Other Test Method – 42: Sampling, Sample Preparation and Operation of the Fluidized Bed Asbestos Segregator

This method is designed to quantify mineral fibers present in soil or other solid media such as vermiculite. Respirable mineral fibers such as asbestos or other unregulated amphibole or zeolite mineral fibers can become airborne and present a potential for inhalation exposure if the soil or other solid media is disturbed. The fluidized bed asbestos segregator (FBAS) is a sample preparation instrument that utilizes air elutriation (separating particles based on size, shape, and density, using a stream of gas or liquid flowing in a direction usually opposite to the direction of sedimentation) to separate light weight asbestos structures from heavier matrix particles and deposit these structures onto a filter, which can then be analyzed by transmission electron microscopy (TEM). The analytical data resulting from use of this method is presented as either structures per gram of soil (s/g) or as an estimated weight percent. Method detection limits achieved can range from 0.002% to 0.005% by weight, which is approximately 100-times lower than the detection limits that are usually achieved using other analytical methods for asbestos in soil and other solid media. The FBAS unit is compact, fitting into a standard laboratory fume hood, and components of the unit are relatively easy to decontaminate or are disposable. The FBAS unit construction and operation costs are relatively low and sample throughput is high (up to 20 samples per day).

The posting of a test method on the Other Test Method (OTM) page of the EPA's Air Emission Measurement Center (EMC) website is neither an endorsement by EPA regarding the validity of the test method nor a regulatory approval of the test method. The purpose of the OTM portion of the EMC is to promote discussion of developing emission measurement methodologies and to provide regulatory agencies, the regulated community, and the public at large with potentially helpful tools.

Other Test Methods are test methods which have not yet been subject to the Federal rulemaking process. Each of these methods, as well as the available technical documentation supporting them, have been reviewed by the EMC staff and have been found to be potentially useful to the emission measurement community. The types of technical information reviewed include: field and laboratory validation studies; results of collaborative testing; articles from peer-reviewed journals; peer-review comments; and quality assurance (QA) and quality control (QC) procedures in the method itself. A table summarizing the available technical information for each method can be found at the link below. The EPA strongly encourages the submission of additional supporting field and laboratory data as well as comments regarding these methods.

This method may be considered for use in addressing asbestos at Superfund sites as part of a risk-based, site-specific approach for site evaluation. It can be part of a framework that provides a flexible approach to investigating and evaluating asbestos contamination at Superfund removal and remedial sites. Consideration of a method's applicability for a particular purpose should be based on the stated applicability as well as the supporting technical information outlined in the table. The methods are available for application without EPA oversight for other non-EPA program uses.

As many of these OTM are submitted by parties outside the Agency, EPA staff may not necessarily be the technical experts on these methods. Therefore, technical support from EPA for these methods is limited, but contact information for the developers is provided so that you may contact them directly. Also, these methods are subject to change based on the review of additional validation studies or on public comment as a part of adoption as a Federal test method.

Method History

EPA advises all potential users to review the method and all appendices carefully before application of this method.

Draft July 31, 2018

Sampling, Sample Preparation and Operation of the Fluidized Bed Asbestos Segregator

Overview: The ability to quantify the concentration of mineral fibers such as asbestos in a solid media, such as soil, aids in risk determinations at Superfund sites. Detection and quantification of mineral fibers using traditional microscopy methods are often difficult due to interferences from matrix particles of various sizes that can obscure or hide mineral fibers of interest that are present in a sample.To address this problem, EPA has continued development and evaluation of the FBAS, which uses air elutriation to segregate smaller light weight structures and deposits them onto a filter, which can then be analyzed by TEM or other microscopy technique (scanning electron microscopy or phase contrast microscopy).

1.0 Scope and Application

1.1 Pollutant/Measured Parameters

The pollutants of concern for this method are elongated mineral fibers including the regulated forms of asbestos (chrysotile, amosite, crocidolite, tremolite, actinolite, and anthophyllite), other fibrous amphiboles (winchite, richterite, magnesio-arfvedsonite) and fibrous zeolite (erionite).

The purpose of this method is to describe how to collect and process samples in the FBAS. An image of the FBAS is included in Figure 1. A schematic diagram of the FBAS is included in Figure 2.

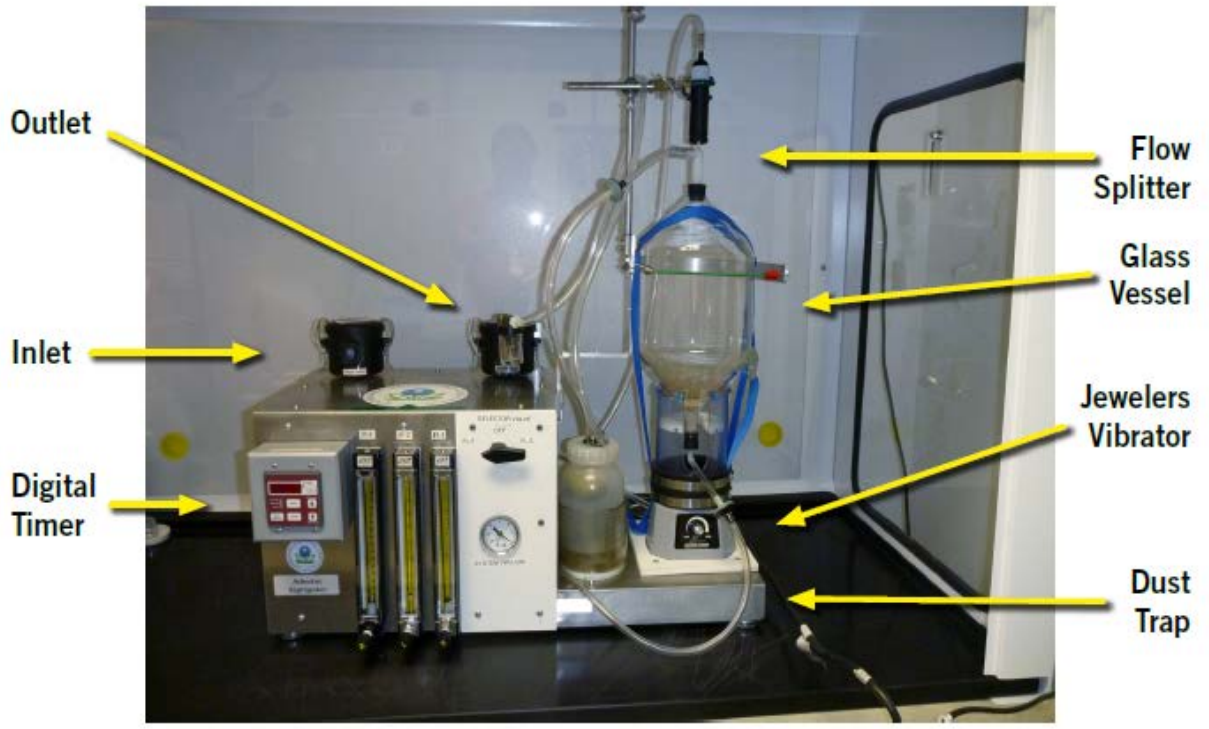

Figure 1 – Image of a fully assembled fluidized bed asbestos segregator.

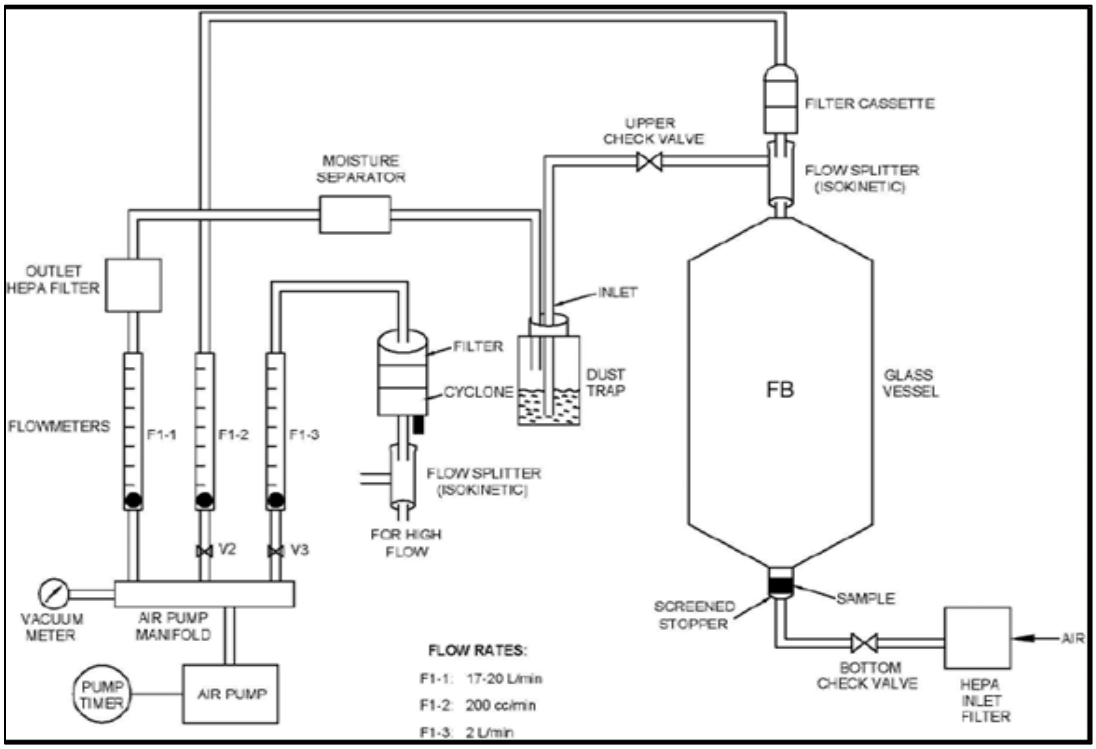

Figure 2 – Schematic of Fluidized Bed Asbestos Segregator.

