EMSL Analytical S.O.P.

Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials with Berman and Kolk Modifications (May 2000)

EPA 540-R-97-028

1.0 Method Description

1.1 Applicable Matrix

This elutriator method is a sampling and analysis method for the determination of the amount of respirable asbestos in soils and bulk materials.

1.2 Scope and application

It is designed to provide results that can be used for risk assessment purposes. Measurements from this method can be combined with published dust emission and dispersion models to provide predictions of airborne asbestos concentrations when asbestos containing bulk materials are disturbed. The Berman and Kolk modifications to the EPA 540 document include changes to the design of the dust generator, changes to the way filters are prepared for TEM analysis, changes to the TEM analysis counting rules, and changes to the calculations for reporting concentrations.

1.3 Summary of Method

Samples submitted for this method are placed in the tumbler of a dust generator. The purpose is to entrain the fine fraction of the sample into the air. The air stream with particulate is passed through a vertical elutriator to isolate the respirable fraction of the particulate. This fraction is then collected on a pre-weighed TEM filter. After collection the filter is weighed to determine the mass of respirable dust that was collected. After weighing, the sample filters are prepped and analyzed as per the ISO 10312 method with analysis restricted to protocol structures i.e. those fibers that are longer than 5 microns and thinner than 0.5 microns. In addition fibers greater than 10 microns in length need to distinguished and enumerated separately. Results are reported as asbestos structures per gram of respirable particulate.

2.0 Interferences

Various interferences may be encountered in both the soil and debris matrices during this analysis. These interferences include (but are not limited to):

2.1 Fibrous materials found in soils such as roots, wood, etc.

2.2 Matrix materials such as titanium dioxide (TiO_2), tar, etc.

2.3 Asbestos mineral "look-alikes" such as talc, attapulgite, halloysite, sepiolite, wollastonite, etc.

2.4 Any material which is long, thin, and small enough to be viewed under the microscope can be considered interference for asbestos.

3.0 Definitions

3.1 Analytical Sensitivity: the calculated asbestos concentration in soil or bulk matrix in asbestos structures/gram of respirable dust equivalent to the counting of one asbestos structure.

3.2 Aspect Ratio: the ratio of length to width of a particle

3.3 Elutriator: a device in which differential flow through a fluid (gas or liquid) against an opposing force (i.e. gravity) is employed to separate particles by size.

3.4 Fiber: an elongated particle that has parallel or stepped sides. In this method only structures meeting the length and width criteria of protocol structures are enumerated.

3.5 Isokinetic Sampling: sampling air in such a manner so as not to disturb the direction or velocity of air flow at the point sampled.

3.6 PCME: Phase Contrast Microscopy (or PCM) Equivalent fibers = structures longer than 5 microns, thicker than 0.25 microns, and have an aspect ratio of at least 3:1

3.7 Protocol Structures: asbestos structures longer than 5 microns and thinner than 0.5 microns

3.8 Respirable Dust: particulate matter in a size range capable of penetrating the deep lung and being deposited in terminal bronchioles and alveoli (beyond the reach of the ciliary escalator). Respirable dust is typically abbreviated as PM_{10}

3.9 Riffle Splitter: a device composed of a hopper and multiple, uniform, parallel chutes that alternately feed from the hopper to opposing receiving trays

4.0 Safety

4.1 All personnel performing preparation and/or analysis of samples must be familiar with the EMSL Chemical Hygiene Plan (EMSLChemHygiene 200.0).

4.2 This test may involve hazardous materials such as asbestos, which is a known carcinogen.

4.3 Sample preparation should be performed in a HEPA filtered negative pressure hood or best management practices should be utilized

4.4 Technicians and analysts should also adhere to good laboratory safety practices when utilizing this protocol (i.e. gloves, safety glasses, proper laboratory attire, etc.).

5.0 Equipment

5.1 Acetone Vapor Generator

5.2 Balance - Analytical - with a resolution of at least 5 decimal places (1×10^{-5}) with a capacity of at least 30 g

5.3 Balance - Micro-Analytical - with a resolution of at least 6 decimal places (1×10^{-6}) with a capacity of at least 200 mg

5.4 Balance - Pan - with a capacity to at least 1500g (reproducibility +/- 0.1g)

5.5 Dust Generator / Elutriator built to the specifications of this method

5.6 Drying oven capable of 50 - 110°C

5.7 Hood (HEPA Laminar Flow)

5.8 Hood (HEPA filtered negative pressure).

5.9 Plasma Asher (low temperature)
5.10 Riffle splitter with at least 24 at least ¾" chutes
5.11 Sieve (ASTM 3/8")
5.12 Transmission Electron Microscope with the following capabilities:
5.12.1 80-120 Kev
5.12.2 fine probe size <250 nm
5.12.3 Energy Dispersive X-Ray Analyzer capable of resolution <175 eV
5.13 Vacuum Evaporator (Carbon Coater) with Graphite or Carbon rods

