



Designation: D7440 – 23

# Standard Practice for Characterizing Uncertainty in Air Quality Measurements<sup>1</sup>

This standard is issued under the fixed designation D7440; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice is for assisting developers and users of air quality methods for sampling concentrations of both airborne and settled materials in characterizing measurements as to uncertainty. Where possible, analysis into uncertainty components as recommended in the International Organization for Standardization (ISO) Guide to the Expression of Uncertainty in Measurement (ISO GUM, (1)<sup>2</sup>) is suggested. Aspects of uncertainty estimation particular to air quality measurement are emphasized. For example, air quality assessment is often complicated by: the difficulty of taking replicate measurements owing to the large spatio-temporal variation in concentration values to be measured; systematic error or bias, both corrected and uncorrected; and the (rare) non-normal distribution of errors. This practice operates mainly through example. Background and mathematical development are relegated to appendices for optional reading.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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.*

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.01 on Quality Control.

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<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3670 Guide for Determination of Precision and Bias of Methods of Committee D22

D6246 Practice for Evaluating the Performance of Diffusive Samplers

D6552 Practice for Controlling and Characterizing Errors in Weighing Collected Aerosols

### 2.2 ISO Standards:<sup>4</sup>

ISO GUM Guide to the Expression of Uncertainty in Measurement, ISO Guide 98, 1995<sup>5</sup>

ISO 7708 Air Quality — Particle Size Fraction Definitions for Health-Related Sampling

ISO 15767 Workplace Atmospheres — Controlling and Characterizing Errors in Weighing Collected Aerosol

ISO 16107 Workplace Atmospheres — Protocol for Evaluating the Performance of Diffusive Samplers

ISO 16702 Workplace air quality — Determination of total organic isocyanate groups in air using 1-(2-methoxyphenyl)piperazine and liquid chromatography

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, see Terminology D1356.

3.2 *Other Terms Defined as Follows are Taken from ISO GUM, Unless Otherwise Noted:*

3.2.1 *accuracy*—closeness of agreement between the result of a measurement and a true value of the measurand.

3.2.2 *combined standard uncertainty,  $u_c$* —standard uncertainty of the result of a measurement when that result is

<sup>3</sup> 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.

<sup>4</sup> BIPM version available for download from <http://www.bipm.org/en/publications/guides/gum.html>. ISO version available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>5</sup> See Ref (1), for an additional measurement uncertainty resource.

obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities.

3.2.2.1 *Discussion*—As within ISO GUM, the “other quantities” are designated uncertainty components  $u_j$  from source  $j$ . The component  $u_j$  is taken as the standard deviation estimate from source  $j$  in the case of a source of random variation.

3.2.3 *coverage factor, k*—numerical factor used as a multiplier of the combined standard uncertainty ( $u_c$ ) in order to obtain an expanded uncertainty ( $U$ ).

3.2.3.1 *Discussion*—The factor  $k$  depends on the specific meaning attributed to the expanded uncertainty  $U$ . However, for simplicity this practice adopts the now nearly traditional coverage factor as the value 2, determining the specific meaning of the expanded uncertainty  $U$  in different circumstances. Other coverage factors if needed are then easily implemented simply by multiplication of the traditional expanded uncertainty  $U$  (see 7.1 – 7.4).

3.2.3.2 *Discussion*—The use of a single coverage factor, often through approximation, avoids the overly conservative use of individual component confidence limits rather than root variance estimates as uncertainty components.

3.2.4 *error (of measurement)*—result of a measurement minus a true value of the measurand.

3.2.5 *expanded uncertainty, U*—quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand.

3.2.5.1 *Discussion*—This definition has the breadth to encompass a wide variety of conceptions.

3.2.5.2 *Discussion*—The expanded uncertainty  $U$  in some cases is expressed in absolute terms, but sometimes as relative to the measurement result. What is meant is generally clear from the context.

3.2.6 *influence quantity*—quantity that is not the measurand but that affects the result of the measurement.

3.2.7 *measurand*—particular quantity subject to measurement.

3.2.8 *measurand value*—(adapted from ISO GUM), unknown quantity whose measurement is sought, often called the true value.

3.2.8.1 *Discussion*—Examples are the concentration ( $\text{mg}/\text{m}^3$ ) of a substance in the air at a particular time and place, the time-weighted average of a concentration at a particular position, or the expected mean concentration estimate as obtained by a reference method at a specific time and position.

