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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:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[4]

D 5755 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations[4]

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[4]

E 456 Terminology for Relating to Quality and Statistics[5]

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

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[1] This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres 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] *Annual Book of ASTM Standards*, Vol 11.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

equal to the decision value, the result would be reported as "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.[6] 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.[7] 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.[8] 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[9] 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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[6] 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.

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

[8] 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.

[9] 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.[10]

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.[11]

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.[12]

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.[13]

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[14] 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.[15] 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.

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

where:

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[10] 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.

[11] 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.

[12] 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.

[13] Background estimation methods are described in 6.4.2.

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

[15] 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*[16]—Let X represent the number of structures counted in a sample based on inspection of a filter area equal to A ($A = \Sigma FOV_1 \cdot FOVA_i$ 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.[17] 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.

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.[18] 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.[19]

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

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

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[16] 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.

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

[18] 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.

[19] 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)
0–2.5×10^{-5}	0	0.000–0.048	1.5×10^{-3}
2.5×10^{-5}–1.8×10^{-4}	0.0005	0.002–0.049	2.4×10^{-3}
1.8×10^{-4}–4.0×10^{-4}	0.0010	0.006–0.049	3.2×10^{-3}
4.0×10^{-4}–6.8×10^{-4}	0.0015	0.010–0.049	3.8×10^{-3}
6.8×10^{-4}–9.9×10^{-4}	0.0020	0.013–0.050	4.6×10^{-3}
9.9×10^{-4}–1.3×10^{-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)
0–2.5×10^{-5}	0	0.000–0.048	2.3×10^{-3}
2.5×10^{-5}–1.8×10^{-4}	0.0005	0.002–0.049	3.3×10^{-3}
1.8×10^{-4}–4.0×10^{-4}	0.0010	0.006–0.049	4.2×10^{-3}
4.0×10^{-4}–6.8×10^{-4}	0.0015	0.010–0.049	5.0×10^{-3}
6.8×10^{-4}–9.9×10^{-4}	0.0020	0.013–0.050	5.8×10^{-3}
9.9×10^{-4}–1.3×10^{-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.[20]

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.[21] 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:

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

where:

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

7.3 *Calculating the DL*:

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[20] 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.

[21] 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.

