigator
3.19.	T	Temperature
3.20.	T/RH	Temperature/Relative humidity

3.21.	TDLAS	Tunable Diode Laser Absorption Spectroscopy
3.22.	USDOT	US Department of Transportation
3.23.	UV-DOAS	Ultraviolet Differential Optical Absorption Spectrometer

4. Health and Safety

4.1. Be sure to use proper protective equipment (Section 8.1) while working at the field site.
4.2. Be careful when using the hinge pivot of the crank-up tower, in order to avoid pinching or other injury during setup and takedown.
4.3. Misuse or mishandling of electrical equipment may cause electrocution, severe burns, injury, or death.

5. Cautions

5.1. The sensors have electrical components, and proper care should be used as with all electrical equipment.
5.2. Firmly stake down the mast to prevent collapse under high winds or heavy ice
5.3. Proper connection of line power is necessary. Carefully crimp cables and connectors.
5.4. Lightning damage is more likely if the datalogger is not properly grounded.
5.5. Tighten all bolts on NEMA box to prevent the box from slipping down or turning on mast.

6. Interferences

6.1. Incorrect sighting of meteorological towers and masts cause measurement inaccuracies.
6.2. Field radio frequency (RF) noise can cause inaccuracies in the measurements if signals are not appropriately filtered and datalogger is not appropriately grounded.

7. Personnel Qualifications

7.1. Personnel must be capable of lifting at least 50 lbs.
7.2. Personnel must be trained in the use of the handheld multi-meter and voltage calibrator.
7.3. Personnel must be trained in the programming and operation of the datalogger and must have read the associated manual
7.4. Personnel must be trained in the programming and operation of the RF modem and must have read the associated manual.
7.5. Personnel must read and understand this SOP and all cited SOPs.

8. Equipment and Supplies

8.1. Safety equipment
  8.1.1. Leather gloves
  8.1.2. Safety goggles
  8.1.3. Ear plugs
  8.1.4. Flashlight/trouble light

- 8.1.5. 8-ft step ladder
- 8.2. Hand tools
  - 8.2.1. Shovel (square mouth)
  - 8.2.2. Level (12″ to 24″)
  - 8.2.3. Tape measures (10 ft and 100 ft)
  - 8.2.4. Conduit and associated tools (as required)
  - 8.2.5. Hammer
  - 8.2.6. Small sledge hammer – to drive stakes or grounding rod
  - 8.2.7. Open-end wrenches (7/16″, 1/2″, 9/16″)
  - 8.2.8. Socket wrench and 7/16″ deep well socket for radiation shields
  - 8.2.9. Allen wrench set (0.05″) for LI2003S – one is supplied with LI2003S
  - 8.2.10. Straight-bit screwdrivers (small, medium, and large)
  - 8.2.11. Phillips screwdrivers (small and medium)
  - 8.2.12. Pliers and needle-nose pliers
  - 8.2.13. Channel locks
  - 8.2.14. (2) Small diagonal side-cutters
  - 8.2.15. Wire strippers
  - 8.2.16. (2) Adjustable (crescent) wrenches
  - 8.2.17. (2) Files – flat rat tail
  - 8.2.18. Hacksaw
  - 8.2.19. Pocket knife
  - 8.2.20. Sheet metal cutters (tin snips)
  - 8.2.21. Nylon cable ties
  - 8.2.22. Enclosure supply kit (CSI P/N 7363)
  - 8.2.23. Electrician’s putty
  - 8.2.24. Bailing wire
  - 8.2.25. Lag screws to mount enclosure
  - 8.2.26. Wire (multi-color)
  - 8.2.27. Caulking gun and caulk
  - 8.2.28. Long bolts, washers, nuts
  - 8.2.29. Self tapping sheet metal screws
  - 8.2.30. Grabber screws
  - 8.2.31. Spare nuts, washers, U-bolts, etc.
- 8.3. Soldering tools
  - 8.3.1. Soldering iron
  - 8.3.2. Cigarette lighter
  - 8.3.3. Solder
  - 8.3.4. Distilled water
  - 8.3.5. Heat shrink assortment
  - 8.3.6. Electrical tape
- 8.4. Power tools
  - 8.4.1. Variable speed drill
  - 8.4.2. Drill bits and screw/nut driver bits
  - 8.4.3. Hammer drill
- 8.5. Electrical equipment