3.2.9 (*population*) *variance (of a random variable)*—the expectation of the square of the centered random variable.

3.2.10 *random error*—result of a measurement minus the mean that would result from an infinite number of measurements of the same measurand carried out under the same (*repeatability*) conditions of measurement.

3.2.10.1 *Discussion*—Random error is equal to error minus systematic error.

3.2.11 (*sample*) *variance*—the sum of the squared deviations of observations from their average divided by one less than the number of observations.

3.2.11.1 *Discussion*—The sample variance is an unbiased estimator of the population variance.

3.2.12 *standard deviation*—positive square root of the variance.

3.2.13 *symmetric accuracy range A*—the range symmetric about (true) measurand values containing 95 % of measurement estimates.

3.2.13.1 *Discussion*— $A$  is a specific quantification of *accuracy* (2).

**ISO 16107**

3.2.14 *systematic error (bias)*—mean that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions minus a true value of the measurand.

3.2.15 *Type A evaluation (of uncertainty)*—method of evaluation of uncertainty by the statistical analysis of series of observations.

3.2.16 *Type B evaluation (of uncertainty)*—method of evaluation of uncertainty by means other than the statistical analysis of series of observations.

## 4. Background Information

4.1 Uncertainty in a measurement result can be taken as the range about an estimate, corrected for bias if known, containing the true, or mean reference value—in the language of ISO GUM, the *measurand* value at given confidence. Uncertainty accounts not only for variation in a method’s results at application, but also for incomplete characterization of the method when evaluated. In accordance with ISO GUM, uncertainty may often usefully be analyzed into individual components.

4.2 There are several aspects of uncertainty characterization specific to air quality measurements. One of these aspects concerns known, that is, correctible, systematic error or mean bias of a measurement relative to a true measurand value. Several measurement methods exist with such bias left uncorrected because of policy, tradition, or other reason. *Uncertainty* deals only with what is unknown about a measurement, and as such does not include correctible (known) bias. The magnitude of the difference between estimate and measurand value is covered by *accuracy* as defined qualitatively in ISO GUM, rather than *uncertainty*, particularly when the bias is known, but uncorrected. Such methods require specification of both uncertainty and as much as is known of the uncorrected bias, or alternatively the adoption of an accuracy measure.

4.3 Often bias is known to exist, but with unknown value. In the case where only limits may be placed on the magnitude of the bias, ISO GUM generally recommends treating the bias as uniformly distributed within the known limits. Such a distribution refers to independent situations, for example, calibrations, where bias may arise (see 7.4 and Appendix X2), rather than variation at the point of method application. Even though such an equal-likelihood bias distribution may be unrealistic, nevertheless a standard deviation estimate may be

made that reveals the limits on the bias. If the even-distribution approximation is clearly invalid for a relevant set of measurements, the procedure may be adjusted slightly by adopting an accuracy measure tailored to the assumed limits.

4.4 Another issue concerns the distribution of measurements. ISO GUM deals only with normally distributed first-order (that is, “small”) variations relative to measurand values. An example to the contrary is afforded by normally distributed data confounded by a small number of apparent outliers (3), which may not detract from the method performance (see Appendix X4 for details). Another example is the determination of an aerosol concentration at one location (perhaps at a worker’s lapel) as an estimate of the concentration at a separate point (such as a breathing zone). In this case the variations can be of the order of the estimate itself and may have the character of a log-normal distribution.

4.5 The spatial inhomogeneity alluded to in 4.4 relates to another point regarding the focus of this practice. The spatio-temporal variations in air quality characteristics are generally so large (4) as to preclude evaluation of a method during application through the use of replicate measurements. In this case, often an initial single method evaluation is undertaken with the purpose of determining uncertainty present in subsequent applications of the method. Confidence in such an evaluation can be specified and relates to the concept of prediction-intervals (5) (see 7.2).

4.6 A related subject is measurement system control. The measurement system must remain in a state of statistical control if an introductory evaluation is to characterize later practical applications of the method. Measurement system control is evaluated using an ongoing quality control program, testing critical performance aspects for detecting problems which may develop in the method.

