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Standard Practice for Asbestos Detection Limit Based on Counts[1]

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1. Scope

1.1 This practice presents the procedure for determining the detection limit (DL)[2] for measurements of fibers or structures[3] using microscopy methods.

1.2 This practice applies to samples of air that are analyzed either by phase contrast microscopy (PCM) or transmission electron microscopy (TEM), and samples of dust that are analyzed by TEM.

1.3 The microscopy methods entail counting asbestos structures and reporting the results as structures per cubic centimeter of air (str/cc) or fibers per cubic centimeter of air (f/cc) for air samples and structures per square centimeter of surface area (str/cm^2) for dust samples.

2. Referenced Documents

2.1 *ASTM Standards:* [4]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres

D 5755 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations

D 6281 Test Method for Airborne Asbestos Concentration in Ambient and Indoor Atmospheres as Determined by Transmission Electron Microscopy Direct Transfer (TEM)

D 6480 Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis of Asbestos Structure Number Concentration by Transmission Electron Microscopy

E 456 Terminology for Relating to Quality and Statistics

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *average*, *n*—the sum of a set of measurements (counts) divided by the number of measurements in the set.

3.1.1.1 *Discussion*—The *average* is distinguished from the *mean*. The average is calculated from data and serves as an estimate of the *mean*. The *mean* (also referred to as the *population mean, expected value*, or *first moment*) is a parameter of the underlying statistical distribution of counts.

3.1.2 *background*, *n*—a statistical distribution of structures introduced by (*i*) analyst counting errors and (*ii*) contamination on an unused filter or contamination as a consequence of the sample collection and sample preparation steps.

3.1.2.1 *Discussion*—This definition of *background* is specific to this practice. The only counting errors considered in this definition of *background* are errors that result in an over-count (that is, false positives). Analyst counting errors are errors such as, determining the length of structures or fibers and whether, based on length, they should be counted; counting artifacts as fibers; determining the number of structures protruding from a matrix; and interpreting a cluster as one, two, or more structures that should be counted only as zero or one structure. For purposes of developing the DL, assume that background contamination sources have been reduced to their lowest achievable levels.

3.1.3 *blank*, *n*—a filter that has not been used to collect asbestos from the target environment.

3.1.3.1 *Discussion*—Blanks are used in this practice to determine the degree of asbestos contamination that is reflected in asbestos measurements. Contamination may be on the virgin filter or introduced in handling the filter in the field or when preparing it for inspection with a microscope. The data required to determine the degree of contamination consists, therefore, of measurements of field blanks that have experienced the full preparation process.

3.1.4 *decision value*, *n*—a numerical value used as a boundary in a statistical test to decide between the null hypothesis and the alternative hypothesis.

3.1.4.1 *Discussion*—In the present context, the decision value is a structure count that defines the boundary between "below detection" (the null hypothesis) and "detection" (the alternative hypothesis). If a structure count were larger than the decision value, then one would conclude that detection has been achieved (that is, the sample is from a distribution other than the background distribution). If the count were less than or equal to the decision value, the result would be reported as

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[1] This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

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[2] The DL also is referred to in the scientific literature as Limit of Detection (LOD), Method Detection Limit (MDL), and other similar descriptive names.

[3] For purposes of general exposition, the term "structures" will be used in place of "fibers or structures." In the examples in Section 8, the specific term, "fiber" or "structure," is used where appropriate. These terms are defined separately in Section 3.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

"below detection," which means that the sample cannot be differentiated from a sample that would have been collected from the background distribution.

3.1.5 *detection limit*—the mean of a structure count population that is sufficiently large so a measurement from this population would have a high probability (for example, 0.95 or larger) of exceeding the decision value that determines detection.

3.1.5.1 *Discussion*—The DL is the value of a parameter, the true mean of a structure count population in the statistical hypothesis testing problem, that underlies the DL concept. Specifically, it is the true mean of the alternative hypothesis that ensures a sufficiently high power for the statistical test that determines detection.

