

ASBESTOS IN AIR BY TEM / AHERA VERSUS NIOSH 7402: A BRIEF REVIEW

Background: Federal, State, and local asbestos regulations have established criteria for worker exposures, building occupancy, clearance levels, bulk sampling and analysis, and even safe drinking standards for water. These rulings are the keystone upon which laboratory results are interpreted and engineering and/or remediation controls applied. [1] In addition, similar standards for laboratory quality assurance and accreditation programs have continued to refine analytical sensitivities through proficiency testing programs.

Asbestos hazard control issues and asbestos laboratory development have matured over the last several years. Issues that used to cause controversy are now routine. The limitations of fiber counting by Phase Contrast Microscopy (PCM) have been answered by the continued utilization of Transmission Electron Microscopy (TEM) for the quantitation and qualification of suspect fibers in air thus eliminating non-asbestos fibers for critical exposure data. [2] Point Counting regimens are now routinely applied for more precise quantitative results by PLM [3]. Limitations inherent to the examination of floor tiles, roofing samples, and other Non-friable Organically Bound (NOB) materials by Polarized Light Microscopy (PLM), have been overcome with gravimetric reduction techniques as refined by ELAP 198.4 through TEM [4].

Consistent advances also include the development and use of a new improved EPA 600 Method for the analysis of asbestos in bulk building materials [5,6]. This 1993 method far exceeds the Interim Method published in 1982. NESHAP, OSHA, and other regulating bodies have posted rulings or interpretations regarding sheetrock and joint compound analyses. Methods (EPA 100.1 and 100.2) have been published that refine field and analytical approaches to asbestos in drinking water and wastewater. [7,8]

There are several alternatives available for the analytical professional to choose from when sampling for asbestos in air. Each method incorporates field sampling protocols, laboratory regimens, and data interpretation. The choices, all based upon TEM capabilities, include ASTM D22.02 (02-01), ISO 13012, CARB 427, Superfund Method, EPA Level II, AHERA 40CFR763, and NIOSH 7402. The comparison of the AHERA and NIOSH methods is the basis of this discussion. The full NIOSH 7402 method and the analytical portion of the AHERA method can be found on the IATL website under Request Info.

Why select AHERA Methodology? Most asbestos related issues center around regulatory and potential liability driven decisions. This may also be the case with the AHERA method. This is the method most often cited as the benchmark analytical method for measuring asbestos in air for the purpose of clearing an area for reoccupancy. It is mandated in public schools. It has unique components concerning how to set-up and sample in the field, how to prepare and analyze in the laboratory, and how to interpret those results leading usually to specific actions. Does this mean that it is the best method for determining asbestos in air? No, in fact, there are other more comprehensive analytical methods available (eg: ISO 13012). What AHERA provides is also familiarity. Since its inception, remediation contractors, air monitoring field technicians, and laboratories have become very familiar with the requirements of this method. This may also be a factor in keeping laboratory costs reasonable.

AHERA: In the Field. This method goes into great detail about how to construct area containments. The method lists how these containments, used as barriers during remediation, are to be maintained and evaluated. AHERA lists criteria for the inspection of a site previous to

sampling the air. There is a strict regimen that needs to be followed when sampling the air in these containments. Consideration is given to how to aggressively disturb the inside of the containment to entrain any asbestos that might remain after the remediation. The method specifies the exact type of filter material to be employed (0.45 μm MCE) and the configuration of each air monitoring cassette, how many sampling units (cassettes) are to be employed, and at what sampling flow rates. Minimum final air volumes (~1200 Liters) are required to achieve certain sensitivity in the testing procedure. The method calls for simultaneous testing of the air outside of the work area containment. It also stipulates the number and type of blank samples that should be submitted with each sampling event. There are instructions to field personnel concerning the recording of all of the field data and the manner in which the samples should be packaged and shipped to a NVLAP accredited TEM laboratory.

AHERA: In the Laboratory. The method continues with very specific instructions to the laboratories regarding the preparation and analysis of the air samples collected in the field. The laboratories are required to reject samples that do not have the above field documentation. The laboratory must also reject samples submitted that have been previously opened, tampered with (a portion of the filter material missing), where the wrong cassettes have been used, where the filter material has been damaged, where there is an overloading of particulate matter (10% of filter surface area), and where bulk asbestos samples may have been shipped in the same sample package as the air cassettes. If the samples meet these requirements the laboratory will document their receipt.

Trained laboratory technicians then go about a strict protocol to prepare each sample for analysis. With advance notice and technicians and analysts in place, a typical preparation of five AHERA samples could take up to three hours... the analysis up to one to two hours. The prep consists of three major procedures: collapsing of the filter material, carbon coating the filter material, and transferring this carbon replica on to indexed sample grids. The unused portion of the filters in the cassettes are archived for at least a year.