  - 8.5.1. Multi-meter
  - 8.5.2. Calculator
  - 8.5.3. Portable scope
  - 8.5.4. Earth resistance checker
  - 8.5.5. Break-out box
  - 8.5.6. PC with LoggerNet® and DevConfig programs
  - 8.5.7. Spare cables
- 8.6. Calibration equipment
  - 8.6.1. Brunton compass or solar sighter (for wind direction alignment)
  - 8.6.2. Handheld GPS unit
  - 8.6.3. Voltage calibrator
- 8.7. Miscellaneous
  - 8.7.1. Locks and keys
  - 8.7.2. Back-up battery and battery cables
  - 8.7.3. 2 RF antennas
  - 8.7.4. Dessicant packages
- 8.8. Documentation
  - 8.8.1. Data logger program listing, manual and prompt sheet
  - 8.8.2. Sensor manuals
  - 8.8.3. Site monitoring plan
  - 8.8.4. Mounting angle for solar panel
  - 8.8.5. Station log and pen
  - 8.8.6. Felt-tipped marking pen
  - 8.8.7. Declination angle for the site
- 8.9. Communication equipment
  - 8.9.1. Two head-held radios
  - 8.9.2. Cellular phone
  - 8.9.3. Hand set (for site with phone line)
  - 8.9.4. Appropriate phone numbers
- 8.10. Equipment and sensors to be installed
  - 8.10.1. NEMA box with CR1000 data logger, CS100 Barometer, XStream® RS-232/485 RF Modem, and CompactFlash memory card
    - 8.10.1.1. Antenna for modem
  - 8.10.2. LiCOR 200 solar radiation sensor and mounting fixture and bracket (SOP E3)
  - 8.10.3. Vaisala HMP45C RH/T sensor in passive radiation shield (SOP W1)
  - 8.10.4. Surface wetness grid and mounting bracket (SOP W3)
  - 8.10.5. (3) RMYoung 81000 sonic anemometers, each with XStream® RS-232/485 RF Modem and antenna (SOP W2)
  - 8.10.6. CSI 107-L Thermistor (SOP L2)
  - 8.10.7. Innovative Sensors CSIM11 ORP redox state sensor (SOP L3)
  - 8.10.8. Innovative Sensors CSIM11 pH sensor (SOP L1)

9. Procedures

- 9.1. Acceptance
  - 9.1.1. Criteria for the following sensors are specified in their respective SOP.
    - 9.1.1.1. Barometric pressure (SOP W5)
    - 9.1.1.2. Surface wetness (SOP W3)
    - 9.1.1.3. Solar radiation (SOP W4)
    - 9.1.1.4. Air T/RH (SOP W1)
    - 9.1.1.5. Lagoon T (SOP L1)
    - 9.1.1.6. Lagoon pH (SOP L2)
    - 9.1.1.7. Lagoon redox state (SOP L3)
    - 9.1.1.8. Sonic anemometer (SOP W2)
  - 9.1.2. Criteria for data logger (Fig. 1)
    - 9.1.2.1. Startup self-test of data logger indicates satisfactory operation (See manual).
    - 9.1.2.2. Use voltage calibrator.
    - 9.1.2.3. Check data logger output for an 5 VDC input on each SE analog channel. Most measurements are single-ended voltage measurements.
      - 9.1.2.3.1. Manufacturer's accuracy specifications
        - 9.1.2.3.1.1. At 5V, accuracy is ±0.1% of reading +1.332 mV (offset for single-ended measurement) across -25° to +50°C. For 5VDC input, factory accuracy is +/- 6.3 mV
      - 9.1.2.3.2. Set data logger to read single-ended voltages at 5-s intervals for ≥5 min and report mean of measurements.
      - 9.1.2.3.3. Attach high and low end of voltage calibrator to each a ground terminal and a single-ended input terminal
        - 9.1.2.3.3.1. Record mean value on Calibration form (Appendix A).
        - 9.1.2.3.3.2. Calculate error as (5 V - mean output voltage), and record on form.
      - 9.1.2.3.4. Repeat 9.1.2.3.2 until all single-ended measurement channels are evaluated.
      - 9.1.2.3.5. If output on all channels is within specifications, the data logger is acceptable.
      - 9.1.2.3.6. If output on any channel is outside specifications, contact the manufacturer for resolution of problem.
- 9.2. Installation
  - 9.2.1. Configure data logger:
    - 9.2.1.1. Uses LoggerNet® software
    - 9.2.1.2. Connect data logger RS232 and PC COM port with modem cable.
    - 9.2.1.3. Set team 'East' or 'West' as "Station Name".
    - 9.2.1.4. Set and record passwords for three levels of security, and send to PM, DM and QAM.
    - 9.2.1.5. Conduct configuration procedures according to data logger manual.
  - 9.2.2. Configure RF Modems
    - 9.2.2.1. Connect a serial cable from XStream Modem to COM port of a laptop PC.
    - 9.2.2.2. Provide power to radio modem by connecting power cable to power connector and switching on power switch (Fig. 2).