1.2 Applicability

This method should be used as a sample preparation technique that segregates asbestos or other mineral fibers from solid media, such as soil, and deposits these fibers onto an air filter, allowing an analysis of the filter to determine releasable asbestos content of the soil. Performance evaluation (PE) studies have shown that analyses of filters prepared using the FBAS technique are able to detect asbestos at levels that are much lower than most standard analytical methods for the analysis of asbestos in bulk materials.[1] There is an approximately linear relationship between the concentration of asbestos in the PE standard (as mass percent) and the mean concentration estimated by the TEM analysis following preparation by FBAS, expressed as asbestos structures captured on the filter per gram of test material (s/g).

1.3 Data Quality Objectives

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. EPA has developed a seven-step process for establishing DQOs and developing a quality assurance project plan (QAPP) to help ensure that data collected during FBAS studies that will be adequate to support reliable decision-making.[2,3]

1 Januch J, Brattin W, Woodbury L, Berry D (2013) Evaluation of a fluidized bed asbestos segregator preparation method for the analysis of low-levels of asbestos in soil and other solid media. Anal Methods 5:1658–1668

2 EPA. 2006. G*uidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4.* U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-06/001. February. http://www.epa.gov/quality/qs-docs/g4-final.pdf.

3 EPA. 2001. *EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5.* U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-01/003. March. http://www.epa.gov/quality/qs-docs/r5-final.pdf.

2.0 Summary of Method

Soil, or other solid media, is collected from an area of interest. It is size-segregated by sieving and the fine fraction is then homogenized and fluidized in the FBAS. Small particles are elutriated from the bulk material and collected on a filter. The filter is then analyzed by TEM for identification and quantitation of fibers. The fibers are counted, typically using the counting rules specified in International Standards Organization analytical methods for TEM analysis.[4,5] The concentration of fibers in the soil can be expressed as either asbestos structures per gram (s/g) of soil or as mass percent (g of asbestos per 100 g of soil).

3.0 Definitions

Asbestiform – A specific type of mineral form in which fibers and/or fibrils possess high tensile strength and flexibility.

Asbestos – Asbestiform varieties of chrysotile (serpentine), crocidolite (riebeckite), amosite (cummingtonite-grunerite), anthophyllite, tremolite, and actinolite.[6] For the purposes of this OTM, the definition also applies to the asbestiform amphibole minerals winchite, richterite, and magnesio-arfvedsonite, the combination of which is commonly referred to as Libby Amphibole (LA).

Erionite – An aluminum silicate zeolite mineral that can occur with a fibrous or blade-like morphology. Erionite has been associated with mesothelioma and is classified as a carcinogen by the World Health Organization.[7]

Fiber – An elongated particle which has parallel or stepped sides. A fiber will have a length greater than 0.5 µm, with a width between 0.25 and 3 µm, and have an aspect (length: width) ratio greater than or equal to 3:1.

Fiber Bundle – A structure composed of parallel, smaller diameter fibers attached along their lengths. A fiber bundle may exhibit diverging fibers at one or both ends.

Structure – A single fiber or fiber bundle. The FBAS data is typically presented using the unit asbestos structures per gram of soil (s/g).

4.0 Interferences

High levels of non-asbestos dust particles may overload the surface of the filter and obscure asbestos fibers in the microscope field of view. Overloading can be minimized by monitoring the particulate load on the filter by screening the filter with a phase contrast microscope (PCM) - see Section 8.2.

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4 International Organization for Standardization, *Ambient Air - Determination of Asbestos Fibres – Direct Transfer Transmission Electron Microscopy Method*, Reference Number ISO 10312:1995(E), May 1995.

5 International Organization for Standardization, *Ambient Air – Determination of Asbestos Fibres – Indirect Transfer Transmission Electron Microscopy Method*, Reference Number ISO 13794:1995(E), July 1995.

6 Title 40 of the Code of Federal Regulations (CFR) 763.83.

7 IARC. 2012. Arsenic, metals, fibres, and dusts. In IARC monographs on the evaluation of carcinogenic risks to human, vol. 100C. IARC Press, 150 Cous Thomas, 69372 Lyon cedex 08, Lyon, France.

Adjusting the sample amount placed in the FBAS and sampling duration accordingly to achieve optimum particulate load on the filter, typically between 10% and 25%, allows the user to maximize the process.

5.0 Safety

Inhalation of asbestos fibers will increase the risk of lung cancer, mesothelioma, and non-malignant lung and pleural disorders including asbestosis, pleural plaques, pleural thickening, and pleural effusions. When handling materials suspected of containing asbestos, precautions should be taken to avoid inhalation exposures. Conduct work with materials suspected of containing asbestos in a safety hood equipped with a negative pressure high-efficiency particulate arrestance (HEPA) filter system.

Engineered safety features of the FBAS include: 1) a vacuum pump to maintain the system under negative pressure and contain asbestos contamination within the system; 2) the sample filter and a HEPA filter upstream of the vacuum pump to remove asbestos prior to the pump exhaust; 3) a diaphragm-style check valve to prevent the asbestos-containing materials (ACM) from draining out of the dust collector bottle; and 4) disposable components and tubing (with the exception of the glass vessel and cyclone attachment - if used) that contact asbestos to minimize handling and cleaning for contaminated equipment.

6.0 Equipment and Supplies

Sample Collection and Preparation

- Negative flow HEPA workstation or proper personal protective equipment, including respirators for handling unconfined material suspected of containing asbestos
- 25-millimeter (mm) mixed cellulose ester (MCE) filter composed of mixed cellulose ester having a pore size equal to 0.8 µm (such as part number 225-231 from SKC) for TEM analysis
- Notebook for recording sampling information
- Small flat-bottomed metal scoop (do not use a rounded-bottom scoop)
- 250 milliliter (ml) or larger beaker with 50 ml (or finer) gradations
- 8-inch sieves (brass or steel); 6.3 mm opening (¼-inch sieve) and 0.85 mm opening (U.S. Standard Sieve No. 20), receiver pan and lid
- Balance capable of weighing 1-20 g to ± 0.01 g
- Troemner or equivalent NIST traceable balance calibration weight set, range 1 mg to 100 grams
- U.S. Silica ASTM 20/30 sand or equivalent Restek Ottawa sand
- Sample containers (e.g., 250 ml glass bottles with caps)
- Metal spatula
- Drying oven capable of operating at 60 ^{o}C ± 5^{o}C
- Soil moisture meter
- (Optional) Phase Contrast Microscope (PCM) and sample preparation kit

Fluidized Bed Segregator Unit Parts

- Inlet HEPA filter (such as part number 5169K72 (housing) and 9179K14 (HEPA) from McMaster Carr)
- Bypass HEPA filter (such as part number 5169K72 (housing) and 9179K14 (HEPA) from McMaster Carr)
- Polyvinyl chloride (PVC) tubing, ¼-inch inner diameter (ID) (such as McMaster-Carr part number 5231K161)
- PVC tubing, 3/8-inch ID (such as VWR part number 60985-544)
- 32-ounce (oz.) Multipurpose calibration jar, polypropylene - used

for oversize dust collection (SKC part no. 225-111)

- Jewelers Vibrator (such as Investment Vibrator 110-volt AC, 60 Hertz [Hz], 0.7 amp (A))
- Allegro Industries vacuum pump, Part Number 9804-88, oil-less rotary vane, 1/10 horsepower (HP) 115-volt AC, 60 Hz.