6.0 Reagents and and Supplies

6.1 Acetone-reagent grade or better
6.2 Di-Methyl Formamide (DMF)
6.3 Glacial Acetic Acid
6.4 Filters - 0.45 micron MCE
6.5 Filters - 0.4 micron PC
6.6 filters - 0.2 micron PC
6.7 Forceps
6.8 Glass Petri Dishes
6.9 Glass microscope slides
6.10 Grids, Grid Clips and Grid Storage Boxes
6.11 Jaffe Wick Washer
6.12 Kim wipes or alternative paper
6.13 Sample containers (should be plastic to avoid breakage during shipping)
6.14 Weighing dishes

7.0 Sample Collection, Preservation, Shipment and Storage

7.1 Sample Collection

7.1.1 Determining when, where and how many soil samples to collect at a site can be a complex issue. A well developed field sampling plan is generally necessary to obtain accurate and representative results. These issues are the responsibility of the client.

7.1.2 For individual samples the target *minimum* sample size to be collected in the field is 1 kg. Larger samples may be required if particularly large (> 4 or 5 cm) rock or debris is present as these will be removed by sieving.

7.1.3 Field Preparation of samples is necessary prior to sending them to the lab. The steps are described in section 9.2

7.1.4 Only the fine fraction samples need to be submitted to the lab however the weight of the course fraction if any should be provided to the lab as well.

7.2 Sample Preservation and Shipment

7.2.1 Sample containers should be sealed, labeled, and cleaned with a damp cloth.
7.2.2 Record the date and time of each sample collection.
7.2.3 Fill out an appropriate Chain of Custody completely.

7.2.4 Samples should be delivered or overnight shipped to the lab in a cooler with ice to limit bacterial growth.

7.2.5 If the samples are not going to be processed immediately they should be stored at ice temperature.

8.0 Calibration and Standardization

8.1 Instrument calibration procedures can be found in the EMSLQCSOP, the analytical method, the AHERA method and the NVLAP handbook.

9.0 Procedure

9.1 Sample Receipt

9.1.2 Upon receipt of samples, check that the sample information on the Chain of Custody (COC) matches the information on the samples and other paperwork.

9.1.3 Any discrepancies must be resolved before proceeding.

9.1.4 If the samples do not have a COC, then one must be completed at time of log in. The client should fill out the necessary information completely.

9.1.5 See EMSL's QA Manual for complete description of EMSL's log in procedures and requirements.

9.1.6 Wipe the samples clean with a moist cloth prior to handling or storage.

9.2 Preliminary (Field) Preparation of Samples

All of the steps outlined in this section (9.2) should be performed in the field by the sampling personnel but often times are not. The laboratory can perform these (for a fee) as an extra service to the client. Call your sales rep for pricing.

9.2.1 Sample Types

9.2.1.1 *Consolidated samples* (rock) need to be course crushed so that a majority of the sample passes through a 3/8" (1 cm) sieve. The material can then be treated as per section 9.2.2

9.2.1.2 *Unconsolidated, homogenous, fine material* generally needs no field preparation. However if the sample size is greater than 70 grams the sample should be split with a riffle splitter as per section 9.2.4 until the final pair of splits is between 40 and 70 grams.

9.2.1.3 *Unconsolidated, heterogeneous material* tends to be the most common sample type. Proceed to section 9.2.2

9.2.2 **Weigh** the initial sample on a scale with precision to at least +/- 10 grams.

9.2.3 **Separate** into course and fine fractions by passing through a 3/8 inch (1 cm) sieve. Push clods of dirt and other soft aggregates through the sieve by hand. Stones and debris remaining on the sieve that cannot be hand crushed shall be transferred to a separate container (course fraction).

9.2.4 **Weigh** the course and fine fractions on a scale with precision to at least +/- 10 grams.

9.2.5 **Homogenize** the fine fraction with a riffle splitter with chutes 3/4 to 1" in size. This must be done in a full size lab hood. If the sample is wet or even visibly moist, the sample should be dried in a 60°C drying oven prior to riffle splitting.

9.2.5.1 Place the entire fine fraction of the sample into a dispensing pan or empty receiving tray.
9.2.5.2 Place two empty receiving trays under the splitter to receive the sample as it pours through the chutes.
9.2.5.3 Place the long edge of the dispensing pan or receiving tray against the inside long edge of the hopper. Slowly rotate the tray so that the container empties into the hopper and flows through the chutes.
9.2.5.4 Tap the sides of the riffle splitter to dislodge any material that failed to pass through the chutes.
9.2.5.5 Remove both receiving trays from under the splitter and slowly combine the contents into one tray.
9.2.5.6 Repeat steps 9.2.5.1 through 9.2.5.5 for at least 5 total iterations.