- 9.2.2.3. Verify power to modem by checking red light on modem (Fig. 2):
- 9.2.2.4. Using Windows HyperTerminal® program on the PC, verify Modem configuration: 8 data bits, 1 stop bit, no parity, 19,200 baud.
- 9.2.2.5. Press program button on modem for one second, and set modem channel to address "0" (Table 1) under selection #1 "Standard settings" See RF400 Radio modem instruction manual (Pg. 21, Section 5.3.1).
- 9.2.2.6. Press "5" to save parameters and exit.
- 9.2.2.7. Disconnect modem cable from PC and modem.
- 9.2.2.8. Remove power from modem.

9.2.3. Install 3-m meteorological mast (SOP U5)

- 9.2.3.1. In horizontal position install sensors on mast as follows:
  - 9.2.3.1.1. Solar radiation sensor and mounting arm (SOP W4) at 3-m height.
  - 9.2.3.1.2. RM Young sonic anemometer (SOP W2) at the top of mast (SOP U5). It communicates directly with the 'LAN server' PC in the instrumentation trailer (SOP U4) according to SOP W2.
  - 9.2.3.1.3. Lightning rod and ground wire.
  - 9.2.3.1.4. RF antenna and RF signal wire to antenna.
- 9.2.3.2. With mast in vertical position, install NEMA box at 1.5-m height.
  - 9.2.3.2.1. Install data logger and CompactFlash card in NEMA box.
  - 9.2.3.2.2. Install barometric pressure sensor in NEMA box (SOP W5).
  - 9.2.3.2.3. Install RF modem into NEMA box.
  - 9.2.3.2.4. Connect data logger to RF Modem.
  - 9.2.3.2.5. Install radiation shielded RH/T probe (SOP W1), at 2-m height (1 m above berm).
  - 9.2.3.2.6. Install wetness grid (SOP W3) facing north at 2-m height.
  - 9.2.3.2.7. Connect all cables for the following sensors to the data logger in accordance with their SOPs (Section 9.1.1, Table 2): barometric pressure, surface wetness, solar radiation, air RH/T, lagoon T, lagoon pH, and lagoon redox state.
  - 9.2.3.2.8. Connect RF cable to RF modem.
  - 9.2.3.2.9. Install ground rod with sledge hammer, connect ground wire from ground rod (using football clamp) to data logger, and test quality of earth ground with earth resistance tester.
    - 9.2.3.2.9.1. If resistance exceeds 2 ohms, check wire connections and firmness of ground rod in ground. Tighten or replace as indicated.
  - 9.2.3.2.10. Attach 120VAC to data logger according to data logger manual. Establish communications.

9.2.4. Verify communications between data logger and LAN server computer (SOP D1).

- 9.2.4.1. Using LoggerNet® software, establish communications with data logger.
  - 9.2.4.1.1. Link COM port, modem, and data logger.
  - 9.2.4.1.2. Set data logger clock and automatic clock update schedule.
    - 9.2.4.1.2.1. Clock will be automatically updated to LAN server computer time each night.
  - 9.2.4.1.3. Set automatic 1-h download cycle from data logger to LAN server computer.
  - 9.2.4.1.4. Set filename for data downloads.

      - 9.2.4.1.5. Load data logger program (Appendix B) into data logger.
  - 9.2.5. Verify communications between anemometer and LAN server computer (SOP D1), following the procedure in SOP W2.
- 9.3. Operation
  - 9.3.1. Data logger measurements of most parameters are made at 5-s intervals.
    - 9.3.1.1. The following measurements are sampled every 5 s, and averaged every 5 min: barometric pressure, surface wetness, solar radiation, air T, lagoon T, lagoon pH and lagoon redox state.
    - 9.3.1.2. The following measurements are sampled every 5 min: air RH/T.
    - 9.3.1.3. Measurements are stored on data logger CompactFlash memory (SOP D1).
      - 9.3.1.3.1. CompactFlash memory mounted in Compact Flash Module (Fig. 3).
      - 9.3.1.3.2. There is one LED that indicates module status (Fig. 3).
      - 9.3.1.3.3. LED flashes red when card is being accessed.
      - 9.3.1.3.4. LED is solid green when it is OK to remove compact card.
      - 9.3.1.3.5. LED is solid orange if there is an error in operation.
    - 9.3.1.4. Averaged and sampled measurements transmitted to ‘LAN server’ (SOP D1).
    - 9.3.1.5. Values are visualized on LoggerNet® software on ‘LAN server’ PC
  - 9.3.2. Wind component measurements are made at 16 Hz.
    - 9.3.2.1. Measurements of the x (U), y (V), and z (W) wind direction components
    - 9.3.2.2. Measurements are conducted as described in SOP W2.
    - 9.3.2.3. Data are transmitted to ‘LAN server’ using XStream Modems (SOP D1).
      - 9.3.2.3.1. Measurements are stored on ‘LAN server’ PC (SOP D1).
    - 9.3.2.4. Values can be visualized on LabView® software on ‘LAN server’ PC
- 9.4. Downloading data from data logger
  - 9.4.1. LoggerNet® software will automatically download data every hour into LAN server computer (SOP D1).
    - 9.4.1.1. Software controls data download
- 9.5. Exchanging compact flash cards.
  - 9.5.1. Removing compact flash card.
    - 9.5.1.1. Press CONTROL button on Compact Flash Module (Fig. 3).
    - 9.5.1.2. Status LED will turn green when ready.
    - 9.5.1.3. Unscrew Compact Flash Module lid with screwdriver or hand.
    - 9.5.1.4. Remove card.
    - 9.5.1.5. If card is not removed within 20 s, it will be reactivated, and data on card will be written over. Remove card before this occurs.
  - 9.5.2. Loading compact flash card.
    - 9.5.2.1. Open Compact Flash Module lid with screwdriver or hand (Fig. 3).
    - 9.5.2.2. Insert Compact Flash Card into Compact Flash Module.
    - 9.5.2.3. Close Compact Flash Module and tighten screw by hand.
- 9.6. Troubleshooting
  - 9.6.1. If communications between data logger and trailer PC has failed:
    - 9.6.1.1. Check power to modems, PC, and data logger. If no power, check circuit breakers in trailer; reset breaker if off.