Sample Processing

- Calibrated barometer and thermometer to measure local barometric pressure and temperature.
- Fluidized bed chamber assembly:
- Glass vessel (per diagram in Figure 3) – Replacements available through Precision Glassblowing of Colorado, 14775 East Hinsdale Avenue, Centennial, Colorado 80112-4243, phone (303) 693-7329
- Two check valves (plastic diaphragm, 5/16-inch tube ID, such as McMaster-Carr part number 47245K24)
- No. 2 or No. 3 black rubber stopper with standard ¼ inch hole, with nylon mesh (20
- µm to 45 µm) covering hole (Use No. 3 stopper if stopper is to be removed after use, otherwise, use No. 2 stopper, such as McMaster-Carr part number 9545K27.)
- Nylon elbow connectors, ¼-inch ID (Such as Cole–Parmer Part# L0-4NN)
- Isokinetic sample flow splitter assembly (per diagram in Figure 3) – replacements available through Cascade Plastics Corporation, Inc. 7009 45th Street Ct. East, Fife, Washington 98424, phone 1 (800) 699-3460
- MCE filter cassettes with 25-mm, 0.8 µm pore size (such as part number 225-231 from SKC)
- Washed (3X in deionized water) and dried (12 hours at 60°C) ASTM 20/30 quartz sand (e.g., U.S. Silica brand or Restek Ottawa Sand), 15-19 g per sample depending on the results from the test sample described in Section 8.3 of this OTM
- 16-ounces of Mineral Oil (such as Vi-Jon, Inc., Swan Mineral Oil U.S.P., Item#831)
- 1-gallon plastic garden sprayer filled with fiber free amended soapy water (Laboratory grade de-ionized water with 4 drops of Alconox liquid soap or equivalent)
- Nilfisk (or equivalent) HEPA vacuum with hose and brush attachment
- Digital timer

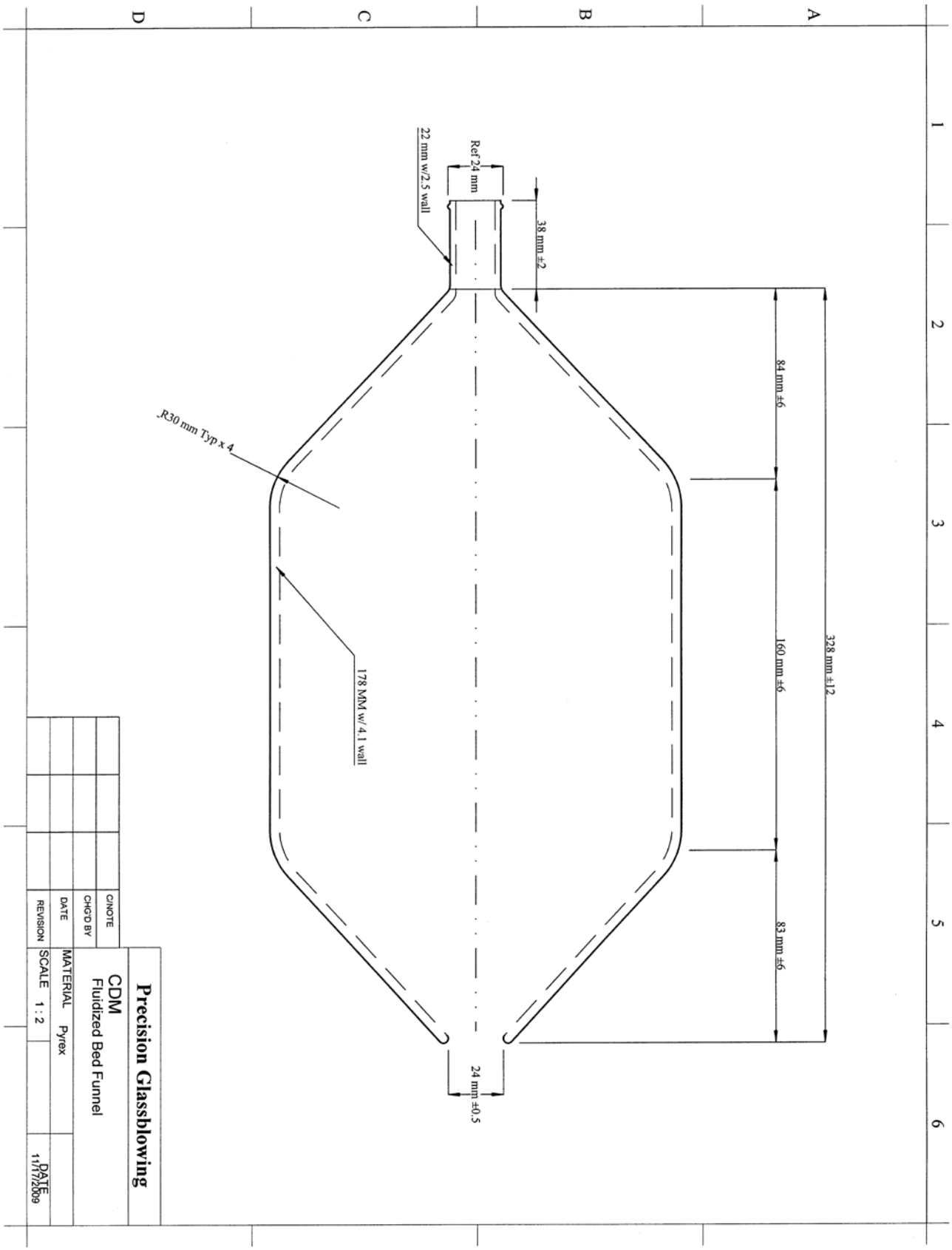

Figure 3 – Diagram of glass vessel.

7.0 Reagents and Standards

Reference materials for asbestos are available through a variety of sources including the National Institute of Standards and Technology (NIST) and the Union of International Cancer Control (UICC). NIST Standard reference material (SRM) 1866 is Common Commercial Asbestos and includes specimens of chrysotile, amosite, and crocidolite. NIST SRM 1867 is Uncommon Asbestos and includes anthophyllite, actinolite, and tremolite. If NIST SRM's are unavailable, then UICC reference material (available commercially) or another substitute of known quality should be used to prepare performance evaluation (PE) samples for QA purposes.

8.0 Sample Collection

8.1 Set up the FBAS Sampling Assembly

1. Place the FBAS unit inside the HEPA filtered safety hood.
2. Plug in the vacuum pump and vibration unit into the outlet mounted on the back of the FBAS unit.
3. Place HEPA filters into the black colored inlet and outlet filter housings located on top of the unit.
4. Fill the dust collector bottle with a maximum of 1-2 inches of mineral oil.
5. Connect a 1-foot section of clear PVC tubing, 1/4-inch ID, 1/16-inch wall, from dust collector bottle cap outlet (nipple with shorter tube on the underside of the cap) to the 1/4-inch barbed adapter on the moisture separator located in front of the outlet HEPA filter.
6. Assemble the isokinetic splitter assembly (see Figure 4) by connecting a 6-inch section of clear PVC tubing, 1/4-inch ID, 1/16-inch wall to a check valve on one end and insert the other free end into a 2-inch section of clear PVC tubing, 3/8-inch ID, 1/16-inch wall mounted on the barbed fitting of a plastic isokinetic splitter (see Figure 5).
7. Cut a second section of tubing 1½ feet long and connect one end to the dust collector bottle cap inlet and the other end to the check valve on the isokinetic splitter assembly.

Caution – If you are replacing a previously used HEPA filter or tubing, you should assume that these items, as well as the contents of the dust collector bottle and the inlet filter housing, are contaminated with asbestos.

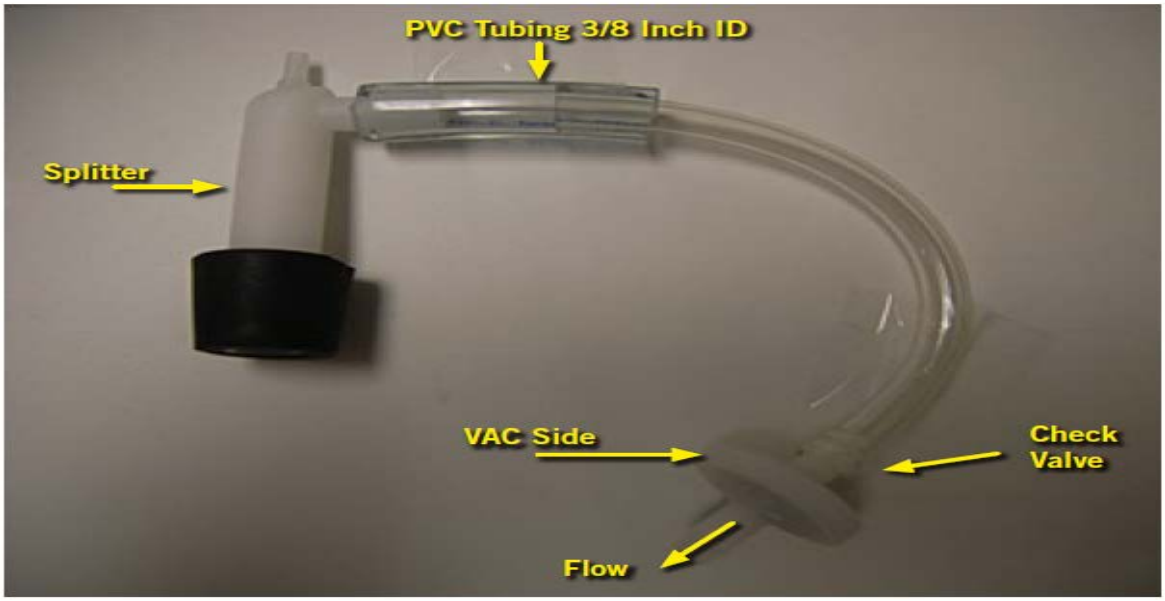

Figure 4 – Isokinetic splitter attached to a check valve.

