Preparation of Lead Filter Audit Strips from NIST SRM and Glass Fiber Filters

Standard Operating Procedure (SOP)

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1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) is intended as a guide for the preparation of lead spiked glass fiber filters for use as laboratory audit filter strips using National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) by an experienced laboratory chemist. This SOP is not intended to address the extraction or analysis of filter strips.

2.0 SUMMARY OF METHOD

This method describes the preparation of 1" X 8" glass fiber filter strips spiked with varying amounts of lead from NIST SRM 3128[1] using calibrated mechanical pipettes. Filter strips are cut from unexposed filters, placed on open faced petri dishes, spiked with a working standard prepared from NIST SRM 3128 using a mechanical pipette, dried in a HEPA-filtered laminar flow hood, and packaged. A subset of the filters prepared is extracted and analyzed to ensure the correct spiking level.

3.0 DEFINITIONS

Pb – Elemental or ionic lead

NIST – National Institute of Standards and Technology

D.I. water - Deionized water

RTI – RTI International

SOP – Standard Operating Procedure

SRM – NIST Standard Reference Material

USEPA – U.S. Environmental Protection Agency

v/v – volume to volume ratio

4.0 INTERFERENCES

4.1 Glassware, plasticware, and other sample processing hardware have the potential to contaminate the filter audit strips. All labware and hardware used must be soaked or rinsed in 1% (v/v) nitric acid, rinsed with deionized water, and completely dried prior to use.

5.0 HEALTH AND SAFETY CAUTIONS

5.1 This method does not address all the possible safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and compliance

with all OSHA regulations. Material Safety Data Sheets (MSDS) for all chemicals used should be readily available to all personnel involved with the procedure.

5.2 The NIST SRM 3128 ampoules contain a lead solution in 10% HNO_3. Nitric acid is a strong, corrosive, oxidizing agent that requires protection of the eyes, skin, and clothing. Items to be worn during use of this reagent include:

- Safety goggles (or safety glasses with side shields)
- Acid resistant rubber gloves
- A protective garment such as a laboratory apron. Nitric acid spilled on clothing will destroy the fabric and result in a hole; contact with the skin underneath will result in a burn.

It is also essential that an eye wash fountain or eye wash bottle be available during performance of this method. An eye wash bottle has a spout that covers the eye. If acid or any other corrosive gets into the eye, the water in this bottle is squirted onto the eye to wash out the harmful material. Eye washing should be performed with large amounts of water immediately after exposure. Medical help should be sought immediately after washing. If nitric acid is spilled onto the skin, wash immediately with large amounts of water. Medical attention is not required unless the burn appears to be significant. Even after washing and drying, the nitric acid may leave the skin slightly brown in color, this will heal and fade with time.

5.3 Lead (Pb) salts and lead solutions are toxic. Great care must be taken to ensure that samples and standards are handled properly; wash hands thoroughly after handling.

5.4 Care must be taken when opening the glass ampoules. The chemist must follow the instructions contained in the NIST certificate of analysis, INSTRUCTIONS FOR USE section, that accompany the SRM.

6.0 EQUIPMENT AND SUPPLIES

6.1 Materials

- Plastic petri dish, Pall Life Sciences, VWR Catalog No. 28145-473 or equivalent.
- Pipette, Rainin EDP2, 10-100 µL, with disposable tips, or equivalent.
- Pipette, Rainin EDP2, 1000 µL, with disposable tips, or equivalent.
- Plastic tweezers; VWR Catalog No. 89026-420, or equivalent.
- Plastic Ruler or block, 1” in width.
- Laboratory marker.
- Ceramic knife, Kyocera LK-25, or equivalent.
- Blank labels or labeling tape, VWR Catalog No. 36425-045, or equivalent.
- Volumetric flask, 100mL, VWR Catalog No. 89001-898, or equivalent.

- Glass fiber filters, unexposed, Whatman EPM2000, or equivalent.
- Millipore Element deionized water system, or equivalent, capable of generating Type I water (>17.9 MΩ-cm).
- Mettler XP205 Balance or equivalent with readability of at least 0.01mg, detectability of at least 1.6mg + $1.2X10^{-5}$ R_{gr}, and repeatability of at least 0.008mg + $6X10^{-8}$ R_{gr} where R_{rg} = gross weight.

7.0 REAGENTS AND STANDARDS

7.1 Reagent or trace metals grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

7.2 Concentrated nitric acid, 67-70%, SCP Science Catalog No. 250-037-177, or equivalent.

7.3 Deionized water – All references to deionized water in the method refer to Type I deionized water with a resistivity >17.9 MΩ-cm.

