



# Standard Guide for Reporting Uncertainty of Test Results and Use of the Term Measurement Uncertainty in ASTM Test Methods<sup>1</sup>

This standard is issued under the fixed designation E 2655; 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 guide provides concepts necessary for understanding the term “uncertainty” when applied to a quantitative test result. Several measures of uncertainty can be applied to a given measurement result; the interpretation of some of the common forms is described.

1.2 This guide describes methods for expressing test result uncertainty and relates these to standard statistical methodology. Relationships between uncertainty and concepts of precision and bias are described.

1.3 This guide also presents concepts needed for a laboratory to identify and characterize components of method performance. Elements that an ASTM method can include to provide guidance to the user on estimating uncertainty for the method are described.

1.4 The system of units for this guide is not specified. Dimensional quantities in the guide are presented only as illustrations of calculation methods and are not binding on products or test methods treated.

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

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

**E 29** Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

**E 122** Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

**E 141** Practice for Acceptance of Evidence Based on the Results of Probability Sampling

**E 177** Practice for Use of the Terms Precision and Bias in ASTM Test Methods

**E 456** Terminology Relating to Quality and Statistics

**E 691** Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

**E 2554** Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method in a Single Laboratory Using a Control Sample Program

### 2.2 Other Standard:

**ISO 17025** General Requirements for the Competence of Testing and Calibration Laboratories<sup>3</sup>

## 3. Terminology

3.1 *Definitions*—Additional statistical terms are defined in Terminology **E 456**.

3.1.1 *accepted reference value,  $n$* —a value that serves as an agreed-upon reference for comparison, and which is derived as: (1) a theoretical or established value, based on scientific principles, (2) an assigned or certified value, based on experimental work of some national or international organization, or (3) a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group. **E 177**

3.1.2 *error of result,  $n$* —a test result minus the accepted reference value of the characteristic.

3.1.3 *expanded uncertainty,  $U, n$* —uncertainty reported as a multiple of the standard uncertainty.

3.1.4 *random error of result,  $n$* —a component of the error that, in the course of a number of test results for the same characteristic, varies in an unpredictable way.

3.1.4.1 *Discussion*—Uncertainty due to random error can be reduced by averaging multiple test results.

3.1.5 *sensitivity coefficient,  $n$* —differential effect of the change in a factor on the test result.

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee E11 on Quality and Statistics and is the direct responsibility of Subcommittee E11.20 on Test Method Evaluation and Quality Control.

Current edition approved Oct. 1, 2008. Published November 2008.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3.1.6 *standard uncertainty,  $u$ ,  $n$* —uncertainty reported as the standard deviation of the estimated value of the quantity subject to measurement.

3.1.7 *systematic error of result,  $n$* —a component of the error that, in the course of a number of test results for the same characteristic, remains constant or varies in a predictable way.

3.1.7.1 *Discussion*—Systematic errors and their causes may be known or unknown. When causes are known, systematic error can sometimes be reduced by incorporating corrections into the calculation of the test result.

3.1.8 *uncertainty,  $n$* —an indication of the magnitude of error associated with a value that takes into account both systematic errors and random errors associated with the measurement or test process.

3.1.9 *uncertainty budget,  $n$* —a tabular listing of uncertainty components for a given measurement process giving the magnitudes of contributions to uncertainty of the result from those sources.

3.1.10 *uncertainty component,  $n$* —a source of error in a test result to which is attached a standard uncertainty.

## 4. Significance and Use

4.1 Part A of the “Blue Book,” Form and Style for ASTM Standards, introduces the statement of measurement uncertainty as an optional part of the report given for the result of applying a particular test method to a particular material.

4.2 Preparation of uncertainty estimates is a requirement for laboratory accreditation under **ISO 17025**. This guide describes some of the types of data that the laboratory can use as the basis for reporting uncertainty.

