

METHOD 8430

ANALYSIS OF BIS(2-CHLOROETHYL) ETHER AND HYDROLYSIS PRODUCTS BY DIRECT AQUEOUS INJECTION GAS CHROMATOGRAPHY/FOURIER TRANSFORM- INFRARED

1.0 SCOPE AND APPLICATION

1.1 This method provides procedures for the identification and quantitation of bis(2-chloroethyl)ether and its hydrolysis compounds in aqueous matrices by direct aqueous injection (DAI) into a gas chromatograph with detection by a Fourier transform infrared spectrometer (GC/FT-IR). The following compounds can be determined by this method:

Compound Name	Abbreviation	CAS Number ^a
Bis(2-chloroethyl)ether	BCEE	111-44-4
2-Chloroethanol	CE	107-07-3
2-(2-Chloroethoxy)ethanol	2CEE	628-89-7
Diethylene glycol	DEG	111-46-6
Ethylene glycol	EG	107-21-1

^a Chemical Abstract Service Registry Number.

1.2 Although the initial study upon which this method is based targeted only the bis(2-chloroethyl)ether and its hydrolysis compounds, it has been suggested that this method can be used for the identification of compounds that are generally non-extractable, highly water soluble, thermally stable, and do not co-elute with water from the gas chromatography (GC). Possible analytes include ethers and alcohols.

1.3 The minimum identifiable quantities (MIQ) for the five compounds in organic-free reagent water range from a low of 46 ng for BCEE, to a high of 120 ng for EG (See Sec. 13.2 for MIQ definition). The MIQ for a specific sample may differ depending on the nature of the interferences in the sample matrix and the amount of sample used for the analysis.

1.4 This method is restricted to use by or under the supervision of analysts experienced in the use of a GC, the interpretation of FT-IR spectra and the use of continuous data collection systems. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Water samples are filtered through a 0.45- μ m filter, and 1- μ L aliquots are injected directly into a GC. The GC is equipped with two detectors, a thermal conductivity detector (TCD) and an FT-IR. During analysis, the analyst disconnects the FT-IR from the system to prevent aqueous degradation of the potassium bromide (KBr) window, using an eight-port switching valve. Further analysis of either the solid precipitate from the filtering step or the aqueous filtrate for trace amounts of non-water soluble compounds may be done by extracting the samples using appropriate 3500 series methods.

3.0 DEFINITIONS

Refer to Chapter One, the individual determinative methods, and the manufacturer's instructions for definitions that may be relevant.

4.0 INTERFERENCES

4.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks.

4.1.1 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. It should then be drained dry, and heated in a laboratory oven at 130 °C for several hours before use. Solvent rinsing with methanol may be substituted for the oven heating. After drying and cooling, glassware should be stored in a clean environment to prevent any accumulation of dust or other contaminants.

4.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

4.2 Matrix interferences may be caused by contaminants that are in the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the matrix being sampled. If significant interferences occur in subsequent samples, additional cleanup may be necessary.

4.3 The extent of interferences that may be encountered in this method has not been fully assessed. Although the GC conditions described allow for a unique resolution of the specific compounds covered by this method, other matrix components may interfere.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals and instrumentation included in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/FT-IR system

6.1.1 GC – Temperature-programmable oven equipped with a cool on-column injection port, a purged splitless injection port, or an equivalent suitable for capillary glass columns

6.1.2 Column – 30 m DB-wax, 1.0 μm film, Megabore (J&W Scientific), or equivalent

6.1.3 Detectors

6.1.3.1 TCD – Must be able to handle temperatures up to 300 $^{\circ}\text{C}$

6.1.3.2 FT-IR spectrometer – System should be equipped with a mercury-cadmium-telluride detector, using a light-pipe interface with KBr windows (i.e., Digilab Model GC/C32 or equivalent). Resolution of 8 cm^{-1} and a range of 4,000 to 7,000 cm^{-1} is required. The light-pipe interface and transfer lines should be contained in a heated system (up to 250 $^{\circ}\text{C}$) to prevent sample condensation. Extra transfer lines will be needed for the switching system.