5. Summary of Practice

5.1 The essential idea behind ISO GUM is the analysis to the fullest extent practical of the elemental sources of what is unknown in the estimate of a measurand value. This contrasts with a global or top-down determination of uncertainty, which could for example be done ideally by comparing replicate estimates to known measurand values over all conditions expected in application of the method. Although a global uncertainty evaluation may sometimes seem inexpensive, there is a difficulty in covering essential contingencies of the method application.

5.2 Uncertainty component analysis further has several specific advantages over global analysis. The results may be applicable to a variety of situations. For example, an aerosol sampler might be (globally) evaluated as to particle-size-dependent error by side-by-side comparison to a reference sampler in several coal mines. The knowledge obtained may not be as easily applied for sampler use in iron mines, for example, as more detailed information on how the sampler performs over given dust size distributions may be needed. Furthermore, specific problem areas of a given method may be pinpointed. The detailed itemization of uncertainty sources leads to a transparency in covering the essential problems of a

measurement method. Examples of potentially significant uncertainty components are listed in Table 1.

5.3 Type A and B Uncertainty Components:

5.3.1 Components that have been statistically evaluated during method application may be classified as Type A. (See Section 7 for specific examples.)

5.3.2 Some components are often statistically evaluated during an initial method evaluation, rather than at application. Also acknowledged is a common situation that components may not have been characterized in a statistically valid manner and therefore may require professional judgment for itemizing. Such components are termed Type B uncertainties. Type B uncertainties are often associated with unknown systematic error or bias; however, random variation may also fall into this category. For example, a common assumption regarding personal sampling in the workplace is that the relative standard deviation associated with personal sampling pump variations is <5 % at essentially 100 % confidence.

5.4 Intrinsic versus Environmentally Associated Components: Influence Quantities:

5.4.1 Some uncertainties may be intrinsic to a method. For example, estimates from aerosol samplers may depend critically on sampler dimensions, which if variable leads to intersampler estimate variation.

5.4.2 On the other hand, a sampler’s performance may depend on the environment. For example, suppose a sampler is sensitive to temperature changes that are impractical to measure in the field; that is, sampler estimates are not temperature-corrected. Then measurement of this sensitivity during method evaluation together with knowledge of the temperature variation expected for a given field application can be used to determine the uncertainty associated with this effect.

TABLE 1 Common Potential Uncertainty Components

Sampling
Personal sampling pump flow rate: setting the pump and subsequent drift
Sampling rate of diffusive sampler
Sampler dimension (aerosol and diffusive sampling)
Collection efficiency of a sampler or sampling medium (also, see (6))
Analytical
Aerosol weighing
Recovery (for example, chromatographic or spectroscopic methods)
Poisson counting (for example, in XRD methods)
Instrument or sensor variation
Operator effects giving inter-lab differences (if data from several labs are to be used)
Sample
Sample stability
Sample preparation (for example, handling silica quasi-suspensions)
Sample loss during transport or storage
Evaluation
Calibration material uncertainty
Evaluation chamber concentration uncertainty
Other bias-correction uncertainty
Environmental Influence Parameters
Temperature (inadequacy of correction, if correction is made as with diffusive samplers)
Atmospheric pressure
Humidity
Aerosol size distribution (if not measured by a given aerosol sampling method)
Ambient wind velocity
Sampled concentration magnitude itself (for example, sorbent loading)

5.4.3 A quantity such as the temperature is known as an *influence quantity*. A common example where influence variables are important involves diffusive monitors, where wind velocity, temperature, pressure, and fluctuating workplace concentrations can affect diffusive monitor uptake rates (Practice D6246, ISO 16107).

5.4.4 Situations exist for which the distribution of an influence quantity is unknown. For example, the deviation between aerosol concentration estimates and samples taken according to accepted convention (for example, ISO 7708) generally depend on the aerosol size distribution sampled. Only limits on the distribution of size distributions (the influence quantity) may be known. In this case, the ISO GUM approach is generally to assume a uniform distribution (see 7.4).

5.4.5 On the other hand, the size distribution may be known to be constant over a set of measurements. In this case, the constant-distribution assumption leads to an abstract performance characterization. Alternatively, a quantity known as the *symmetric accuracy range A* (Appendix X1 and Section X4.2) in the case of unknown, but large limited *lbiasl*, may be used to establish intervals bracketing the (true) values of measurand and thus represents the *expanded uncertainty*.