8. Connect one end of a 6-inch section of PVC tubing to the bottom check valve and the other end to a Nylon barbed elbow connector, ¼-inch ID, that's been inserted into a Number 2 screened bottom stopper (see Figure 6).

9. Push the stopper into the bottom opening of the glass vessel.

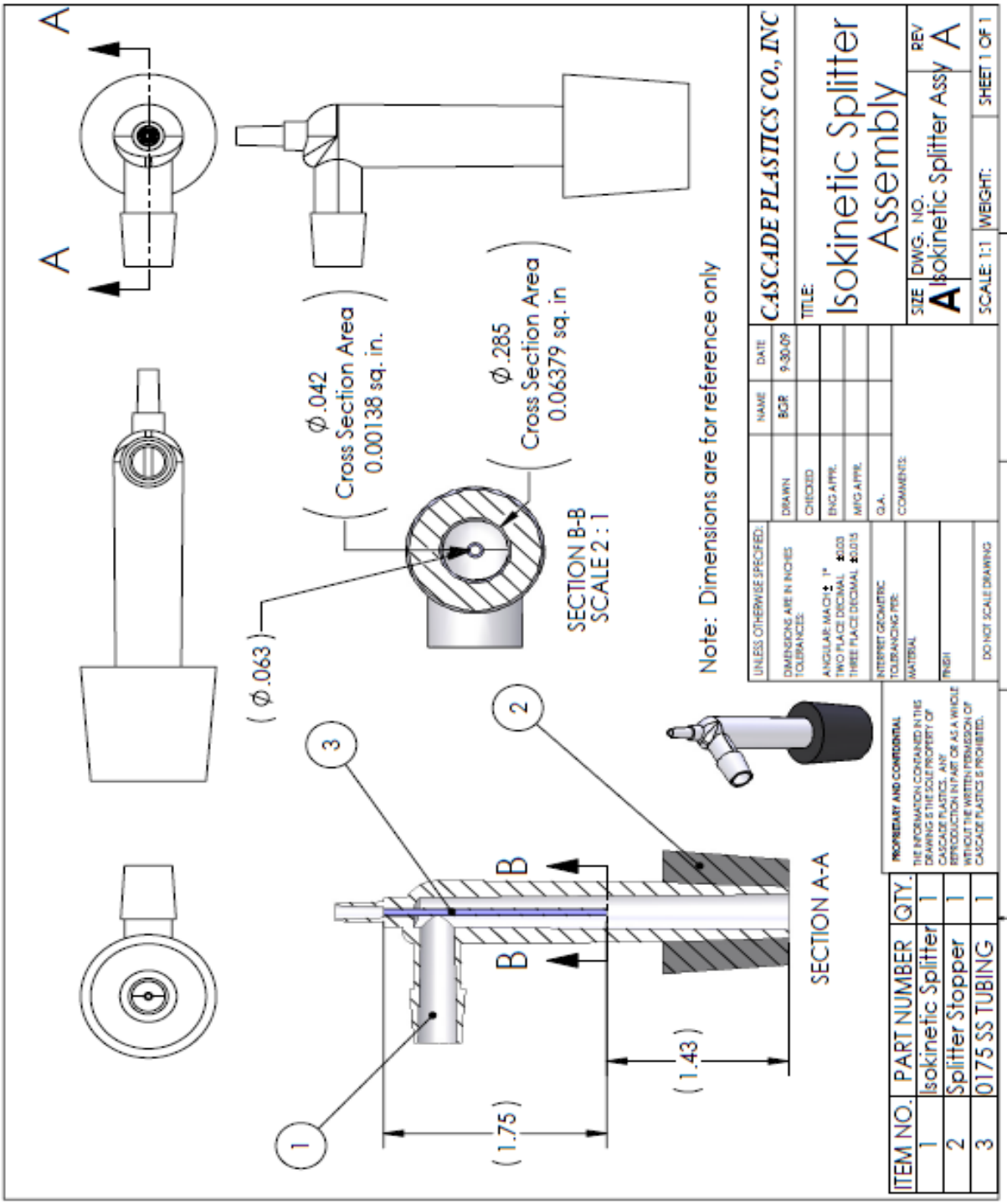

Figure 5 – Isokinetic Splitter attached to a check valve

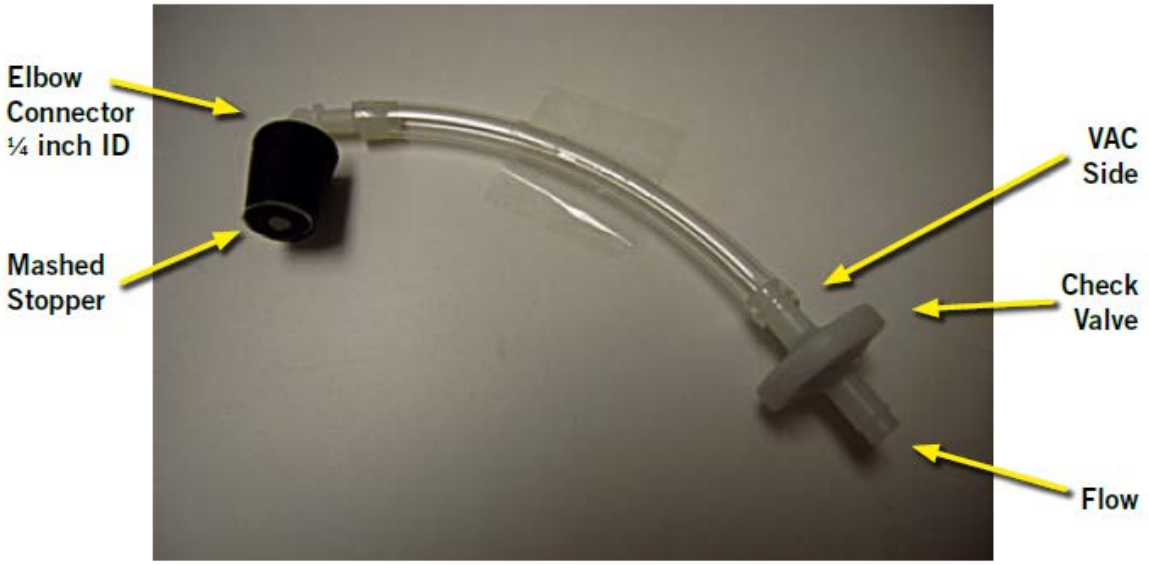

Figure 6 – Meshed stopper connected to check valve

Mount a clean glass vessel onto the plastic holder connected to the top surface of the vibrator unit. Fasten the safety belt and the front elastic cord to secure the glass vessel.

10. Seat the isokinetic splitter stopper into the top opening of the glass vessel so the outlet tube is facing up.
11. Remove the cap on the bottom of the conductive cowl of a filter cassette and insert an additional cowl (stack one cowl on top of the other) and cap the bottom of the additional cowl (see Figure 7).
12. Remove the red colored plugs from the inlet and outlet of the sample cassette assembly.
13. Connect the PVC tubing marked "Filter" to the top outlet tube of the filter cassette outlet.
14. Seat the bottom inlet tube of the filter cassette assembly to the top outlet tube of the isokinetic splitter.

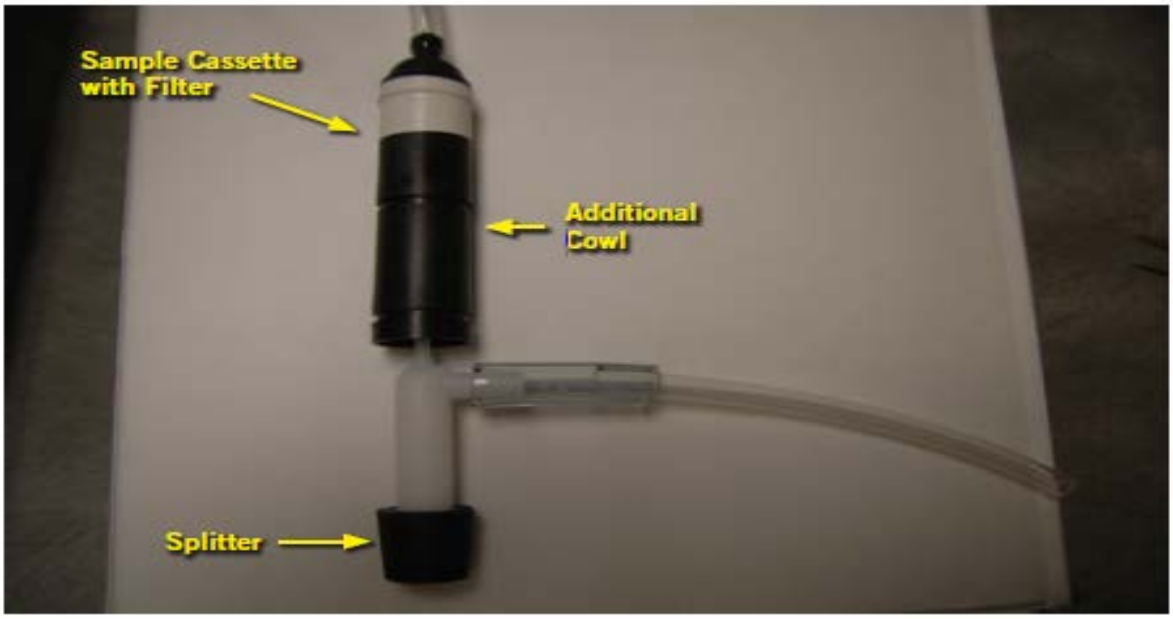

Figure 7 – Stacked conductive filter cowls connected to isokinetic splitter.