6.1.4 Detector switching system – Eight-port stainless steel GC rotary switching valve, with an inert interval surface (e.g., front end processor (FEP)) and capable of withstanding temperatures up to 300 $^{\circ}\text{C}$. Ideally, the valve should be mounted inside the GC oven with external control. If internal mounting is not possible, then use of an external heated valve enclosure may be employed. This allows direct injection of aqueous samples by using the valve system to route water away from the FT-IR KBr window, which water rapidly destroys.

6.1.5 Data collection – Each detector should have its own signal recorder.

6.1.5.1 TCD signal – Either an analog strip chart recorder or a digital computerized data collection system is acceptable.

6.1.5.2 FT-IR signal – The continuous collection of the spectrometer's signal will require a computerized data system with the ability to continuously collect spectra at a rate of 4 scans/sec and add the 4 scans to produce a data point for each second. To confirm the identity of target compounds, a library of vapor phase spectra should be established.

6.2 Glassware

6.2.1 Glass funnels

6.2.2 Volumetric flasks (square) – various sizes

6.2.3 Pipettes (A grade) – various sizes

6.2.4 Sample vial with polytetrafluoroethylene (PTFE)-lined lids

6.3 Syringes – 10 μL , suitable for GC work

6.4 Analytical balance, accurate to ± 0.0001 g

6.5 Vacuum filtration apparatus – 0.45 μm filter and clean glass flasks that are able to hold at least 1 L of liquid

7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water – All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

7.3 Helium gas – Suitable for gas chromatography

7.4 Methanol, CH₃OH – Pesticide reagent grade, nanograde, or equivalent

7.5 Stock standard solutions

7.5.1 Prepare, in organic-free reagent water, a stock standard solution containing all of the target analytes at a concentration of 1000 mg/L. Record the actual weight of each compound added and calculate the actual concentration of each component of the solution.

7.5.2 Transfer the stock standard solution into a PTFE-sealed screw-cap bottle for storage. Store at ≤6 °C and protect from light. Check the solution periodically for signs of degradation or evaporation. This solution must be replaced every three months, or when any sign of degradation or evaporation is observed.

7.6 Calibration standards

7.6.1 Calibration standards at 500, 250, 100, 50, and 25 mg/L, from the aqueous stock standard solution, by appropriate volumetric dilutions with water are suggested. Store as specified in Sec. 7.5.2. The calibration solutions should not be made by serial dilution of a single solution.

7.6.2 Since the levels of detection for the target compounds in water have not been established, the suggested calibration curve concentrations may be modified, depending on matrix interferences and sensitivity of equipment. In general, the calibration curve should span at least one order of magnitude and the working range should bracket the analyte concentration.

7.7 Spiking solution – The analyst should monitor the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix, by spiking a known amount of the target analytes into blanks or into matrix spike samples. A suggested preliminary concentration of the spiking stock solution is 100 g/L. It is recommended that the spiking concentration should be set at 1 to 5 times higher than the background concentration determined for that matrix.

7.8 Surrogate spiking solution – To monitor the performance of the method for all samples, a minimum of one surrogate compound should be chosen by the laboratory. This compound should be diluted with water to an appropriate concentration and added to all samples, method blanks, matrix spikes, and calibration standards.

8.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

- 8.1 See Chapter 4, Organic Analytes, Sec. 4.1 for more information on this topic.
- 8.2 Samples should be stored at ≤ 6 °C and protected from light.
- 8.3 Samples should be filtered and analyzed within 14 days of collection.

9.0 QUALITY CONTROL

9.1 General guidance

Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving collection of analytical data should include development of a structured and systematic planning document, such as a quality assurance project plan (QAPP) or a sampling and analysis plan (SAP), which translates project objectives and specifications into directions for those who will implement the project and assess the results.

Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged, as described in Sec. 9.6 of Method 8000. Use of instrument-specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and QC data should be maintained for reference or inspection. QC to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

9.2 Initial demonstration of proficiency (IDP)

Prior to implementation of a method, each laboratory must perform an IDP consisting of at least four replicate reference samples spiked into a clean matrix taken through the entire sample preparation and analysis. Whenever a significant change to instrumentation or procedure occurs, the laboratory must demonstrate that acceptable precision and bias can still be obtained by the changed conditions. Whenever new staff members are trained, an analyst IDP must be performed.

9.2.1 Demonstration of proficiency for new analysts

Each laboratory should have a training program which documents that a new analyst is capable of performing the method, or portion of the method, for which the analyst is responsible. This demonstration should document that the new analyst is capable of successfully following the standard operating procedure (SOP) established by the laboratory.