3.1.6 *count*, *n*—the number of fibers or structures identified in a sample.

3.1.7 *fiber*, *n*—any of various discrete entities with essentially parallel sides counted by a particular method that specifies length, width, and aspect ratio.

3.1.7.1 *Discussion*—The definitions of "fiber" and "structure" are similar because the measurement method employed specifies the shape, length, width, and aspect ratio.

3.1.8 *mean*, *n*—the mean value of the number of structures in the population of air or dust sampled.

3.1.8.1 *Discussion*—The *mean* in this definition is intended to be the population mean, expected value, or first moment of a statistical distribution. It is a theoretical parameter of the distribution that may be estimated by forming an average of measurements (refer to Terminology E 456 for definition of population).

3.1.9 *power*, *n*—the probability that a count exceeds the decision value for a sample that was obtained from a population other than the background population.

3.1.9.1 *Discussion*—Power is the probability of selecting, based on a statistical test, the alternative hypothesis when it is true. In the present context, this means the probability of making the correct decision to report a structure concentration for a sample that was collected from a population other than the background population. The *power* of the statistical test equals 1 minus the *type II error rate*.

3.1.10 *replicate*, *n*—a second measurement is a replicate of the initial measurement if the second measurement is obtained from an identical sample and under identical conditions as the initial measurement.

3.1.10.1 *Discussion*—"Identical," as applied to sample, can mean" same subsample preparation," "separate preparation of a distinct subsample," or a distinct sample obtained from the same population as the initial sample. For this practice, "identical" means distinct sample obtained from the same population as the initial sample.

3.1.11 *sample*, *n*—the segment of the filter that is inspected, and thereby, embodies the air or dust that was collected and the subset of structures that were captured on the portion of the filter subjected to microscopic inspection (also, see Terminology D 1356).

3.1.12 *sensitivity*, *n*—the structure concentration corresponding to a count of one structure in the sample.

3.1.13 *structure*, *n*—any of various discrete entities counted by a particular method that specifies shape, length, width, and aspect ratio.

3.1.14 *type I error*, *n*—choosing, based on a statistical test, the alternative hypothesis over the null hypothesis when the null hypothesis is, in fact, true; a false positive outcome of a statistical test.

3.1.14.1 *Discussion*—A type I error would occur if the count for a sample exceeded the decision value, but the sample was, in fact, obtained from the background population. The analyst erroneously would be led by the statistical test to report a structure concentration (that is, choose the alternative hypothesis of the statistical test), where the result should be reported as "below the detection limit" (that is, the null hypothesis of the statistical test is true).

3.1.15 *type II error*, *n*—choosing, based on a statistical test, the null hypothesis over the alternative hypothesis when the alternative hypothesis is, in fact, true; a false negative outcome of a statistical test.

3.1.15.1 *Discussion*—A type II error would occur if the count for a sample does not exceed the decision value, but the sample was, in fact, obtained from a population other than the background population. The analyst would erroneously be led by the statistical test to report a "below the detection limit" result (that is, choose the null hypothesis of the statistical test), where the result should be reported as a structure concentration (that is, the alternative hypothesis of the statistical test is true).

3.1.16 *type I error rate*, *n*—the probability of a type I error (also referred to as the *significance level,* α*-level*, or *p-value* of the statistical test).

3.1.17 *type II error rate*, *n*—the probability of a type II error (also referred to as the β-level of the statistical test).

3.1.18 λ—lambda, the Greek letter used to represent the population mean of a Poisson distribution.

3.1.19 λ_0—the population mean of the Poisson distribution of *background* counts.

3.1.19.1 *Discussion*—λ_0 is the population mean of the Poisson distribution under the null hypothesis in the statistical hypothesis testing problem that defines the DL.

3.1.20 λ_1—the population mean of the Poisson distribution under the alternative hypothesis in the statistical hypothesis testing problem that defines the DL (DL = λ_1).