7.4 NIST SRM 3128 Working Standard. The chemist must exactly follow the NIST directions for "Preparation of Working Standard Solution by Volume" as detailed in the NIST certificate. The chemist must prepare the solution on a weight/volume basis with the concentration of the working standard close to a nominal value of 1000 µg/mL. Based on the current NIST certified value of 9.987mg/g, a 10-fold dilution is required. The exact solution concentration must be determined following the instructions in the NIST "Preparation of Working Standards by Volume" instructions on the certificate that accompanies the SRM.

8.0 FILTER STRIP PREPARATION

The ceramic knife, all plasticware (trays, ruler, tweezers), and glassware used in the preparation procedure must be rinsed or soaked in 1% HNO_3 (v/v) and then rinsed with deionized water and dried prior to use. Record all preparation steps in a laboratory notebook or appropriate electronic laboratory records system.

8.1 The mechanical pipette to be used for spiking the audit strips must be verified at the volumes to be used for spiking. The analyst must use deionized water and a calibrated balance readable to at least a 4 decimal places. NOTE: Mechanical pipettes should be set to the desired volume and then activated through one draw and dispense cycle before any liquids are pipetted. A small disposable polypropylene beaker or other container must be placed on the balance and tared. The analyst must pipette the selected volume of deionized water into the container and record the weight, tare the balance, and repeat until five replicate weights have been recorded.

The average of the five replicates is calculated by dividing the sum of the weights by 5. The relative percent error is calculated by Equation 1 below.

Equation 1. Relative Percent Error

$$\text{Relative \% Error} = ((M - T)/T) \times 100$$

Where M = average of five replicate weights

T = target weight

The relative percent error must be less than 1 at each volume to be used for spiking.

8.2 Place a cleaned plastic tray in the laminar flow hood. Select a filter from a box of new, unexposed, 8" X 10" glass fiber filters. Carefully fold the filter in half along the 10" side. Place the plastic ruler or 1" plastic guide block on the folded filter aligning the edge of the ruler/block with the edge of the filter. Hold the ceramic knife so that it is at approximately a 30° angle to the filter, apply light pressure, and draw the knife straight back slowly to complete the cut. NOTE: Examine the ceramic blade carefully before use for nicks or chips. Do not use a knife with any defects in the blade edge to avoid tearing the glass fiber filter during sectioning. Use plastic tweezers to place the strip on a clean plastic tray. Repeat the sectioning steps until the required number of filter strips have been cut.

8.3 Using plastic tweezers, carefully select one filter strip at a time and place the strip so it is centered on a labeled, open faced petri dish. (See Figure 1)

8.4 The amount of NIST working standard prepared in Section 7.4 to be used for spiking must be calculated. Equation 2 below shows the calculation for determining the amount of spike to add to achieve the desired percentage of the NAAQS standard.

Equation 2.

$$\frac{\mathbf{T}\ \mu g}{m^3} \times \frac{2000\ m^3}{\text{filter}} \times \frac{\text{filter}}{\mathbf{X}\ \text{strips}} \times \frac{mL}{\mathbf{C}\ \mu g} = \_\_\_\_\_ mL$$

Where

T = the concentration in $\mu g/m^3$ (desired percentage in decimal form X 0.15 $\mu g/m^3$)

X = the number of filter strips (9 for 1", 12 for ¾")

C = the concentration of the NIST working standard prepared in Section 7.4

For 37% of the NAAQS standard on a 1" strip using a 1024 µg/mL working standard, the calculation would be:

$$\frac{\textbf{0.0555}\ \mu g}{m^3} \times \frac{2000\ m^3}{filter} \times \frac{filter}{\textbf{9}\ strips} \times \frac{mL}{\textbf{1024}\ \mu g} = 0.012mL$$

The calculation of the exact µg/strip based on 0.012mL of working standard is shown in Equation 3 below.

Equation 3.

$$\frac{A}{0.012\ mL} \times \frac{B}{\frac{1024\ \mu g}{mL}} \times \frac{}{1\ Strip} = \frac{C}{\frac{12.288\ \mu g}{strip}}$$

Where:

A = the mL of NIST working standard added

B = the concentration of the NIST working standard prepared in Section 7.4

C = the final concentration in µg/strip

8. 5 Set the mechanical pipette to the desired volume and activate it through one draw/dispense cycle. Place a tip on the pipette and draw the desired volume of NIST working standard prepared in Section 7.4. NOTE: The analyst must visually inspect the pipette tip after drawing spiking solution to ensure that there are no droplets on the exterior of the tip. The analyst should also note the liquid level inside the tip to compare to all subsequent draws.

