



Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method in a Single Laboratory Using a Control Sample Program¹

This standard is issued under the fixed designation E2554; 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 describes techniques for a laboratory to estimate the uncertainty of a test result using data from test results on a control sample.

1.2 Uncertainty as defined by this practice applies to the capabilities of a single laboratory. Any estimate of uncertainty determined through the use of this practice applies only to the individual laboratory for which the data are presented.

1.3 The laboratory uses a well defined and established test method in determining a series of test results. The uncertainty estimated using this practice only applies when the same test method is followed. The uncertainty only applies for the material types represented by the control samples, and multiple control samples may be needed, especially if the method has different precision for different sample types or response levels.

1.4 The uncertainty estimate determined by this practice represents the intermediate precision of test results. This estimate seeks to quantify the total variation expected within a single laboratory using a single established test method while incorporating as many known sources of variation as possible.

1.5 This practice does not establish error estimates (error budget) attributed to individual factors that could influence uncertainty.

1.6 This practice describes the use of control charts to evaluate the data obtained and presents a special type of control chart to monitor the estimate of uncertainty.

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

1.8 *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 appro-*

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

2. Referenced Documents

2.1 ASTM Standards:²

D5184 Test Methods for Determination of Aluminum and Silicon in Fuel Oils by Ashing, Fusion, Inductively Coupled Plasma Atomic Emission Spectrometry, and Atomic Absorption Spectrometry

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

E2282 Guide for Defining the Test Result of a Test Method

2.2 ASTM Publication:

Manual on Presentation of Data and Control Chart Analysis, 7th Edition²

2.3 ISO Standard:

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories³

3. Terminology

3.1 Definitions:

3.1.1 The terminology of Terminology **E456** applies to this practice except as modified herein.

3.1.2 *control sample, n*—sample taken from a stable, homogeneous material for the purposes of monitoring the performance of a test method in a laboratory.

3.1.2.1 *Discussion*—The control sample material is representative of the product typically tested in the laboratory by the given test method. A control sample is run periodically using the complete test method protocol to develop a test result. Such test results may be statistically evaluated to monitor test method performance over time. It is not necessary to have an

¹ This practice 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.

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² 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 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

accepted reference value assigned to the control sample material. When the current material is nearly consumed, a replacement material should be run in parallel with the current material to ensure continuity in the control sample program.

3.1.3 *check sample, n*—see *control sample*.

3.1.4 *uncertainty control chart, n*—control chart that includes control limits based on the variation attributed to the uncertainty of the test method.

3.1.5 *intermediate precision, n*—the closeness of agreement between test results obtained under specified intermediate precision conditions. **E177**

3.1.5.1 *Discussion*—The specific measure and the specific conditions must be specified for each intermediate measure of precision; thus, “standard deviation of test results among operators in a laboratory,” or “day-to-day standard deviation within a laboratory for the same operator.”

3.1.5.2 *Discussion*—Because the training of operators, the agreement of different pieces of equipment in the same laboratory and the variation of environmental conditions with longer time intervals all depend on the degree of within-laboratory control, the intermediate measures of precision are likely to vary appreciably from laboratory to laboratory. Thus, intermediate precisions may be more characteristic of individual laboratories than of the test method.

3.1.6 *test result, n*—the value of a characteristic obtained by carrying out a specified test method. **E2282**

3.1.7 *repeatability, n*—precision under repeatability conditions. **E177**

4. Summary of Practice

4.1 A standard material or control sample is measured repeatedly over time. The presumption of this practice is that the variation experienced on this material will be indicative of the laboratory total expected variation. Incorporation of specific known or potential sources of variation in the testing program is encouraged.

4.2 A control chart is prepared and the results are evaluated to identify short-term variation and longer-term variation. These data can then be used to determine an estimate of uncertainty standard deviation.