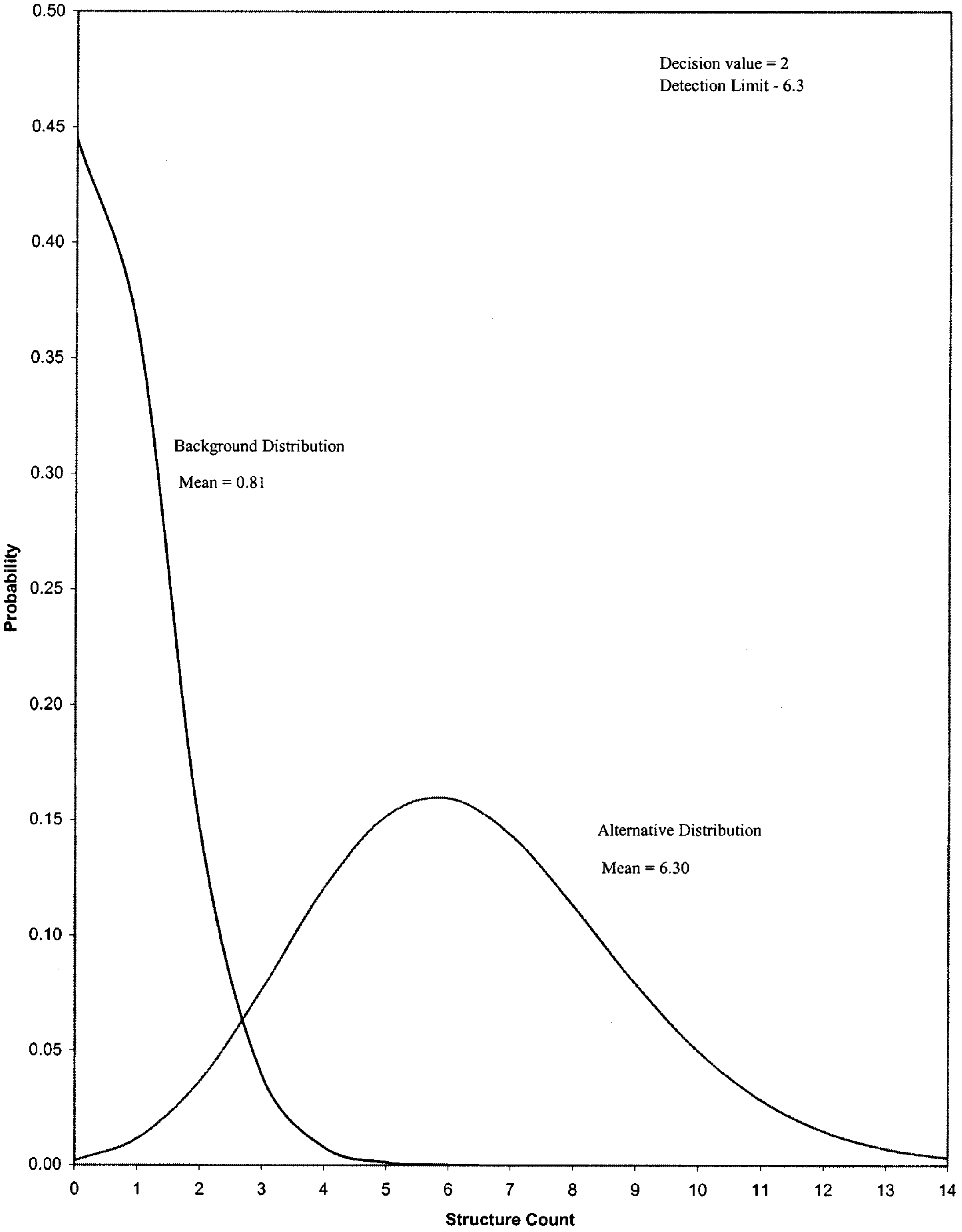

FIG. 1 Background Distribution, Decision Value, and Detection Limit

7.3.1 *Background Mean Known*—The background sources for dust measurements are the same general sources identified as sources for air measurements, that is, analyst error and laboratory error. The laboratory component for dust measurements may be larger than the laboratory component for air measurements due to additional steps in the preparation process (refer to Test Methods D 5755 and D 6480). For purposes of demonstrating the DL for dust measurements, assume the same range of background means used for air measurements applies.

TABLE 5 Detection Limits for TEM (Direct Preparation) Measurement Units—str/cc (Sensitivity = 0.0064; Nominal α = 0.05; Power = 0.95)

Background Mean (str/cc)	Decision Value (str/cc)	Actual Type I Error Rate (α)	Detection Limit (str/cc)
$0–3.2\times10^{-4}$	0	0.000–0.048	1.9×10^{-2}
$3.2\times10^{-4}–2.4\times10^{-3}$	0.0064	0.002–0.049	3.0×10^{-2}
$2.4\times10^{-3}–5.2\times10^{-3}$	0.0128	0.006–0.049	4.0×10^{-2}
$5.2\times10^{-3}–8.7\times10^{-3}$	0.0196	0.010–0.049	4.9×10^{-2}
$8.7\times10^{-3}–1.3\times10^{-2}$	0.0256	0.013–0.050	5.8×10^{-2}
$1.3\times10^{-2}–1.7\times10^{-2}$	0.0320	0.016–0.050	6.7×10^{-2}

TABLE 6 Detection Limits for TEM (Direct Preparation) Measurement Units—str/cc (Sensitivity = 0.0064; Nominal α = 0.05; Power = 0.99)

Background Mean (str/cc)	Decision Value (str/cc)	Actual Type I Error Rate (α)	Detection Limit (str/cc)
$0–3.2\times10^{-4}$	0	0.000–0.048	3.0×10^{-2}
$3.2\times10^{-4}–2.4\times10^{-3}$	0.0064	0.002–0.049	4.2×10^{-2}
$2.4\times10^{-3}–5.2\times10^{-3}$	0.0128	0.006–0.049	5.4×10^{-2}
$5.2\times10^{-3}–8.7\times10^{-3}$	0.0196	0.010–0.049	6.4×10^{-2}
$8.7\times10^{-3}–1.3\times10^{-2}$	0.0256	0.013–0.050	7.4×10^{-2}
$1.3\times10^{-2}–1.7\times10^{-2}$	0.0320	0.016–0.050	8.4×10^{-2}