8.2 Sampling Considerations

QA and QC measures for sampling and analysis should be performed according to an approved QAPP. Specific sampling procedures should be performed according to this method. If procedures to obtain samples differ from this method, the changes should be well documented in the project notebook.

Samples should be examined during test sampling and periodically during regular sample collection to determine the degree of filter loading. Filters can be examined visually, or with the aid of a PCM (preferable). If a PCM is used, follow the sample preparation and analytical instructions found in the National Institute of Occupational Safety and Health (NIOSH) Method 7400, Issue 2.

8.3 Obtain a Test Sample

The purpose of the test sample is to determine how much soil sample mass is necessary to properly load the collection filter on the FBAS.

1. Use the flat-bottomed scoop to collect about 250 ml (enough to fill as 8-ounce glass sample jar) of representative soil sample. Collect the sample from the uppermost 1 inch of surface soil or as otherwise specified in a site-specific QAPP. Take the entire soil column (rectangular cross-section for entire linear scoop). Move the scoop slowly (<2 cm/s) to reduce segregation at the cutter edges.

NOTE: Proper sub-sampling technique is critical to obtain representative samples of particulate solids. Do not use a rounded-bottom scoop for soil sampling.

2. If the soil is saturated with water and does not disaggregate into individual grains easily, it should be dried before proceeding. Dry the soil in a laboratory drying oven at 60° C for 12 hours or as long as it takes to achieve disaggregation of soil particles. The type of soil (e.g., high clay content) may require additional drying if it does not disaggregate. Measure the soil moisture using the soil moisture meter and record the value in the laboratory log book.
3. Assemble the sieves so that the largest opening (6.3 mm) sieve is on top of the 0.85 mm sieve and the collection pan is on the bottom.
4. Put the sample on the top sieve of the sieve stack. Place the lid on the top sieve and shake the sieves by hand back and forth for 2-5 minutes. Alternatively, an automatic sieve shaker can be used to increase shaking time and improve sorting.
5. Use the material collected in the bottom pan for the test sample.
6. Weigh out at least three replicate 1-5 g portions of sample and 15-19 g portions of sand to help determine the optimum sample/sand combination that results in optimum filter loading. An average estimate of loading between the three replicate tests per combination will ensure that optimum filter loading has been achieved.
7. Combine the sample and the sand so that the combined weight of the mixture equals 20 g.
8. Record the weight of the sample and the weight of the sand in the notebook.
9. Remove the isokinetic splitter stopper and place a combined mixture of sample/sand into the glass vessel, then replace the isokinetic splitter stopper.
10. Set the digital timer for a minimum of 3 minutes.
11. Press the “on” button on the digital timer, which will engage the vacuum pump and the vibration unit. The pump and vibration unit will shut off after sample time (typically 3 minutes) has elapsed.
12. Examine the filter using PCM (preferable) or visually when the segregator completes the sample. If the segregator did not clog and the filter is optimally loaded (between 10% and 25% particulate load), run the remaining test samples. Use a new filter cassette assembly and a clean glass vessel for each sample.
13. Repeat steps 9-12 with the different sample/sand combinations, increasing or decreasing the soil

mass as appropriate. Compare the filters to determine which will give the optimum filter loading. Try different amounts of soil, but make certain to use a balance of sand so that the sample/sand combination equals 20 g.

When the optimum soil sample mass for the FBAS has been determined, make a note in the notebook as to the proper mass and continue with formal sample collection (Section 8.5).

8.4 Decontamination of the FBAS

1. Remove the glass vessel from the vibration unit and lay it on its side in front of the FBAS.
2. Remove the isokinetic splitter and the meshed stopper from the ends of the glass vessel and discard in the asbestos equipment waste container.
3. Pour the spent sample/sand material into the asbestos sample waste container.
4. Use the Nilfisk HEPA vacuum to evacuate fibers and other loose debris by placing the HEPA vacuum hose over one of the ends of the glass vessel to extract the airborne fine particulate matter and visible spent sample material off the inner surface of the vessel. This should only take 15-30 seconds.
5. Remove the HEPA vacuum hose.
6. Place the wand of a garden sprayer (filled with amended –soapy water) into the glass vessel and wet the inside of the vessel. Seal the openings of the vessel with tape (either lab tape or duct tape).
7. Remove the vessel from the HEPA hood and clean with soap and water in a wash basin. Use a flask brush to scrub the inside of the vessel.
8. Use warm tap water to rinse the soap out of the interior and off the outer surface of the glass vessel. Thoroughly rinse the interior and exterior surfaces of the glass vessel with deionized water.
9. The glass vessel can either be air dried on a drying rack or can be placed inside a drying oven and dried at 60^{o} C for approximately 1 hour.
10. Remove the glass vessel from the drying oven and allow it to cool. Then seal all openings with tape to avoid contamination before next use.
11. Between sample sets, use a HEPA vacuum to clean the outer surface of the FBAS frame, including the pump, vibrator, time, and flow meters.

8.5 Collecting a Sample

1. Combine the appropriate amount of sample/sand, based on the test sample findings from Section 8.3, and place it inside a clean glass vessel that has been mounted on the segregator as described in Section 8.1.
2. Insert the isokinetic splitter stopper into the top opening of the glass vessel.
3. Remove the red plugs from the inlet and outlet of the filter cassette.
4. Attach a filter cassette to the top post of the isokinetic splitter.
5. Attach the PVC tubing from the bulkhead barb labeled “from sample filter” on the top of the segregator to the top outlet peg on top of the filter cassette.
6. Set valves to achieve the desired flow rate. There are two flow setting options. The low-flow setting accommodates collecting samples at a flow rate of approximately 200 cubic centimeters of air per minute (cc/min) onto a standard 25-mm MCE filter. The adjustment knob on the bottom of the rotameter should be set accordingly to achieve a flow rate of 200 cc/min.
7. Set the digital timer for a minimum of 3 minutes.
8. Press the “on” button on the digital timer, which will engage the vacuum pump and the vibration unit.
9. Monitor the flow and vacuum gauges and adjust as necessary to maintain settings. If the system is operating properly, readjustments will not be necessary. Record instrument readings in the notebook.

10. After the vacuum pump and vibrator unit are shut off, disconnect the filter cassette, remove the additional cowl, and replace the cap and end plugs of the sample cassette.

8.6 Post-Sampling Decontamination

1. Repeat steps in Section 8.4.
2. In addition, if mineral oil in the dust collector needs to be replaced (oil will appear dark with sediment settled on the bottom), empty the contents of into a rigid disposable container and dispose of it as asbestos waste material.

9.0 Record Keeping, Chain of Custody, and Shipping

Sampling records should be maintained in a logbook in the format equivalent to the example provided in Appendix 1. To correct errors in the logbook, make a one-line mark through the error and initial and date the change.

Use the FBAS Chain of Custody (COC) Form or equivalent COC form to record sample information and analysis required for the laboratory. An example of the form is included in Appendix 2. Containers used to transport/ship sample cassettes should be secured with a chain of custody seal, or equivalent. An example of the seal is included in Appendix 3.

Ship air sampling cassettes in a rigid container with cassettes in an upright position (filter facing upward) with the cowl attached. Use packing material to prevent jostling or damage during shipment. Do not use untreated polystyrene foam as packing because electrostatic forces may cause fiber loss from the filter.

The condition of samples and the custody seals should be noted on the COC form upon arrival at the laboratory.