9.3 It is recommended that the laboratory adopt additional QA practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.4 Lower limit of quantitation (LLOQ)

The laboratory shall establish the LLOQ as the lowest point of quantitation, which in most cases, is the lowest concentration in the calibration curve. LLOQ verification is recommended for each project application to validate quantitation capability at low analyte concentration levels. This verification may be accomplished with either clean control material (e.g., reagent water, solvent blank, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix, free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated data quality objectives (DQOs).

9.5 QC necessary to evaluate the GC system operation is found in Method 8000 Sec. 11 under Retention Time Windows, Calibration Verification and Chromatographic Analysis of Samples. Refer to Table 1 for FT-IR spectrometer QC requirements.

9.6 Sample QC for preparation and analysis – The laboratory should also have established procedures for documenting the effect of the matrix on method performance (i.e., precision, accuracy, and sensitivity). At a minimum, this includes a method blank, matrix spike, duplicate, and laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample(s).

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate should be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair.

9.6.2 A LCS should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

9.6.3 See Method 8000, Sec. 9 for the details on carrying out sample QC for preparation and analysis.

9.7 Surrogate recoveries – The laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. Currently, surrogate compounds have not been selected for this procedure. See Method 8000, Sec. 9 for information on evaluating surrogate data and developing and updating surrogate limits.

9.8 It is recommended that the laboratory adopt additional QA practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 CALIBRATION AND STANDARDIZATION

10.1 External calibration – Calibration standards may be prepared using the suggested concentrations in Sec. 7.6. Matrix interferences may prevent quantitation at the suggested concentrations. When necessary, the lowest concentration of the calibration curve should be adjusted to be at, or near, the LLOQ. Refer to Method 8000, Sec. 11 for proper external calibration procedures.

10.2 Calibration must take place using the same sample introduction method that will be used to analyze actual samples.

11.0 PROCEDURE

11.1 GC/FT-IR conditions (recommended):

Flow Rate:	2.4 mL/min of helium carrier gas
Run Time:	approximately 15 min
Injection Volume:	1 μ L
Valve Switch Time:	4 min
Temperature Program:	80 °C to 220 °C at 15 °C/min, hold at 220 °C for 10 min
TCD Temperature:	290 °C
GC/FT-IR Interface, Transfer Lines, and Light Pipe Temperature:	220 °C
Scan Time:	4 scans/sec

NOTE: Modifications of these parameters may be necessary to facilitate the separation of certain compounds depending on matrix interferences encountered.

11.2 Refer to Method 8000, Sec. 11 for the establishment of retention time windows.

11.2.1 Analyze a solvent blank to ensure that the system is clean and interference free.

11.2.2 Analyze the 5 calibration standards, starting with the lowest concentration and ending with the highest concentration.

11.2.3 Tabulate the IR absorbance peak area along with the calibration factor (CF) for the analyte at each concentration. Refer to Sec. 11 of Method 8000 for linear and non-linear calibration acceptance criteria. It should be noted that IR transmission is not directly proportional to concentration.

11.2.4 Recheck the instrument calibration each day, before and after an analysis is performed, by analyzing one or more calibration standards. The response obtained should fall within ± 15 percent of the expected value or the instrument must be recalibrated.

11.2.5 After the analysis of 10 or fewer samples, one of the calibration standards must be reanalyzed to ensure that the retention times and the CFs of the target analytes remain within the QC requirements.

11.3 Sample spiking and filtering

11.3.1 Allow the sample to come to ambient temperature. Mark the water meniscus on the side of the 1-L sample bottle for later determination of exact sample volume.

11.3.2 Add 2 mL of the spiking solution to the spiked blank and the matrix spike sample. The final concentration of the added analytes should be approximately 200 mg/L.

11.3.3 Vacuum filter the sample through a 0.45- μ m filter that has been rinsed with organic-free reagent water. The filtrate should be collected in a clean glass bottle. Any particulate collected by the filtering process may be discarded or extracted for analysis of trace amounts of non-water soluble compounds using an appropriate 3500 series method.