TABLE 9 Detection Limits for Dust Measurements by TEM Measurement Unit Equals STR/cm^2 (Nominal α = 0.05; Power = 0.99)

Background Mean (μ_0 in STR/cm^2)	Decision Value y_0	Actual Type I Error Rate (α)	Detection Limit μ_1 in STR/cm^2
0–50	0	0.000–0.048	4610
50–350	1000	0.002–0.049	6640
350–810	2000	0.006–0.049	8410
810–1360	3000	0.010–0.049	10050
1360–1970	4000	0.013–0.050	11610
1970–2610	5000	0.016–0.050	13110

TABLE 7 Rule for Selecting x_0 Based on Measurements from 100 Blank Filters

x_0	Total Structure Count in 100 Blank Filter Measurements
0	0–5
1	6–34
2	35–78
3	79–132
4	133–194
5	195–269

7.3.1.1 *Sensitivity*—A typical value of sensitivity discussed for dust sampling and analysis methods is 1000 str/cm^2.[22] Using values for λ_0 and λ_1 from Tables 1 and 2, Tables 8 and 9 display DLs in units of str/cm^2 for dust measurements associated with various background means.

7.3.2 *Background Mean Unknown*—Follow the procedure described in 6.4.2 for air samples. In addition, blank filters used for estimating the background level should go through the entire applicable preparation process.

8. Examples

AIR MEASUREMENTS

8.1 *PCM*:

8.1.1 *Estimate Background Mean, Decision Value, and DL*—Compile the measurement results for 100 blank MCE 25 mm diameter filters with EFA 385 mm^2. The count for each filter should reflect the number of fibers with a 3:1 aspect ratio and length of 5 µm or longer in 100 graticule fields each with area 0.00785 mm^2. Use NIOSH 7400 counting rules. Tabulate the total number of fibers counted across the 100 filters.

8.1.2 *Example 1*—Analysis of 100 blank filters yields 150 fibers. Based on Table 7, the decision value, x_0, for detection is four, and from Table 1 the detection limit is 9.15.[23]

8.1.2.1 *Sampling and Analysis*—Using a 25-mm diameter MCE filter, collect a 960-L air sample. Prepare the filter and count the number of fibers in 100 fields each with an area of 0.00785 mm^2. Sensitivity for this measurement is 0.0005 f/cc. The DL, stated in f/cc units, is 9.15·0.0005 = 0.0046 f/cc. If the number of fibers counted in the sample is larger than 4, multiply the count by 0.0005 and report the result as f/cc. If the number of fibers counted in the sample is less than or equal to four, report the measurement as "below the detection limit of 0.0046 f/cc (<0.0046 f/cc)."

8.1.2.2 Assume five fibers are counted. The airborne fiber concentration estimate is reported as 0.0025 f/cc. This value is an estimate of the mean concentration for the environment that is sampled. The statistical uncertainty associated with this estimate of the mean can be assessed by calculating the 95 % upper confidence limit (95 % UCL) for this result. The 95 % UCL corresponding to a count of 5 is 10.51, and the 95 % UCL for the concentration is 0.0053 f/cc (see Table 10). If the number of fibers counted in the sample were less than or equal to the decision value of 4, the measurement would be reported as "below detection." For example, if three fibers are counted, the result would be reported as "below the detection limit of 0.0046 f/cc" or "<0.0046 f/cc." If the estimated f/cc value for this sample were calculated, it would be 3·0.0005=0.0015 f/cc with a 95 % UCL of 0.0038 f/cc; however, the 0.0015 f/cc estimate will not be reported. Record the result only as "below the detection limit of 0.0046 f/cc" because the count does not exceed the decision value.