## 5. Concepts for Reporting Uncertainty of Test Results

5.1 Uncertainty is part of the relationship of a test result to the property of interest for the material tested. When a test procedure is applied to a material, the test result is a value for a characteristic of the material. The test result obtained will usually differ from the actual value for that material. Multiple causes can contribute to the error of result. Errors of sampling and effects of sample handling make the portion actually tested not identical to the material as a whole. Imperfections in the test apparatus and its calibration, environmental, and human factors also affect the result of testing. Nonetheless, after testing has been completed, the result obtained will be used for further purposes as if it were the actual value. Reporting measurement uncertainty for a test result is an attempt to estimate the approximate magnitude of all these sources of error. In common cases the measurement will be reported in the form  $x \pm u$ , in which  $x$  represents the test result and  $u$  represents the uncertainty associated with  $x$ .

5.2 Practice **E 177** describes precision and bias. Uncertainty is a closely related but not identical concept. The primary difference between concepts of precision and of uncertainty is the object that they address. Precision (repeatability and reproducibility) and bias are attributes of the test method. They are estimates of statistical variability of test results for a test method applied to a given material. Repeatability and intermediate precision measure variation within a laboratory. Reproducibility refers to interlaboratory variation. Uncertainty is an

attribute of the particular test result for a test material. It is an estimate of the quality of that particular test result.

5.3 In the case of a quantity with a definition that does not depend on the measurement or test method (e.g., concentration, pH, modulus, heat content), uncertainty measures how close it is believed the measured value comes to the quantity. For results of test methods where the target is only definable relative to the test method (e.g., flash points, extractable components, sieve analysis), uncertainty of a test result must be interpreted as a measure of how closely an independent, equally competent test result would agree with that being reported.

5.4 In the simplest cases, uncertainty of a test result is numerically equivalent to test method precision. That is, if an unknown sample is tested, and the test precision is known to be sigma, then uncertainty of the result of test is sigma. The term uncertainty, however, is correct to apply where variation of repeated test results is not relevant, as in the following examples.

5.4.1 *Example*—The Newtonian constant of gravitation,  $G$ , is  $6.6742 \times 10^{-11} \pm 0.0010 \times 10^{-11} \text{ m}^3\text{kg}^{-1}\text{s}^{-2}$  based on 2002 CODATA recommended values (**1**).<sup>4</sup>  $0.0010 \times 10^{-11} \text{ m}^3\text{kg}^{-1}\text{s}^{-2}$  is the standard uncertainty. The value and the uncertainty together represent the state of knowledge of this fundamental physical constant. It is not naturally thought of in terms of variation of repeated measurements. Both  $G$  and its uncertainty are derived from the analysis and comparison of a variety of measurement data using methods that are an elaboration of those presented in this guide.

5.4.2 *Example*—A length is measured but the result only reported to the nearest inch (for example, a measuring rod graduated in inches was used to obtain the measurement). Precision of the reported value, in the sense of variation of repeated measurements, is zero when all reported lengths are the same. In this case it is not possible to detect random variation in the series of repeated measurements. Uncertainty of the length is primarily composed of the systematic error of  $\pm 0.5$  inch due to the resolution of the measurement apparatus.

5.5 The goal in reporting uncertainty is to take account of all potential causes of error in the test result. In many cases, uncertainty can be related to components of variability due to sampling and to testing. Both of these should be taken into account for the uncertainty of the measurement when the purpose of the result is to estimate the property for the entire lot of material from which the sample was taken. Uncertainty of the lot property value based on a single determination is then  $\sqrt{s_1^2 + s_2^2 + u_3^2}$  where  $s_1$  is an estimate of the sampling standard deviation,  $s_2$  is an estimate of the standard deviation of the test method, and  $u_3$  is standard uncertainty due to factors that affect all measurements under consideration.