3.1.21 x_0—decision value for determining detection. If the count in a measurement is not greater than x_0, the measurement is reported as "below detection."

3.1.22 *X*—a Poisson distributed random variable used to denote the number of structures (fibers) counted in a sample.

3.1.23 *A*—the area of the filter inspected to obtain a structure count.

3.1.24 $P(X>x/\lambda, A)$—the Poisson probability of a structure count exceeding x structures (fibers) when the population mean is equal to λ and an area, *A*, of the filter is inspected.

4. Significance and Use

4.1 The DL concept addresses potential measurement interpretation errors. It is used to control the likelihood of reporting a positive finding of asbestos when the measured asbestos level cannot clearly be differentiated from the background contamination level. Specifically, a measurement is reported as being "below the DL" if the measured level is not statistically different than the background level.

4.2 The DL, along with other measurement characteristics such as bias and precision, is used when selecting a measurement method for a particular application. The DL should be established either at the method development stage or prior to a specific application of the method. The method developer subsequently would advertise the method as having a certain DL. An analyst planning to collect and analyze samples would, if alternative measurement methods were available, want to select a measurement method with a DL that was appropriate for the intended application.[5] The most important use of the DL, therefore, takes place at the planning stage of a study, before samples are collected and analyzed.

5. Descriptive Terms and Procedures

5.1 *Introduction*:

5.1.1 The DL is one of a number of characteristics used to describe the expected performance of a measurement method.[6] The DL concept addresses certain potential measurement interpretation errors. Specifically, a measurement is reported as being "below the DL" if the measured level cannot be distinguished from zero or from the randomly varying background contamination level. Stated differently, the DL provides protection against a false positive finding. When a measured value is less than an appropriately specified decision value, the analyst is instructed to disregard the measured value and report the result only as "below the DL."

5.1.2 The DL concept for asbestos measurements, which are based on microscopy, is simpler than the DL concept for measurement methods that depend, for example, on spectroscopy. For asbestos, the measurement is derived from a direct count of discrete structures using a microscope. For spectroscopy methods, the measurement is indirect requiring a calibration curve, and is subject to interferences and unspecified background signals that could be responsible for measurement values that are false positives.

5.1.3 The sources of false positives for asbestos counts are (*i*) analyst errors (for example, determining the length of structures or fibers and whether, based on length, they should be counted; counting artifacts as fibers; determining the number of structures protruding from a matrix; interpreting a cluster as one, two, or more structures that should be counted only as zero or one), and (*ii*) contamination (for example, virgin filter contamination or contamination introduced during sample collection or sample preparation). Collectively, these sources are referred to subsequently as "background." For purposes of developing the DL, assume that each background source has been reduced to its lowest achievable level.

5.2 *DL—General Discussion*:

5.2.1 DLs often have been misspecified and misinterpreted because the DL concept has not been defined with sufficient clarity for translation into operational terms; however, the DL concept and operational implementation have been presented correctly in the scientific literature by a number of authors.[7] These authors describe the DL as a theoretical value, specifically the true mean concentration of a substance in a sampled medium. This true mean, the DL, must be large enough to ensure a high probability (for example, 0.95 or larger) of concluding based on one or more measurements from a sample of the medium that the true concentration in the medium is, in fact, greater than zero or greater than an appropriately defined background level. The DL, therefore, is a parameter in the statistical decision that determines whether the concentration of a substance in a sample is consistent with the background level, which may be zero, or is greater than the background level.

5.2.2 Determining whether the mean concentration of a substance in a sample is consistent with the background concentration or is greater than the background concentration is a statistical decision problem. Due to statistical variation, replicate measurements of a sample or measurements from replicate samples do not yield identical results; thus, a measurement may exceed the true background mean level even if the sample were collected from the background distribution. Differences in replicate results are characterized as statistical variation. Values of replicate measurements are described by a probability distribution. The decision concerning whether or not a measurement is consistent with the background concentration fits the standard hypothesis testing framework in statistics. The statistical testing problem, therefore, provides the necessary structure for determining a numerical value for the DL, as well as a rule for reporting measurements as "below the DL."