For MCE filter material, each sample is initially collapsed on to glass slides using hot acetone vapor. These portions of filter material are then introduced into a low temperature asher (or plasma etcher) for further controlled collapsing of the organic filter material. This second process may also remove some of the organic material collected on the filter. The slides are then placed into a high vacuum evaporator where a fine coating of pure carbon is deposited on to each sample. A portion of these carbon replicas are then transferred to indexed grids. The grids supporting each sample are then set on a wick assembly where an organic solvent gently removes the remaining remnant of any filter material.

The analysis of each sample is strictly guided by the AHERA method. It includes very strict requirements for analyst training, instrument calibration and performance, and quality control routines to assure accuracy and precision by the analyst and the laboratory. The individual sample analysis concerns examining a known area of filter (usually <0.070 square millimeters). This is done at magnifications routinely at 20,000. Here individual asbestos fibrils (the smallest denomination) can be resolved and identified.

The qualification procedure requires three criteria: size and morphology, crystalline diffraction pattern, and elemental identification. The method lists strict counting and recording rules. Not just fibers, but assemblages of fibers (structures) are counted if certain size and aspect ratios are met. All data is then broken down into those structures greater than 0.5 μm and less than or equal to 5.0 μm , and also those greater than 5.0 μm . An electron diffraction pattern is obtained and may be recorded on film to document the crystalline properties of the suspect asbestos mineral.

AHERA requires a certain amount of photo documentation for every asbestos structure detected. Finally, a chemical fingerprint of the suspect asbestos structure is acquired using the energy dispersive x-ray microanalysis unit ancillary to the TEM. Only when these data meet certain criteria can a suspect mineral be classified as asbestos.

The analyst may spend up to an hour per sample depending upon how much particulate is present on the sample. The sample grids are archived for at least three years. The data and associated documentation is then used to calculate results to the client. AHERA requires archiving all paperwork and data for three years.

All TEM Laboratories that analyze air samples using the AHERA method are certified by NIST/NVLAP. The requirements for this accreditation are beyond the scope of this discussion.

AHERA: Results Interpretation. Limits of Detection for this method are required to be <0.0050 s/cc. The results calculated from raw data by TEM are presented in the following manner: Asbestos Structures per millimeter square area of the filter examined (mm^2) and Asbestos Structures per volume of air sampled (cc). The report also expands on the size ranges noted above. AHERA lists passing and failure criteria based upon the average of the inside work area samples. In order to clear an area for reoccupancy, this average may not exceed 70 s/ mm^2 . For samples with 1200 Liters of volume this is equivalent to about 0.02 s/cc.

If the samples fail this test AHERA specifies alternatives. Many times the area is cleaned again, inspected, aggressively sampled again, and then analyzed again by the laboratory. AHERA also allows for a Z-test. Here, inside and outside area averages can be used to pass an area based upon the outcome of the arithmetic comparison of both areas.

Can AHERA be used for other situations outside of clearance testing? Yes, many times just the analytical regimen is employed without the field requirements. The sample results are still valid, just not for clearing an area for reoccupancy.

Why select NIOSH 7402 Methodology? Prior to its release in 1989, there was only a few analytical protocols established by the environmental community for the analysis of asbestos in air by TEM. The NIOSH 7400 method existed for the analysis of fibers in air by Phase Contrast Microscopy. Environmental professionals and laboratories wanted a companion technique to qualify those fibers identified by PCM. Hence the issue of NIOSH 7402. The method clearly states “is used to determine asbestos fibers in the optically visible range and is intended to complement the results obtained by PCM Method NIOSH 7400.”

Why might the environmental professional consider employing this method? First because they will be using it as the method stated above, to compare these results with PCM air testing results. In some cases, the project manager may select NIOSH because the AHERA requirements (i.e. no containment in place) can not be achieved.

NIOSH 7402: In the Field. The field sampling protocol is more tolerant of air volumes gathered, the pump flow rates utilized, and the filter type and configuration allowed. Indeed 25mm cassettes of between 0.45 to 1.2 μm pore sizes are acceptable. Flow rates of 0.5 to 16 L/min are also listed as acceptable. Finally, the final volumes that the method recommends range from a minimum of 400 L to 10000 L.

Unlike the AHERA protocol, there is no need for containment construction, inspections, and aggressive sampling techniques. Again, many times this method is selected as a diagnostic tool for asbestos in air surveys.

