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LEAD (Pb) AUDIT

by

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CAUTION

Disclaimer: This Standard Operating Procedure has been developed for use by ManTech Environmental Technology, Inc. in support of the National Performance Audit Program (NPAP) under contract to the U.S. Environmental Protection Agency and may not be applicable to the activities of other organizations.

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1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the preparation, packaging, and shipment of lead (Pb) audit samples in support of the Environmental Protection Agency's (EPA) National Performance Audit Program (NPAP).

2.0 SUMMARY

Quartz fiber filter strips are spiked once per year for quarterly audits and stored until the scheduled audit. For each audit, two target concentrations (levels), usually in the ranges of 100-300 and 600-1000 : g/strip, are requested by the EPA. Solutions containing specified amounts of lead are spiked onto filter strips using an automatic pipetter. Each filter strip has a maximum capacity of 0.5 mL which is taken into account in the preparation of the spiking solutions. The spiked strips are dried, placed in labeled plastic bags, and the plastic bags heat sealed. At the appropriate time, the sealed bags are placed in labeled mailing envelopes and shipped to audit participants.

3.0 SAFETY PRECAUTIONS

Standard practices for the safe handling of chemicals, including wearing safety glasses and disposable gloves, must be followed at all times.

4.0 FACILITY REQUIREMENTS

This procedure requires a laboratory equipped with both a sink and source of deionized (DI) water. Sufficient counter space should also be available for the automatic pipetter and incubator.

5.0 INTERFERENCES

Contamination from dirty glassware, equipment, and storage bags is a primary source for interferences in the analytical method. Improper handling of the filter strips is also a source for contamination. Chemical reagents of improper quality produce errors in the actual concentration values spiked.

6.0 APPARATUS

- Hamilton Microlab[®] automatic pipetter with foot switch
- LabLine Instruments incubator
- Audio Elextro bag sealer
- Analytical balance, readable to ± 0.1 mg

7.0 REAGENTS/MATERIALS

- Lead nitrate, $\text{Pb}(\text{NO}_3)_2$, ACS reagent grade
- Filter cutter template
- Pizza cutter and/or razor blade
- Lead-free, quartz-fiber high volume filters (cut into 3/4" x 10" strips)
- Chromatography clips
- Glass rods (to hang the length of the incubator)
- Plastic tubing, 2" wide (to make 2" x 6" bags)
- Plastic tubing, 4-5" wide (to make 4-5" x 7" bags)
- Sequentially numbered labels
- Laboratory standard glassware (cleaned and rinsed with DI water): 1 L and 500 mL volumetric flasks
- Disposable gloves
- Weighing boats and spatula
- S/P Micro All-Purpose Cleaner, or an equivalent synthetic, non-fatty acid based cleaner
- DI water: obtained from IonPure water purification system consisting of the following: activated charcoal, mixed bed resin, and cation resin.
- Drain rack

8.0 PROCEDURE

CAUTION

Always wear disposable gloves when handling the filters.

8.1 Prepare Glassware

1. Do not use abrasive cleansers on glassware, especially volumetric ware. Inspect cleaning brushes and discard any that have sharp points which may scratch the glassware. Contaminants may accumulate in scratches and scratches may also prevent proper drainage.
2. Never lift a volumetric flask by the neck because the neck is very fragile and breaks or cracks may occur. Take extra care when the flask is full.
3. Clean glassware promptly after use. Rinse the dirty glassware with DI water. . This removes contaminants from the glassware and also prevents contamination of the soaking or washing solution. If dirty glassware cannot be cleaned after this rinse, soak it in the standard cleaning solution of 75 cc (2.5 fluid ounces) S/P[®] MICRO cleaner per gallon of DI water.
4. Rinse off the drain rack with DI water prior to use.
5. Rinse the cleaning solution from the glassware with tap or DI water. Follow with a minimum of three rinses of DI water.
6. Check the glassware to see that the surface wets evenly after rinsing. If the water beads into droplets, repeat the cleaning procedure.
7. Invert all glassware, except pipets (See 8 below), on the pegs of the drain rack to dry. Do not allow glassware to touch because breaking or scratching could result. Store glassware in a cabinet or drawer.
8. Pipets:
 - a. Rinse the pipet once with DI water immediately after use.
 - b. After cleaning, rinse the pipets with a minimum of three cycles of DI water.
 - c. Inspect the pipets for water droplets which indicate that the glassware is still dirty. Repeat the cleaning cycle as often as necessary until an unbroken film of water indicates that the pipets are clean.
 - d. Place the cleaned pipets tips up in a plastic-coated test tube rack. Drain and dry the pipets at room temperature. Store in a cabinet or drawer.

