UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 1 - OFFICE OF ENVIRONMENTAL EVALUATION & MEASUREMENT
60 Westview Street, Lexington, Massachusetts 02421

LATEST REVISIONS: December 5, 1997

THE PROTOCOL FOR SCREENING SOIL AND SEDIMENT SAMPLES FOR ASBESTOS CONTENT USED BY THE US ENVIRONMENTAL PROTECTION AGENCY, REGION 1 LABORATORY

BACKGROUND: This protocol is not a "reference" method. It was developed out of necessity to facilitate finding asbestos fibers in a soil or mud (sediment) sample that does not contain any obvious asbestos fibers or asbestos-containing building or product materials when examined dry (or wet) using a stereo microscope at 10X or 20X magnification. It has been used successfully to screen several thousand soil and sediment samples for asbestos content in order to help delineate contaminated areas. It has proven to be an extremely sensitive method capable of finding very small amounts of asbestos fibers in a soil or sediment matrix.

Glassware or other materials, supplies, etc. mentioned below in the procedures are those being used in this laboratory. Other materials may be substituted as long as the primary purpose of the protocol is followed, namely: to find asbestos fibers in the sample. Identification of fibrous components is accomplished by the routine **Polarized Light Microscopy (PLM)** (with dispersion staining) method. (See EPA approved method references on last page.)

SAMPLE HANDLING AND PREPARATION

All samples are received and signed for using chain of custody procedures. The samples are usually contained in individual plastic "Ziplock" freezer bags or other sealed, clear plastic sample bags and subsequently assigned a laboratory identification number.

ANALYTICAL PROCEDURES

1. A representative portion of the sample is removed from the sample container after thoroughly mixing for homogeneity. Because asbestos fibers usually cannot be seen because of the composition of the sample matrix such as the dirt, sand, mud, vegetation, water, etc., steps must be taken to clean up the sample to the point where the asbestos fibers, if any, may be seen using the stereomicroscope at 10X to 20 X magnification. **A stereomicroscope is mandatory for this protocol.**

2. To eliminate interfering particles, a 16mm ID by 150mm long, good quality PYREX or KIMAX test tube (not a fragile disposable tube) is used to remove portions of the well-mixed soil/sediment sample from several places in the sample container by pushing it into the sample to accumulate a sample depth of about 2.5 inches (65mm) in the test tube. A glass or plastic stirring rod is used to push the sample down into the tube and fiber-free (tap) water is added for shaking purposes. The soil and water mixture is shaken vigorously to loosen and separate the fines and other components of the sample and the contents of the test tube are then poured into a 3 inch ID, 60 mesh (250 micrometers) sieve. This serves to eliminate, or greatly reduce, colloidal material, fine sand, silt and other non-fibrous particulates from the sample. More water is added to the tube, shaken and dumped into the sieve. Repeat this step until the tube is clean. The sample in the sieve is then rinsed until clean (clear water running through the sieve) with a fairly fine, pressurized stream of water from a plastic wash bottle.

All of the material remaining in the sieve is then washed from the sieve screen using a stream of water from the rinse bottle into a square plastic weighing dish of about 100ml liquid capacity. Use just enough water to completely cover the sample in the dish about 1/8th inch or so for examination with the stereo microscope.

After the cleaned sample is transferred to the weighing dish for examination, thoroughly rinse the sieve and test tube under running tap water (preferably aerated to minimize splashing) and carryover will not be a problem from sample to sample. It is a good idea to carry out all washing of the sample fines over a plastic dishpan or other container set into the sink basin in order to capture the fines and keep them from clogging the sink drain trap. After a settling period, the overlying water may be poured off and the fines/mud disposed of separately.

NOTE:
Since the purpose of the test is to find out if the soil or mud sample contains a significant amount of asbestos (>1%) that can be identified using the PLM technique, **all portions of the sample are to be examined except those fines which pass through the sieve.**

3. After examining them for asbestos fibers, floating pieces of organic material such as roots, sticks, leaves, etc., may be removed to get a better view of the rest of the sample in the dish. Frequently, root structures found in surface soils will

trap asbestos fibers during the shaking process and are a good place to look for the fibers. The sample is then carefully and systematically examined under the stereo microscope at 10X-20X magnification for visible asbestos fibers and fiber bundles. A good, bright, focused light source such as a Nicholas transformer-base external illuminator is very helpful here. The fibers tend to stand out, shine, flash, etc., in the clean water matrix. Poking and stirring the sample with forceps and/or dissecting needles will help to locate the fibers. If no fibers are seen, gently shaking the weighing dish to redistribute particles will sometimes turn up previously hidden fibers when scanning the sample a second time. Suspect fibers are removed with sharp forceps and placed upon a clean microscope slide.