TABLE 8 Detection Limits for Dust Measurements by TEM Measurement Unit Equals STR/cm^2 (Nominal α = 0.05; Power = 0.95)

Background Mean (μ_0 in STR/cm^2)	Decision Value y_0	Actual Type I Error Rate (α)	Detection Limit μ_1 in STR/cm^2
0–50	0	0.000–0.048	3000
50–350	1000	0.002–0.049	4740
350–810	2000	0.006–0.049	6300
810–1360	3000	0.010–0.049	7750
1360–1970	4000	0.013–0.050	9150
1970–2610	5000	0.016–0.050	10510

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[22] See Test Method D 5755. If 100 cm^2 were vacuumed, EFA = 1320; GOA = 0.01; and 4 mL out of 100 mL were deposited on the filter, then inspecting 30 GOs would yield a sensitivity of approximately 1000 STR/cm^2.

[23] Recall that the DL is the population mean of a fiber count distribution, and, therefore, is unlikely to be an integer value. The DL of 9.15, which is found in Table 1, corresponds to a 0.95 probability that a measurement larger than the decision value of four fibers does not belong to the background distribution. If a probability of 0.99 would be a more acceptable representation of measurement uncertainty relative to the background distribution, the DL would be 11.61 (Table 2).

TABLE 10 Upper Confidence Limits for the Poisson Distribution[A]

Count	95 % UCL	99 % UCL
0	2.996	4.605
1	4.744	6.638
2	6.296	8.406
3	7.754	10.045
4	9.154	11.605
5	10.513	13.108
6	11.842	14.571
7	13.148	16.000
8	14.435	17.403
9	15.705	18.783
10	16.962	20.145
11	18.208	21.490
12	19.443	22.821
13	20.669	24.139
14	21.886	25.446
15	23.097	26.743
16	24.301	28.030
17	25.499	29.310
18	26.692	30.581
19	27.879	31.845
20	29.062	33.103
21	30.240	34.355
22	31.415	35.601
23	32.585	36.841
24	33.752	38.077
25	34.916	39.308
26	36.077	40.534
27	37.234	41.757
28	38.389	42.975
29	39.541	44.190
30	40.691	45.401

[A] Calculations based on formulas in *Biometrika Tables for Statisticians*, Vol 1, Cambridge University Press 1954.

8.1.2.3 A full accounting of the uncertainty in the two measurement results described above is displayed in Table 11[24].

8.1.3 *Example 2*—Analysis of 100 blank filters yields 50 fibers. Based on Table 7, the decision value for detection is two and the detection limit is 6.30 fibers (Table 1).[25]

TABLE 11 Example Demonstrating Application and Interpretation of Detection Limits

		Calculated Results		Reported Results	
Sample ID	Fiber Count	Concentration Estimate (f/cc)	95 % UCL (f/cc)	Concentration Estimate (f/cc)	95 % UCL (f/cc)
1	5	0.0025	0.0053	0.0025	0.0053
2	3	0.0015	0.0039	<0.0046	NA

8.1.3.1 *Sampling and Analysis*—Using a 25-mm diameter MCE filters, collect a 960-L air sample. Prepare the filter and count the number of fibers in 100 fields, each with area 0.00785 mm^2. Sensitivity for this measurement is 0.0005 f/cc. The DL, stated in f/cc units is 6.30·0.0005 = 0.0032 f/cc. If the number of fibers counted in the sample is larger than two, multiply the count by 0.0005 and report the result as f/cc. If the number of fibers counted in the sample is less than or equal to two, report the measurement as "below the detection limit of 0.0032 f/cc (<0.0032 f/cc)."

8.2 *TEM*:

8.2.1 *Estimate Background Mean, Decision Value, and DL*—Compile the measurement results for 100 blank MCE 25 mm diameter filters with EFA 385 mm^2. (The count for each filter should reflect the number of asbestos structures with a 5:1 aspect ratio and length of 0.5 µm or longer in 10 grid openings each with area 0.01 mm^2. Use EPA AHERA counting rules. Tabulate the total number of asbestos structures counted across the 100 filters.

8.2.2 *Example 1*—Analysis of 100 blank filters yields seven structures. Based on Table 7, the decision value for detection is one and the detection limit 4.74 structures (Table 1).