8.2 Prepare Quartz Filter Strips

1. Determine the number of filter strips to be cut:
 - a. multiply the number of participants by the number of levels.

e.g., 80 participants x 2 levels = 160

- b. multiply the number of levels by 20 to account for the QA samples
e.g., 2 levels x 20 = 40
 - c. add the results of (a) and (b) to get the number of filter strips needed
e.g., 160 + 40 = 300 filter strips
2. Fold the 8" x 10" quartz fiber filter top to bottom.
 3. Place one folded filter in the filter cutter template.
 4. Use the pizza cutter or razor blade, pre-cleaned with alcohol, to cut strips 3/4" wide. Cut as many strips as possible from the filter. Discard the section of the filter containing the identification number.
 5. Store the strips in one of the original filter boxes.

8.3 Prepare Plastic Bags

8.3.1 Small Bags for Single Strips

1. Prepare the same number of small bags as the number of filter strips prepared.
2. Set the Audio Elextro bag sealer heater timer to approximately five seconds.
3. Use rolled 2" wide plastic tubing. Pull the tubing down to the 6" mark on the bag sealer plate.
4. Lower the heater arm and wait for the buzzer to sound. When the buzzer sounds (approximately five seconds, if working properly), slide the cutter lever across the top of the heater arm and back to its starting position.
5. Raise the heater arm and remove the bag.
6. Continue with this procedure until enough bags are made for the audit.

8.3.2 Large Bags for Sample Sets

1. Make the same number of large bags as the number of participants plus twenty for the QA samples.
2. Set the timer on the Audio Elextro bag sealer heater to eight seconds. A longer or shorter time may be needed depending on the thickness of the plastic material.
3. Use the large (4-5" wide) rolled plastic tubing. Pull the tubing down to the 7" mark on the bag sealer plate.
4. Lower the heater arm and wait for the buzzer to sound.
5. Slide the cutter lever across the top of the heater arm and back to its starting position.
6. Raise the heater arm and remove the bag.
7. Continue this procedure until the required number of bags have been made.

8.4 Prepare Stock Solution

Prepare 1 liter of 10,000 ppm stock standard solution for concentration levels of 200-3000 ppm by following these steps:

1. Weigh out 15.9844 grams of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) to the nearest 0.1 mg.
2. Transfer the lead nitrate quantitatively to a 1-liter volumetric flask.
3. Dilute to the mark with DI water and mix thoroughly.

8.5 Prepare Target Level Solutions

1. Calculate the volume of stock standard solution needed to prepare 500 mL of each target level by using the equation in **Section 9.0 Calculations**.
2. Prepare the low level first.
 - a. Pipet the calculated volume of stock solution into a 500 mL volumetric flask.
 - b. Fill the volumetric flask to the mark with DI water

c. Mix thoroughly.

3. Prepare each subsequent level.

8.6 Prepare and Verify the Hamilton Microlab® Pipetter

Note: Store the pipetter with the Cornwall ball in DI water.

8.6.1 Prepare the Hamilton Microlab® Pipetter

1. Turn on the power at the Hamilton Microlab® Pipetter keyboard (on the right side) and have the Cornwall ball in DI water.
2. Program the auto pipetter to hold a total volume of **5000 : L** and dispense **500 : L** in **10** steps by entering the following program (see Figure 1):

* , RDIS, 5, 0, 0, 0 (total capacity of syringe in : l) E
* , RDIS, 5, 0, 0 (volume of each aliquot in : l), E
1, 0 (number of steps), E

Press the foot pedal three times to progress the program and then enter:

* , SPEED, 0 (slowest speed), E (Default is 4)

(The syringe of the auto pipetter will automatically fill.)

3. Remove the pipetter from its special support stand and point the syringe needle upwards. Tap the syringe gently to get all bubbles to rise to the top.
4. Press the foot pedal to dispense the liquid upwards into a large KimWipe® until the syringe is free of bubbles.
5. Place the pipetter on its stand.
6. Using the foot pedal to control the pipetter action, fill the syringe with DI water and dispense the entire contents 2-3 times to thoroughly rinse the syringe.