NIOSH 7402: In the Laboratory. The procedure for the preparation of samples is similar to that listed for AHERA. There is, however, a greater degree of tolerance for the acceptance and rejection of samples, the use of particulate interference techniques, and the grids employed. The AHERA method calls for the use of the LTA to assist in reducing organic and filter interference. The NIOSH method does not incorporate this preparation stage. NIOSH will allow non-indexed sample grids. NIOSH, though mentioning avoiding particulate interference in the sampling regimen, does not clearly state the threshold to reject a sample. IATL uses a 25% of the filter surface as this criteria.

The analysis of each sample is strictly guided by the NIOSH 7402 method. It includes *no* requirements for analyst training. There are some instrument calibration and performance requirements. NIOSH does not specify quality control routines to assure accuracy and precision by the analyst and the laboratory.

The individual sample analysis concerns examining a known area of filter depending on particulate loading. This ranges from 6 to 40 grid openings or 0.078 mm² to 0.52 mm². The cost of a 40 grid opening count is generally four to five times greater than an AHERA analysis. IATL employs a grid opening minimum to achieve <0.0030 s/cc sensitivity (usually 6-10 grid openings).

Similar to AHERA, the qualification procedure requires three criteria: size and morphology, crystalline diffraction pattern, and elemental identification. The method lists strict counting and recording rules. Unlike AHERA, only fibers with >3:1 aspect ratio are counted. Furthermore only those fibers greater than 5 µm are counted.

The analysis requires magnifications routinely at 1,000 with exceptions for magnifications at 10,000. This is 2- 20 times lower than the AHERA requirement. Will asbestos structures be missed at these magnifications? Yes (see the AIHA Journal January 1996 The Yant Award Lecture Series). Here individual asbestos fibrils (the smallest denomination) can not be resolved and identified. In fact long fibers >10 µm in length may be missed if their diameter is less than 0.25 µm.

It is for these 'deficiencies' in magnification and recorded fiber length that many labs offer modified NIOSH analysis. IATL offers NIOSH with analytical sensitivity down to 0.0030 s/cc, using magnifications at 20,000 and IATL counts fibers from 0.5 µm to less than or equal to 5.0 µm and fibers greater than 5.0 µm.

The analyst may spend up to an hour per sample depending upon how much particulate is present on the sample. There is no provision for sample grid archiving. The data and associated documentation is then used to calculate results to the client.

All TEM Laboratories that analyze air samples using the NIOSH method need not be certified by NIST/NVLAP. Generally AIHA Industrial Hygiene Laboratory certification is favored. The requirements for this accreditation are beyond the scope of this discussion.

NIOSH 7402: Results Interpretation. Limits of Detection for this method may vary depending on the sample volume provided. Again, IATL attempts <0.0030 f/cc as the LOD. The results calculated from raw data by TEM are presented in the following manner: Asbestos Fibers per millimeter square area of the filter examined (mm²) and Asbestos Fibers (>5.0 µm) per volume of air sampled (cc). The IATL report also expands on the size ranges noted above.

The NIOSH method also has provisions for reporting the fraction of optically visible asbestos fibers on the filter (f/mm²). This measurement compares the total non-asbestos fiber count (>5.0 µm at <10,000 magnification) with the asbestos fibers counted (>5.0 µm at <10,000 magnification).

Though there are no 'failure' and 'passing' guidelines, the method does list OSHA, MSHA, NIOSH, and ACGIH values for fiber levels considered exceeding the carcinogenic level. These range from 0.1 to 2.0 f/cc for those fibers greater than 5.0 µm long.

IATL and Asbestos in Air Issues. In summary, AHERA is strictly outlined as to field set-up, sampling, laboratory analysis, and data interpretation. It requires a vast amount of quality assurance activity and the on-going certification of laboratories. The magnification and structure counting requirements clearly set AHERA apart from NIOSH. The NIOSH 7402 method, can be used as a diagnostic tool for asbestos in air surveys. The methods, though comparable, have many areas where they deviate from each other in intent and content.

IATL employs a fully trained, full time TEM staff of four along with five other technicians for two shifts per day, seven days per week. Most of our asbestos in air work concerns the AHERA method. We offer NIOSH and a full range of other analytical protocols to our clients.