4. After picking as many suspect fibers or other material from the sample as necessary to determine its content, the slide preparation is allowed to dry and prepared for PLM analysis using an appropriate high-dispersion refractive index liquid and coverslip.

5. Next, the slide preparation is examined with a polarized light microscope (PLM) with dispersion staining to identify any fibers found. Standard, EPA approved PLM procedures are used to identify any asbestos fibers found as to specific type and form. The identification of asbestos using PLM is rapid and unequivocal due to the unique optical crystallographic properties of morphology, refractive indices, elongation, angle of extinction, dispersion and birefringence. Slide preps are examined for each sample with suspect fibers to confirm the presence of asbestos.

6. If asbestos fibers are identified, return to the sieved sample under the stereo microscope, observe the remaining asbestos fibers and bundles of fibers, and make a visual estimate of the percentage asbestos content in the **whole sample including the material previously washed through the sieve.** (This is all based on asbestos fibers seen using 10X to 20X magnification under the stereomicroscope.) Obviously, many of the finest fibers pass through the sieve and the finest ones remaining can't be seen at 20X magnification, but, this protocol is not meant to be used as a quantitative method. It is useful, however, to determine whether or not the soil or sediment is contaminated with significant amounts of asbestos. (> than 1 % by volume).

7. As a rule, if asbestos fibers and/or fiber bundles can't be found relatively quickly and easily (one to two minutes) in the cleaned up sample under the stereo microscope, the percent asbestos content is most likely less than 0.1% and certainly less than 1.0% ! One should be able to find asbestos fibers in a sample containing more than 1.0% in a few seconds up to a one

minute examination under the stereo microscope.

8. Usually, as much of the original sample as possible is returned to the original sample container. If saving the fines is required for further examination by other methods, use individual beakers to catch the sieve washings containing the sand and silt components.

9. The sample containers are then resealed before storage, disposal or return to the organization that requested the analyses.

10. If further information is required on this protocol or if anyone has found a better way to find and estimate asbestos fibers in muds or soils, please contact:

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APPROVED EPA BULK ANALYSIS PROTOCOL:

40 CFR PART 763, SUBPART F, APPENDIX A

or, "ASBESTOS IDENTIFICATION"- Walter C. McCrone, 1987, McCrone Research Institute, Chicago, Ill.

See also, "Asbestos Content In Bulk Insulation Samples: Visual Estimates and Weight Composition"- US EPA, EPA-560/5-88-011, September, 1988.

ADDENDUM.TO "PROTOCOL FOR SCREENING SOIL.AND.SEDIMENT SAMPLES FOR ASBESTOS CONTENT USED BY THE U.S. ENVIRONMENTAL PROTECTION AGENCY, REGION I LABORATORY"

Addendum dated: August 1997

This addendum can be used to more accurately quantitate the volume of asbestos in soil and sediment samples. It is meant to give the analyst a good visual estimate of the fine materials volume which pass through the mesh sieve relative to the original sample volume analyzed. It must be used in conjunction with the above mentioned protocol.

Sample Preparation Analytical Procedure:

1. Transfer a well mixed portion of homogenized soil into the plastic weigh dish. Cover the bottom of the dish with a thin (1cm) layer. Quantitatively transfer the soil/sediment into the test tube using a wide mouth funnel. Fiber-free (tap) water can be used to help wash fines into the test tube. A glass or plastic stirring rod is used to push the sample down into the test tube end and to break-up any soil clumps. Wash the test tube sides down with a stream of water. Let the soil/water mix settle such that the volume of material in the test tube can be measured. Measure from the bottom of the test tube to the top of the settled soil with a ruler and record the value (i.e., 4.5cm).

   After the soil volume measurement, continue the analytical procedure (i.e., shake vigorously to loosen and separate the fines, pour contents into sieve for clean-up, transfer sample component left in sieve to weigh dish for examination, etc.)

   After complete examination and determination of asbestos content of the sample portion in the weigh dish which did not pass through the sieve (using stereo microscope and PLM), the sample in the weigh dish is quantitatively transferred back into the test tube and allowed to settle. After settling, the volume of material in the test tube is again measured and recorded (i.e., 2.0cm).

Determine the asbestos content of the sample as follows:

$$\text{\% asbestos in sample portion which did not pass through the sieve} \times \frac{\text{Volume of sample which did not pass through sieve}}{\text{Initial sample volume}}$$