5.2.3 The DL is determined by formulating the statistical testing problem as follows.

5.2.3.1 Consider a statistical test, based on one measurement, of the null hypothesis that the true mean concentration, λ, of substance in a sample is equal to the background mean, λ_0, versus the alternative hypothesis that λ is greater than λ_0. The typical decision rule leads to a choice of $\lambda>\lambda_0$ over $\lambda=\lambda_0$ if a standardized measurement[8] is larger than a specified decision value for the statistical test. The decision value is chosen to control the Type I error rate (also referred to here as the false positive rate) of the statistical test. The false positive rate is the probability that a measurement will exceed the

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[5] For example, the purpose of the measurements might be to assess differences in the levels of a substance between two sources. If it were anticipated that the levels associated with each source are likely to be less than the DL of a particular measurement method, that method would not be appropriate for the intended application.

[6] Other characteristics are precision, bias, and for asbestos measurements, sensitivity.

[7] Clayton, C. A., Hines, J. W., and Elkins, P. D., "Detection Limits with Specified Assurance Probabilities," *Analytical Chem.* 59, 1987, 2506–2514; Currie, L. A., "Limits of Qualitative Detection and Quantitative Determination: Application to Radiochemistry," *Analytical Chem.*, Vol 40, 1968, 586–593; Currie, L. A., "Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements," National Bureau of Standards Report, 1984; Fowler, D. P., "Definition of Lower Limits for Airborne Particle Analyses Based on Counts and Recommended Reporting Conventions," *Ann. Occup Hyg.*, Vol 41 Supplement 1, 1997, 203–209.

[8] In this statistical context, a standardized measurement is calculated as the measurement minus the background mean divided by the standard deviation of the background distribution.

chosen decision value, leading to acceptance of $\lambda>\lambda_0$, when the true mean concentration is, in fact, λ_0.[9]

5.2.3.2 The DL concept, although providing protection against false positives in measurement systems, also requires consideration of probabilities associated with true positives. A high degree of confidence (that is, a high probability) is required that decision in favor of $\lambda>\lambda_0$ over $\lambda=\lambda_0$ is correct. In statistical hypothesis testing terminology, this probability is referred to as the "power of the statistical test."

5.2.3.3 The power of a statistical test is the probability that a measurement exceeds the decision value (that is, the probability that the measurement leads to the choice, $\lambda>\lambda_0$) when the true mean concentration is a value larger than λ_0. The power of the test is an increasing function of the true mean, λ. The DL is the value of λ that makes the power sufficiently large. EPA definitions of the DL indicate that power, the probability of a true positive result, should be 0.95 or greater.

5.2.4 Based on the structure outlined in 5.2.3.3 reporting measurements subject to DL considerations would be implemented as follows:

5.2.4.1 Determine the decision value in the statistical test for determining if a measurement is large enough to conclude that $\lambda>\lambda_0$ is correct and determine the value of λ, say λ_1, to achieve sufficient power. λ_1 is the DL.

5.2.4.2 If the measured value exceeds the decision value, report the measured value. If the measured value is less than or equal to the decision value, report that the measurement is "below the DL."

6. Application to Air Samples

6.1 The statistical hypothesis testing formulation described above and the Poisson distribution are employed to define and calculate DLs for measurements of airborne structure concentrations.

6.2 For the DL concept to have meaning there must be a background distribution of structure measurements. The background distribution consists of sources of structures that are not the measurement targets of interest but cannot be eliminated or further reduced.

6.2.1 The background distribution for airborne structure measurements is a combination of (*i*) analyst error and (*ii*) contamination (filter or laboratory).