10.0 Calculations and Reporting Results

The analytical laboratory will generally record the results of the TEM analysis of the filter in the format included in the analytical method.[4,5] These data can be used to estimate the concentration of asbestos in the soil prepared using the FBAS. Alternatively, the laboratory may use the FBAS specific National Asbestos Data and Evaluation Sheet for TEM. This electronic data deliverable can be found at the link below:

https://response.epa.gov/site/doc_list.aspx?site_id=4525

The concentration of asbestos in soil reported from the analysis of a filter may be expressed in two alternate ways:

1. **Structures of asbestos per gram of soil (s/g).** The basic formula for calculating concentration in these units is as follows:

C_{soil} (s/g) = N · S

where:

N = number of asbestos structures counted (s)

S = analytical sensitivity (g^{-1})

The analytical sensitivity (S) is calculated as follows:

(g^{-1}) = EFA / (GOx · Ago · M · Q_R)

where:

EFA = effective filter area (square millimeters [mm^2])

Q_R = flow ratio; this is the fraction of air passed through the soil sample (V_{total}) that is captured on the air filter (V_{filter}), and is calculated as:

$$Q_R = V_{filter}/V_{total}$$

GOx = Number of grid openings evaluated

Ago = Area of one grid opening (mm^2)

M = mass of asbestos-containing soil placed in the FBAS (g); does not include the mass of sand

2. **Mass percent (grams of asbestos per 100 grams of soil).** In order to express soil concentration as mass percent, the mass of each asbestos structure observed is estimated from its dimensions. In the absence of detailed data on the true geometry of each particle, the mass is approximated by assuming a simple rectangular solid shape, as follows:

$$m_i = l_i \cdot w_i^2 \cdot \delta \cdot 1E\text{-}12$$

where:

l_i = length of structure *i* (um)

w_i = width of structure *i* (um)

δ = density of asbestos (e.g., LA = 3.1 g/cm^3, chrysotile = 2.6 g/cm^3)

1E-12 = conversation factor (cm^3 per um^3)

The concentration, expressed as mass percent (grams of asbestos per 100 grams of soil), is then calculated as follows:

$$C_{soil} \text{ (mass percent)} = \Sigma m_i \cdot S \cdot 100$$

11.0 QA/QC Evaluation

Accuracy, precision, and detection limit have not been completely evaluated for this method.

11.1 Negative Controls

Lot Blanks

A minimum of two lot blanks from each filter lot used will be analyzed to determine the mean asbestos structure loading. If the mean count of all types of asbestos structures is > 10 structures per square millimeter (s/mm^2), or if the mean count of asbestos fibers and/or bundles longer than 5 µm is > 0.1 s/ mm^2, the filter lot should be rejected. The analysis of blanks shall be performed in such a manner as to achieve an equivalent number of grids counted as to be comparable to those of the sample set.

Preparation Blanks

Preparation blanks shall be submitted for analysis at a frequency specified in a site/project specific QAPP and will be evaluated according to acceptance criteria specified in the QAPP. A preparation blank is a filter that is left uncovered on the bench top inside the FBAS hood during processing of soil samples with the FBAS. It is a measure of general laboratory cleanliness.

Sand Blanks

A sand blank shall be submitted for analysis at a frequency specified in a site/project specific QAPP and will be evaluated according to acceptance criteria specified in the QAPP. A sand blank consists of a filter generated from operating the FBAS as described in Section 8.5 with 20 g clean sand added to a clean glass vessel, but without adding a soil sample.

11.2 Performance Testing Samples

Soil samples prepared as PT samples may be spiked with known concentrations of asbestos reference materials (see Section 7). Samples should be collected in accordance with this method. Sampling conditions such as flow rate, sampling duration, sample size, etc. should be documented in the project notebook – per Section 8.2.

11.3 Calibration

The balance used to weigh samples should be checked daily with a set of NIST traceable metric test weights to verify accuracy. The flow meters on the FBAS should be checked with a rotameter calibrated to a primary standard at least once a week. Replace the inlet and bypass HEPA filters whenever they are observed to be excessively discolored or the total flow diminishes appreciably. Always change the HEPA filters before total flow approaches 15 liters per minute; the total flow should not be allowed to fall below 15 liters per minute. Replace both check valves when the HEPA filters are changed. The check valves should also be replaced if the FBAS between sample preparation projects from different sites.

12.0 Method Performance

EPA has conducted three primary studies to evaluate the performance of the FBAS preparation method to support validation. A study conducted by EPA in 2011 used PE standards, which were prepared by combining different matrix materials (soil and vermiculite) and different types of asbestos (chrysotile and amphibole). The nominal chrysotile levels in the soil PE's were 0.0001%, 0.001%, 0.01% and 0.1% by mass. Another set of soil samples was spiked with amphibole asbestos from the Libby Superfund Site.[1] A second study involved testing PE standards prepared by spiking soil with different concentrations of erionite ranging from 0.1%, 0.01%, 0.001%, and 0.0001% erionite by weight.[8] A third interlaboratory study conducted in 2015 used PE standards consisting of Arvada, Colorado soil containing either fibrous Libby amphibole structures, amosite asbestos, or chrysotile asbestos, at concentrations of 0.01%, 0.005%, 0.001% and 0.0005% by weight.[9] The analytical results of all three studies illustrate an approximate linear relationship between the nominal concentration of asbestos or erionite (as % by weight) in the PE standard and the concentration estimated by TEM analysis expressed as asbestos or erionite structures per gram of test material (s/g). The method detection limits in each of these studies fall within 0.002% to 0.003% by weight, which is approximately 100-times lower than typical detection limits for soils by PCM.

The graphs in Figures 8, 9, and 10 summarize the average results of analysis for each type of PE standard combing the results of the 2011 and 2015 studies. Mean concentrations were computed across all filter replicates for each PE standard, using a value of zero for filter replicates where no structures were observed. Mean concentrations are shown as structures per gram. Figure 11 shows the percent recovery for the analysis of asbestos when results from the 2015 study are calculated as mass percent. Figure 12 displays within-laboratory variability for the 2015 study (based on structures/gram). Figure 13 shows a graph summarizing the average results of analysis of erionite in the 2013 study.

8 Berry, David; Januch, Jed; Woodbury, Lynn; Kent, Douglas (submitted for publication March 2018). Detection of Erionite Fibers in Soils Using the Fluidized Bed Preparation Methodology.

9 Fluidized Bed Asbestos Segregator Interlaboratory Study (Manuscript in process).

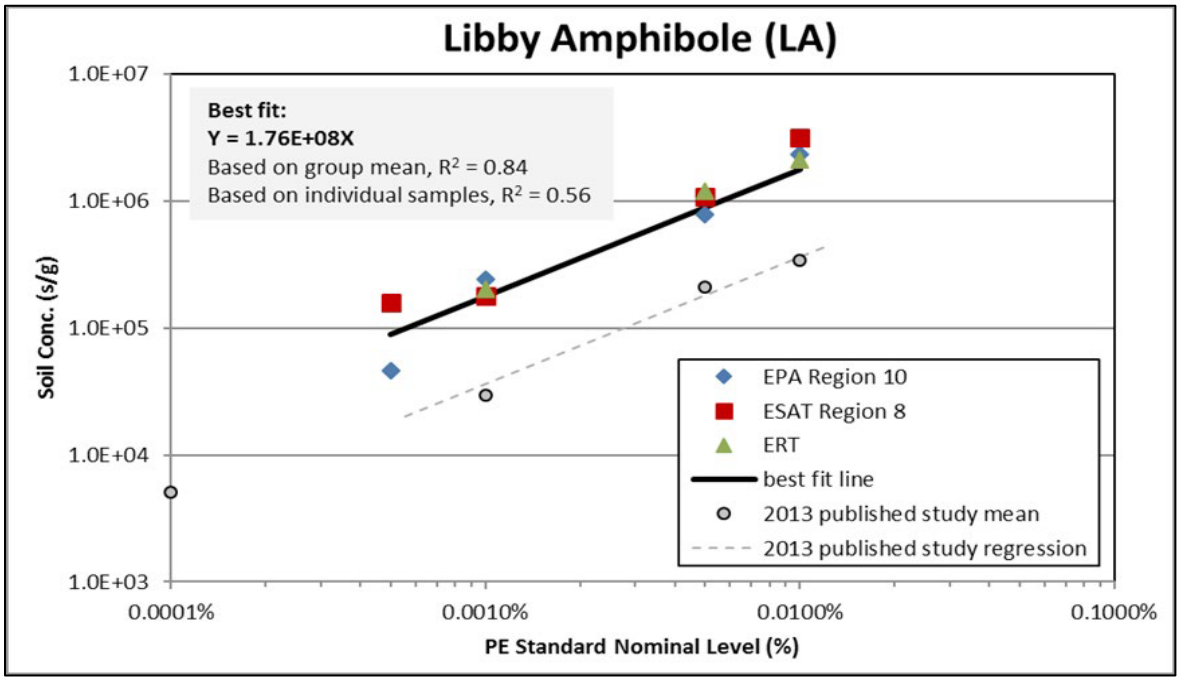

Figure 8 – PE sample results – Average Libby Amphibole Results from 2011 and 2015 studies.

