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TECHNICAL NOTES

Asbestos Quantification Using

Point Counting and Other methods

by Jim Richards

In analysis quantification is necessary and expected. In asbestos management there are rules and regulations that apply to materials that contain certain quantities of asbestos. The quantification is most critical at the 1 percent level as this is the determination point as to whether a material is or is not legally considered asbestos containing[1]. It makes little difference if a material is determined to be 10 percent or 11 percent asbestos, however, it is significant if the level is determined to be less than one percent or exactly one percent.

Polarized Light Microscopy is the EPA approved method[1] for the analysis of building materials suspect to contain asbestos Point counting was developed for the quantification of minerals in rock specimens using PLM. In point counting a graticule is placed in the ocular (eye piece) of the microscope. The graticule contains a crosshair. The place at which the crosshairs meet is known as the “point”. When the slide containing the sample preparation is moved into the field of view of the microscope the “point” may fall on a particle. If the particle is identified as an asbestos fiber the point is counted as a asbestos point. If the particle is identified as a non-asbestos particle then the point is counted as a non asbestos point. If the point falls on an empty area of the slide nothing is counted and the slide is moved to a new field of view. The process is repeated until 400 to 1000 asbestos and non asbestos points are counted. The total number of asbestos points is found and is divided by the number of non asbestos points and asbestos points

counted. The result is multiplied by 100. This gives the percent of asbestos in the sample.

The size of the asbestos fibers themselves play an important role in quantification. For example, in floor covering products such as tile and sheet vinyl, the asbestos fibers must be very small so that the mechanical properties of the flooring are not deteriorated by the use of the asbestos filler[2]. The fiber size used was often in the range of 10 micrometers in length and corresponding width of less than 0.5 micrometers which is below the limit of resolution of an optical microscope[3]. Therefore, even if the flooring sample contains the typical 20 to 30 percent asbestos[4] the optical microscope cannot resolve these fibers making identification impossible. To the microscopist these particles do hot exhibit the optical properties of asbestos and therefore can't be positively determined to be asbestos. Because they do not appear to be fibrous they are overlooked. Point counting would be of no use because the analyst can't identify the particles as being asbestos whether they fall on a point or not. As a result, the point counting of a floor covering product is useless. Analysis by PLM is also useless for the same reason. There are other materials such as plaster, joint compound and stucco that may have many very short fibers and therefore are not possible to analyze using PLM.

Point counting is statistical. Obviously one can not look at the entire sample to determine the concentration of asbestos. Therefore, the field technician must take a ***representative*** sample of the material and send that to the lab. Once the sample is in the laboratory the analyst must prepare slides that are ***representative*** of the sample sent in by the client. If the fiber size is large enough to be observed in an optical microscope, point counting analysis can begin. As mentioned before, according to regulations if the sample is 1 percent or greater than it is considered to be an asbestos containing material.

When performing a 400 point count analysis of an asbestos sample that contains 1% asbestos we should count 4 asbestos particles in 400 total particles because there is a probability in 400 trials that 4 particles should be asbestos and 396 should not. The 95% confidence level of the sample containing 1% asbestos would be 4 asbestos particles +/- 3.9 asbestos particles. Therefore we are 95% certain that our analysis of a sample that actually contains 1% asbestos will give a result between 0 to 2% asbestos[5]. For a 1000 point count of a 1% asbestos sample we should count 10 asbestos particles. The 95% confidence level of this will be smaller because the number of points counted is higher. Therefore, we would expect to count 10 +/- 6.17 asbestos particles for a range of 1.6% to 0.4% asbestos. In either case we can't be exactly certain that the sample contains 1% asbestos but that it falls into a range between the 95% upper confidence limit and the 95% lower confidence limit. When interpreting the results of analysis one must keep this fact in mind.

Again, it is very important to remember that there is a high probability that the validity of all quantitative analysis of asbestos samples may be in doubt due to not having taken truly homogeneous samples from the field site. This is true of point counting as well as any other method. There should be a ***statistically significant*** collection technique developed by the field inspector and put into practice during all sample collection. If this is not done than the integrity of the sample is dubious and therefore so will the results of the analysis.

Gravimetry is a technique that enhances PLM in the quantitation of asbestos in bulk building materials. The sample is first dried and then weighed. It is then subjected to high temperature (between 300 deg C to 500 deg C) in a furnace for a period of approximately 6 hours. During this process organic material is oxidized (burned). After cooling the sample is re-weighed and the weight loss recorded. Further treatment with dilute hydrochloric acid may be used to remove carbonates and other acid soluble substances in the sample. The remaining material is then washed with plain water, dried and weighed. This weight loss is also recorded. The residue is now examined by PLM or electron microscopy for the presence of asbestos fibers. Using PLM point counting is performed to determine the concentration of asbestos fibers in the residue. Because we know the weight of the residue we can calculate the concentration of asbestos in the original sample. Gravimetry improves the ability to detect asbestos fibers in the sample and also improves the quantification. This is so because the residue is very often granular particulates rather than a wide variety of materials of vastly different density and mass thereby making more representative slides and improving point counting statistics..