6.2.1.1 Analyst errors are errors such as: determining the length of structures or fibers and whether, based on length, they should be counted; counting artifacts as fibers; determining the number of structures protruding from a matrix; interpreting a cluster as one, two, or more structures that should be counted only as zero or one.[10]

6.2.1.2 Filters may become contaminated from impurities that are inherent in their production or in the laboratory during filter preparation for analysis in the laboratory. Filter contamination should be minimized by laboratory QA/QC procedures.[11]

6.2.2 All background sources should be reduced to their lowest achievable levels. From an empirical perspective, it is neither practical nor necessary to quantify the background sources separately. The background level may be determined by analyzing blanks without attempting to differentiate among sources.[12]

6.3 *Characterization of Sampling and Analysis to Measure Airborne Asbestos*—As an aid in the subsequent discussion, a simplified characterization of air sampling and analysis for measuring airborne asbestos concentrations is used. Although this characterization of the measurement process may lack important details from a microscopist's perspective, it is adequate for describing how to calculate a DL (refer to D 6281 and NIOSH 7400[13] for additional details).

6.3.1 Air sampling is accomplished by drawing air through a filter at a specified rate for a specified period of time. Airborne particles consisting of asbestos and other matter are deposited on the filter. After air sampling has been completed, a section of the filter is prepared for inspection by microscopy. A specified number of fields of view of known size (that is, graticule fields for PCM and grid openings for TEM), are randomly selected and inspected microscopically. The particles found in each field of view are classified as fibers for PCM or asbestos structures for TEM and a count is recorded. The count obtained from the fields that were inspected is increased by an appropriate factor to produce an estimated count for the total filter. This estimate is divided by the volume of air collected during sampling. The resulting measurement is interpreted as an estimate of the asbestos concentration in the air, and is reported in units of fibers/cc of air (f/cc) for PCM or structures/cc of air (str/cc) for TEM.

6.3.2 The information described in 6.3.1 that is needed to address DLs can be summarized as a single number-measurement "sensitivity." Sensitivity is a characteristic that applies to individual measurements.[14] Sensitivity is defined as the structure concentration corresponding to a count of one structure in the sample. Sensitivity, therefore, depends on air volume and the fraction (a proportion) of the filter that is inspected. The fraction depends on the size of the effective filter collection area, the size of the fields of view, and the number of fields of view that are inspected.

$$\text{Sensitivity }(S) = [(EFA/(FOV*FOVA)]/V \qquad (1)$$

where:

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[9] This probability also is referred to as the significance level or *p*-value of the test and typically is selected to be 0.05, but could be larger or smaller to reflect the gravity of the consequences of a false positive.

[10] Misclassification of a nonasbestos structure as an asbestos structure is not treated as a false positive in the present discussion of DLs. For purposes of defining a DL, consider only the background sources described above as contributing to false positives.

[11] QA/QC procedures include: testing a sample of filters from a new supply before the new supply is used in the field; and diligently eliminating sources of asbestos contamination from the laboratory.

[12] Background estimation methods are described in 6.4.2.

[13] Asbestos and Other Fibers by PCM, "NIOSH Manual of Analytical Methods, Fourth Edition, 8/15/94.

[14] The sensitivity concept also may be applied to averages of multiple measurements in situations where "a measurement" always means the average of a specified number of independent replicate measurements. This application of sensitivity is not discussed here.

EFA = the effective filter collection area in square millimeters (mm^2);
FOV = the number of fields of view;
FOVA = the average field of view area in mm^2; and,
V = air volume in cubic centimeters (cc).

6.3.3 Given any value as a requirement for sensitivity, the air volume, field of view size, and number of fields of view may be varied to achieve the required value.

NOTE 1—Typical *EFA*s are 385 mm^2 for a filter with a 25-mm diameter and 855-mm^2 for a filter with a 37-mm diameter.

6.3.4 From the definition of sensitivity, it follows that the structure concentration measurement for a sample is the number of structures counted multiplied by sensitivity:

$$str/cc = (\#\ \text{structures})*S \tag{2}$$

6.4 Based on the usual assumption that the structure count from an air sample is described by the Poisson probability distribution, equations were developed for calculating DLs. The DL is stated as a mean structure count. The mean structure count may subsequently be translated to concentration units (str/cc) through multiplication by the sensitivity of the measurement as shown in Eq 2.