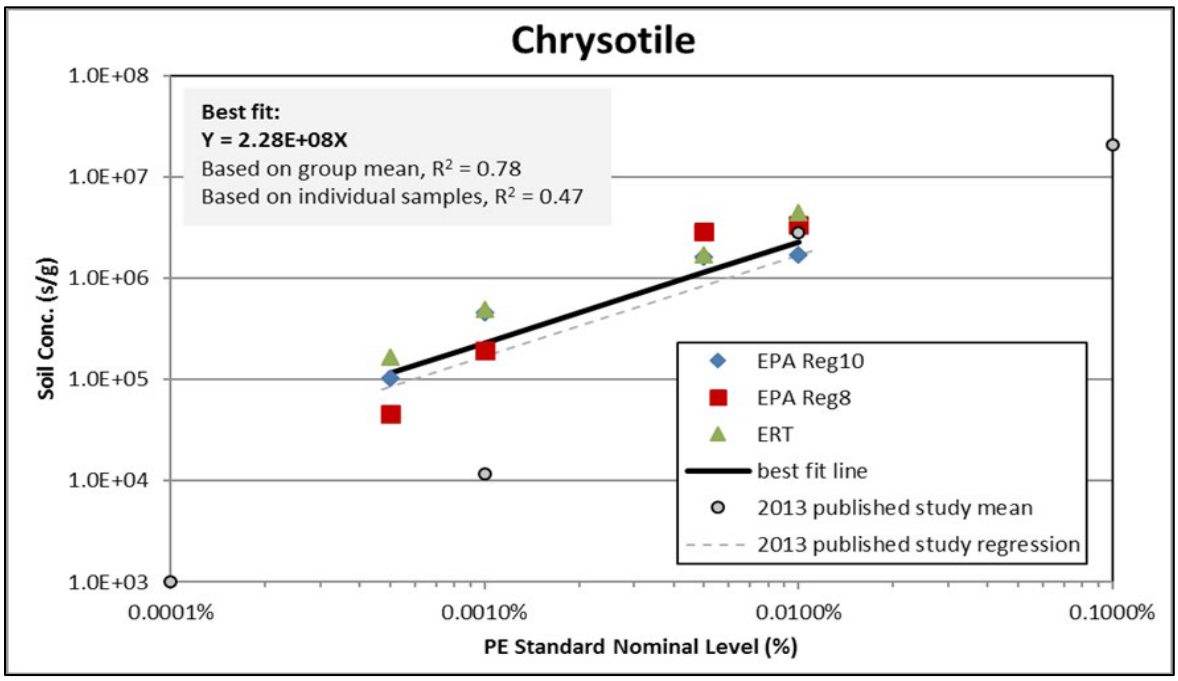

Figure 9 – PE sample results – Average Chrysotile Results from 2011 and 2015 studies.

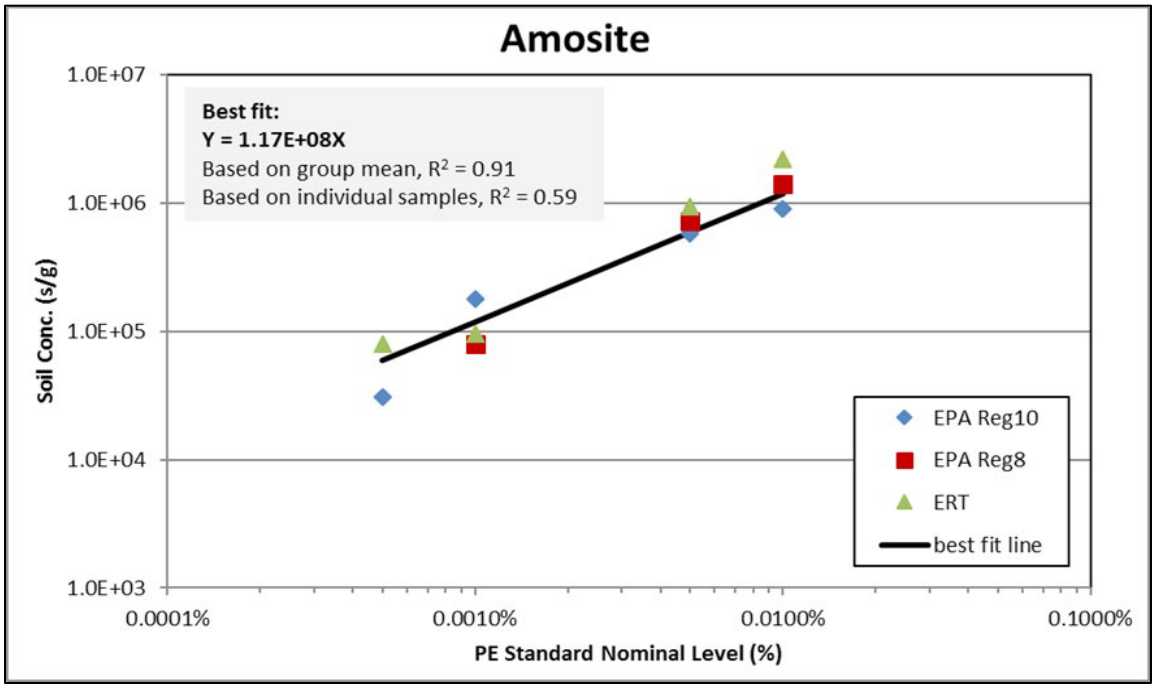

Figure 10 – PE sample results – Average Amosite Results from 2015 study.

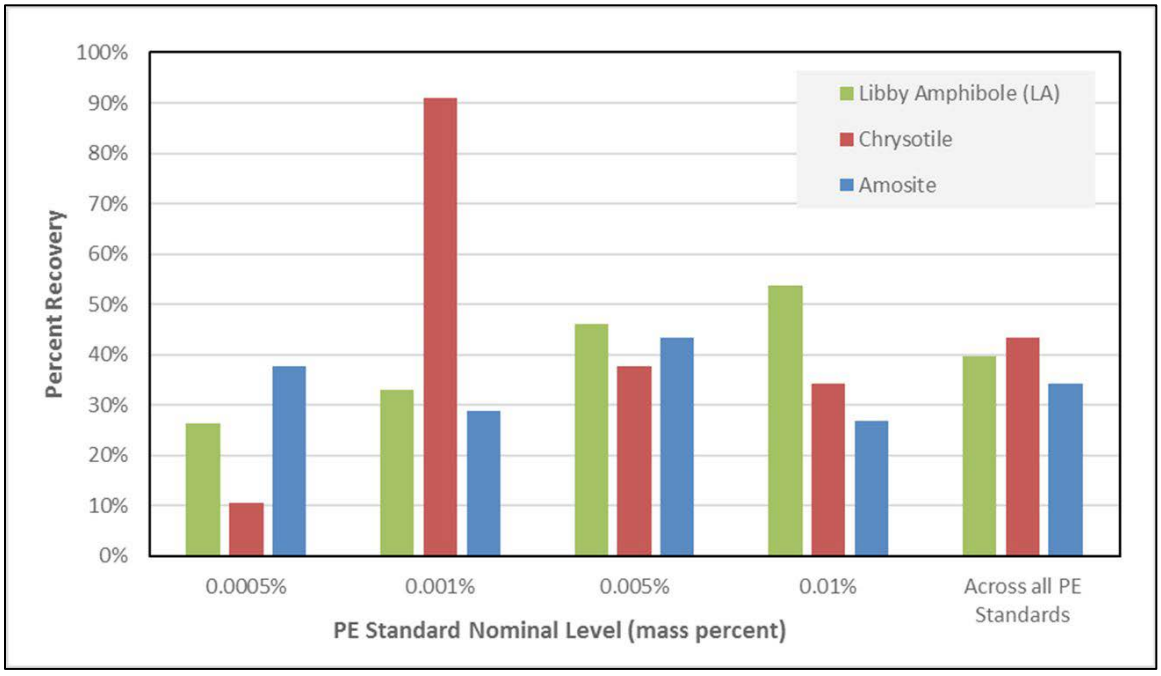

Figure 11 – PE sample results – Percent Recovery from 2015 study.

