Methods for the Analysis of Carpet Samples for Asbestos

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Assessing asbestos fiber contamination in a carpet is complicated by the nature of the carpeting — because of the pile's rough surface and thickness, samples cannot be collected directly from carpet for analysis by TEM. Two indirect methods are currently used by laboratories when preparing samples for measuring the amount of asbestos present in carpet material. One is an ultrasonic shaking technique which requires that a portion of the carpet be cut out and sent to the laboratory. The other is a micro-vacuuming technique which has been used generally in the assessment of asbestos in settled dust in buildings. It is not destructive to the carpet. Both methods utilize TEM to identify, measure and count the asbestos fibers found. Each can provide important but different information when an assessment of the level of contamination of carpeting is being made. The ultrasonic shaking (bulk-carpet sonication) technique gives an index of the asbestos contamination throughout the entire carpet piece and the micro-vacuuming technique gives an index of the readily releasable asbestos fiber from the carpet surface.

A major concern in buildings that contain asbestos-containing material (ACM) is the extent to which the carpet may serve as a reservoir of asbestos fibers that have been released from the ACM by one mechanism or another.[1] The Asbestos Hazard Emergency Response Act (AHERA) requires that all carpet in areas of school buildings in which ACM is present be cleaned with either a high-efficiency particulate air (HEPA) filtered vacuum cleaner or a hot water extraction cleaner (steam cleaner). The potential for airborne asbestos fiber reentrainment during carpet cleaning activities was shown in studies in which airborne asbestos concentrations were found to be between two and four times greater during cleaning than before the carpet cleaning activities.[2, 3]

There are currently two methods used by the authors' laboratories for collecting and indirectly preparing samples to evaluate the amount of asbestos in or on a carpet. Direct collection and direct preparation procedures such as tape lift sampling have been investigated and cannot be used effectively with carpeting. There are currently no standard EPA methods for assessing asbestos in carpeting. One of the methods which is used is an ultrasonic extraction procedure in which a square piece cut from a carpet is mildly sonicated in water with surfactant to release asbestos fibers both on the surface and embedded in the carpet. This method is similar to one that has been published about how to measure asbestos fibers in clothing and fabric materials.[4] The other method is a vacuuming procedure which uses a modified air sampling cassette to vacuum a sample of dust primarily from the surface of the carpet. The microvacuuming technique is non-destructive to the carpet. Both methods use the particle dispersion techniques developed over the years for the analysis of asbestos in drinking water.[5,6]

The Ultrasonic Preparation Procedure

Samples of carpet are collected by cutting a piece (usually 10 centimeters by 10 centimeters) from the carpet with a razor blade or utility knife and placing it in a wide-mouth polyethylene jar or zip-loc bag. In the laboratory, five (5) centimeter by five (5) centimeter squares of carpet are cut and placed carpet-side down in a 1,000 milliliter beaker containing 100 milliliters of 0.1% solution

of the surfactant aerosol OT or a 0.002% solution of the surfactant methyl cellulose in particle-free water. The beaker is placed in an ultrasonic bath for 30 minutes. The carpet piece is removed and rinsed into the beaker with 100 milliliters of particle-free water. The entire suspension (200 milliliters) is then shaken vigorously by hand to disperse the particles and then allowed to sit for two minutes to allow the denser particles to sink and the light particles to float to the top. At this time, three measured aliquots of different volumes (usually one (1), ten (10) and fifty (50) milliliters) are extracted with disposable graduated pipettes 1/4 to 1/2 inch below the water surface in the beaker. The aliquot is mixed with particle-free water to make 50 milliliters and filtered through a 0.22 µm pore size mixed cellulose ester filter or a 0.2 µm polycarbonate filter backed by an 0.45 µm pore size cellulose ester filter. If by visual observation, the initial beaker contains large non-asbestos fibers (carpet material), the entire suspension is passed through a coarse stainless steel mesh screen. The filters are dried and prepared for TEM analysis according to the NIOSH 7402 preparation procedure (cellulose ester filter)[7] or the Yamate, et al., procedure (polycarbonate filter).[8] At least two TEM grids from different areas of the filter are prepared for each sample. After the three filtrations are completed, the remaining suspension is transferred to a graduated cylinder and the volume recorded. This volume is added to the volumes of the measured aliquots to obtain the total volume of the sample. This accounts for the variable amount of water absorbed in the carpet during processing. A sample blank is prepared in an identical way as the sample, although no carpet segment is actually used in the ultrasonic procedure.