8.2.2.1 *Sampling and Analysis*—Using a 25 mm diameter MCE filters, collect a 2400-L air sample. Prepare the filter and count the number of asbestos structures in 10 grid openings, each with area 0.01 mm^2. Sensitivity for this measurement is 0.0016 str/cc. The DL stated in str/cc is 4.74 × 0.0016 = 0.0076 str/cc. If the number of structures counted in the sample is larger than one, multiply the count by 0.0016 and report the result as str/cc. If the number of structures counted in the sample is either zero or one, report the measurement as "below the detection limit of 0.0076 str/cc (<0.0076 str/cc)."

8.2.3 *Example 2*—Analysis of 100 blank filters yields five structures. Based on Tables 5 and 6, the decision value for detection is zero and the detection limit 3.00 structures (Table 1).

8.2.3.1 *Sampling and Analysis*—Using a 25-mm diameter MCE filter, collect a 2400 liter air sample. Prepare the filter and count the number of asbestos structures in ten grid openings, each with area 0.01 mm^2. Sensitivity for this measurement is 0.0016 str/cc. The DL, stated in str/cc units is 3.00 × 0.0016 = 0.0048 str/cc. If the number of structures counted in the sample is larger than 0, multiply the count by 0.0016 and report the result as str/cc. If the structure count in the sample is 0, report the measurement as "below the detection limit of 0.0016 str/cc (<0.0016 str/cc)."

8.3 *Dust Measurements*:

8.3.1 *Estimate Background Mean, Decision Value, and DL*—Following the procedure described in Test Method D 5755, prepare 100 blank polycarbonate (PC) 47 mm diameter filters for evaluation with a TEM. Count the number of asbestos structures (5:1 aspect ratio; length of 0.5 µm or longer) in 30 grid openings each with area 0.01 mm^2. Use Test Method D 5755 counting rules.

8.3.2 *Example 1*—Analysis of 100 blank filters yields seven structures. Based on Table 7, the decision value for detection is one and the detection limit 4.74 structures (Table 1).

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[24] The "reported" concentration estimates in Column 5 of Table 11 might be viewed as nonintuitive or inconsistent because a fiber count of 5 leads to a concentration estimate (0.0025) that is smaller than the estimate for a fiber count of 3 (<0.0046). These "reported results," nevertheless, are correct. The ordering of the concentrations for "calculated results" shows what would be expected; however, the sample with the fiber count of 3 does not satisfy the test for detection. Therefore, the result for that sample is an upper bound, namely the DL of 0.0046 f/cc. The concentration for that sample can be reported, at best, as a range of uncertainty between 0 f/cc and 0.0046 f/cc. The sample with the count equal to 5 satisfies the test for detection. It has a point estimate concentration of 0.0025 f/cc and a range of uncertainty that includes, as an upper bound, 0.0053 f/cc with 95 % confidence.

[25] The DL of 6.30, which is found in Table 1, corresponds to a 0.95 probability that a measurement larger than the decision value of 4 fibers does not belong to the background distribution. If a probability of 0.99 would be a more acceptable representation of measurement uncertainty relative to the background distribution, the DL would be 8.45 (Table 2).

8.3.2.1 *Sampling and Analysis*—Vacuum dust in a 100-cm^2 area. Prepare the sample, which is redeposited on 47 mm diameter polycarbonate filter. Count the number of asbestos structures in 30 grid openings of area 0.01 mm^2. Sensitivity for this measurement is approximately 1000 str/cm^2. The DL, stated in str/cm^2 units, is $4.74 \times 1000 = 4740$ str/cm^2. If the number of structures counted in the sample is larger than 1, multiply the count by 1000 and report the result as str/cm^2. If the number of structures counted in the sample is either zero or one, report the measurement as "below the detection limit of 4740 str/cm^2 (<4740 str/cm^2)."

8.3.3 *Example 2*—Analysis of 100 blank filters yields five structures. Based on Table 7, the decision value for detection is zero and the detection limit 3.00 structures (Table 1).