9.2.6 **Split** the now homogenized fine fraction with a riffle splitter to produce 2 samples weighing between 40 and 70 grams each. This is done as follows:
9.2.6.1 Repeat steps 9.2.5.1 9.2.5.4. DO NOT perform step 9.2.5.5.
9.2.6.2 Pour the contents of one of the trays into a new unused gallon sized plastic bag.
9.2.6.3 Repeat steps 9.2.5.2 through 9.2.5.4. until the mass of material in one pan after one cycle is between 80 and 140 g.

Note: It may be helpful to obtain a tare weight on one of the receiving trays so that you can quickly weigh the sample after each cycle.

9.2.6.4 Riffle split one more time. This should create 2 subsamples weighing 40-70 grams each
9.2.6.5 Weigh **both** of the final (40-70 gram) samples (subtract the tare weights), their weights, and place each subsample in a separate, unused jar or plastic bag.

Note: Samples should always be submitted to the lab as duplicate splits.

9.3 Lab Preparation of Samples

9.3.1 Once the above field prep procedures have been carried out, whether by the client or the lab you should now have 2 bags or containers of soil for each. One is held for archival and/or QC purposes. The other is prepped (conditioned) for the elutriator.
9.3.2 If each of the two sample splits is between the target of 40 and 70 grams, skip section 9.3.3 and 9.3.4 and proceed to step 9.3.5.
9.3.3 *Dilution of Samples < 40g with play sand*
If the sample submitted to the lab yields < 40 grams after the preparation steps to this point, then the sample must be diluted with dry, washed play sand for a total mass of approximately 70 grams (soil and sand).

9.3.3.1 If dilution with play sand is necessary then minimum particle size should be 0.5 cm not the regular 3/8" (1cm) recommended minimum. Sieve if necessary to ensure this is the case.

9.3.3.2 Weigh and record any fraction that does not pass through the 0.5 cm sieve

9.3.3.3 Within a hood, combine the sample (everything < 0.5 cm) and sand in a clean plastic bag and shake to homogenize. Its dry weight should be between 40 and 70 grams.

9.3.3.4 Record initial weight, weight of play sand, and final weight.

9.3.3.5 At this point the sample is ready to be conditioned in the elutriator.

9.3.4 *Homogenization and Splitting of Samples > 70 grams*

If the sample submitted to the lab weighs > 70 grams, then the samples must be homogenized and split to obtain the targeted 40-70 grams.

9.3.4.1 Follow steps 9.2.5 for homogenization.

9.3.4.2 Follow steps 9.2.6 for splitting the sample.

9.3.5 Checking the samples for moisture content.

9.3.5.1 Samples need to be as close to 50% humidity as possible. A Soil moisture meter can be used to determine if samples need to be dried prior to conditioning. Fully insert the rods of the soil moisture meter into the soil. Both rods must be completely covered with soil to obtain an accurate reading.

9.3.5.2 If the volumetric water content (VWC) reads 40 % or less, than the sample is ready to condition (section 9.6.2). If the VWC is greater than 40 %, the sample must be dried in a 60 degree Celsius oven until its VWC is below 40%.

9.3.5.3 Some clients require moisture content to be determined regardless of the moisture content. If this is the case, samples must be dried in a 60 degree Celsius oven overnight. The pre- and post- drying weights must be recorded.

9.4 Conditioning and Pre-weighing Sample Filters

9.4.1 Mixed Cellulose Ester (MCE) Sampling Filters

Prior to using the dust generator, a supply of <u>at least</u> 15 MCE filters (0.45 micron pore size, 25 mm diameter), all from the same filter lot, must be conditioned overnight to 50% Relative Humidity (RH).

9.4.1.1 Expose the filters in a dessicator with the saturated $CaNO_3 * 4H_2O$ solution or alternatively in an environmental chamber held at 50% RH.

9.4.1.2 Pre-weigh each filter to a minimum precision of plus or minus 0.000002g

9.4.1.3 Place each pre-weighed filter in a plastic petri dish with the filter weight marked on the lid.

9.4.2 Polycarbonate (PC) Filters

Pre-weigh at least 10 PC (polycarbonate) filters of either 0.1 or 0.2 micron pore size

9.4.2.1 These filters are not sensitive to humidity changes in the 40% to 55% Relative Humidity (RH) range. This range can be easily achieved in an air air conditioned office environment.
9.4.2.2 The 0.2 micron pore size filters are suitable when only protocol structures are of interest.
9.4.2.3 The 0.1 micron pore size filters are recommended if fibers down to 0.5 microns in length are to be collected and counted.
9.4.2.4 Weigh ten of the appropriate pore size PC filters on a microbalance with a precision of $\pm$ 0.000002g .
9.4.2.5 Place each pre-weighed filter in a plastic petri dish with the filter weight marked on the lid.

NOTE: In practice it is most efficient to build up a large inventory of conditioned and pre-weighed MCE and PC filters.