The Microvac Technique

Samples which are collected by microvacuuming are commonly referred to as microvac samples or microvac dust samples. They are collected by vacuuming a 100 cm^2 area (or other known area) of carpet with a membrane filter air-sampling cassette and vacuum pump. The sampling assembly consists of a 25-mm diameter mixed cellulose ester filter contained in a three-piece standard AHERA[9] air cassette with a one (1) inch piece of tubing attached to the face cap as a nozzle[10]. The end of the nozzle is cut at 45 degrees. The cassette is connected to a personal sampling pump with flexible tubing. The pump and cassette assembly are calibrated to 2 L/min. The 100-cm^2 area is vacuumed by moving the filter cassette nozzle across the carpet to agitate the carpet pile. The carpet is vacuumed for approximately 30 seconds in one direction, then another 30 seconds in a direction 90 degrees to the first. After one minute of vacuuming, the pump is turned off and the filter cassette nozzle is plugged and the cassette is labeled.

In the laboratory, the unopened microvacuuming cassettes are wet-wiped and then prepared for analysis under clean room conditions. The cassettes and filters are rinsed out with particle-free water and refiltered through a second filter which is used in the analysis. The original filter is washed during the sample preparation procedure but otherwise is not used in the preparation.

Specifically, the plug from the nozzle of the cassette is removed and the cassette is filled with approximately 10 ml of prefiltered water. The plug is replaced and the cassette is shaken vigorously by hand fortwo to three seconds. The entire cap of the cassette is removed and the suspension poured into a pre-cleaned 200 ml glass medical specimen bottle. All visible traces of the sample are rinsed into the specimen bottle with a plastic squirt bottle of filtered water. This procedure is repeated two additional times for a total of three washings. Next, the nozzle is rinsed two or three times into the specimen bottle. Typically, the total amount of water used in the rinse is approximately 70 to 75 ml. The water level in the specimen bottle is then carefully adjusted to 100 ml with prefiltered water. The pH of the water is adjusted to 3-4 using a 1.0% HCl solution. The sample container is capped and ultrasonicated for three minutes to make a uniform suspension. After two minutes of settling, a measured volume of suspension is extracted with a graduated pipette inserted halfway into the sample solution. The aliquot is mixed with particle-free water in the filter funnel and filtered through a 0.22 µm pore size mixed cellulose ester filter backed by an 5.0 µm pore size cellulose ester filter. The filter is dried and prepared according to the AHERA air sample preparation procedure. A sample blank is prepared in an identical way as the sample, although no carpet segment is actually vacuumed.

Asbestos Counting

In the transmission electron microscope, the number of each type of asbestos structures, chrysotile or amphibole, is determined by examining a known area on the grid in terms of a number of grid openings. The asbestos fibers are identified on the basis of morphology, selected area electron diffraction (SAED) and/or energy dispersive X-ray spectrometry (EDS).

The samples are counted following the EPA (Yamate) Provisional Method or the AHERA counting rules. The choice of counting methods depends on the particular interest of the analyst. The Yamate counting rules provide more information about the sizes of structures found by the analyst than do the AHERA counting rules. The amount of asbestos in a given sample is expressed as structures per square foot, structures per square centimeter or structures per square meter. The value is calculated using the following equation:

$$\frac{EFA \times 100 \times NOSTR}{GO \times GOA \times SPL \times V} = \text{STRUCTURES /AREA OF THE CARPET}$$

NOSTR = Number of asbestos structures counted in the analysis

EFA = Effective filter area of the final sampling filter in square millimeters

GO = Number of grid openings analyzed

GOA = Average area of one TEM grid opening in square millimeters

SPL = Amount of carpet area sampled (in square feet or square centimeters)

V = Volume of sample filtered

Table I—Results of Carpet Analyses for Asbestos Samples Randomly Chosen in Cafeteria Carpet After Conventional (Dry) Vacuuming (all values in asbestos structures per square centimeter)

Sample #	LAB A (Sonication)	LAB B (Sonication)	LAB B (Microvac)
1	5,400,000	4,800,000	21,000
2	3,050,000	3,300,000	30,000
3	*68,000	*5,400	*<350
4	3,600,000	3,800,000	74,000
5	3,400,000	3,000,000	50,000
6	4,300,000	2,500,000	95,000
7	3,200,000	3,600,000	18,000
8	2,000,000	4,700,000	35,000

