Designation: D 6620 – 06

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

This standard is issued under the fixed designation D 6620; 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.

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 Surface Loading

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 for Asbestos Structure Number Surface Loading by Transmission Electron Microscopy

E 456 Terminology 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

[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.

Current edition approved Oct. 1, 2006. Published October 2006. Originally approved in 2000. Last previous edition approved in 2000 as D 6620 – 00.

[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.

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.

3.1.5 *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

[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.