- 9.6.1.2. Check connections between RF modems and antennae for firm connection. Correct as needed.
  - 9.6.1.2.1. Check communications.
  - 9.6.1.2.2. Verify communications link between radio modems (Yellow and Green lights intermittently lit during communications) on modems (Fig. 2).
  - 9.6.1.2.3. Verify firm connections for power, signal, and antenna for serial communications. Correct as needed.
  - 9.6.1.2.4. Verify power at modems in trailer and at anemometer, as indicated by a lit red light on the modems (Fig. 2).
    - 9.6.1.2.4.1. If not within acceptable range, check continuity of wires and tightness of cable connections. Correct as needed.
    - 9.6.1.2.4.2. If all connections are tight and of good continuity, send sensor to manufacturer for repair and/or replacement of the modem.
  - 9.6.1.2.5. Verify destination address of radio modems (Table 1). Correct as needed.
  - 9.6.1.2.6. Check that the correct baud rate is selected (Section 9.2.2). Correct as needed.
- 9.6.2. If communications between data logger and PC is working, but data logger is not indicating data being logged:
  - 9.6.2.1. Check program (See Appendix B) for accuracy. If necessary, reload program according to data logger manual
  - 9.6.2.2. Check battery voltage of data logger.
    - 9.6.2.2.1. If necessary, replace battery and check for problem with sensor wiring.
    - 9.6.2.2.2. If problems persist, contact manufacturer for repair or replacement.
  - 9.6.2.3. If input table of data logger indicates invalid measurements, Check appropriate sensor SOP for possible corrective measures.
- 9.6.3. If communications between sonic anemometer and trailer PC is not working, follow procedures in SOP W2.
- 9.6.4. If communications between sonic anemometer and trailer PC is working, but no data is being collected at the PC, follow procedures in SOP W2.

9.7. Data acquisition, calculations & data reduction requirements

- 9.7.1. Data from RH, air T, lagoon T, barometric pressure, surface wetness, solar radiation, lagoon pH and lagoon redox state will be logged on the CR1000 data logger.
  - 9.7.1.1. Calculations and data reduction for each sensor are indicated in SOPs W1, W3, W4, W5, L1, L2, and L3.
  - 9.7.1.2. Data logger is programmed using a BASIC-like language specifically designed by the manufacturer.
  - 9.7.1.3. Communications between data logger and trailer PC (radio telemetry, SOP D1).
- 9.7.2. Data from sonic anemometers is stored on ‘LAN server’ PC.
  - 9.7.2.1. Calculations and data reductions for each sensor are in SOP W2.
  - 9.7.2.2. Communications between sensors and trailer PC uses radio telemetry (SOP D1).

9.8. Computer hardware & software

- 9.8.1. Data processing is conducted by the network LAN server (SOP D1).
  - 9.8.1.1. Processing of measurements reported by data logger uses LoggerNet® program.
  - 9.8.1.2. Processing of measurements reported by the sonic anemometers uses Labview® program.

10. Data and Records Management

10.1. Data logger
  10.1.1. Measurements of RH, air T, lagoon T, barometric pressure, surface wetness, solar radiation, lagoon pH and lagoon redox state
  10.1.2. Data is stored in a FIFO memory and on a 64Mb CompactFlash® memory card, which is typically filled in about 20 d.
  10.1.3. Three 5-digit security passwords are needed to change the data acquisition program, download data, or change the clock.
  10.1.4. The NEMA box is locked to limit unauthorized entry.
10.2. Network LAN server-data acquisition task
  10.2.1. Measurements from sonic anemometers (SOP W2) are stored in on-site network server PC.
  10.2.2. Data is stored in the LAN server, and transferred to PAML (SOP D1).