5.6 A commonly cited definition (**2, 3**) defines uncertainty as “a parameter, associated with the measurement result, or test result, that characterizes the dispersion of values that could reasonably be attributed to the quantity subject to measurement or characteristic subject to test.” This definition emphasizes

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

uncertainty as an attribute of the particular result, as opposed to statistical variation of test results. The uncertainty parameter is a measure of spread (for example, the standard deviation) of a probability distribution used to represent the likelihood of values of the property.<sup>5</sup>

5.7 The methodology for uncertainty estimates has been classified as Type A and Type B as discussed in (4). Type A estimates of uncertainty include standard error estimates based on knowledge of the statistical character of observations, and based on statistical analysis of replicate measurements. Type B estimates of uncertainty include approximate values derived from experience with measurement processes similar to the one being considered, and estimates of standard uncertainty derived from the range of possible measurement values for a given material and an assumed distribution of values within that range. See Practice E 122 for examples (e.g., rectangular, triangular, normal) where a standard deviation is derived from a range without data from samples being available. Complex estimates of test result uncertainty are calculated by combining Type A and Type B component standard uncertainties for factors contributing to error (see Section 8).

#### 5.8 Forms of Uncertainty Expression:

5.8.1 *Standard Uncertainty*—The uncertainty is reported as the standard deviation of the reported value. The report  $x \pm u$  implies that the value should be between  $x - u$  and  $x + u$  with approximate probability two-thirds, where  $x$  is the test result.

5.8.2 *Relative Standard Uncertainty*—The uncertainty is reported as a fraction of the reported value. For a measured value and a standard uncertainty,  $x \pm u$ , the relative standard uncertainty is  $u/x$ . This method of expressing uncertainty may be useful when standard uncertainty is proportional to the value over a wide range. However, for a particular result, reporting the value and standard uncertainty is preferred.

5.8.3 *Expanded Uncertainty*—The uncertainty is reported as  $x \pm U$ , where the value of  $U$  is a multiple of the standard uncertainty  $u$ . The most common multiple used is 2, which is approximately equal to the 1.96 factor for a 95 % two-sided confidence interval for the mean of a normal distribution (see 5.8.4).

5.8.4 *Confidence Intervals*—A confidence interval for a parameter (the actual value of the material property subject to measurement) consists of upper and lower limits generated from sample data by a method that ensures the limits bracket the parameter value with a stated probability  $1 - \alpha$ , referred to as the confidence coefficient.

5.8.4.1 From statistical theory, a 95 % confidence interval for the mean of a normal distribution, given  $n$  independent observations  $x_1, x_2, \dots, x_n$  drawn from the distribution, is  $\bar{x} \pm ts / \sqrt{n}$  where  $\bar{x}$  is the sample mean,  $s$  is the standard deviation of the observations, and  $t$  is the 0.975 percentile of the Student's  $t$  distribution with  $n-1$  degrees of freedom. Because Student's  $t$  distribution approaches the Normal as  $n$  increases, the value of  $t$  approaches 1.96 as  $n$  increases. This is the basis for using the factor 2 for expanded uncertainty.

5.8.5 *Measurement Uncertainty*—Measurement uncertainty is uncertainty reported for a test result without taking into account sampling variation or heterogeneity of the material of interest. The report of measurement uncertainty then refers specifically to the particular sample presented for analysis.

5.8.6 *Reporting Uncertainty with a Bias Component*—Good measurement practice requires that biases due to environmental and other factors should be corrected in the reported result when there is a sound basis for correction and the error in the correction terms themselves is not greater than the bias. Such corrections are part of the calculation of the result within the test method. The symmetrical form of reporting a measurement with standard uncertainty,  $x \pm u$ , is adequate for measurements where bias is absent or corrected. If the measurement process has a bias for which there is an estimate of magnitude and it is not corrected in the reported value  $x$ , a form of reporting should be used making clear both bias and random components. A typical form to highlight the asymmetry caused by bias is  $x - u_l / + u_h$ , where  $u_l$  = bias – standard uncertainty and  $u_h$  = bias + standard uncertainty.

5.8.7 Bias estimates are often subjective or based on weak information. When bias is present, but magnitude and direction are unknown, the uncertainty of the bias is an important part of uncertainty as a whole and should be combined with random components. The overall root mean square uncertainty is then  $u = \sqrt{u_{\text{bias}}^2 + \sigma^2}$ .