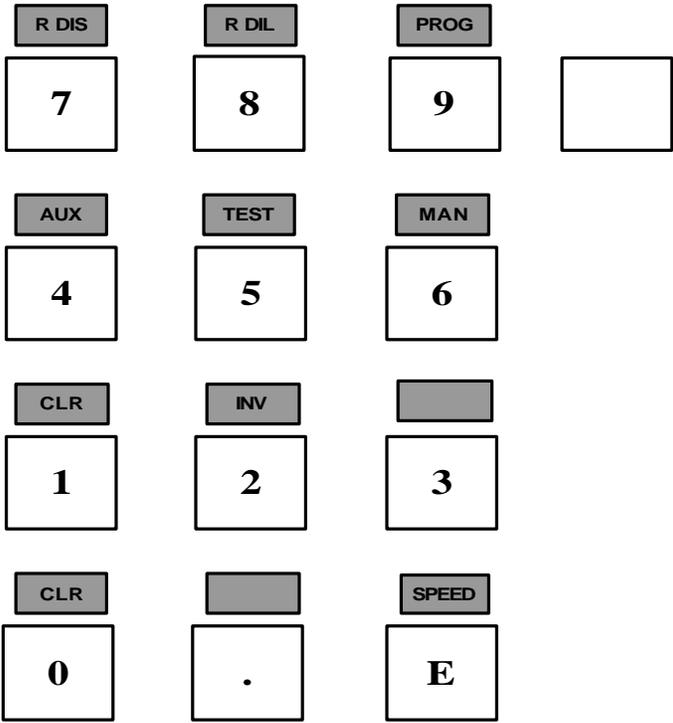


Figure 1. The Hamilton Microlab® Pipetter Keyboard

8.6.2 Verify the Hamilton Microlab® Pipetter

Note: Reproducibility of Hamilton Microlab® Pipetter is 1%.
1 mL H₂O = 1 g

1. Verify the Microlab® Pipetter annually.

CAUTION

Always wear disposable gloves when handling volumetric flasks during the verification process.

2. Select ten clean, dry volumetric flasks, preferably 5 mL to 25 mL volume. The flasks do not need to be labeled. A total of ten aliquots will be taken and weighed. This will require two fillings of the syringe since only the middle eight aliquots will be used. The first and last aliquots are discarded.
3. Place the volumetric flask on the weighing platform and tare the balance.
4. With a full syringe of DI water, dispense the first aliquot into a waste container or onto a KimWipe®.
5. Place the needle of the syringe into the neck of the volumetric flask so that all fluid goes into the flask. Dispense one aliquot into the flask.
6. Place the flask on the weighing platform of the balance and record the weight to three decimal places.
7. Tare another volumetric flask, dispense one aliquot, and record the weight.
8. Repeat **Step 7** until eight flasks have been used.
9. Dispense the tenth aliquot into a waste container or onto a KimWipe®. The syringe will automatically refill.

10. Repeat **Steps 4 through 7** for the ninth and tenth aliquot to be weighed.
11. Use a computer program such as Excel[®] to calculate the average and standard deviation of the data set.
 - a. Accept the verification if the mean is within ± 0.005 g of the theoretical standard mass of 0.5000 g/0.5 mL for water.
 - b. Reject the verification if the mean exceeds ± 0.005 g of the theoretical standard mass of 0.5000 g/0.5 mL for water.
 - c. Check the syringe for bubble accumulation and the pipetter for leaks. Repeat the verification. Accept the verification if the criteria is met.
 - c. If the pipetter again exceeds the criteria, send the pipetter to ManTech Environmental QA laboratory in-house repair.

8.7 Prepare Spiked Filter Strips

CAUTION

Always wear disposable gloves when handling the filters.

8.7.1 Dispense Lead Solution onto the Filter Strips

1. Turn on the incubator and warm it to a few degrees above room temperature.
2. Place the sample line of the auto pipetter in a beaker filled with the target value solution for Level 1.
3. Fill and completely empty the syringe two to three times so that the syringe is thoroughly rinsed with the solution.

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4. Place approximately 20 chromatography clips on a horizontal glass rod near the pipetter.
5. Begin with a full syringe and dispense the first aliquot of solution into a waste beaker to eliminate possible discrepancies due to bubbles in the syringe.

Note: As an additional precaution, the first and last aliquot in the syringe are **always** dispensed into a waste beaker and the filters are spiked with **only** the middle eight aliquots.
6. Wearing disposable gloves, pick up a single filter strip, and evenly dispense the Pb solution lengthwise on the filter. **Be extremely careful not to touch the moist section.**
7. Hang the filter strip lengthwise from a chromatography clip on the glass rod.
8. Follow **Steps 6 and 7** to spike each of the remaining filter strips in this group of eight. Hang each strip from a chromatography clip **MAKING SURE** that the strips do not touch.
9. Press the foot pedal to dispense the last step into the beaker and to start the pipetter refilling for another set of ten aliquots.
10. Repeat **Steps 5 through 9** until the glass rod is filled.
11. Repeat **Steps 4 through 11** until all filters for this level are spiked and are hung in the incubator.
12. Turn off the incubator and allow the filters to dry overnight in the closed incubator.
13. Place the sample line in a volumetric flask containing DI water. Rinse the sample line 2-3 times. Store the line in the volumetric flask until the next usage.