6.4.1 *Background Mean Known*[15]—Let X represent the number of structures counted in a sample based on inspection of a filter area equal to A ($A = \Sigma FOV_I \cdot FOVA_i$ where FOV_i is the number of grid openings with area $FOVA_i$). Let λ be the true average structure count. To establish the DL, set up a statistical test of the hypothesis H_0: $\lambda=\lambda_0$ versus the alternative H_1: $\lambda>\lambda_0$ as described in 5.2.3. λ_0 is the true mean count of structures for the background distribution when an area, A, of the filter is inspected.[16] The decision value, x_0, is defined as the solution to $P(X>x_0|\lambda=\lambda_0, A)=\alpha$ (α is the significance level or Type I error rate of the statistical test). The power of the statistical test is calculated as $P(X>x_0|\lambda=\lambda_1, A)=1-\beta$. β is the Type II error rate of the test and $1-\beta$ is the value specified as the power of the test. The DL is the value of λ_1 that satisfies the equation for the power of the test.

6.4.1.1 The equations for calculating the DL are as follows: Solve

$$P(X>x_0)|\lambda=\lambda_0, A) = \alpha \tag{3}$$

to determine the decision value, x_0.

Then solve

$$P(X>x_0)|\lambda=\lambda_1, A) = 1 - \beta \tag{4}$$

for λ_1, which is the DL.

6.4.1.2 Calculate the probabilities indicated in Eq 3 and 4 using the following:

$$P(X>x \mid \lambda, A) = 1 - \Sigma\lambda^t \cdot e^{-\lambda}/t! \tag{5}$$

where the index t in the sum takes the values 0, 1, 2, ..., x.

6.4.1.3 *Numerical Examples of DLs for Airborne Asbestos*—Based on assumptions about the true value of the underlying background mean, decision values and DLs have been determined and are recorded in Tables 1-4. The examples in Tables 1 and 2 have been developed for a statistical test of $\lambda=\lambda_0$ versus $\lambda>\lambda_0$ with the nominal significance level of α = 0.05 and nominal powers equal to 0.95 and 0.99, respectively. Because of the discrete nature of structure counts and the discrete nature of the Poisson distribution, it is not possible to achieve the nominal value of α = 0.05 exactly. For each case in Tables 1 and 2, x_0 was chosen to correspond to the largest value of α that is less than or equal to 0.05. The actual values of α are shown in the tables.

TABLE 1 Detection Limits for Different Background Means Measurement Unit Equals Number of Structures (Nominal α = 0.05; Power = 0.95)

NOTE 1—"Structures" applies both to structures and fibers depending on the measurement protocol.

Background Mean (λ_0)	Decision Value x_0	Actual Type I Error Rate (α)	Detection Limit (λ_1)
0.00–0.05	0	0.000–0.048	3.00
0.05–0.35	1	0.002–0.049	4.74
0.35–0.81	2	0.006–0.049	6.30
0.81–1.36	3	0.010–0.049	7.75
1.36–1.97	4	0.013–0.050	9.15
1.97–2.61	5	0.016–0.050	10.51

6.4.1.4 Fig. 1 displays an example of the two Poisson distributions that determine the detection limit. This example is taken from Table 1. The background mean, λ_0, is 0.81, the decision value is 2, and the detection limit is 6.30.

6.4.1.5 It is extremely important to recognize that the background mean, λ_0, and therefore, the DL depend on the area of the filter that is inspected to produce a measurement. For example, if λ_0 equals 0.60 for measurements based on inspecting A mm^2 of the filter, λ_0, would be expected to be 1.20 for measurements based on inspecting $2 \cdot A$ mm^2 of the filter. The corresponding DLs would be 6.30 and 7.75, respectively, (Table 1) or 8.41 and 10.05 respectively (Table 2).