11. Quality Control and Quality Assurance

11.1. Perform the following QC actions after the initial setup of the site, before departing grounds for an extended period, and in the event of a failure of any piece of equipment that requires technical personnel to be dispatched to the site or results in a critical loss of data (including failures caused by severe weather, vandalism, lightning/power surges, or negligence):
  11.1.1. Inspection of site grounds near instrument
    11.1.1.1. Visually check the site grounds around the tower for hazards such as rodent burrows, buried conduit trench settling, and insect nests.
    11.1.1.2. Log inspection results by checklist with comments and remediation.
  11.1.2. Tower structures
    11.1.2.1. Check that all masts and towers are vertical and all guy wires, turnbuckle and tower base bolts are tight.
    11.1.2.2. Check for broken or cracked tower structure components. Inspect lightning rod ground wire for tightness and damage.
    11.1.2.3. Log inspection results by checklist with comments and remediation.
  11.1.3. Sensors
    11.1.3.1. Check that all sensors are tightly attached to towers at appropriate heights.
    11.1.3.2. Check broken, pinched, or cracked cables between sensors and NEMA box.
    11.1.3.3. Log inspection results by checklist with comments and remediation.
  11.1.4. NEMA box
    11.1.4.1. Check that the box is locked
    11.1.4.2. Check that the box is secured tightly to the meteorological tower.
    11.1.4.3. Check for wet dessicant in box and replace if indicator is pink.
    11.1.4.4. Check for any indications of broken or cracked NEMA box.
    11.1.4.5. Inspect grounding wire and lightning rod ground wire for tightness and damage.
    11.1.4.6. Log inspection results by checklist with comments and remediation.

11.1.5. Data logger

11.1.5.1. Check that all wires are correctly and firmly connected to the correct terminals of the data logger (Table 2).

11.1.5.2. Check that correct destination address (Table 1) is in RF Modem.

11.2. Perform the following QC actions every six months, and in the event of a failure of the data logger that requires technical personnel to be dispatched to the site or results in a critical loss of data from severe weather, lightning or power surges:

11.2.1. Evaluate accuracy of all single-ended voltage measurement channels according to Section 9.1.2.3..Record results on Calibration Form (Appendix A).

11.2.1.1. If problem cannot be resolved in field, then return datalogger to manufacturer for repair and recalibration.

12. References

12.1. ARM, Atmospheric Radiation Monitoring Network: Standard Operating Procedures

12.2. Campbell Scientific Inc. 2004. CR1000 Measurement and Control System Operators Manual, preliminary, Logan, UT.

12.3. Campbell Scientific Inc. 2005. LoggerNet Instruction Manual. Logan, UT.

12.4. Tripod Manual. 2005. Tripod installation manual, Models CM110, CM115, CM120, Campbell Scientific Inc., Logan, UT.

12.5. Crank-Up Tower Manual. Model LPT Towers Manual. FORCE12, Inc., Pas Robles, CA.

12.6. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV-Meteorological Measurements, U.S. EPA, August 1989.

12.7. IMPROVE, Particulate Monitoring Network: Standard Operating Procedures, University of California, Davis.

12.8. Xstream Model Manual. 2005. XStream-PKG-R RS-232/485 RF Modem. Product Manual v4.2B, Maxstream, Lindon, UT.

12.9. SOP D1. 2006. Management of Open Source, Weather and Lagoon-Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.

12.10. SOP E3. 2006. The Measurement of Solar Radiation with the Licor Model 200SL Silicon Pyranometer. Standard Operating Procedure E3. Purdue Ag Air Quality Lab.

12.11. SOP L1. 2006. Measurement of Lagoon pH with Innovative Sensors Model CSIM11 Sensor. Standard Operating Procedure L1. Purdue Applied Meteorology Lab.

12.12. SOP L2. 2006. Measurement of Lagoon Temperature with Campbell Scientific Model 107-L Thermistor. Standard Operating Procedure L2. Purdue Applied Meteorology Lab.

12.13. SOP L3. 2006. Measurement of Lagoon Redox State with Campbell Scientific CSIM11_ORP Sensor. Standard Operating Procedure L3. Purdue Applied Meteorology Lab

12.14. SOP U4. 2006. Open-source Instrument Trailer. Standard Operating Procedure U4. Purdue Applied Meteorology Lab.

12.15. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.