5.9 The repeatability and reproducibility values published for an ASTM method are derived from an interlaboratory study following Practice E 691 or a similar procedure. Repeatability and reproducibility values given for ASTM test methods are intended to estimate the variability of test results for competent laboratories (see Practice E 177). Reproducibility measures variability of test results on identical samples derived independently by different laboratories. This reproducibility is a good guide to the uncertainty level that it is possible to achieve for measured values obtained using the method. It may be useful to a user of test results from the method in the absence of a more definite uncertainty estimate. However, a laboratory generating test results using the test method should derive the value to quote for its test results based on its own methodology and experience, which are not necessarily equivalent to the laboratories that participated in the original interlaboratory study.

5.9.1 Variability of samples, when the quantity is a property of a heterogeneous material, is part of uncertainty for the measurement. This component of variability is not usually included in reproducibility because interlaboratory evaluation of test methods uses test materials that are as uniform as possible.

5.10 Certified reference values for standard materials that cannot be made to a known value are often obtained by interlaboratory testing. The average of test results for participating laboratories becomes the “consensus” accepted reference value. The standard uncertainty of the consensus value is  $s / \sqrt{n}$ , where  $s$  is the standard deviation of results reported by the  $n$  laboratories.

5.11 Practice E 29 describes evaluation of conformance of a material with a specification by comparing the test result with specification limits. Some proposals (5) use uncertainty values

<sup>5</sup> A probability distribution representing the likelihood of property values given data is known in statistical theory as the Bayes posterior distribution of the property value.

in an alternative procedure for evaluating conformance with specifications. Compliance of the material with specifications is demonstrated if the entire expanded uncertainty interval is contained within the specification range. Noncompliance is demonstrated when the entire uncertainty interval is outside the specification range. Where the uncertainty interval straddles a specification limit, the test result is indecisive.

5.11.1 If this method for evaluating conformance is used, the test method shall include an explicit procedure for calculating the uncertainty interval.

## 6. Uncertainty for Estimates Based on Probability Samples

6.1 Classical statistical methods for estimation apply directly to the estimation of uncertainty provided the underlying distribution assumptions are met. Probability sampling (see Practice E 141) is a procedure for ensuring that statistical methods are applicable and provide valid estimates of their uncertainty. Measurement tasks to which probability samples apply include determining the proportion of items in a specified set having a qualitative observable characteristic, the average of a quantitative characteristic which may be non-uniform over a prescribed area, or the aggregate of a property for a lot of material which may be non-uniform.

### 6.2 Simple Statistical Applications:

6.2.1 The value to be reported is an average of  $n$  measurements each of which has standard deviation  $\sigma$ , bias is presumed to be absent, and the measurements are mutually independent. Then uncertainty of the average value is  $\sigma / \sqrt{n}$ .

6.2.2 The value to be reported is an average of measurements that are not independent. This situation occurs when some uncertainty components are shared among all measurements. If standard deviations are respectively  $\sigma_1$  and  $\sigma_2$  for the shared components and unshared (independent for different measurements) components, the uncertainty of the average of  $n$  such correlated measurements is  $\sqrt{\sigma_1^2 + \sigma_2^2 / n}$ . This example shows that, when measurements in a group share some sources of error, the average has a residual uncertainty that cannot be removed by increasing the number of the measurements in the group.

6.2.3 The measurement is made by comparison against a reference material similar in kind to the sample. The data for measurements of this type are responses  $x$  and  $y$  for sample and reference material respectively, and an accepted reference value  $Y$  of the reference having standard uncertainty  $u_Y$ . The measurement result is then calculated as  $X = (x/y) \times Y$ . Validity of this result depends critically on response being directly proportional to the quantity, which must be demonstrated for the method. Uncertainty of the result depends on variability of responses and on the uncertainty of the reference value. Then the standard uncertainty of the determination based on responses  $x_1, x_2, \dots, x_n$  of sample and  $y_1, y_2, \dots, y_m$  of reference material is:

$$u_x = X \sqrt{\frac{s_x^2}{n\bar{x}^2} + \frac{s_y^2}{m\bar{y}^2} + \frac{u_Y^2}{Y^2}}$$

## 7. Uncertainty Estimation by the Control Sample Approach

7.1 A measure of intermediate precision within the laboratory can be used as the basis for routine reporting of uncertainty for measurements when a control sample is run together with routine samples. Such control materials are used to monitor performance of the method using control charts. Practice E 2554 describes generation of uncertainty estimates from control sample test results. The intermediate precision is the standard deviation of the control sample measurements, taken over an extended period of time. It measures variability due to a subset of factors contributing to uncertainty, and is within the capability of the laboratory to generate. It does not include uncertainty components due to constant bias sources within the laboratory or due to heterogeneity of samples. The following conditions should also be met for intermediate precision to be applied.