NOTE: MCE filters are preferred over PC filters. Despite their insensitivity to humidity, PC filters have a high static charge and are extremely flimsy. PC filters are very susceptible to folding in on themselves. This disrupts the uniformity of the deposited sample and can create sample loss. Properly conditioned MCE filters are easier to handle and yield better preps.

9.5 Setting Up the Dust Generator

9.5.1 Initiating Humidity Control

Humidity control is very important to the standardization of the entire sampling process. In addition to the the pre-conditioning of the filters and samples prior to tumbling, the actual sampling of the air during the tumbling process and separation of the respirable from non respirable particles in the elutriator must all be performed at as close to 50% RH as possible. This is achieved with a combination of environmental controls in the room and saturated solution of $CaNO_3$ * $4H_2O$ in the environmental chamber.

9.5.1.1 *Room humidifiers/dehumidifiers* should have a rated output appropriate for the size of the enclosed room. They should also have an electronic humidistat that turns the unit on and off at specific set humidities.

9.5.1.2 *Moisture chambers* should be enclosed chambers of plexiglass, glass or other similar construction with two exposed containers of a saturated $CaNO_3$ * $4H_2O$ solution.

9.5.1.2.1 Prepare the salt solution by placing 1000g of the $CaNO_3$ * $4H_2O$ into a 1 Liter container. Add filtered, distilled water to fill the container.
9.5.1.2.2 With a marker, mark the level of solid salt, as well as the level of total solution on the container. The container should be capped except when adding additional water or salt on succeeding days as necessary.
9.5.1.2.3 The solution has stabilized when there is no apparent change in the relative amount of salt and water from day to day. Equilibrium can

be achieved faster by agitation, however, no water or salt should be added less than 24 hours from the point of use.

9.5.1.2.4 To load the salt solution, remove the trays from the humidity chamber (dessicator).

9.5.1.2.5 Fill each tray with the saturated salt solution making sure to get approximately 25% by volume of undissolved salt into each pan.

9.5.1.2.6 Replace the pans back into the humidity chamber and close the door. The primary entrance of air should be through the enclosure's top openings.

NOTE : It is important to keep the door shut on the humidity chamber at all times. This helps ensure the salt solution maintains 50% RH in the chamber.

9.5.2 Adjusting the Initial Air Flow

The correct air flow within all components of the dust generator and elutriator is critical so that the respirable particles are properly separated.

9.5.2.1 The proper linear flow rate in the elutriator shall be set at 0.31 cm/s as per the method. Based on the construction of the elutriator as laid out in the method this corresponds to the following flow rates:

9.5.2.2 The flow rate for the IST opening should be calibrated to 72 ml/min

9.5.2.3 The flow rate for the ME opening should be calibrated to 1,430 ml/min

NOTE: Air flow requirements need to be calculated.

$$V_v = 81.1 * V_1$$

Where:

V_l = estimated linear flow rate required to separate respirable particles
This is given by the method to be 0.31 cm/s

81.1 = cross sectional area of the 4" diameter elutriator tube
Pi (r^2)= (3.14)((4)(2.54)) = 81.1

V_v = corresponding volumetric flow rate (cm^3/s)

Therefore: $V_v = (81.1)(0.31\ cm/s)$

$\mathbf{V_v = 25.14\ cm^3/s}$

This flow rate (25.14 cm^3/s) needs to be divided between the IST and the ME openings

% area of IST opening vs entire cross section of elutriator = Area of IST ÷ 81.1 = 4.7%

Then 4.7% of 25.14 = 0.047(25.14) = 1.19 cm^3/s = 72 ml/min

Note: cm^3 = ml

Remaining 95.3% flow goes through the ME port
Then 95.5% of 25.14 = 0.953(25.14) = 23.9 cm^3/s = **1,430 ml/min**

9.5.3 Determining Proper Air Flow for a Dust Generator Run

Before beginning a run, the elutriator must be set-up properly. Filter cassettes should be placed in the top of the elutriator and appropriate filters should be mounted in each cassette (0.45 micron ME filters with backing pads over the ME openings and 0.2 micron PC filters with backing pads over the IST openings). Air flow is calibrated while performing leak checks (Section 9.5.4).

9.5.3.1 The ME airflow is to be adjusted to 1430 ml/min. Calibrate this airflow as close as possible. There is an acceptable variance of ± 20 ml/min.

9.5.3.2 The IST airflow is to be adjusted to 72 ml/min. Airflow may be higher than this value, but MUST NOT be lower.

9.5.4 Checking for Leaks

The filter assembly over the IST opening is very sensitive to leaks due to the low air flow. Therefore, it is important to check for leaks before every run. Leaks do not substantially affect the ME opening due to the higher air flow, but should still be checked for.

9.5.4.1 *IST Leak Checks and Flow Calibration*

9.5.4.1.1 To check for leaks on the IST opening, disconnect the tubing junction to the left of the elutriator stand that is labeled IST. Pull the tube with the clamp off of the barb. Place tubing connected to the suction end of a dry-cal onto the barb.