11.3.4 Using a pipette, withdraw a 5-mL aliquot of the sample (aqueous filtrate) and place it into a clean glass sample vial with a PTFE-lined lid. This 5-mL aliquot will be the sample used for the direct aqueous injection of the water sample. Store sample extracts at ≤ 6 °C in the dark. The remainder of the aqueous filtrate may be saved in a glass bottle with a PTFE-lined lid until after the analysis is complete or extracted for analysis of trace amounts of non-water soluble compounds using an appropriate 3500 series method.

11.4 GC analysis

11.4.1 Method 8000, Sec. 11 provides instructions on calibration, establishing retention time windows, the analysis sequence, appropriate dilutions, and chromatographic identification criteria.

11.4.2 Determining the valve switching time – The valve switch time must be determined before proceeding with the analysis of samples.

11.4.2.1 Place the GC switching valve in the position which routes the sample to the TCD and away from the FT-IR spectrometer and inject a 1 μ L aliquot of a reagent blank to determine the retention time of water in the system. Use this retention time to determine the optimum valve switch time to cycle water away from the KBr window loop. At the optimum valve switch time, the eight-port valve may be moved to the position which allows use of the FT-IR detector and the TCD in tandem after the bulk of the aqueous portion of the sample has been diverted away from the KBr window.

11.4.2.2 Once the optimum valve switch time is established, the system may be re-evaluated with one of the aqueous calibration standards to assure complete separation of all target analytes.

NOTE: Traces of water in the GC/FT-IR interface are acceptable. However, the repeated injection of 1.0- μ L aqueous samples may eventually destroy the KBr window used for FT-IR detection. Care should be taken in deciding the valve switching time so that the KBr window is exposed to only trace amounts of water and target analytes that elute with retention times just after water are not missed.

11.4.2.3 If the TCD is proven to have adequate sensitivity for a particular analysis, it may be used as the prime detector once the target analytes are identified using the FT-IR detector. When the TCD is used as the prime

detector, the switching valve should remain in the position which diverts the sample away from the KBr window.

11.4.3 Sample analysis – Analyze the samples, blanks, spiked blanks, and spiked matrix samples by injecting 1- μ L aliquots into the GC and switching to the FT-IR spectrometer at the previously determined time. Dilution of the sample may be necessary to adjust the analyte concentration to within the working range of the calibration curve. If dilution is necessary, note which samples were diluted in the final report and make the appropriate calculation adjustments.

11.4.4 GC/FT-IR identification – Visually compare the analyte IR spectrum versus the search library spectrum of the most promising on-line library search hits. Report, as identified, those analytes with IR frequencies for the five (maximum number) most intense IR bands ($S/N \geq 5$) which are within $\pm 5.0 \text{ cm}^{-1}$ of the corresponding bands in the library spectrum. Choose IR bands which are sharp and well resolved. The software used to locate spectral peaks should employ the peak "center of gravity" technique. In addition, the IR frequencies of the analyte and library spectra should be determined with the same computer software.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculations

12.1.1 Calculate the CF for each calibration standard and determine the percent relative standard deviation (%RSD) using the external standard calibration procedure in Sec. 11 of Method 8000.

12.1.2 Calculation of the concentration of analytes using the external standard calibration procedure is provided in Sec. 11 of Method 8000.

12.1.3 Results should be reported in units commensurate with their intended use and all dilutions should be taken into account when computing final results.

13.0 METHOD PERFORMANCE

13.1 No LLOQ data are available.

13.2 Minimum Identifiable Quantities

13.2.1 The MIQ is defined as the minimum quantity that must be injected to result in a spectral match that has the correct compound identification in the top 5 spectral matches. The MIQ will vary depending on instrument sensitivity and sample matrix effects.

13.2.2 The MIQ range for CE, BCEE, EG, 2CEE, and DEG in organic-free reagent water by direct aqueous injection goes from a low of 46 ng for BCEE to a high of 120 ng for EG.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The Environmental Protection Agency (EPA or the Agency) has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, a free publication available from the ACS, Committee on Chemical Safety, http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf.

15.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations and complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the ACS at the web address listed in Sec. 14.2 above.

16.0 REFERENCES

1. Payne, W.D. and Collette, T.W., "Identification of Bis(2-chloroethyl)ether Hydrolysis Products by Direct Aqueous Injection GC/FT-IR," *J. of High Res. Chrom.*, 12, 693-696, 1989.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method.