7.1.1 The control sample should be similar to routine samples and have approximately the same value for the characteristic. Alternatively, if it is known that relative standard deviation is constant for the test method, or a similar relation between the test result and its variability holds, the relative standard deviation for control samples may be applied to test results for routine samples.

7.1.2 The control samples should be run on an ongoing basis. It is not useful to cite a standard deviation of controls run in the past and subsequently unused.

7.1.3 The controls should be run under the same range of environmental conditions, on the same testing equipment, and by the same personnel, as routine samples.

7.1.4 The control chart should indicate that the testing process is statistically stable.

7.2 When samples come from inhomogeneous lots of material and the measurement result is intended to apply to the entire lot, an additional uncertainty component due to sampling variability can be added to the estimate of measurement variability. Uncertainty due to variability of samples should be combined with intermediate precision estimated from control sample test results. The combined uncertainty is  $\sqrt{s_1^2 + s_2^2}$ , where  $s_1$  is an estimate of the component of variability due to sampling and  $s_2$  the standard deviation of controls.

7.2.1 To estimate the sampling component  $s_1$ , an experiment should be carried out making multiple determinations on each of several independent samples (say,  $n \geq 2$  tests on each of  $k$  samples). Estimate the sampling component of variance using analysis of variance.

## 8. Propagation of Uncertainty

8.1 The propagation of uncertainty method and its associated tabular form, the uncertainty budget, is a tool for determining uncertainty of test results by combining uncertainty attributable to reference materials, precision of observation, environmental factors, and other sources of error in the test result. The method may also be used as a guide to specifying the required precision of measurements, in order to achieve a desired precision of the test result. Evaluation of a test method, to identify principal sources of error, requires a high level of expertise in the technology of the measurement. Historically,

experience has been that uncertainties are underestimated by this procedure, as errors of components tend to be underestimated and unknown error components left out of the tabulation (6).

8.2 To apply the method, an explicit equation is written that relates the test result to underlying quantities, either measurements for which uncertainties are in hand, or factors affecting the measurement for which variation has been assessed. Uncertainty or variability of the independent variables is combined using the law of propagation of errors to form an estimate for uncertainty of the result. In particular, represent the test result as a function of variables  $z_i$

$$x = f(z_1, z_2, \dots)$$

8.2.1 If  $b_i$  is the systematic error (bias) and  $u_i$  the standard uncertainty (or standard deviation) for variable  $z_i$ , then the bias and standard uncertainty components of the calculated result are given by the following approximation, which is derived from the linear expansion of the function.

$$bias_x = \sum_i \left( \frac{\partial f}{\partial z_i} \right) b_i$$

$$u_x^2 = \sum_i \left( \frac{\partial f}{\partial z_i} \right)^2 u_i^2$$

8.2.2 This form assumes that the uncertainty components  $z_i$  are uncorrelated. In the case of correlated uncertainty components, the combined standard uncertainty also depends on correlations  $\rho_{ij}$  between components.

$$u_x^2 = \sum_i \left( \frac{\partial f}{\partial z_i} \right)^2 u_i^2 + 2 \sum_{i < j} \left( \frac{\partial f}{\partial z_i} \right) \left( \frac{\partial f}{\partial z_j} \right) \rho_{ij} u_i u_j$$

8.3 Calculations using propagation of errors are most conveniently arranged in a tabular form. The uncertainty budget is a table listing the factors affecting result uncertainty, their biases ( $b_i$ ) and standard uncertainty values ( $u_i$ ), the sensitivity coefficient  $c_i = (\partial f / \partial z_i)$ , and the bias and standard uncertainty components for the measurement,  $c_i b_i$  and  $c_i u_i$ . An essential part of the uncertainty budget is documentation of the basis for component bias and standard uncertainty values.

