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

This standard is issued under the fixed designation D6620; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 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.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*



## 2. Referenced Documents

2.1 *ASTM Standards:*[4]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D5755 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading



D6281 Test Method for Airborne Asbestos Concentration in Ambient and Indoor Atmospheres as Determined by Transmission Electron Microscopy Direct Transfer (TEM)



D6480 Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Surface Loading by Transmission Electron Microscopy

~~E456~~D7712 Terminology ~~Relating to Quality and Statistics~~for Sampling and Analysis of Asbestos

## 3. Terminology

3.1 *Definitions:*

3.1.1 For terms not defined in this practice, see Terminologies D1356 and D7712.

3.2 *Definitions of Terms Specific to This Standard:*

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

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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~~Sampling, Analysis, Management of Asbestos, and Other Microscopic Particles.

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[2] The DL also is referred to in the scientific literature as ~~Limit~~limit of ~~Detection (LOD), Method Detection Limit~~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.

3.2.1.1 *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.

3.2.2 *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.

3.2.2.1 *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.

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

3.2.3.1 *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.~~background levels (also referred to as contamination) for asbestos measurement methods. Blanks are analyzed by the same method employed to analyze filters used to collect particulate from the target population.



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

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

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

3.2.5.2 *Discussion—*

The decision value is a threshold for measured values (or averages of measured values). If the measured value (or average) exceeds this threshold, one can conclude that asbestos has been detected in the sampled material. Measured values (or averages) below the decision value may be reported but flagged to indicate that they fall below the decision value. Alternatively, they may be censored (that is, actual measured values not reported) and simply reported as non-detects. Then users of the data will be able to specify in advance whether values below the decision value are to be censored.

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

3.2.6.1 *Discussion—*

The detection limit (DL) is determined through the statistical hypothesis test described in 3.2.5.1. 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~~ statistical distribution of counts for a structure population, that is statistically larger than the mean of the ~~alternative hypothesis that ensures a sufficiently high power for the statistical test that determines detection.~~statistical distribution for the background population of structures. The DL is the smallest of the means for which the power of the statistical test is sufficiently large (for example, 0.95 or larger). The DL is not used to make decisions about individual measurements (for example, to decide whether they are detects or non-detects). The DL may be used to make decisions about methods, laboratories, or measurement systems. For example, to identify trace levels of asbestos a measurement method with a suitably small DL would be required.

3.2.7 *fiber, n*—any of various discrete entities with essentially parallel sides counted by a particular method that specifies length, width, and aspect ratio. (This definition of fiber, although adequate for purposes of this standard, Practice D6620, is not to be confused with the mineralogical term fiber.)

3.2.7.1 *Discussion—*

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

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

3.2.8.1 *Discussion—*

The *mean* in this definition is intended to be the population mean, mean (equivalently expected value, or first momentmoment) 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 measurements. E456 for definition of population).

3.2.9 *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.given that the count is from a distribution whose mean exceeds the background mean by a specified amount.

3.2.9.1 *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*.

3.2.10 *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.



3.2.10.1 *Discussion—*

"Identical," as applied to sample, can mean" samemean "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.

3.2.11 *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 total particulate collected on a filter or dust collected on a wipe from a population of structures; also, the portion a filter or wipe that is analyzed by microscopy (also, see Terminology D1356).

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

3.2.13 *statistical distribution, n*—the set of probabilities of structure count outcomes (0, 1, 2, 3, …).

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

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

3.2.15.1 *Discussion—*

A When testing a material that truly has zero or background levels of asbestos, 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 occurs when a measured value falls above the decision value. The analyst, based on the data, 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).detection."

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

3.2.16.1 *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 analyst, based on the data, would be led by the statistical test to report a "below the detection limit" result (that is, choose the null hypothesis of the statistical test), detection" result where the result should be reported as a structure concentration (that is, the alternative hypothesis of the statistical test is true).concentration.

3.2.17 *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).

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

3.2.19 $\lambda$—lambda, the Greek letter used to represent the population mean of a Poisson distribution.

3.2.20 $\lambda_0$—the population mean of the Poisson distribution of *background* counts.

3.2.20.1 *Discussion*—

$\lambda_0$ is the population mean of the Poisson distribution under the null hypothesis in the statistical hypothesis testing problem that defines the DL.

3.2.21 $\lambda_1$—the population mean of the Poisson distribution under the alternative hypothesis in the statistical hypothesis testing problem that defines the DL (DL = $\lambda_1$).

3.2.22 $x_0$—decision value for determining detection. If the count in a measurement is not greater than $x_0$, the measurement is reported as "below detection."

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

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

3.2.25 $P(X > x \mid \lambda, A)$ $P(X>x/\lambda, A)$— —the Poisson probability of a structure count exceeding x structures (fibers) when the population mean is equal to $\lambda$ 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



[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.

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 typically 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.~~authors **(1-4).**[7] These authors describe the DL as a theoretical value, ~~specifically~~usually the true mean concentration of a substance in a sampled ~~medium.~~population. This true mean, the DL, must be large enough to ensure a ~~high~~large probability (for example, 0.95 or larger) ~~of concluding based on~~ that results for one or more ~~measurements from a sample of the medium~~ samples of the substance from the population will be larger than the *decision value,*~~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.~~ which leads to the conclusion that the samples were not from the background population (that is the results were not below detection). The DL should be the smallest value that ensures a high probability of detection.

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, $\lambda_0$, versus the alternative hypothesis that $\lambda$ is greater than $\lambda_0$. 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~~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 chosen decision value, leading to acceptance of $\lambda>\lambda_0$, when the true mean concentration is, in fact, $\lambda_0$.[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 a 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 $\lambda_0$. The power of the test is an increasing function of the true mean, $\lambda$. The DL is the value of $\lambda$ 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 $\lambda$, say $\lambda_1$, to achieve sufficient power. $\lambda_1$ 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."

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[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.~~ The boldface numbers in parentheses refer to a list of references at the end of this standard.

[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.

[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.

## 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.~~ analysis. 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 Test Method D6281 and NIOSH 7400 (**5**) 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.[13] 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.



~~Sensitivity (S) = [(EFA)/(FOV*FOVA)]/V (1)~~

~~where:~~

~~*EFA* = the effective filter collection area in square millimeters (mm²);~~
~~*FOV* = the number of fields of view;~~
~~*FOVA* = the average field of view area in mm²; and,~~
~~*V* = air volume in cubic centimeters (cc).~~

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

where:

*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.

[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] 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.