9.5.4.1.2 Use the dry-cal and IST rotameter valve to adjust the airflow to a minimum of 72ml/min. The air flow MUST be above 72ml/min. Record the air flow and disconnect dry-cal. Replace the initial tubing back onto the barb and tighten the clamp.

9.5.4.1.3 Remove the top of the elutriator and place a No. 3 stopper with tygon tubing into the bottom of the isokinetic tube. This tubing should be connected to the suction end of a dry-cal.

9.5.4.1.4 The air flow here should read within 7% of the initial airflow. If not, ensure that all tubes are tightly connected and that they are correctly configured and repeat the leak test.

9.5.4.2 *ME Leak Checks and Flow Calibrations*

9.5.4.2.1 To check for leaks on the ME opening, disconnect the tubing junction to the right of the elutriator stand that is labeled ME. Pull the tube with the clamp off of the barb. Place tubing connected to the suction end of a dry-cal onto the barb.

9.5.4.2.2 Use the dry-cal and ME rotameter valve to adjust the airflow to

1430 ml/min (± 100ml/min). Record the air flow and disconnect dry-cal. Replace the initial tubing back onto the barb and tighten the clamp.

9.5.4.2.3 Ensure that the Elutriator is completely assembled. Connect tubing from the suction end of a dry-cal to the air inflow valve at the bottom of the Elutriator stack.

9.5.4.2.4 The air flow should read within 7% of the initial airflow. If not, make sure all tubes are tightly connected and correctly configured. Also ensure all clamps and seals are in place and fastened securely. Repeat the leak test.

9.6 Dust Generator Operation

Preparing samples using the dust generator requires loading the tumbler, conditioning the sample, allowing the dust generator to stabilize, and collecting appropriately loaded filters, which are analyzed using direct transfer techniques and TEM.

9.6.1 Loading the Tumbler

9.6.1.1 Ensure the tumbler is completely dry before using. If not, the sample will become moist and may clump, preventing respirable particles from reaching the filters.

9.6.1.2 Under the hood, place the tumbler on a support surface with the lid removed.

9.6.1.3 Gently transfer the 40-70g sample into the tumbler against the inner lip. Tilt the container so that the sample pours smoothly into the tumbler.

9.6.1.4 Move the container back and forth to ensure uniform distribution. Tap the empty container to ensure quantitative transfer. Place the lid on the tumbler and secure it by clamping down the four levers.

9.6.1.5 Ensure the rubber gasket is present and properly situated in the end of the tumbler.

9.6.1.6 Attach the tumbler to the D.C. motor (see Appendix A of Berman and Kolk 1997).

9.6.2 Conditioning the Sample

The sample is conditioned by flowing humidity controlled air over the sample for several hours. Before conditioning the sample, ensure that there are two pans, filled with saturated salt solution, in the constant humidity chamber; that appropriate filters have been mounted into the four cassettes; that air flow valves have been adjusted; and that all air lines, flow valves, and pumps are properly configured.

9.6.2.1 To begin conditioning, turn on all pumps and begin airflow through the elutriator.

9.6.2.2 DO NOT turn on the tumbler motor.

9.6.2.3 Allow the air to flow through the tumbler for *at least* 2 hours.

Note: It may be acceptable to condition samples in the hood overnight.

9.6.3 Initiating a Run

Once the sample is conditioned, the tumbler motor can then be started and a run initiated.

9.6.3.1 Set the tumbler motor to 30 rpm and turn it on.

9.6.3.2 Perform an RPM test to ensure the tumbler is rotating at 30 rotations per minute. Record the tumbling speed.

9.6.4 Monitoring to Confirm Stable Dust Generation

During each run a series of filters is collected continuously from the top of the ME (main exit) openings. These filters are to be pre-weighed so that total dust accumulation can be determined. The rate at which respirable particles are released is monitored at the beginning of the sample run over the ME opening. This rate will indicate when conditions have stabilized, and when IST filters may be collected.

9.6.4.1 Initially change the ME filters every 10-20 minutes.

9.6.4.2 To change the filter, adjust the stopcocks so that both ME valves are open. Move the slider mechanism left so that the new filter is switched over the ME opening and the old filter is out of alignment with the air flow. Turn off the airflow to the unaligned ME cassette.

9.6.4.3 Dismount and replace the old filter with a new, pre-weighed filter from the stock pile.

9.6.4.4 Turn on both stopcocks, move slider right, begin timer for the new filter, and turn off airflow to the unaligned ME cassette. For a more detailed description of this process, see Berman and Kolk (1997) Section 9.4.4

9.6.4.5 Weigh each filter after dismounting. Record the pre and post weight on the Elutriator worksheet. The time interval for which the filter was mounted should also be recorded.