*at detection limit

Data on Precision and Percent Recovery

Studies on the precision and level of recovery of asbestos of the bulk-carpet piece ultrasonic shaking (sonication) technique and the micro-vacuuming technique were performed as part of the 1988 EPA study.[9] Six samples were collected using each method from carpet artificially contaminated with approximately 1 billion (1×10^9) asbestos structures per square foot (s/ft) or 1.08×10^6 str/cm^2. The artificial contamination was accomplished by spraying a known area of a carpet with a water solution of known asbestos concentration. Because there was no independent way to measure the concentration of asbestos on the carpet, no accuracy determination could be made. However, the relative efficiency of recovery of one method to the other could be assessed. The mean asbestos recovery using the microvacuuming technique was 2.3×10^7 s/ft^2 (2.5×10^4 s/cm^2). This was approximately 3% of the mean recovery of the bulk-carpet sonication extraction technique which was 7.9×10^8 s/ft^2 (8.5×10^5 s/cm^2). The calculated coefficient of variation (CV) for the microvacuum technique was 166%. The CV for the bulk-carpet sonication procedure was 43%. It should be noted that the values given in reference 2 for structures per square foot are the correct values.[11] There was an English/metric conversion error in the article which provided incorrect values for structures per square meter throughout the paper.

A similar set of tests using "real world" carpeting that had been contaminated over a 15 -year period of normal use has been performed by the authors' laboratories. The carpet samples were collected from a cafeteria in the Social Security Administration in Baltimore, Maryland The cafeteria had an acoustical plaster ceiling containing 1 to 5% chrysotile. All furnishings had been removed from the cafeteria and the area had been vacuumed with a conventional dry vacuum cleaner twice before the samples were collected. Previous use and traffic patterns were not taken into account in collecting the samples. The samples were collected in a random manner and some samples may have been from areas where an appliance such as a soft drink machine may have stood previously. The data which include some interlaboratory comparisons are presented in Table I. Although there appears to be one outlier in the data set, the relative stands deviation was calculated using all eight data points for each method. The S_r for the microvacuum technique was 77% and for the bulk-carpet sonication procedure was 51% for Lab A and 47% for Lab B. The mean recovery of the microvacuuming technique compared to the bulk-carpet sonication procedure was 1%.

Discussion

The answer to the question about which method is best for assessing the asbestos level in carpeting depends on the specific question being asked. In the 1988 EPA carpet study the authors concluded that sonication of bulk-carpet samples provided a more precise and accurate estimate of asbestos concentrations in carpet than the micro-vacuuming sampling technique. Their conclusion was based in part on data which showed that the microvacuuming technique was shown to recover significantly less asbestos from the carpet than the ultrasonic extraction technique.

The fact that one technique is more efficient in recovering fibers than the other may be important for some studies investigat-

ing the cleaning of the total carpet. However, there is reason to believe that carpets can act as a trap for asbestos fibers and that activities short of carpet removal may not disturb asbestos fibers which have worked their way deep into the carpet pile.

For those fibers which can be readily reentrained into the air from the carpet surface during cleaning activities the microvacuuming procedure may be more appropriate. This is reasonable considering that the microvacuuming procedure collects fibers from the top layer of the carpet and the bulk-carpet sonication procedure shakes out more fibers which may be embedded deep in the carpet pile. The data show that the sonication of bulk-carpet samples is a more precise procedure than the microvacuuming procedure. However, the microvacuuming procedure has an advantage of being non-destructive. While some building owners may be willing to have a piece of carpeting cut from their building if the carpet is to be removed, it is less likely that a piece may be cut if the intent is to study the effectiveness of various cleaning procedures.

It is probable that a relationship exists between carpet contamination and air levels of asbestos produced during cleaning and other activities. For a given carpet, a higher level of asbestos in the carpet would be expected to produce a higher level in the air for a particular activity. The two methods described here will provide the basis for evaluating the level of asbestos contamination in carpeting to be compared with levels of asbestos in the air produced during studies of re-entrainment.

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16. ABSTRACT

Because of the nature of carpet pile, no samples can be directly prepared from carpet for analysis by transmission electron microscopy (TEM). Two indirect methods are currently used by laboratories when preparing samples for measuring the amount of asbestos present in carpet material. One is an ultrasonic shaking technique which requires that a portion of the carpet be cut and sent to the laboratory. The other is a micro-vacuuming technique which has been used generally in the assessment of asbestos in settled dust in buildings. It is not destructive to the carpet. Both methods utilize TEM to identify, measure and count the asbestos fibers found. Each can provide important but different information when an assessment of the level of contamination of carpeting is being made.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Asbestos Transmission Electron Microscopy Carpet Indirect Preparation	Asbestos (GS- Minerals) (RT- Sertentine) Electron Microscopy (GS-Microscopy) (RT-Microanalysis) Particulate Sampling (GS-Sampling; RT-Concentration)	

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