5.5 *Combined and Expanded Uncertainty*—The essential ISO GUM approach then is to obtain estimates  $u_j$  of the standard deviation (often designated as  $s$  as computed on most handheld calculators) associated with the  $j$ th uncertainty source. The estimates  $u_j$  may be designated as *uncertainty components*. Then if the sources are independent, that is, if the variations are uncorrelated, a *combined standard uncertainty*  $u_c$  estimating the net standard deviation may be computed as:

$$u_c = \sqrt{\sum_j u_j^2} \quad (1)$$

5.5.1 Finally, an *expanded uncertainty*  $U$  is calculated at coverage factor  $k$  as:

$$U = k \cdot u_c \quad (2)$$

5.5.2 The purpose of the expanded uncertainty  $U$  is to bracket the unknown measurand value. For example, for an unknown mass  $M$ , given an estimate  $m$ , a coverage factor could be selected so that:

$$m - U < M < m + U \quad \text{for 95 \% of estimates } m \text{ of measurand value } M \quad (3)$$

5.5.3 However, this practice suggests use of the nearly traditional value  $k = 2$ , permitting the meaning in terms of confidence levels to float.

## 6. Significance and Use

6.1 A primary use intended for this practice is for qualifying ASTM International Standards as Standard Test Methods. In the past, a “Precision and Bias” report has been required. However, recently a statement of uncertainty has become an acceptable alternative to Guide D3670. Inclusion of such a statement with a method description simplifies comparison of ASTM Test Methods to analogous ISO and Committee for European Normalization (CEN) standards, now required to have uncertainty statements.

6.2 Standardizing the characterization of sampling/analytical method performance is expected to be useful in other applications as well. For example, performance details are a necessity for justifying compliance decisions based on experimental air quality assessments (7). Documented uncertainty can form a basis for specific criteria defining acceptable sampling/analytical method performance.

6.3 Furthermore, high quality atmospheric measurements are vital for making decisions as to how hazardous substances are to be controlled. Valid data are required for drawing reasonable epidemiological conclusions, for making sound decisions as to acceptable limits, as well as for determining the efficacy of a hazard control system.

6.4 Finally, because of developing world-wide acceptance of ISO GUM for detailing measurements when statistics are simple, the practice should be useful in comparing ASTM International Test Methods to other published methods. The codification of statistical procedures may in fact minimize the difficulty in interpreting a plethora of individual, albeit possibly valid, approaches.

## 7. Specific Examples

NOTE 1—Some of the above concepts can be illuminated through example. Application to more complicated situations is then possible.

### 7.1 Standard Deviation $\sigma$ Known Exactly:

7.1.1 Suppose the method yields unbiased estimates  $m$  in measuring unknown  $M$  so that:

$$m = M + M \cdot \varepsilon \quad (4)$$

where  $\varepsilon$  is normally distributed about 0 with known standard deviation  $\sigma$ , sometimes designated the *true relative standard deviation TRSD*. For example, suppose the method has been evaluated with essentially an infinite number of measurements of a calibration standard, giving a tight estimate of  $\sigma$ . Then estimates  $m$  are distributed normally about  $M$  so that:

$$M - 1.960 \times M \cdot \sigma < m < M + 1.960 \times M \cdot \sigma \quad \text{at probability} = 95 \% \quad (5)$$

7.1.2 Thus, to first order in  $\sigma$ , the true value  $M$  is bracketed by:

$$m - 1.960 \times m \cdot \sigma < M < m + 1.960 \times m \cdot \sigma \quad \text{at probability} = 95 \% \quad (6)$$

7.1.3 Therefore, the (relative) *expanded uncertainty*  $U$  would be consistent with Eq 3, if the coverage factor  $k$  is chosen as:

$$k = 1.960 \quad (7)$$

as a factor of *combined standard uncertainty*  $u_c$ :

$$u_c = \sigma \quad (8)$$

in other words:

$$U = 1.960 \times \sigma \quad (9)$$

7.1.4 Eq 7 is consistent with the traditional selection  $k = 2$ .

NOTE 2—Although the measurement variation depicted in Eq 4 is very common in air quality measurements, at decreasing values of  $M$ , generally a constant variation (that is, independent of  $M$ ) becomes significant, leading to non-zero limits of quantitation and detection. (See, for example, ISO 15767 and Practice D6552.)