6.4.1.6 The corresponding DLs may be stated in concentration units by multiplying the values in Tables 1 and 2 by measurement sensitivity. One example for PCM and one example for TEM are provided. For PCM, the results are displayed in Tables 3 and 4 for a measurement sensitivity of 0.0005 f/cc.[17] For TEM, the DLs stated in concentration units (str/cc) are displayed in Tables 5 and 6 for sensitivity, S, equal to 0.0064 str/cc.[18]

6.4.2 *Background Mean Unknown*—It is unlikely that the background mean would be known with certainty. The background mean may be estimated from data collected by analyzing blank filters. The estimate would have statistical error associated with it that must be accounted for in the DL determination. The magnitude of statistical error in the estimate of the background mean varies inversely with the number

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[15] One may assume that the background mean, λ_0, has a known value based on a long history in the laboratory of consistent results obtained for blank filters. It is more likely that the mean would be estimated from a recent, fixed number of blank filter analyses. The latter case is discussed in 6.4.2.

[16] λ_0 is an increasing function of the filter area, A, that is inspected.

[17] A PCM measurement based on an 8-h sample at 2 L/min, a filter with effective collection area of 385 mm^2, and inspection of 100 graticule fields of size 0.00785/mm^2 has sensitivity equal to 0.0005 f/cc.

[18] A TEM measurement based on ten grid openings each 0.006-mm^2 on a filter with effective collection area equal to 385 mm^2 (that is, a 25-mm diameter MCE filter), and 1000 L of air would, by Eq 1 have S = 0.0064 str/cc.

TABLE 2 Detection Limits for Different Background Means Measurement Unit Equals Number of Structures (Nominal α = 0.05; Power = 0.99)

NOTE 1—"Structures" applies both to structures and fibers depending on the measurement protocol.

Background Mean (λ_0)	Decision Value x_0	Actual Type I Error Rate (α)	Detection Limit (λ_1)
0.00–0.05	0	0.000–0.048	4.61
0.05–0.35	1	0.002–0.049	6.64
0.35–0.81	2	0.006–0.049	8.41
0.81–1.36	3	0.010–0.049	10.05
1.36–1.97	4	0.013–0.050	11.61
1.97–2.61	5	0.016–0.050	13.11

TABLE 3 Detection Limits for PCM Measurement Units—f/cc (Sensitivity = 0.0005; Nominal α = 0.05; Power = 0.95)

Background Mean (f/cc)	Decision Value (f/cc)	Actual Type I Error Rate (α)	Detection Limit (f/cc)
$0–2.5\times10^{-5}$	0	0.000–0.048	1.5×10^{-3}
$2.5\times10^{-5}–1.8\times10^{-4}$	0.0005	0.002–0.049	2.4×10^{-3}
$1.8\times10^{-4}–4.0\times10^{-4}$	0.0010	0.006–0.049	3.2×10^{-3}
$4.0\times10^{-4}–6.8\times10^{-4}$	0.0015	0.010–0.049	3.8×10^{-3}
$6.8\times10^{-4}–9.9\times10^{-4}$	0.0020	0.013–0.050	4.6×10^{-3}
$9.9\times10^{-4}–1.3\times10^{-3}$	0.0025	0.016–0.050	5.3×10^{-3}

TABLE 4 Detection Limits for PCM Measurement Units—f/cc (Sensitivity = 0.0005; Nominal α = 0.05; Power = 0.99)

