

ENVIRONMENTAL LABORATORY APPROVAL PROGRAM
CERTIFICATION MANUAL

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decreasing precision should not cause problems with higher concentrations of asbestos since materials containing these are definitely ACM and will require treatment as ACM, regardless of the accuracy in quantitation.

1.4.2. Scanning Electron Microscopy (SEM). Because the wavelength of an SEM's 10-40 keV electron beam is smaller than that of visible light, electron microscopy has potential for better resolution than PLM. In SEM, a narrow electron beam is rastered across a specimen while concurrently emitted secondary electrons are collected and synchronized to present a CRT image of the specimen surface. The elaborate collection and presentation of SEM video images against noisy backgrounds, however, often reduces contrast and produces practical resolution that is only marginally better than PLM (Steel 1988). An SEM interfaced with an energy-dispersive x-ray (EDX) spectrometer allows determination of elemental composition of particles of interest. X rays emitted from shifting orbital electrons are collected, sorted, and identified according to their energy. While this provides semi-quantitative composition for elements constituting greater than 1% of the specimen, SEM lacks the ability to determine crystalline structure. Hence, non-asbestos fibers with elemental compositions similar to asbestos can be misidentified as asbestos by SEM. These shortcomings can lead

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to false positive or false negative results. At present, there are no widely accepted routine methods for bulk sample analysis by SEM.

1.4.3. X-Ray Diffraction (XRD). Any solid crystalline substance will diffract impinging x rays under certain conditions. Characteristic diffraction patterns, a function of crystalline lattice spacings and their orientation, can be identified through careful control of an x-ray beam and incident angles. XRD is a reliable workhorse in mineralogy and has been reported to yield chrysotile detection limits as low as 2 μg (<1% w/w in certain samples (Puleda and Marconi, 1990; Lange and Haartz, 1979)). However, its application to routine asbestos identification in bulk samples is still limited by several factors which can lead to false negative or false positive results:

- XRD cannot determine morphologies of identified minerals, making it impossible to differentiate asbestos types from their non-asbestiform mineral varieties, e.g., chrysotile vs. lizardite, crocidolite vs. riebeckite, etc.

- XRD is also incapable of consistently differentiating the five amphibole asbestos types.

- Several materials produce diffraction peaks which may interfere with identification of asbestos peaks.

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Section 2.4 of the Draft Interim Method (Perkins and Harvey, 1991) provides some details on XRD's application to bulk sample analysis while Pierce (1990) details results from VATs. However, given the deficiencies outlined above and the wide variation in instrumental capabilities and methods, there are no widely accepted routine protocols for bulk sample analysis by XRD.

1.4.4. Transmission Electron Microscopy (TEM). TEM's extremely short wavelength of 80-100 keV electrons coupled with simple image presentation yields resolvable images of even the smallest ($\sim 0.02 \mu\text{m}$) asbestos fibrils. Furthermore, identification of chrysotile or amphibole crystalline structure can be consistently produced via the electron-diffraction capabilities of modern TEMs. Finally, the five amphibole types can be differentiated based on their elemental composition when EDX is utilized. The major drawback to TEM is accurate and reproducible subsampling. Since only $\sim 10^{-6}$ of the original sample is viewed via TEM, extreme precautions must be taken to prevent potentially enormous subsampling bias. ELAP is approving a quantitative TEM method for NOBs (ELAP Certification Manual Item 198.4).

2. Application. Methods outlined herein are applicable to most types of bulk materials. Friable materials must be analyzed by one of the point-counting methods while NOBs must be analyzed by the gravimetric

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matrix reduction method. Quantitative TEM (ELAP Item 198.4) is the only NOB method that can be used to report non-ACM results to clients (Section 6.5).

Sample types not specifically addressed in this method (Item 198.1) can probably be analyzed by either the friable materials method or the NOB method. Paint chips or rope, for example, could be analyzed through application of gravimetrically tracked matrix reduction.

3. Equipment and Supplies The following items must be available for sample preparation and analysis in laboratories that analyze bulk samples, regardless of analytical method(s) employed:

3.1. HEPA-ventilated, negative-pressure sample preparation work area. This can be a laminar-flow safety cabinet or a similar enclosure which draws all air from the enclosure through a HEPA filter. This should minimize cross contamination and maintain a safe work environment. A flow rate of at least 75 fpm should be maintained at the opening.

3.2. Low-power (10-45X) stereobinocular microscope with external light source for gross examination.

3.3. Forceps, dissecting needles, probes, scalpel or razor blades, etc. for manipulating bulk sample.

3.4. Homogenization equipment:

3.4.1. Mortar and pestle

3.4.2. At least one of the following:

3.4.2.1. Mini-blender (approximately 30 ml capacity)