7.2 Standard Deviation  $\sigma$  Estimated Initially by  $n$  Replicates (Type B Uncertainty):

7.2.1 Almost as simple as 7.1 is the situation in which a (relative) standard deviation estimate  $s$  is obtained through an initial  $n$  measurements of a calibration standard prior to the method's multiple subsequent uses without re-evaluation. Variations of this situation are common in air quality measurement. For example, diffusive samplers may be evaluated initially by a vendor followed by many applications without re-evaluation (see ISO 16107 or Practice D6246). Suppose Eq 4-6 still hold, except that that now  $\sigma$  is unknown but is estimated by  $s$  with  $\nu = n - 1$  degrees of freedom. What is known is that  $\sigma$  is limited by:

$$\sigma < (\nu/\chi_{\nu, 0.05}^2)^{1/2} s \tag{10}$$

at 95 % confidence in the evaluation/calibration experiment, where  $\chi_{\nu, 0.05}^2$  is the chi-square 5 % quantile at  $\nu$  degrees of freedom (obtainable from statistics tables or programs). Therefore, at 95 % confidence in the evaluation, the unknown  $M$  is bracketed by:

$$m - 1.960(\nu/\chi_{\nu, 0.05}^2)^{1/2} \times m \cdot s < M < m + 1.960(\nu/\chi_{\nu, 0.05}^2)^{1/2} \times m \cdot s \tag{11}$$

for greater than 95 % of measurements.

7.2.2 In this case, the combined (relative) uncertainty  $u_c$  is:

$$u_c = s \tag{12}$$

but if the meaning of Eq 3 is sought, the coverage  $k$  factor in Eq 11 is now:

$$k = 1.960(\nu/\chi_{\nu, 0.05}^2)^{1/2} \tag{13}$$

7.2.3 In Fig. 1 the coverage factor  $k$  of Eq 13 is plotted versus degrees of freedom  $\nu$  and is seen to approach 1.960 as  $\nu \rightarrow \infty$  corresponding to 7.1. However, Fig. 1 indicates that over a wide range of degrees of freedom adopted in practical method evaluations,  $k$  is of the order of 3 in order to achieve 95 % evaluation confidence.

NOTE 3—Specification of an evaluation confidence level together with coverage probability (both taken here to equal 95 %) relates to the statistical theory of tolerance or prediction intervals (5).

7.3 Continual Method Evaluation (Type A Uncertainty):

7.3.1 Preferred, though often not practical in air quality measurements, is an  $n$ -measurement calibration giving an estimate  $s$  for  $\sigma$  with  $\nu = n - 1$  degrees of freedom every time a practical method is applied. Then it is possible to show that the true value  $M$  is bracketed by:

$$m - t_{\nu, 0.975} \times m \cdot s < M < m + t_{\nu, 0.975} \times m \cdot s \text{ at probability} = 95 \% \tag{14}$$

where  $t_{\nu, 0.975}$  is the student-t 97.5 % quantile at  $\nu$  (also found in statistical sources).

7.3.2 Therefore the coverage factor  $k$  is now given by:

$$k = t_{\nu, 0.975} \tag{15}$$

7.3.3 In Fig. 1 this coverage factor is plotted versus the number  $\nu$  of degrees of freedom in the evaluation. As can be seen from the figure, with continual method evaluation, the