ANALYSIS OF BIS(2-CHLOROETHYL)ETHER AND HYDROLYSIS PRODUCTS
BY DIRECT AQUEOUS INJECTION

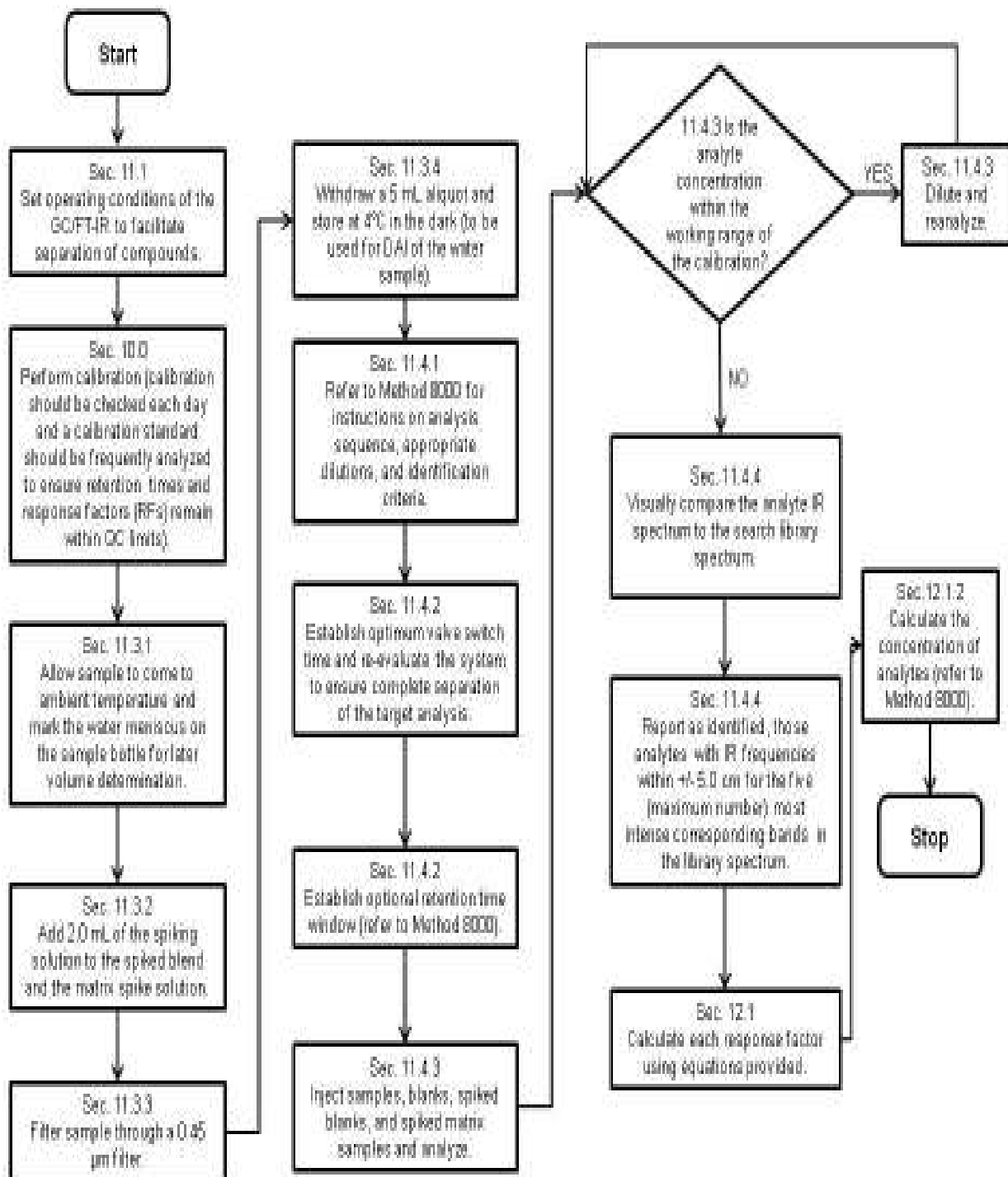


TABLE 1

QC of FT-IR Spectrometer

T.1 Equipment Adjustments and Maintenance

T.1.1 Mirror alignment – Adjust the interferometer mirrors to attain the most intense signal. Data collection should not be initiated until the interferogram is stable. If necessary, align the mirrors prior to each GC/FT-IR run.