8.6 Hold the pipette over the center of the strip and activate the dispense cycle. The pipette tip should be <u>just</u> above the strip, but not touching the strip. For volumes over 25uL, the analyst should move the pipette along the center of the filter strip to deposit the spiking solution in a stripe instead of a spot. NOTE: The orientation of the tip to the audit strip is critical to ensure that all the spiking solution drawn is deposited on the strip. The analyst must visually inspect the pipette tip after deposition to ensure that all solution was deposited and no droplet remains in the tip. If any liquid remains in the pipette tip, the pipette tip and filter strip should be discarded and the spiking repeated on a new filter strip.

8.7 The steps in 8.5, 8.6, and 8.7 are repeated until all strips have been spiked.

8.8 The audit strips must be allowed to dry for a minimum of 8 hours in a HEPA filtered laminar flow hood.

8.9 Once the audit strips have dried for at least 8 hours, the individual strips are sealed in plastic bags that have been screened for the presence of lead.

8.10 Label the audit strips so that the concentrations can be identified by the laboratory preparing the strips but not by outside laboratories.

9.0 QUALITY CONTROL

9.1 A minimum of seven audit strips at each concentration prepared must be selected at random from all the audit strips prepared for extraction and analysis. The actual number of strips will be dependant on the total number prepared and the desired confidence level. The strips selected must have been taken through the entire preparation process including bagging and labeling.

9.2 Extract and analyze the selected strips by a method appropriate for the analysis of lead on TSP filters. This SOP is specific to the preparation of the filter strips and not intended to cover the extraction and analysis of filter strips.

9.3 The analysis results must be within 5% bias of the expected result. The expected result is calculated by Equation 3 as shown in Section 8.4. If the average of the seven filter strips is outside ± 5% bias, the lot must be rejected and the entire preparation process repeated.

10.0 METHOD PERFORMANCE

Information in this section is an example of typical performance results achieved by following this SOP. Actual performance must be demonstrated by each individual laboratory and instrument.

10.1 Recovery tests with filter strips spiked with NIST SRMs were performed using the ultrasonic/ nitric and hydrochloric acid filter extraction method and measurement of the dissolved lead with ICP-MS. Table 1 shows recoveries for two spiking levels. The recoveries for the audit strips are all within ± 5% bias for each strip and the average.

11.0 POLLUTION PREVENTION

11.1 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. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. The sources of pollution generated with this procedure are waste acid extracts and lead-containing solutions.

11.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St. N.W., Washington, D.C., 20036, www.acs.org.

12.0 WASTE MANAGEMENT

12.1 Laboratory waste management practices must be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect air, water, and land by minimizing all releases from hood and bench operations, complying with the letter and spirit of any sewer and discharge permits and regulations, and by complying with all solid and hazardous waste regulation. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society listed in Section 14.2.

12.2 Waste nitric acid, hydrochloric acid, and solutions containing these reagents and/or Pb must be placed in labeled bottles and delivered to a commercial firm that specializes in removal of hazardous waste.

13.0 REFERENCES

[1] NIST, *Certificate of Analysis: Standard Reference Materials 3128, Lead Standard Solution, Nominal 10 mg/g Lead,* National Institute of Standards and Technology, Gaithersburg, MD, 2005.

14.0 TABLES AND FIGURES

Table 1. Recoveries of Lead from NIST SRM Spiked Glass Fiber Filter Audit Strips.

Low Audit Strip (12.288 µg/strip)	µg/strip	% Bias
1	12.24	-0.4
2	12.33	0.3
3	12.37	0.7
4	12.31	0.2
5	12.49	1.7
6	12.28	-0.1
7	12.42	1.1
Average	**12.35**	**0.5**
Standard Deviation	0.09	-0.4
%RSD	0.704	
High Audit Strip (71.68 µg/strip)	**µg/strip**	**% Bias**
1	71.40	-0.4
2	70.40	-1.8
3	70.44	-1.7
4	70.68	-1.4
5	70.32	-1.9
6	71.24	-0.6
7	70.16	-2.1
Average	**70.66**	**-1.4**
Standard Deviation	0.48	-0.4
%RSD	0.675	

Figure 1. Filter Strip on Petri Dish