12.16. SOP W1. 2006. Measurement of Atmospheric Temperature and Humidity with the Vaisala Model HMP45C Sensor and Solar Shield. Standard Operating Procedure W1. Purdue Applied Meteorology Lab.
12.17. SOP W2. 2006. Measurement of Wind with the RM Young Model 81000 3-Dimensional Sonic Anemometer. Standard Operating Procedure W2. Purdue Applied Meteorology Lab.
12.18. SOP W3. 2006. Measurement of Wetness with the Campbell Scientific Model Resistance Grid. Standard Operating Procedure W3. Purdue Applied Meteorology Lab.
12.19. SOP W5. 2006. The Measurement of Barometric Pressure with the Setra Model 278 (Campbell Scientific CS100) Barometer. Standard Operating Procedure W5. Purdue Applied Meteorology Lab.

13. Contacts

13.1. Data logger manufacturer: Campbell Scientific, Inc.
Address: 815 W 1800 N, Logan, UT 84321
Technical support: 435-753-2342
13.2. Modem manufacturer: Maxstream, Inc.
Address: 355 S 520 W, Suite 180, Lindon, UT 84042
Phone: (801) 765-9885
Fax: (801) 765-9895
E-mail: rf-xperts@maxstream.net

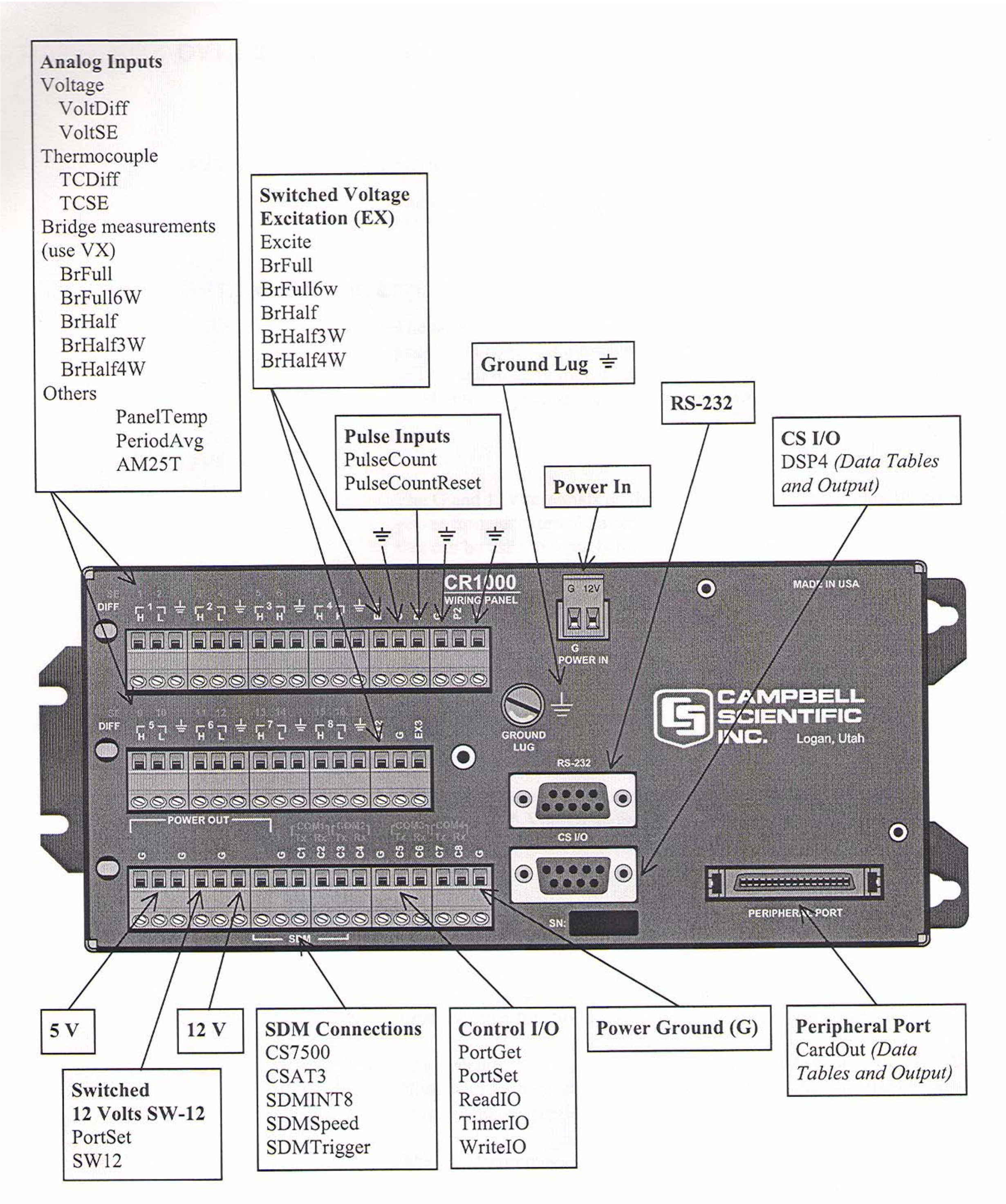

Figure 1. CR1000 Data Logger layout.