9.6.4.6 After collecting two or three ME filters, the time interval for collection can be optimized. Do not allow more than 0.006g to accumulate on any filter.

9.6.4.7 Use Excel to plot the filter weights during the run to determine when dust generation has stabilized and when IST filters can be collected. This typically occurs one and a half hours after the run begins.

9.6.5 Collecting Filters for Asbestos Analysis

Once the ME filter weights have stabilized and while the tumbler continues to run, a second set of filters is collected, this time from the Iso-kinetic Sampling Tube or "IST" port. The flow rate for these samples should be 72 ml/minute. Filter cassettes are loaded with 0.45 MCE filters or 0.2 micron Polycarbonate filters. Before collecting IST filters, determine the time required for optimal deposition.

$$t_{opt} = .002 / D_{rate}$$

Where t_{opt} represents the time needed for optimal deposition and D_{rate} is the deposit rate from the last ME filter collected, expressed in grams / second.

Note: In order to collect optimally loaded filters, it is possible to increase the flow rate through the IST opening upto 70%. This will not impact the elutriator's performance.

9.6.5.1 Respirable particles are collected over the IST opening in the same way the ME filters are collected (see section 9.6.4.2 to 9.6.4.4).
9.6.5.2 Polycarbonate (PC) filters are to be used over the IST opening because they are less affected by humidity than MCE filters.
9.6.5.3 Immediately after dust collection, the filter is to be weighed on a microbalance to determine whether or not the target dust mass was collected (0.100g - 0.150mg). Sample filters should be weighed to determine the mass of particulate on the filter prior to the direct preparation technique following the ISO 10312 method
9.6.5.4 Before filters are weighed, electrostatic charges on the filters must be neutralized. This is accomplished by passing the filter over an antistatic bar.
9.6.5.5 Collect five good IST filters (0.100g – 0.150mg with even dust distribution). Two filters are sent to analysis. The rest are kept with the ME filters as back-up filters.
9.6.5.6 The run may be terminated after 5 hours so long as 5 good IST filters and 8 good ME filters are collected and so long as the curve for cumulative dust mass over time is satisfactory.

9.6.6 Cleaning the Elutriator

The design of the elutriator allows for quick assembly and disassembly which helps facilitate cleaning.

9.6.6.1 After all necessary ME and IST filters are collected, turn off the motor. Ensure stopcocks are open for the ME and IST cassettes in line of the air flow.
9.6.6.2 Allow air to run through the system for 30 minutes.
9.6.6.3 Turn off all stopcocks and pumps and carefully remove the tygon tubing from the top of the elutriator. Remove all filters from the cassettes and carefully discard of them in asbestos waste.
9.6.6.4 Disassemble each section with caution. Be careful not to disturb or spill any dust that may remain in the elutriator. Mist, with water, inside each piece of the elutriator before carrying it to the cleaning room.
9.6.6.5 Discard remaining soil inside the tumbler into asbestos waste.
9.6.6.6 Wash all pieces with biodegradable detergent and asbestos-free water.
9.6.6.7 Pieces may air dry in room air or be force dried under a HEPA hood.

9.6.7 Preparation of Specimen Grids for TEM Analysis

9.6.7.1 Filters collected over the IST opening are prepared for TEM analysis using the direct transfer technique.

9.7 Sample Analysis

9.7.1 After prep, the ISO 10312 analysis protocol is followed with the following modifications:

9.7.1.1 Only protocol structures are counted. That is structures >5 microns in length and less than 0.5 microns in width.
9.7.1.2 Analysis is typicall performed at 10,000X to 20,000x
9.7.1.3 The target analytical sensitivity is 3×10^6 structures/gram $_{pm10}$

9.7.1.4 Enough grid openings have been analyzed to reach the target analytical sensitivity OR that grid opening where the 25th asbestos protocol structure that is longer than 10 microns is counted is completed

10.0 Calculations

10.1 The asbestos concentrations determined by this method are reported as the ratio of asbestos to dust with the units of Structures per gram of respirable dust (S/g_{pm10}).

$$C_{dust} = S_c .* A_f \div (N_{go} * A_{go} .* \Delta M_f)$$

C_{dust}	=	Concentration of asbestos per unit mass of respirable dust
S_c	=	Number of structures counted
A_f	=	effective filtration area of filter
N_{go}	=	grid openings analyzed
A_{go}	=	area of a grid opening
ΔM_f	=	mass of respirable dust deposited on the filter

10.2 Chi Square the test for uniform fiber distribution.
This test is performed to quantify the randomness of the fibers distributed on the filter surface. It is calculated with the following equation:

$$\sum_{i=1}^{i=k} \frac{(n_i - np_i)^2}{np_i}$$

Where (p_i); is the grid opening area divided by the total area of the TEM grid examined. (See10.1 above)

Where n_i is the observed number of primary structures on that grid opening.
Where n is the total number of primary structures observed in the sample.