For further information please contact:

IATL at iatl.com,
Frank Ehrenfeld at frankehrenfeld@iatl.com,
International Asbestos Testing Laboratory
16000 Horizon Way, Unit 100
Mt. Laurel, NJ 08054
(856) 231-9449

References:

- 1 Rook, H. L., and Beard, M. E., "Advances in Environmental Measurement Methods for Asbestos", ASTM STP 1342, *American Society for Testing and Materials*, 1999.
- 2 Verma, D. K., and Clark, N. E., "Relationships Between Phase Contrast Microscopy and Transmission Electron Microscopy Results of Samples From Occupational Exposure to Airborne Chrysotile Asbestos," *American Industrial Hygiene Association Journal*, Vol. 56 September 1995.
- 3 Chatfield, E. J., "A Validated Method for Gravimetric Determination of Low Concentrations of Asbestos in Bulk Materials" *Advances in Environmental Measurement Methods for Asbestos*", ASTM STP 1342, H.L. Rook and M. E. Beard Eds., American Society for Testing and Materials, 1999.
- 4 Webber, J. S. and Jackson K. W., "Transmission Electron Microscope Method for Identifying and Quantitating Asbestos in Non-Friable Organically Bound Bulk Samples. Method 198.4" *In Environmental Laboratory Approval Program Certification Manual*. Revision March 1, 1997.
- 5 United States Environmental Protection Agency. "Interim Method for the Determination of Asbestos in Bulk Samples. EPA 600/M4-82-020. 1982.
- 6 Perkins, R.L. and Harvey, B. W., "Test Method: Method for the Determination of Asbestos in Bulk Building Materials." EPA 600/R-93/116 1993.
- 7 Chatfield, E. J., and Dillion, M. J., "Determination of Asbestos Structures in Drinking Water." Method 100.1, EPA 600/4-83/043. 1983
- 8 Brackett, K. A., Clark, P. J., and Millette, J. R., "Determination of Asbestos Structures Over 10mm in Length in Drinking Water." Method 100.2, EPA 600/R-94/134. 1994
- 9 Rook, H. L., and Beard, M. E., "Overview Section in Advances in Environmental Measurement Methods for Asbestos", ASTM STP 1342, *American Society for Testing and Materials*, 1999.
- 10 Occupational Safety and Health Administration Field Operations Manual. "Instruction CPL 2-2.0A, March 1984, Chapter VII: Sampling for Surface Contamination. 1.h."
- 11 Ewing, Wm., E. and Alber, G. P., "Observations of Settled Asbestos Dust in Buildings." *In Environmental Information Association Technical Journal*, Summer 1996.
- 12 Crankshaw, O. S., Perkins, R. L., and Beard M. E., "An Overview of Settled Dust Analytical Methods and Their Relative Effectiveness." *In Advances in Environmental Measurement Methods for Asbestos*", ASTM STP 1342, H.L. Rook and M. E. Beard Eds., American Society for Testing and Materials, 1999.
- 13 Crankshaw, O. S., Perkins, R. L., and Beard, M. E., "Quantitative Evaluation of the Relative Effectiveness of Various Methods for the Analysis of Asbestos in Settled Dust," *Environmental Information Association Technical Journal*, Vol. 4, No. 1, Summer 1996.
- 14 Millette, J. R., and Hays, S. M., "Settled Dust Sampling and Analysis." Lewis Publishers 1994.
- 15 Lee, R. J., Stewart I. M., and Van Orden D. R., "Dust and Airborne Concentrations – Is There a Correlation? Presentation at *Joint ASTM Committee D-22 and EIA Conference: Advances in Environmental Measurement Methods for Asbestos*, 1997 Boulder Colorado.

- 16 Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentration. *American Society for Testing and Materials*. ASTM Method D5755-95
- 17 Millette, J. R., and Mount M. D., "Applications of the ASTM Asbestos Dust Method D5755". In *Advances in Environmental Measurement Methods for Asbestos*", ASTM STP 1342, H.L. Rook and M. E. Beard Eds., American Society for Testing and Materials, 1999.
- 18 Beard, M. E., "Monitoring Strategies and Data Interpretation for Asbestos in Settled Dust: Options and Recommendations". Presentation at *ASTM Technical and Professional Training Course Asbestos Control* PCN#36.003022.00 Version 1.1, June 2000.
- 19 Millette, J. R., Hays, S. M., and Ewing Wm., M., "Dust Sampling Studies and Recommended Action Levels." Presentation at *ASTM Technical and Professional Training Course Asbestos Control* PCN#36.003022.00 Version 1.1, June 2000.
- 20 Ewing, Wm. M., "Further Observations of Settled Asbestos Dust in Buildings." In *Advances in Environmental Measurement Methods for Asbestos*", ASTM STP 1342, H.L. Rook and M. E. Beard Eds., American Society for Testing and Materials, 1999.
- 21 Standard Test Method for Wipe Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentration. *American Society for Testing and Materials*. ASTM Method D6480-99.
- 22 CARB
- 23 ASTM
- 24 Superfund
- 25 EPA II
- 26 ISO 13012
- 27 AIHA Journal Yang Lecture
- 28