There are other techniques such as x-ray diffraction and electron microscopy that can be employed in asbestos identification and quantification.

X-ray diffraction is a technique in which a monochromatic x-ray beam is diffracted by the planes in a solid crystal lattice of a material. The diffracted angle can be measured with great accuracy and is unique for each crystalline material. X-ray diffraction unfortunately cannot differentiate between asbestos polymorphs (non fibrous forms) that are not regulated minerals. Therefore this technique is not valid unless the particles can be identified by other methods as being fibrous. In the case of tile, the fiber size is too small to be identified by optical microscopy therefore X-ray diffraction is not a viable alternative. Furthermore, the method detection limit is not always low enough to detect even 1 percent asbestos in a sample[6].

Electron microscopy has the unique ability to determine if a particle is a fiber even when it is as small as 0.02 microns (150 times smaller than an optical microscope). Identification of the morphology is very easy as one can simply see that the particle is of fibrous habit or not. Furthermore the electron microscope has two types of confirmatory analysis that may

be undertaken to prove that the object is a regulated mineral or not. These methods are energy dispersive x-ray analysis and electron diffraction. Electron diffraction is similar to x-ray diffraction in that each diffraction pattern produced is unique for the mineral that generated the pattern. Quantitative electron microscopy is performed following gravimetric reduction. The sample is then placed in an aqueous suspension using a suitable surfactant and then filtered through a MCE filter. The filtering takes place in a special apparatus that helps to make a uniform distribution of particles on the filter. The sample is then prepared exactly the same as an air TEM sample. The fibers length and width are measured within a known area of the filter. Chrysotile is tubular and therefore the volume of a cylinder is calculated. By converting the fiber area into volume and knowing the density the mass of the fibers on the filter can be calculated. Because the area of the filter that was observed in the TEM is known the total mass of asbestos on the filter can be calculated and then in turn the total mass of asbestos in the whole sample can be calculated.

In summary none of these methods are the equal of other laboratory analysis as regards precision and accuracy. However, there are no alternative methods available. Therefore it is important to remember that the concentration of asbestos measured my vary considerably from one analysis to another even of the same exact sample. This is not because of incompetence but rather the practical limitations of statistical analysis techniques. It is possible to determine the concentration of asbestos in many samples to very great accuracy, however, in most cases this would take extensive laboratory time and would result in very high cost. For example, using point counting and counting 13,000 points would still not significantly improve the quantification. The range for a 1 percent asbestos sample would be from 0.83% to 1.17%. This would take 11 worker hours to count and cost more than $500 to perform. This is the same range that is normally expected in the ELLPAT proficiency testing rounds required for lead analysis of paint chips[7]. In atomic absorption spectroscopy the analysis takes only a few minutes. Therefore the industrial hygienist must use good judgement in evaluating the results of analysis and the specific situation. If a sample is analyzed and found to contain 0.8 percent asbestos it is very possible that another analysis or laboratory may report 1.2 percent and both would be correct within the limits of the methods. How does one then decide that the sample is asbestos containing material or not? This is the realm of the industrial hygienist to determine. The same problems occur when, for example, a lead sample is at or very near the action level.

References:

1 EPA 600/M4-82-020, December 1982. (40 CRF Part 763, Appendix A to Subpart F)

2 PolyVinyl Chloride, Harold A. Sarvetnick, van Nostrand Reinhold, New York, 1969,

LCCCN (Library of Congress Card Catalog Number) 69-18948, page 108

3 The Stabilization of PolyVinyl Chloride, Fernand Chevassus, Translated from the French by C. John R. Eichhorn and Esteban E. Sarmiento, St. Martin's Press, Inc., New York. 1963 (LCCCN (Library of Congress Card Catalog Number) 63-19023) page 303.

4 Optics and Optical Instruments, B.K. Johnson, Dover Publications, New York, ISBN 0-486-60642-2, page 89

5 If x represents the number of successes in n independent trials of an experiment for which π is the probability of success in a single trial, then the variable $x-n\pi / \sqrt{n\pi(1-\pi)}$ has a distribution that approaches the standard normal distribution as the number of trials becomes increasingly large.

6 US EPA Method 600/R-93/116, July 1993, "Method for the Determination of Asbestos in Bulk Building Materials", page 35.

7 AIHA (American Industrial Hygiene Association) ELLPAT Proficiency Round 022 3/18/98 for paint chip sample one with a range of 1.2043 to 1.7171 percent.