Background Mean (f/cc)	Decision Value (f/cc)	Actual Type I Error Rate (α)	Detection Limit (f/cc)
$0–2.5\times10^{-5}$	0	0.000–0.048	2.3×10^{-3}
$2.5\times10^{-5}–1.8\times10^{-4}$	0.0005	0.002–0.049	3.3×10^{-3}
$1.8\times10^{-4}–4.0\times10^{-4}$	0.0010	0.006–0.049	4.2×10^{-3}
$4.0\times10^{-4}–6.8\times10^{-4}$	0.0015	0.010–0.049	5.0×10^{-3}
$6.8\times10^{-4}–9.9\times10^{-4}$	0.0020	0.013–0.050	5.8×10^{-3}
$9.9\times10^{-4}–1.3\times10^{-3}$	0.0025	0.016–0.050	6.6×10^{-3}

of blank filters analyzed to form the estimate. If the number of blank filters employed is large enough to render the statistical error negligible, the DL would be obtained from Tables 1 and 2 by using the estimate as if it were the true value of λ_0. Otherwise, the magnitude of the DL varies directly with the statistical error in the estimate of the background mean, and therefore, inversely with the number of blank filters used to estimate the background mean. In 6.4.2.1, this relationship is discussed and guidance is provided for the number of blank filters that should be analyzed. After estimating the background mean, a quality assurance program including standard quality control measures should be employed to maintain the lowest achievable level of filter contamination.[19]

6.4.2.1 *Method*—The correct value for λ_0, depends on the true value λ_0. Analysis results for blank filters may be used to estimate λ_0, which, in turn, leads to a value for x_0. The estimate of λ_0 is an interim calculation on the way to determining x_0; therefore, the method for determining x_0 presented here is based directly on the blank filter analysis results and does not require that the estimate of λ_0 be calculated. The method should have a high probability of determining the correct value of x_0 and a low probability of indicating a wrong value of x_0. As the number of blank filters that are analyzed to determine x_0 is increased, the probability of a correct determination approaches 1.0 (100 %). There is, however, a cost-accuracy tradeoff between the number of blank filters analyzed to determine x_0 and the degree of error that can be tolerated in x_0.

6.4.2.2 Use 100 blank filter analyses to determine the value of x_0.[20] The 100 blank filter measurements for a particular laboratory may be selected from recent historical blank analysis results obtained in that laboratory. The rule for determining x_0 based on n = 100 blank filter analyses is shown in Table 7.

6.4.2.3 Using a value of x_0 from Table 7, refer to Tables 1 and 2 for the DL (examples are provided in Section 8).

7. Application to Dust Samples

7.1 The development of a DL for dust measurements is similar to the development for air measurements. The DL for dust measurements is the mean value of the alternative in the statistical hypothesis testing formulation that was described in Section 6 for air measurements. Differences in the sample collection and preparation methods may affect the magnitude of the background mean, which, in turn, affects the magnitude of the DL. Also, the calculation of sensitivity for dust measurements is different than for air measurements because of different process steps.

7.2 *Dust Measurement Characterization*—Dust is collected from a surface using either a microvac or a wipe (see Test Methods D 5755 and D 6480). Sample preparation involves various steps including suspension of particles in liquid and filtration. Structures are counted by TEM.

7.2.1 *Sensitivity*—The initial liquid volume and the volume deposited on the filter affect the sensitivity of the measurement. Sensitivity is calculated as follows:

$$S = [EFA/(GO\cdot GOA)]\cdot(100/V)/SPL \tag{6}$$

where:

EFA = effective filter area for the secondary filter (mm^2);
GO = number of grid openings counted;
GOA = average grid opening area (mm^2);
V = volume of sample filtered representing the actual volume taken from the original 100-mL suspension (mL); and,
SPL = the area of the surface vacuumed or wiped.

It follows that the asbestos structure concentration in dust, STR/cm^2, is:

$$STR/\mathrm{cm}^2 = \#STR\cdot S \tag{7}$$

where:

$\#STR$ = number of asbestos structures counted in the sample.

7.3 *Calculating the DL*:

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[19] The blank filters under discussion here are those used to establish the background mean. They are not the blanks, typically one for every field batch of filters, that are part of the ongoing QC program. The blanks used in the QC program are intended to flag gross contamination or identify a change in the previously established background mean.

[20] Rules for determining a value for x_0 based on analyzing n = 10, 25, 50, 100, and 200 blank filters have been developed and evaluated. The rules are discussed in the appendix to this practice.