8.3.1 Standard uncertainty for the test result is calculated from uncertainty components  $c_i u_i$  as  $u = \sqrt{\sum (c_i u_i)^2}$ .

8.3.2 The fraction of uncertainty for the test result contributed by the  $i$ -th component is frequently useful to identify the major causes of uncertainty. The fraction contributed by component  $i$  is  $(c_i u_i)^2 / \sum (c_j u_j)^2$ .

8.3.3 If a desired standard uncertainty for the result is given, required uncertainty or precision of factors can be back calculated using the uncertainty budget. A method designer uses this approach to specify accuracy of parts of a test method, the degree of control over environmental factors required to achieve a target bound for combined uncertainty from the measurement.

8.4 An example applying the method is described in Appendix X1. Several additional examples are given in References (3, 7).

## 9. Keywords

9.1 measurement error; precision; propagation of errors; random error; systematic error; test results; Type A; Type B; uncertainty

## APPENDIX

### (Nonmandatory Information)

#### X1. EXAMPLE OF UNCERTAINTY CALCULATION BY PROPAGATION OF UNCERTAINTY

X1.1 This example illustrates use of an uncertainty budget for evaluating uncertainty of a test result, in which part of the information required to evaluate uncertainty becomes available only during the testing process.

X1.2 In the determination of moisture content of a dry powder, the procedure is to extract water from a weighed portion of sample using a dry solvent, and to measure the amount of water in the extract by Coulometric Karl Fischer analysis. The equation for moisture content is:

$$\% \text{moisture} = 100 \frac{C_{\text{sample}} - C_{\text{solvent}}}{w} \times k$$

where  $C_{\text{sample}}$  and  $C_{\text{solvent}}$  represent current used in titration of the extract and an equal volume of extraction solvent (a blank), and  $w$  is the sample weight. Currents are converted by the instrument into units of weight water using a calibration factor  $k$ . The test result reported will be the average of determinations made on a number  $n$  of samples drawn from the lot.

X1.3 For simplicity, the uncertainty budget for a single

determination is considered first. Uncertainty of a test determination depends on the following components:

X1.3.1 Variability of the actual volume of extraction solvent, variability in the amount of moisture that might be absorbed by the sample from the air, and variability of operations for the instrument. These factors affect currents,  $C_{\text{sample}}$  and  $C_{\text{solvent}}$  and are not distinguishable from one another.

X1.3.2 Variability of sample weighing.

X1.3.3 Uncertainty for the calibration factor  $k$ .

TABLE X1.1 Single Determination Uncertainty Budget

| Source                    | Value | Sensitivity<br>$c_i$ | Uncertainty<br>$u_i$ | Contribution<br>$c_i u_i$ | Fraction |
|---------------------------|-------|----------------------|----------------------|---------------------------|----------|
| $C_{\text{sample}}$ (mg)  | 0.826 | 1.9268               | 0.041                | 0.07958                   | 85.1%    |
| $C_{\text{solvent}}$ (mg) | 0.329 | -1.9268              | 0.016                | 0.03170                   | 13.5%    |
| $w$ (mg)                  | 51.9  | -0.0185              | 0.200                | 0.00369                   | 0.2%     |
| $k$                       | 1     | 0.9476               | 0.010                | 0.00958                   | 1.2%     |
| determination             |       |                      |                      | combined uncertainty      |          |
| 0.96%                     |       |                      |                      | 0.086%                    |          |

X1.4 Sensitivity coefficients are found by differentiating the formula for the test result with respect to each component value:

$$\begin{aligned}\frac{\partial(\%M)}{\partial C_{\text{sample}}} &= \frac{100}{w} k \\ \frac{\partial(\%M)}{\partial C_{\text{solvent}}} &= -\frac{100}{w} k \\ \frac{\partial(\%M)}{\partial w} &= -100 \frac{C_{\text{sample}} - C_{\text{solvent}}}{w^2} k \\ \frac{\partial(\%M)}{\partial k} &= 100 \frac{C_{\text{sample}} - C_{\text{solvent}}}{w}\end{aligned}$$