8.3.3.1 *Sampling and Analysis*—Vacuum dust in a 100-cm^2 area. Prepare the sample, which is redeposited on 47-mm diameter polycarbonate filter. Count the number of asbestos structures in 30 grid openings of area 0.01 mm^2. Sensitivity for this measurement is approximately 1000 str/cm^2. The DL, stated in str/cm^2 units, is $3.00 \times 1000 = 3000$ str/cm^2. If the number of structures counted in the sample is larger than 0, multiply the count by 1000 and report the result as str/cm^2. If the number of structures counted in the sample is zero, report the measurement as "below the detection limit of 3000 str/cm^2 (<3000 str/cm^2)."

9. Keywords

9.1 asbestos; detection limit; fiber count

APPENDIX

(Nonmandatory Information)

X1. ESTIMATING THE BACKGROUND MEAN, λ_0, AND THE DECISION VALUE, x_0

X1.1 If the value of the background mean, λ_0, is known, the decision value, x_0, is uniquely determined (refer to 6.4); however, it is unlikely that the background mean would be known with certainty. 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 that is not needed to determine x_0; therefore, the method for determining x_0 presented here is based directly on the blank filter analysis results and does not require calculation of the interim estimate of λ_0.

X1.2 The method for determining x_0 should have a high probability of indicating 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 the estimate of x_0.

X1.3 The rules for determining x_0 based on n = 10, 25, 50, 100, and 200 blank filters have been evaluated. With the exception of n = 100 and 200, the probabilities of incorrectly determining the value of x_0 are unacceptably large. The rules for determining the value of x_0 are displayed in Table X1.1 for n = 100 and n = 200.

X1.4 The probabilities of correctly and incorrectly determining x_0 are displayed in Table X1.2. To interpret these entries, consider as an example the case where the correct value of x_0 is two and apply the rule based on data for n = 100 blank filters. The ideal rule for determining x_0 would have high probabilities for correctly determining x_0 and low probabilities for incorrect determinations of x_0. Due to the discrete nature of the Poisson distribution, any improvement of the probability in one cell in Table X1.2 degrades the probability in another cell. When true value of x_0 is 2, the probability that x_0 would be incorrectly determined as 1 is 0.05 (5 %); the probability that x_0 would be incorrectly determined as three is 0.11 (11 %). The probability that x_0 is determined correctly as two is 0.85 (85 %). These probabilities do not add to 1.00 exactly due to rounding errors.

TABLE X1.1 Rule for Selecting x_0 Based on Blank Filter Measurements

x_0	Total Structure Count in 100 Blank Filter Measurements	Total Structure Count in 200 Blank Filter Measurements
0	0–5	0–12
1	6–34	13–71
2	35–78	72–161
3	79–132	162–270
4	133–194	271–394
5	195–269	395–529

X1.5 On balance, the rules for n = 100 and n = 200 are both reasonable and not substantially different. This follows because the two types of errors in determining x_0, selecting a value too small or selecting a value too large, are not equally important. The DL concept is intended to provide protection against false positive errors. Choosing a value for x_0 that is larger than the correct value will provide additional protection against false positives, and, therefore, is the less significant of the two errors. Upon reinspection of the entries in Table X1.2, note that the probabilities of selecting a value for x_0 that is too small is no greater than 0.05 (5 %) both for the rule based on n = 100 and for the rule based on n = 200. From this assessment, the rule based on n = 100 is adequate.

TABLE X1.2 Probabilities of Determining Values for x_0

Correct Value of x_0	Rule	Data for Blanks Indicates x_0= 0	Data for Blanks Indicates x_0= 1	Data for Blanks Indicates x_0= 2	Data for Blanks Indicates x_0= 3	Data for Blanks Indicates x_0= 4	Data for Blanks Indicates x_0= 5	Data for Blanks Indicates x_0= 6
0	n = 100	0.88	0.12					
	n = 200	0.95	0.05					
1	n = 100	0.04	0.87	0.09				
	n = 200	0.04	0.91	0.05				
2	n = 100		0.05	0.85	0.11			
	n = 200		0.04	0.90	0.06			
3	n = 100			0.04	0.84	0.12		
	n = 200			0.04	0.89	0.07		
4	n = 100				0.05	0.84	0.11	
	n = 200				0.05	0.89	0.06	
5	n = 100					0.07	0.88	0.05
	n = 200					0.06	0.89	0.05

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