The value from the above calculation is compared with the significance points of the Chi Square table, having (k-1) degrees of freedom at the 0.001 significance level. Where k is the total number of grid openings analyzed.

11.0 Reporting

In addition to the reporting requirements of the ISO 10312 method, the following items must be reported for each sample:

11.1. Total asbestos concentration for protocol asbestos structures as defined in this SOP meeting minimum Identification required, typically CD and ADX

11.1.1 For sample where 3 or less structures were counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.

11.1.2 For samples where 4 or greater structures are counted report the mean asbestos structure concentration.

11.2 Chrysotile asbestos concentrations are reported as follows: (For protocol asbestos structures as defined in this SOP)

11.2.1 Determine which identification and structure categories will be used to calculate the concentration. This is specified by the client. If it is not specified by the client, use all Total structures identified as CD or better.

11.2.2 For sample where 3 or less chrysotile structures were counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.

11.2.3 For samples where 4 or greater structures are counted report both

11.2.3.1 The mean chrysotile concentration

11.2.3.2 The upper and lower chrysotile concentrations based on the corresponding two-sided Poisson 95% confidence intervals.

11.3 Total amphibole asbestos concentrations are reported as follows: (For protocol asbestos structures as defined in this SOP)

11.3.1 Determine which identification and structure categories will be used to calculate the concentration. This is specified by the client. If it is not specified by the client, use all Total structures identified as ADX or better.

11.3.2 For samples where 3 or less amphibole structures were counted, report the concentration as less than the corresponding one-sided upper 95% confidence limit for the Poisson distribution.

11.3.3 For samples where 4 or greater structures are counted report both

11.3.3.1 The mean amphibole concentration

11.3.3.2 The upper and lower amphibole concentrations based on the corresponding two-sided Poisson 95% confidence intervals.

11.4 Reference to this method

11.5 Sample ID and batch number for the sample

11.6 Sampling date and site from which the sample was collected

11.7 The weights and identities of the course and fine fractions of the sample an the sub-sample of the fine fraction sent for analysis

11.8 Laboratory receive date

11.9 Analysis Date

11.10 Concentration of asbestos protocol structures (both total and >10 microns in length) reported in structures/gram$_{PM10}$

11.11 Chi Square value (See section 10.6 of ISO 10312 SOP)

11.12 Each sample is entered into the most current version of the Elutriator spreadsheet. The spreadsheet records the following stages of the sample:

11.12.1 Sample Receiving

11.12.2 Sample Prep (Drying, Splitting, Percent Moisture)

11.12.3 Sample Analysis using modified ISO 10312 analysis

11.12.4 Sample reporting

12.0 Method Performance

12.1 MDL Method performance has not yet been determined. Standards and a Round Robin inter- laboratory QC program must be developed to this end.

12.2 DOC's

12.3 PT's There are currently no proficiency testing samples available for asbestos in soils.

12.4 Accuracy Analysts performing this analysis must have successfully completed TEM qualifications training (initial TEM Certification and ISO 10312) and must have been authorized for analysis by a Laboratory Manager, Regional Manager or National Director.

12.5 Precision

12.5.1 In addition to the precision studies performed with the initial elutriator method, further studies were done using this modified version. The results of this study indicate that for protocol structures, counts derived from replicated filters cannot be distinguished from one another (I.e. they can be adequately described by a single poisson distribution) for 8 of the 10 pairs tested.

13.0 Quality Control

The QC requirements for the analysis of samples is indicated in the ISO 10312 method and SOP. In addition to these requirements the following blank and duplicate/replicate schedule shall be employed when running samples using this elutriator method.

13.1 Filter Lot Blanks

13.1.1 2 unused filters per lot of 50 filters (4%) are analyzed prior to sampling.

13.1.2 The background contamination must be < 0.2 fiber/mm^2 or the lot is rejected for use

13.2 Field Blanks

13.2.1 The number of field blanks collected as well as the QC criteria to be applied is to be determined by the client as part of the sampling plan.

13.3 Lab Blanks

13.3.1 A sufficient number of lab blanks shall be collected, prepared using a direct transfer technique, and analyzed to show that the elutriator prep room air is suitable.

13.3.2 A blank filter is left exposed in an open petri dish in proximity to the sampling ports of the elutriator throughout the prep process. This filter will be included with the set of filters generated from each sample.

13.3.3 This lab blank need not be analyzed unless there is a question of contamination.

13.3.4 If a lab blank exceeds 10 structures/mm^2 (the filter lot blank acceptance criteria) the blank shall be considered contaminated.

