ANALYTICAL SENSITIVITY OF BULK ASBESTOS ANALYSIS

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THERE IS ALSO ANALYTICAL SENSITIVITY FOR BULK ASBESTOS ANALYSIS

For airborne asbestos analysis by TEM (transmission electron microscopy), the AHERA (Asbestos Hazard Emergency Response Act) protocol requires that a minimum area or a minimum number of grid openings on the prepared TEM grids is to be analyzed so that the AS (analytical sensitivity) is no greater than 0.005 structure/cm^3. AS is therefore defined to be *the airborne asbestos concentration represented by one asbestos structure found in the analyzed area of TEM grids prepared from a filter that has collected between 1,200 and 1,800 liters of air*.

Although conventional wisdom tells us that, regardless of the analytical technique used, there must be a minimum amount of sample materials that is to be analyzed in order to ensure the reliable detection of a specified concentration of the target component in a multiple-component system, none of the two EPA bulk asbestos analysis protocols (EPA/600/M4-82-020 and EPA/600/R-93/116) has addressed this critical issue. Nor have the two protocols provided any guidance in the determination of the minimum amount of bulk sample materials that is to be analyzed to ensure the reliable detection of 1%wt. of asbestos component(s).

For bulk asbestos analysis by PLM (polarized light microscopy), the AS can be defined as *the weight percentage of a single asbestos structure in the amount of sample analyzed*. To be specific for the point counting technique used in bulk sample quantification proposed in EPA/600/M4-82-020, the AS is defined to be the weight percentage of a single asbestos structure in the sample material in the total number of FOV's (filed of view) analyzed.

EQUATIONS FOR CALCULATING ANALYTICAL SENSITIVITY OF BULK ASBESTOS ANALYSIS

I have derived a series equations related to the AS in bulk asbestos analysis from the following basic equation:

$$AS(\%) = \frac{W_A}{W_S} * 100\%, \qquad (1)$$

where W_A – the weight a ***single*** asbestos structure;
W_S – the weight of the sample analyzed.

Since W_S equals the sum of W_A and W_M, the weight of matrix materials analyzed, Equation (1) can be expressed as

$$AS(\%) = \frac{W_A}{W_M + W_A} * 100\% \text{ or } AS(\%) = \frac{1}{(\frac{W_M}{W_A}) + 1} * 100\%. \qquad (2)$$

Assuming that the thickness of the particle layer in a microscopic slide preparation equals to the thickness of an average asbestos structure in the sample and the width of the asbestos structure equals its thickness, for practical purpose, the AS can be calculated by

$$AS(\%) = \frac{1}{(\frac{D_M * N_{FOV} * A_{FOV} * C_{FOV}}{D_A * T_A^{\ 2} * AR}) + 1} * 100\%, \qquad (3)$$

where D_M – the average density of the matrix material (g/cm^3);
N_{FOV} – the number of FOV analyzed;
A_{FOV} – the area of FOV (μm^2) ***at the magnification used in point counting***;
C_{FOV} – the average coverage of FOV by soil samples (%);
D_A – the average density of asbestos fibers (g/cm^3);
T_A – the average thickness of asbestos fiber (μm);
AR – the average aspect ratio (length/width) of asbestos fibers.

From Equation (3), the minimum weight of a bulk sample or the minimum number of FOV that must be analyzed to attain a specified AS can be easily calculated. Table 1 lists the calculation results for chrysotile in a matrix with average density of 2.3 g/cm^3.

Table 1. Analytical Sensitivity Calculations for Chrysotile (Density 2.6 g/cm^3)

Fiber Size*		Weight of A Single Fiber (mg)	Minimum Sample Weight (mg) to Achieve Analytical Sensitivity			Minimum No. of FOV to Achieve Analytical Sensitivity**		
Width (mm)	Aspect Ratio		0.5	1	2	0.5	1	2
0.05	20	0.007	***1.3***	***0.7***	***0.3***	***6***	***3***	***1***
0.1	20	0.052	***10***	***5***	***3***	***24***	***12***	***6***
0.2	20	0.416	***83***	***42***	***21***	***96***	***48***	***24***
0.3	20	1.404	***281***	***140***	***70***	***216***	***108***	***54***
0.4	20	3.328	***666***	***333***	***166***	***384***	***192***	***96***
0.5	20	6.5	***1300***	***650***	***325***	***600***	***300***	***150***
1	20	52	***10400***	***5200***	***2600***	***2399***	***1199***	***600***
0.05	100	0.033	***7***	***3***	***2***	***30***	***15***	***7***
0.1	100	0.26	***52***	***26***	***13***	***120***	***60***	***30***
0.2	100	2.08	***416***	***208***	***104***	***480***	***240***	***120***
0.3	100	7.02	***1404***	***702***	***351***	***1079***	***540***	***270***
0.4	100	16.6	***3328***	***1664***	***832***	***1919***	***960***	***480***
0.5	100	32.5	***6500***	***3250***	***1625***	***2999***	***1499***	***750***
1	100	260.0	***52000***	***26000***	***13000***	***11994***	***5997***	***2999***

* Assuming thickness=width (square cross section)

** Assuming A_{FOV} = 3.14 mm^2 (the diameter of Olympus BH-2 PLM's FOV with 10X dispersion staining objective is 2 mm). C_{FOV} = 60%, and D_M = 2.3 g/cm^3.