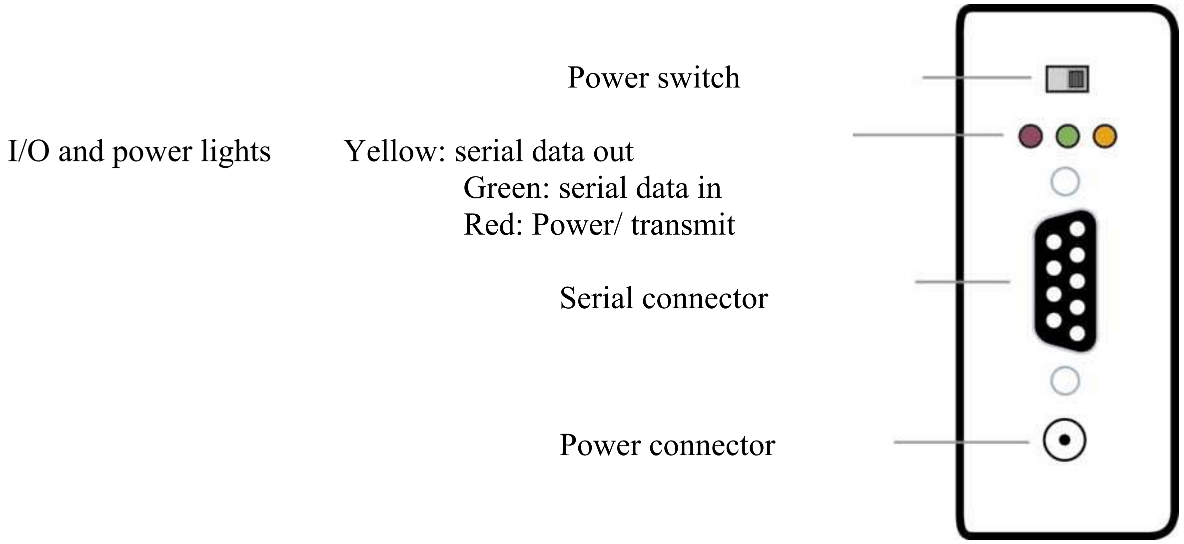

_Figure 2. Radio modem connectors and indicators.

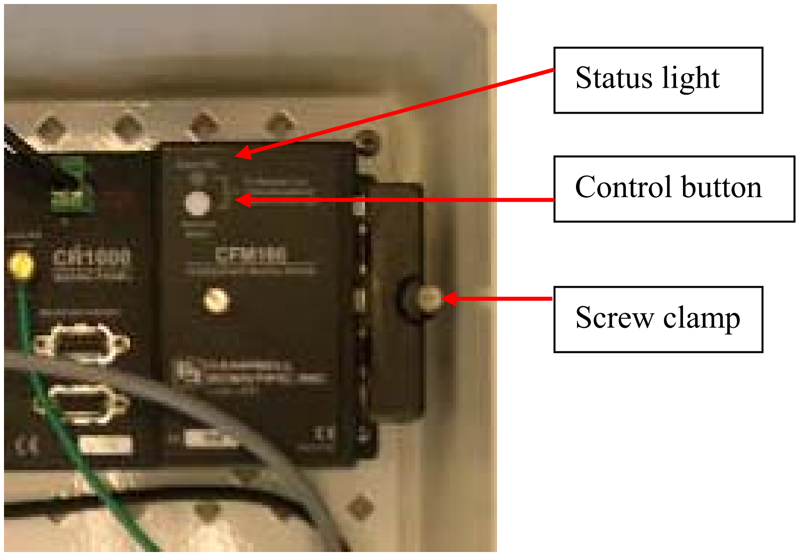

Figure 3. Compact Flash module, as mounted on CR1000 datalogger peripheral port (Fig. 1). Locations of the Status light, control button, and screw clamp are indicated.

Table 1. Radio modem communications channels.

Device Radio Is Connected to	Radio address *
CR1000 data logger	0
2m sonic anemometer	1
8m sonic anemometer	2
16m sonic anemometer	3
UV-DOAS -1	4
UV-DOAS -2	5
TDLAS – 1	6
TDLAS Scanner-1	7
TDLAS – 2	8
TDLAS Scanner-2	9

* Note: leave network address at “0”

Table 2. Wiring diagram for meteorological and lagoon characterization sensors.