8.7.2 **Package the Spiked Filter Strips**

1. Wearing disposable gloves, remove the filters from the incubator, fold them in half, spiked side folded in, and place one filter in each small plastic bag.

Note: When all filters have been packaged, disposable gloves may be removed.

2. Seal the open end of the bags using the bag sealer set on five seconds.
3. Label the bags using the following code:

PB XYZZ

where

PB = the lead audit
X = concentration level (1,2)
Y = audit of calendar year (1,2,3,4)
ZZ = year (e.g., 97, 98,...)

e.g., level 2 lead samples for the second audit of 1997 would have the ID number 2297

8.7.3 **Prepare Remaining Level**

1. Rinse the sample line 2-3 times with DI water.
2. Place the sample line of the auto pipetter in a beaker filled with the target value solution for Level 2.
3. Complete the level following **Steps 3 through 14 in Section 8.6.1 Dispense the Lead Solution onto the Filter Strips**. Package the dried samples following **Section 8.6.2 Package the Spiked Filter Stripes**.

8.7.4 **Prepare Sample Sets for Shipment**

1. Place one packaged filter strip from each level in a large plastic bag.
2. Set the bag sealer for approximately eight seconds.

3. Seal the bag.
4. Place labels on the large bags indicating the audit and lot number. Label the bags using the following code:

PB 0QXX

where

PB = Pb audit
0Q = audit of calendar year (1,2,3,4)
XX = last two digits of the year

e.g., first audit 1997 = PB 0197

8.7.5 Pack Audit Sample Sets

1. Print the Mailing Labels and Mailing List using the NPAP database.
2. Select a participant from the list and locate the corresponding shipping label.
3. Affix the printed label on a ManTech Environmental mailing label.
4. Affix the mailing label to a padded envelope.
5. Place the following in the addressed padded envelope:
 - Large plastic bag containing 1 filter strip for each level
 - Cover letter from ManTech
 - Instructions for the EPA Lead Performance Audit
 - Data sheet
 - Pre-addressed envelope for returning the data sheet to ManTech Environmental
 - Questionnaire
6. Seal the padded envelope with filament tape.
7. Check the participant's name off the shipping list, set the padded envelope aside, and continue until all envelopes are completed.

8. Compare the total envelopes addressed against the shipping list.
 - a. If the counts are the same, mail the Audit Sample sets.
 - b. If the counts are not the same, locate the error and correct it. Verify the counts.

9.0 CALCULATIONS

$$V_1 = \frac{V_2 \times C_2}{C_1}$$

- where
- V_1 = the unknown volume of stock solution
 - V_2 = the volume of the target level (usually 500 mL)
 - C_2 = the concentration of the target level
 - C_1 = the concentration of the standard stock solution (usually 10,000 : g/mL)

For example, if the request is for 300 : g/strip, then the volume would be

$$\frac{(500 \text{ mL})(300 : \text{g/strip})}{(10,000 : \text{g/mL})(0.5 \text{ mL/strip})} = 30 \text{ mL}$$

Dilute 30 mL of the stock solution to 500 mL.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Quality control is maintained by strict adherence to the use of only synthetic non-fatty acid based cleaners and daily checks of the DI water supply. Visual inspection for water breaks or beading after the final rinse is conducted on all glassware. If the water sheets evenly on the glassware, the glassware is considered clean. If the water beads or breaks, then the cleaning procedure is repeated.

As quality control, 20 of the packaged strips from each level are randomly selected and submitted for analysis. The 20 analyzed samples per level must have a 95% or greater recovery with a coefficient of variation of 2% or less.

11.0 CORRECTIVE ACTION

Attempt to identify the cause of error. Discard the sample batch, prepare a new solution, and spike filters with the new solution if a sample level fails the criteria specified in **Section 10.0 QUALITY ASSURANCE /QUALITY CONTROL**.

12.0 DATA REPORTING

Audit data is sent directly to the Data Entry personnel and handled according to **NPAP-SOP-005: Computer Data Entry, Report Printing, and System Maintenance for the NPAP**.

13.0 REFERENCES

MicroLab® P Manual. Hamilton. Reno, Nevada