THE USE OF CHALKLEY POINT ARRAY SHOULD BE STOPPED

Many bulk asbestos laboratories have been using Chalkley point array reticle (Fig. 1) for point counting because it is one of the options specified by EPA/600/M4-82-020. In most majority cases, this reticle is used with 10X dispersion staining objective. For Olympus BH-2 PLM (polarized light microscope), the diameter of FOV with 10X DS objective is approximately 2 mm. Also in most majority cases, only 400 points specified by EPA/600/M4-82-020 are counted, which are equivalent to as little as 8 FOV's if all 25 points in each FOV are non-empty points. If half of the 25 points in each FOV are non-empty points, only 16 FOV's are counted to reach the required 400 points. The calculations presented in Table 1 clearly indicate that if the width of average asbestos fiber in a bulk sample is 200 μm or greater, using Chalkley point array for pointing counting is not going to achieve the AS needed to differentiate ACM (asbestos-containing material) from non-ACM. One fact that is often ignored by analysts is that the area defined by the circle of Chalkley point array is much smaller than the area of FOV. Moreover, the distance between the points or crosses in Chalkley point array is too small to ensure proper sampling of bulk asbestos samples. The practice of using Chalkley point array should be stopped. There are even published or unpublished test methods that recommend the use of point arrays with 100 points or crosses inside the reticle. Needless to say, the authors of those test methods seem to have not read Chayes's classic treatise on the point counting and did not know how to choose the distance between grid points when conducting point counting. Nor did those authors understand the fundamental principles of stereology.

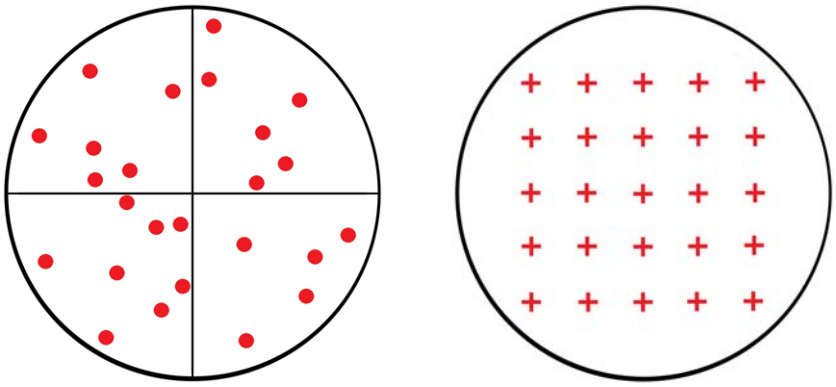

Figure 1. Chalkley point array: 25 points (left) and 25 crosses (right).

EPA'S POINT COUNTING PROCEDURE SHOULD BE REVISED

The EPA point counting procedure detailed in EPA/600/M4-82-020 can be summarized as follows:

– preparing at least 8 slides with proper particle loading (25-50% empty area);
– using either a cross-hair reticle and mechanical stage or a reticle with 25 points (Chalkley Point Array);
– counting 50 non-empty points in each slide;
– counting 400 total points in 8 slides;
– if Chalkley point array is used, counting 2 randomly selected FOV's in each slide.

According to Table 1, which is calculated for chrysotile based on 2 mm diameter of FOV (Olympus BH-2 PLM with 10X dispersion staining objective), 40% empty area preparation based on EPA's recommendation, and 2.3 g/cm^3 matrix density, 8 slides are far from sufficient to reach 1% AS except when chrysotile fibers are extremely fine.

A scientific point counting procedure should be based on the following guidelines:

– determining N_{FOV} to be counted to achieve the target AS based on the average size of asbestos fibers, the density of asbestos fibers, the average density of matrix, the A_{FOV} of the microscope system used, the coverage of sample materials in prepared slides;

– preparing sufficient number of slides based on N_{FOV} to be counted (normally it takes about 3 mg to 7 mg of samples to make a slide with 22 mm square cover glass and 50-75% particle coverage, depending on the density of matrix);

– using only a cross-hair reticle and mechanical stage to conduct point counting;

– the distance between adjacent scanning grid points should be equal for both X and Y directions and based on the average length of asbestos fibers.

REFERENCES

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