T.1.2 Interferometer – If the interferometer is air-driven, adjust the interferometer drive air pressure to manufacturer's specifications.

T.1.3 Lightpipe – The lightpipe and lightpipe windows should be protected from moisture and other corrosive substances at all times. For this purpose, maintain the lightpipe temperature above the maximum GC program temperature but below its thermal degradation limit. When not in use, maintain the lightpipe temperature slightly above ambient. At all times maintain a flow of dry, inert, carrier gas through the lightpipe.

T.1.4 Beamsplitter – If the spectrometer is thermostated, maintain the beamsplitter at a temperature slightly above ambient at all times. If the spectrometer is not thermostated, minimize exposure of the beamsplitter to atmospheric water vapor.

T.2 Centerburst intensity and mercury/cadmium/tellurium (MCT or HgCdTe) detector check

T.2.1 With an oscilloscope, check the MCT detector centerburst intensity versus the manufacturer's specifications. Increase the source voltage, if necessary, to meet these specifications. For reference purposes, laboratories should prepare a plot of time versus detector voltage over at least a five-day period.

T.2.2 If the centerburst intensity is 75 percent or less of the mean intensity of the plot maximum obtained by the procedure, install a new source and check the MCT centerburst with an oscilloscope versus the manufacturer's specifications (if available). Allow at least five hours of new source operation before data acquisition.

T.2.3 Align test – With the lightpipe and MCT detector at thermal equilibrium, check the intensity of the centerburst versus the signal temperature calibration curve. Signal intensity deviation from the predicted intensity may mean thermal equilibrium has not yet been achieved, loss of detector coolant, decrease in source output, or a loss in signal throughput resulting from lightpipe deterioration.

T.3 GC/FT-IR sensitivity

T.3.1 Capillary column interface sensitivity test – Install a 30 m x 0.32 mm fused silica capillary column coated with 1.0 μm of DB-5 (or equivalent). Set the lightpipe and transfer lines at 280 °C, the injector at 225 °C and the GC detector at 280 °C (if used). Under splitless Grob-type or on-column injection conditions, inject 25 ng of nitrobenzene and dissolve in 1 μL of methylene chloride. The nitrobenzene should be identified by the on-line library software search within the first five hits (nitrobenzene should be contained within the search library).

T.3.2 One hundred percent line test – Set the GC/FT-IR operating conditions to those employed for the sensitivity test (see Sec. T.3.1). Collect 16 scans over the entire detector spectral range. Plot the test and measure the peak-to-peak noise between 1800 and 2000 cm^{-1} . This noise should be less than or equal to 0.15 percent. Store this plot for future reference.

T.3.3 If the GC/FT-IR was purchased before 1985, there may be a temperature effect at the interface. To account for this, prepare a plot of lightpipe temperature versus MCT centerburst intensity (in volts or other vertical height units). This plot should span the temperature range between ambient and the lightpipe thermal limit in increments of about 20 °C. Use this plot for daily QA/QC (see Sec. T.2.3). Note that modern GC/FT-IR interfaces (1985 and later) may have eliminated most of this temperature effect.

T.4 Frequency calibration – At the present time, no consensus exists within the spectroscopic community on a suitable frequency reference standard for vapor-phase FT-IR. One reviewer has suggested the use of indene as an on-the-fly standard.

T.5 Single beam test – With the GC/FT-IR at analysis conditions, collect 16 scans in the single beam mode. Plot the co-added file and compare with a subsequent file acquired in the same fashion several minutes later. Note if the spectrometer is at purge equilibrium. Also check the plot for signs of deterioration of the lightpipe potassium bromide windows. Store this plot for future reference.

Appendix A

Changes in this version from the December 1996, Revision 0.

1. Improved overall method formatting for consistency with new SW-846 methods style guidance. The format was updated to Microsoft Word .docx. This includes the additions of all sections required by the new format.
2. Many minor editorial and technical revisions were made throughout to improve method clarity.
3. The revision number was changed to one and the date published was changed to July 2014.
4. This appendix was added showing changes from the previous revision.
5. The "Quality Control of FT-IR Spectrometer" section previously referred to as Appendix A was renamed to TABLE 1 to keep consistency in format with other methods in Update V.
6. The flowchart on page 11 was updated to reflect the current section numbers.