13.4 Method Blanks

A method blank is a filter collected in a mount over one of the openings atop the elutriator while washed play sand is tumbling under conditions appropriate for a routine sample run

13.4.1 Method blanks should be collected and analyzed at a minimum frequency of 1/20 samples analyzed. The cumulative loading of asbestos structures observed on the method blanks must not exceed 0.2 S/mm^2

13.5 Equipment Blanks

An equipment blank is a filter collected from a mount over one of the openings atop the elutriator while air is passed through an empty tumbler assembly and the elutriator

13.5.1 Equipment blanks are interchangeable with method blanks

13.6 Conditioning Filters

One conditioning filter is to be collected at the beginning of every run. These filters do not have to be prepped unless there is a case of contamination. These filters can be analyzed and prove useful for troubleshooting should the need arise (ie. Investigation

of possible laboratory air contamination)

13.7 Spatial Duplicates

These are defined as two samples collected at immediately adjacent locations or two composites composed of an independent, inter-located set of samples representing the same area of volume

13.7.1 These should be submitted to the lab by the client blindly at a rate between 5 and 10%. The frequency is typically defined in the client's sampling plan.

13.7.2 The comparison of the results of these samples gives an indication of the precision of the entire prep and analysis procedure.

13.8 Sample Duplicates

100% of samples submitted to the lab are supposed to be submitted as duplicate pairs. These are homogenized splits of the same sample.

13.8.1 The lab should randomly select 2-3% of these sample duplicates and prepare and analyze them as separate samples.

13.8.2 Comparison of the results of these analyses provides an indication of the precision achieved by the preparation and analysis.

13.8.3 If the variability between sample duplicates exceeds 50% relative percent difference then select samples shall re-analyzed as blind QC either as

- Inter analyst QC
- Intra analyst QC performed on different days

13.8.3.1 Results of these QC samples shall serve to distinguish whether the main source of variability observed among duplicate pairs is due to analysis or to sample preparation. Appropriate corrective action can then be devised.

13.9 Inter-Laboratory Programs

For large scale projects the client should incorporate an inter-laboratory component to their sampling and analysis plan. This will help to gauge the relative performance of the laboratories. The program should include blind field replicates sent occasionally for comparison both between the labs and within each laboratory.

14.0 Data Assessment

14.1 Acceptance criteria for QC measures

Analysis data of the splits will give a measurement of the precision of the method for this laboratory. Evaluate results and determine: Acceptance Criteria and Corrective actions for out of control analysis

14.2 Corrective Actions

Corrective action policies are addressed in the QA manual (EMSLQAMAN).

14.3 Contingencies for handling out-of control or unacceptable data

14.3.1 Any quality control requirements not met must have an explanation to their nonconformance. Corrective action policies are addressed in the QA manual (EMSLQAMAN).

14.3.2 If more than three quality control requirements are not met the entire QC batch can not be reported, and the client notified.

14.3.3 Any quality control requirements not met must have an explanation to their nonconformance.

14.4 The Chi Square uniformity test shall be conducted using the number of primary protocol structures found on individual grid openings (see section 10.6 of the ISO 10312 SOP) This value will be compared with significance points of the Chi Square distribution. If the structure count fails this test, the precision of the result may be in question and if new samples may need to be run or additional grid openings may need to be examined.

15.0 Pollution Prevention / Waste Management

15.1 Pollution Prevention

EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal. EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering.

15.2 Waste Management

The waste generated during prep and analysis will be disposed of following safety procedures outlined in the chemical hygiene plan (EMSLChemHygiene 200.0). Digests from analysis will be disposed of in a Poly-Closed Drum as corrosive waste. Each drum must be properly identified with a hazardous waster sticker when placed into service. Each drum will be analyzed prior to manifesting and removal. A reputable waste hauler will remove full drums from the site within 90 days. All records pertaining to waste shall be kept including the waste manifest. Hazardous waste is classified for proper disposal. A reputable waste hauler will remove waste from the site.

All records pertaining to waste shall be kept including the waste manifest.

16.0 Tables, Diagrams, Flowcharts, and Validation Data

17.0 References

17.1 American Lung Association http://www.lungusa.org

17.2 EMSL EPA Soil Screen Overview Modified to Provide Quantitative Results April 21, 2005 Revision

17.3 EPA 600/R-93/116 Method for the Determination of Asbestos in Bulk Building Materials

17.4 EPA 540/R-97/028 Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Material

18.0 Revision History

Revision #	Date	Revision	Initials
0	March 2007		
1	5/18/09	Reformatted to current SOP Template, various clarifications	RD, MAC
2	1/13/10	Clarifications in Reporting section to include the use of the Elutriator report spreadsheet. Included Chi² in QC section. Verbage change throughout to clarify the procedure.	RD
2.1	6/11/10	Minor changes to the reporting section	RD

Authorizing Signatures

Robyn Denton	Robyn Denton	6/11/2010
Author (print)	Author Signature	Date
Ed Cahill	Edward R Cahill	6/11/2010
Reviewer (print)	Reviewer Signature	Date
Ed Cahill	Edward R Cahill	6/11/2010
Corporate Approval (print)	Corporate Approval Signature	Date