X1.5 Table X1.1 shows an example uncertainty budget for this single determination. For purposes of the example, it is assumed that instrument measurements  $C_{\text{sample}}$  and  $C_{\text{solvent}}$  both have relative standard deviation 5 %, that the weight  $w$  is approximately 50 mg and measured with standard deviation 0.0002 g or 0.2 mg. Uncertainty of the calibration factor  $k$  is assumed to be 1 %. These figures must be derived from data other than a sample analysis. Relative standard deviations of  $C_{\text{sample}}$  and  $C_{\text{solvent}}$  are estimated from variability of water containing standards used as check samples.

X1.6 To derive the uncertainty of the test result, which is an average of  $n$  determinations using a common blank ( $C_{\text{solvent}}$ ) and  $k$ , an additional uncertainty component, the variation between samples within the lot, must be considered. The contribution of variation between samples to the test result uncertainty is  $\sigma/\sqrt{n}$ , where  $\sigma$  is the standard deviation due to variation of samples, and depends on how uniform is the particular lot being tested. The standard deviation  $s$  for the  $n$

samples is used to estimate this component of uncertainty; it also contains components due to instrument variations in  $C_{\text{sample}}$  and to weighing. However, uncertainty due to the common blank and to  $k$  are not accounted for in the standard deviation among samples.

X1.7 To evaluate test result uncertainty in Table X1.2, we suppose that  $n = 3$  sample determinations have been performed with results: 0.95, 1.16, and 0.70 percent moisture. The average and standard deviation of samples are 0.94 and 0.23. The contribution of sampling and ( $C_{\text{sample}}$ ,  $w$ ) factors is estimated as the standard error of the mean,  $0.23/\sqrt{3} = 0.133$ . Average values of  $C_{\text{sample}}$  and weights are used to calculate influence coefficients and contributions to uncertainty for the blank and for  $k$ .

X1.8 A result of this analysis is that variability of samples is the largest single source of uncertainty in the determination for this particular lot. However, uncertainty of the determination for a sample also depends critically on the moisture level of the solvent.

TABLE X1.2 Uncertainty Calculation for Test Result

| Source                    | Value       | Sensitivity<br>$c_i$ | Uncertainty<br>$u_i$ | Contribution<br>$c_i u_i$ | Fraction |
|---------------------------|-------------|----------------------|----------------------|---------------------------|----------|
| Sampling                  | 1           |                      |                      | 0.133                     | 94.2%    |
| $C_{\text{sample}}$ (avg) | 0.819       |                      |                      |                           |          |
| $w$ (avg)                 | 52.0        |                      |                      |                           |          |
| $C_{\text{solvent}}$ (mg) | 0.329       | -1.9231              | .016                 | 0.03163                   | 5.3%     |
| $k$                       | 1           | 0.9423               | .01                  | 0.00942                   | 0.5%     |
|                           | test result |                      |                      | combined uncertainty      |          |
|                           | 0.94%       |                      |                      | 0.137%                    |          |

## REFERENCES

- (1) Mohr, P. J. and Taylor, B. N., CODATA Recommended Values of the Fundamental Physical Constants: 2002, *Reviews of Modern Physics*, Vol 77, pp. 1-107, 2005.
- (2) International Organization for Standardization, *International Vocabulary of Basic and General Terms in Metrology*, Geneva, Switzerland, 1993. (VIM)
- (3) International Organization for Standardization, *ISO Guide 98 — Guide to the Expression of Uncertainty in Measurement*, Geneva, Switzerland, 1995. (GUM).
- (4) Taylor, B. N. and Kuyatt, C. E., NIST Technical Note 1297, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, 1994.
- (5) International Laboratory Accreditation Cooperation, Guidelines on Assessment and Reporting of Compliance with Specification, G8, 1996.
- (6) Youden, W. J., "Enduring Values," *Technometrics*, Vol 14, pp. 1-11, 1972.
- (7) EURACHEM, *Quantifying Uncertainty in Analytical Measurement*, 2nd Ed., 2000.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).