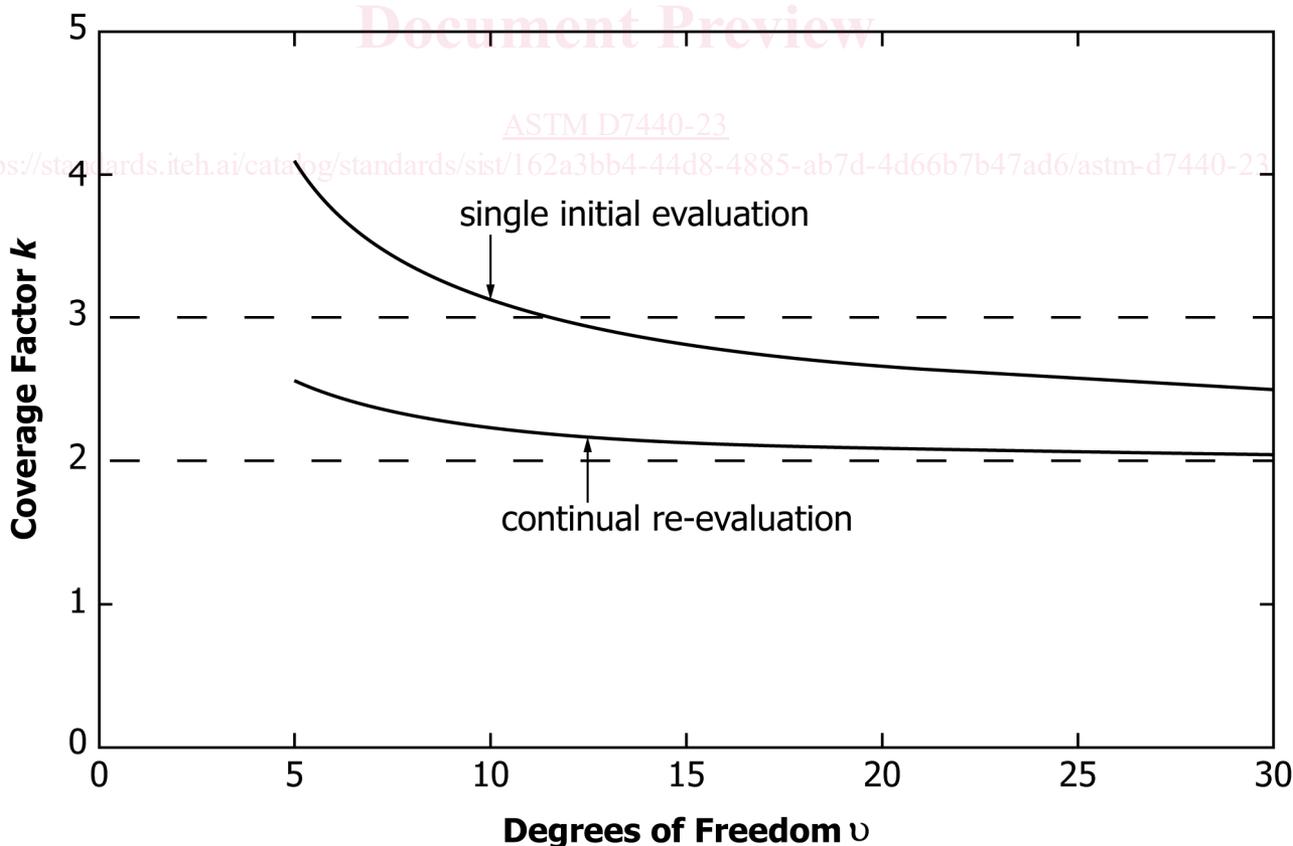


FIG. 1 Comparison of Coverage Factors  $k$  for Single Initial Method Evaluation versus Continual Evaluation with  $\nu$  Degrees of Freedom

coverage factor is close to 2 over a range of values for  $v$ . In fact, this is the reason behind the now nearly traditional use of the value 2 for the coverage factor.

7.3.4 The use of the traditional coverage factor = 2 simply gives intervals bracketing the unknown measurand with interpretation specific to the measurement circumstances. Of course, as alluded to in Section 3, if  $U$  is actually reported with the traditional coverage factor 2, then, if needed, an expanded uncertainty with 95 % evaluation confidence is easily obtained by multiplication (by about 3/2).

#### 7.4 Uncertainty Characterization of Unknown Bias or Systematic Error (Type B Uncertainty):

7.4.1 Unknown systematic error or bias in a measurement may originate in several ways. For example, if a method is not re-calibrated at each application, then error from the finiteness of an initial calibration may be present as a non-random variable in subsequent applications. Even if re-calibrated, bias may result from repeated use of a reference material or method, itself with unknown bias. In either case, the uncertainty component corresponding to uncertain method bias may be taken as the uncertainty in the bias itself.

7.4.2 *Reference Uncertainty*—As an example, suppose a method is repeatedly calibrated by a reference method that is itself biased, though is negligibly variable (as example). Then the estimated mass in measuring unknown  $M$  may be represented as:

$$m = M(1 + \Delta_{ref}) + M \cdot \varepsilon \quad (16)$$

where the standard deviation estimate  $s$  for the normally distributed random variable  $\varepsilon$  may be obtained from the calibration experiment, and where  $\Delta_{ref}$  is the unknown bias of the reference method. (See Appendix X2 for details on a similar situation, including finite-calibration bias.)