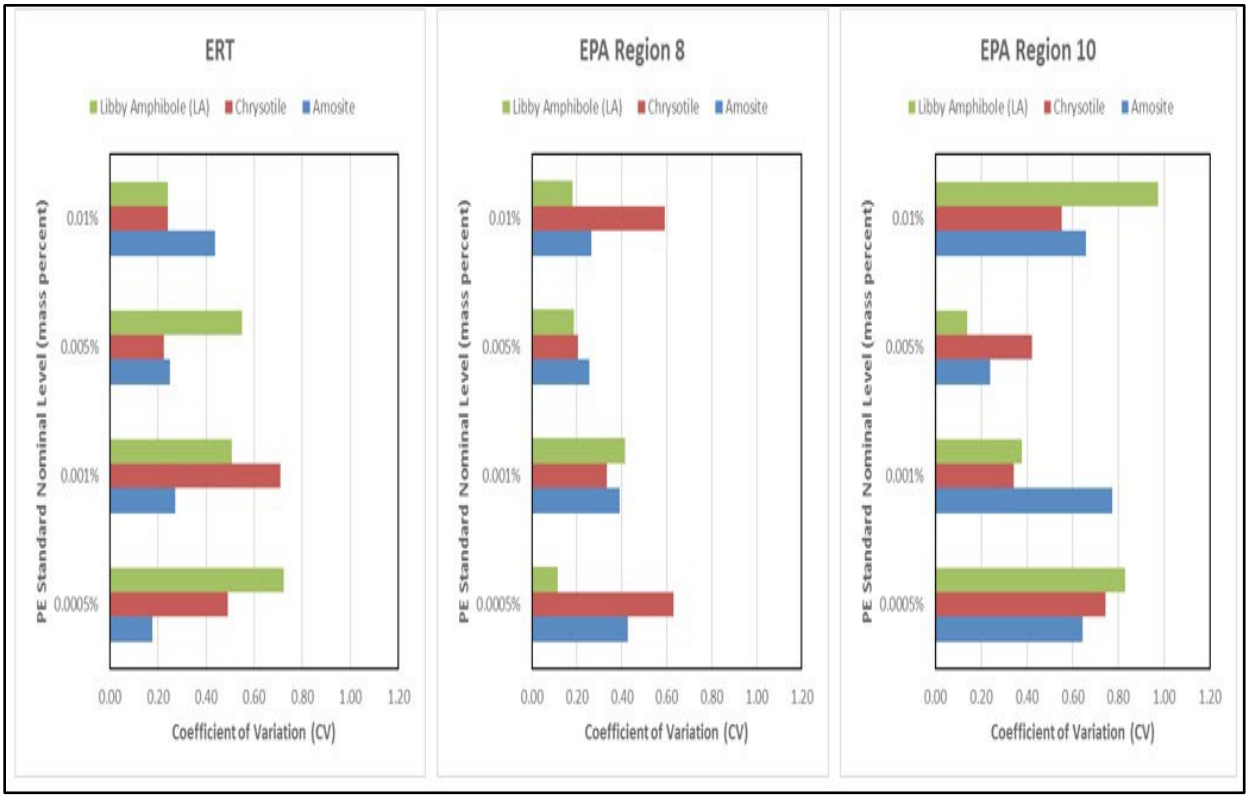

Figure 12 – Within-laboratory variability for 2015 study (based on structures/gram).

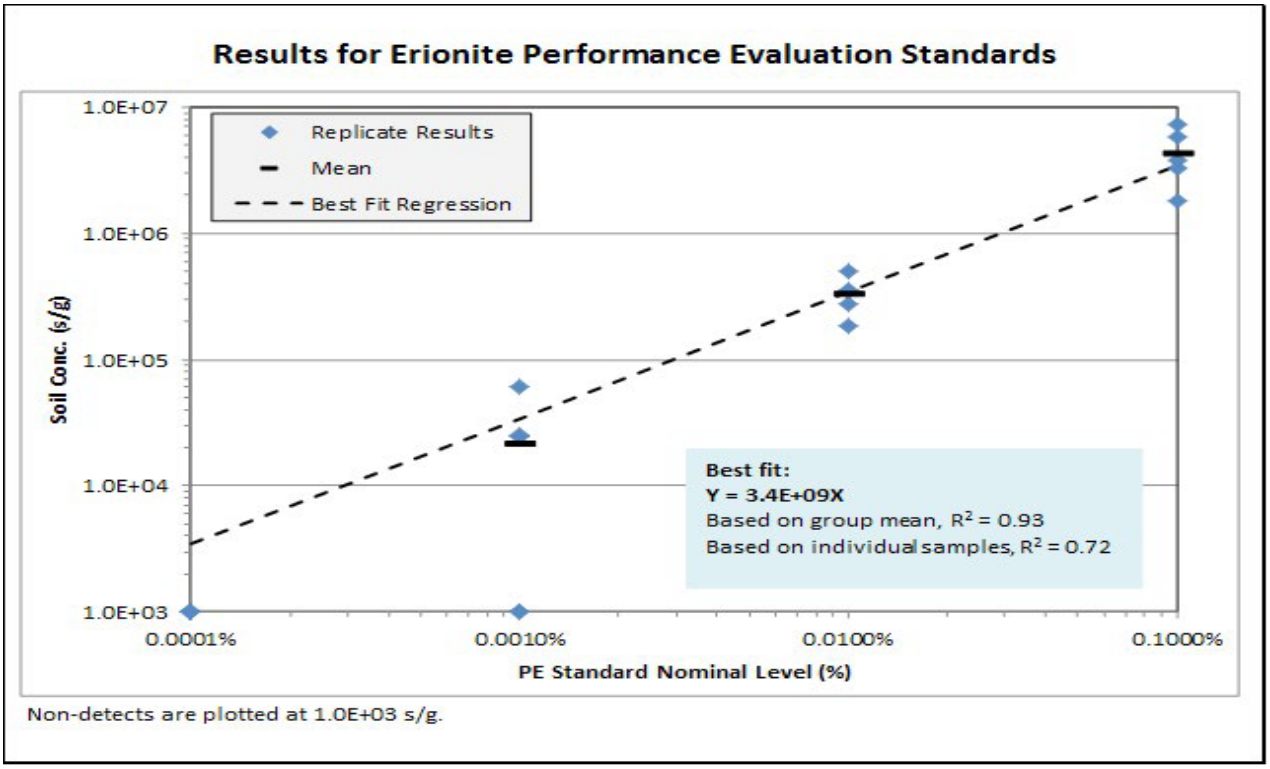

Figure 13 – PE sample results – Average Erionite Results from 2013 study.

13.0 Pollution Prevention

Environmental factors should be considered in acquisition, use, and disposal decisions supporting asbestos analysis activities.

14.0 Waste Management

Waste materials potentially contaminated with asbestos, including used flow splitters, meshed stoppers, spent sample, spent sand, mineral oil from the dust collector, and HEPA filters, should be placed in double zip-top plastic bags and labeled with an asbestos warning labels. Asbestos waste material should be disposed of at an approved asbestos waste disposal facility per local regulations.

15.0 References

1. Januch J, Brattin W, Woodbury L, Berry D (2013) Evaluation of a fluidized bed asbestos segregator preparation method for the analysis of low-levels of asbestos in soil and other solid media. Anal Methods 5:1658–1668.
2. EPA. 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-06/001. February. http://www.epa.gov/quality/qs-docs/g4-final.pdf.
3. EPA. 2001. *EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5.* U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-01/003. March. http://www.epa.gov/quality/qs-docs/r5-final.pdf.
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5. International Organization for Standardization, *Ambient Air – Determination of Asbestos Fibres – Indirect Transfer Transmission Electron Microscopy Method*, Reference Number ISO 13794:1995(E), July 1995.
6. Title 40 of the Code of Federal Regulations (CFR) 763.83.
7. IARC. 2012. Arsenic, metals, fibres, and dusts. In IARC monographs on the evaluation of carcinogenic risks to human, vol. 100C. IARC Press, 150 Cous Thomas, 69372 Lyon cedex 08, Lyon, France.
8. Berry, David; Januch, Jed; Woodbury, Lynn; Kent, Douglas (submitted for publication March 2018) Detection of Erionite Fibers in Soils Using the Fluidized Bed Preparation Methodology.
9. Fluidized Bed Asbestos Segregator Interlaboratory Study (Manuscript in process).

Appendix 1 – Example of a FBAS Sample Log

FBAS Sample Log Sheet **Page____ of ____**

Project Name: ____

Operator(s):____

Sample Date: ____________________ Temperature: ________ Humidity: ________ Q_R: __________

Sample Time	Sample ID	Sample Type Check One					Total Sample Weight (g)		FBAS Sample Weight (g)		Comments
		Field Sample	Sand Blank	Lot Blank	Preparation Blank	FBAS Replicate	>850 µm fraction	<850 µm fraction	Soil	Sand	

Appendix 2 – Example Chain of Custody Form for FBAS

FBAS CHAIN OF CUSTODY RECORD **COC No.:** _____
[FBAS-YYMMDD###]

COC entered by (signature): _____ **COC Page** _____ **of** _____

Project Manager: _____ **Project Identifier:** _____

Method of Shipment: _____ **Shipment Tracking No.:** _____ **Ship Date:** _____

Shipment Destination: _____ **Shipment Origin:** _____

Sample Information		Sample Preparation Request				Filter Analysis Request				FBAS Information			
Sample ID (PE Standard ID or Replicate Filter ID)	**Medium** *[S for soil; A for air filter]*	OEA FIELDSOP-102 (Rev 1)++	with cyclone	without cyclone		TEM, ISO 10312:1995(E)**				**Blank?** *[mark 'x' if yes]*	**FBAS Flow Ratio** (Q_R)	**FBAS Soil Mass** (grams, dry weight)	**Sample Remarks**

RELINQUISHED BY:		RELINQUISHED TO:	
NAME, AFFILIATION	**DATE, TIME**	**NAME, AFFILIATION**	**DATE, TIME**

COMMENTS/CONDITION OF SAMPLES:

++ Prepare replicate filters using fluidized bed asbestos segregator in accordance with SOP.

** Specific recording and stopping rules are specified in a site/project specific QAPP. As needed, grids may be prepared indirectly in accordance with ISO 13794:1999(E).

Appendix 3 – Example Custody Seal