Sensor	Wire color	Connection needed	Terminal on Data logger
Barometric pressure sensor	Red	+12VDC	
	Clear	Analog ground	
	Green	Ext control	C1
	Blue	SE	SE 1 (DE 1 H)
Wetness grid	Black	5VDC excitation	EX1
	Purple	Ground	
	Red	SE	SE 2 (DE 1 L)
LiCOR 200 solar radiation sensor	Red/Center	SE	SE 3 (DE 2 H)
	Black/Shield	Analog ground	
HMP 45C RH and air temperature sensor	Clear	Ground	
	Red	Switched 12VDC	SW12
	Black	Ground	
	Blue	SE	SE 4 (DE 2 H)
	Yellow	SE	SE 5 (DE 3 H)
	White	Analog ground	
Lagoon water temperature	Black	Switched EX	EX2
	Purple	Ground	
	Clear	Ground	
	Red	SE	SE 6 (DE 3 L)
Lagoon Ph sensor CSIM11	Brown	Analog ground	
	Red	+ DE	DE 4 H
	Green	- DE	DE 4 L
Lagoon redox sensor CSIM11-ORP	Brown	Analog ground	
	Red	+ DE	DE 5 H
	Green	- DE	DE 5 L

APPENDIX A
CR1000 Data logger Quality Assurance Form

Instrument Serial Number:__________________________________

Date (MM/DD/YYYY): ____/_____/_____ Calibrator: _______________________________

Begin time (HrHr:MinMin) _____________ End time (HrHr:MinMin) _____________

For 5VDC input, factory accuracy: +/- 6.3 mV

SE Channel	Recorded voltage Mean value	Error (5-mean)	Within factory accuracy (+/- 6.3 mV)? (Y/N)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
12			
13			
14			
15			
16			

APPENDIX B
CR1000 Data logger Program

```
'CR1000
'Created by RHGrant

'Declare Variables and Units
Dim pHMult_10
Public Batt_Volt
Public BP_kPa
Public LW_kOhm
Public SlrW
Public SlrMJ
Public AirTC
Public RH
Public T107_C
Public pH
Public ORP

Units Batt_Volt=Volts
Units BP_kPa=kPa
Units LW_kOhm=kOhms
Units SlrW=W/m²
Units SlrMJ=MJ/m²
Units AirTC=Deg C
Units RH=%
Units T107_C=Deg C
Units pH=pH
Units ORP=mV

'Define Data Tables
DataTable(Table1,True,-1)
Cardout(0,-1)
      DataInterval(0,15,Min,10)
      Average(1,BP_kPa,FP2,False)
      Average(1,LW_kOhm,FP2,False)
      Average(1,SlrW,FP2,False)
      Average(1,AirTC,FP2,False)
      Sample(1,AirTC,FP2)
      Sample(1,RH,FP2)
      Sample(1,T107_C,FP2,False)
      Sample(1,pH,FP2,False)
      Sample(1,ORP,FP2,False)
      sample(1,Batt_Volt,FP2,False)
EndTable

'Main Program
BeginProg
      Scan(5,Sec,1,0)
            'Default Datalogger Battery Voltage measurement Batt_Volt:
            Battery(Batt_Volt)
            'CS100 Barometric Pressure Sensor measurement BP_kPa:
```

```
            PortSet(1,1)
            VoltSE(BP_kPa,1,mV2500,1,1,0,_60Hz,0.2,600.0)
            BP_kPa=BP_kPa*0.1
            '237 Leaf Wetness Sensor measurement LW_kOhm:
            BrHalf(LW_kOhm,1,mV250,2,Vx1,1,250,True,0,250,1,0)
            LW_kOhm=1/LW_kOhm-101
            'LI200S Pyranometer measurements SlrMJ and SlrW:
            VoltDiff(SlrW,1,mV7_5,2,True,0,_60Hz,1,0)
            If SlrW<0 Then SlrW=0
            SlrMJ=SlrW*0.001
            SlrW=SlrW*200.0
            'HMP45C (7-wire) Temperature & Relative Humidity Sensor
measurements AirTC and RH:
            PortSet(2,1)
            Delay(0,150,mSec)
            VoltSE(AirTC,1,mV2500,5,0,0,_60Hz,0.1,-40.0)
            VoltSE(RH,1,mV2500,6,0,0,_60Hz,0.1,0)
            PortSet(2,0)
            If RH>100 And RH<108 Then RH=100
            '107 Temperature Probe measurement T107_C:
            Therm107(T107_C,1,7,1,0,_60Hz,1.0,0.0)
            'CSIM11 pH Probe (ISI M11-pH) measurement pH:
            'Calculate temperature correction multiplier
            pHMult_10=1/(((T107_C+273)/298)*59)
            'Make pH measurement
            VoltDiff(pH,1,mV2500,5,True,0,_60Hz,pHMult_10,7.0)
            'CSIM11 ORP Probe (ISI M11-ORP) measurement ORP:
            VoltDiff(ORP,1,mV2500,6,True,0,_60Hz,1,0.0)
            'Call Data Tables and Store Data
            CallTable(Table1)
      NextScan
EndProg
```