7.4.2.1 Suppose that all that is known about the reference bias  $\Delta_{ref}$  is that it is bounded by a constant positive quantity  $\Delta_{max}$ , often a matter of judgment, so that:

$$|\Delta_{ref}| < \Delta_{max} \quad (17)$$

7.4.2.2 ISO GUM generally suggests handling this situation by approximating (evaluation to evaluation)  $\Delta_{ref}$  as uniformly distributed between  $\pm \Delta_{max}$ . Then it is simple to compute an inter-evaluation variance as:

$$\text{Var}[\Delta_{ref}] = \frac{1}{3} \Delta_{max}^2 \quad (18)$$

7.4.2.3  $\Delta_{max}$  characterizes a shortcoming in the method evaluation, as does an imperfect initial determination of  $\sigma$ , the standard deviation of  $\varepsilon$  (see 7.2). Thus, assuring confidence (for example, 95 %) in the calibration with the same (prediction or tolerance) sense as in 7.2, a coverage factor  $k$  can be selected so that an expanded uncertainty given by:

$$U = k \sqrt{\frac{1}{3} \Delta_{max}^2 + s^2} \quad (19)$$

brackets the unknown  $M$  for a high fraction (for example, 95 %) of measurements (see Appendix X2 for details).

7.4.2.4 In other words, the uncertainty component  $u_{\Delta}$  for the bias is:

$$u_{\Delta} = \sqrt{\frac{1}{3} \Delta_{max}^2} \quad (20)$$

and again the random uncertainty component  $u_{random}$  is:

$$u_{random} = s \quad (21)$$

with combined uncertainty  $u_c$  given by:

$$u_c = \sqrt{u_{\Delta}^2 + u_{random}^2} \quad (22)$$

and:

$$U = k u_c \quad (23)$$

7.4.3 *Finite-Calibration Uncertainty*—Similarly to 7.4.2, correcting bias by a single  $n$ -mean estimate  $m_{ref}$  of a reference mass  $M_{ref}$  (again with estimated corrected standard deviation  $s$ ) and then calibrating subsequent application measurements by a calibration factor  $M_{ref}/m_{ref}$  leads to an uncertainty component  $u_n$  given by:

$$u_n = s/n^{1/2} \quad (24)$$

7.4.3.1 In this case the two components  $u_n$  and  $u_{random}$  are not independent if  $u_{random}$  is given by Eq 21.

NOTE 4—If an  $n$ -measurement calibration is effected at each application measurement, then the value in Eq 24 still appears as part of the calibration uncertainty, but now refers to a random rather than systematic variation.

7.4.4 *Large Bias Magnitude of Unknown Sign*—There are examples in air quality measurement where the range of unknown bias may be large relative to the variable components of uncertainty. For example, aerosol samplers used for measuring dust concentrations according to one of the international sampling conventions (ISO 7708), for example, respirable, thoracic or inhalable, generally differ in particle-size acceptance from convention. Therefore, in sampling a particular site with aerosol of unknown particle size distribution range, an unknown and sometimes large bias relative to convention is possible.

7.4.4.1 With large bias magnitude, the ISO GUM approach of 7.4.2 of combining uncertainty components squared may be replaced by a linear combination of bias magnitude uncertainty and variability uncertainty. On the other hand, the usual ISO GUM approach (with coverage factor  $k = 2$ ) gives similar uncertainty values. For an example, see Section X2.3.

#### 7.5 Analysis of a Round Robin Evaluation:

7.5.1 Analysis of a specific round robin evaluation of a measurement method as applied by several independent labs is presented here, illustrating features of uncertainty characterization. Suppose that the overall method bias magnitude, though unknown, is likely smaller (as can be decided if necessary through a student-t test) than correctable by the round robin itself because of the size of the inter-lab variation and the small number of labs tested. Appendix X1 indicates that in this case, the bias uncertainty component can be taken conservatively as the bias estimate itself. Uncertainty components estimated include  $u_{intra}$  characterizing within-lab method variability,  $u_{inter}$  for the variability between labs, and  $u_{bias}$  for overall bias of the labs (averaged together) relative to spiked or reference values.