4.3 Laboratories already having control chart procedures in place may use existing data.

4.4 Ongoing monitoring of the test method is conducted using an uncertainty control chart.

5. Significance and Use

5.1 Laboratories accredited under ISO 17025 are required to present uncertainty estimates for their test results. This practice provides procedures that use test results to develop uncertainty estimates for an individual laboratory.

5.2 Generally, these test results will be from a single sample of stable and homogeneous material known as a control or check sample.

5.3 The true value of the characteristic(s) of the control sample being measured will ordinarily be unknown. However, this methodology may also be used if the control sample is a

reference material, in which case the test method bias may also be estimated and incorporated into the uncertainty estimate. Many test methods do not have true reference materials available to provide traceable chains of uncertainty estimation.

5.4 This practice also allows for ongoing monitoring of the laboratory uncertainty. As estimates of the level of uncertainty change, possibly as contributions to uncertainty are identified and minimized, revision to the laboratory uncertainty will be possible.

6. General Considerations

6.1 *Materials to be Used:*

6.1.1 This methodology requires a quantity of stable and homogeneous material which will serve as the source of control samples (sometimes called check samples). The material shall be similar in composition to the samples of material routinely analyzed by this test method in this laboratory. By stable it is assumed that the test results obtained from this material should be consistent over the time interval that this material will be used. By homogeneous it is assumed that samples taken from the material source will not have a significant variation in the characteristic measured by the test method.

6.1.2 For destructive testing of control sample materials, provision shall be made for depletion and replacement of the control sample material.

6.1.2.1 In some cases, the test method may be nondestructive and the same material may be reused indefinitely.

6.1.2.2 In other cases, the material may be used up, deteriorate, or otherwise gradually change.

6.1.3 The test method should describe the best practices for preparing and storing the control material and taking the control samples.

6.2 *Test Conditions:*

6.2.1 An uncertainty estimation program should be designed to include all known sources of variation, such as operators (analysts), equipment, reagents, and so forth, and these should be deliberately incorporated into the design of the program. In general, these sources of variation will be defined (including acceptable tolerances) by the test method.

6.2.2 In cases in which control over such variations is not possible or undefined, at least 30 to 50 sampling periods shall be evaluated to permit environmental and other factors to be incorporated in the overall estimate.

7. Overall Procedure—Control Charting Methods

7.1 General concepts of control charts are described elsewhere, such as in Manual 7A.⁴

7.2 The general procedure involves two major phases: Preliminary and Monitoring.

7.2.1 *Preliminary Phase:*

7.2.1.1 This phase begins with an initial collection of test results.

⁴ *Manual on Presentation of Data and Control Chart Analysis: 7th Edition*, ASTM International, West Conshohocken, PA, 2001.

7.2.1.2 Preliminary control charts are then prepared and examined. These charts are evaluated to determine if the process is in a state of statistical control. The usual principles of control charting utilize short-term variability to estimate the limits within which samples of test results should vary. For control sample programs this short-term variability is equivalent to repeatability precision. It is expected, however, that additional contributions to variation will be present over time and therefore additional variation, equivalent to intermediate precision, will be encountered.

7.2.1.3 An estimate of uncertainty standard deviation is developed.

7.2.1.4 An uncertainty control chart is then prepared to monitor future sample results.

7.2.2 *Monitoring Phase:*

7.2.2.1 The proposed uncertainty control chart is used to provide evidence that the estimate of uncertainty is not exceeding the estimated value.

7.2.2.2 The estimate of uncertainty should be periodically re-evaluated.

7.2.2.3 Where appropriate, it is recommended that a standard control chart also be maintained to determine whether the variation over time has been reduced to the level of short-term variation (repeatability).

7.3 Two types of control charting methods are recommended to develop estimates of uncertainty. These include:

7.3.1 Mean (\bar{X}) and range or standard deviation charts are used when multiple test results are conducted in each time period.

7.3.2 Individual charts (IndX) are used when single test results are obtained in each time period.

7.4 *Variation Estimates:*

7.4.1 Either a range chart or a standard deviation chart may be used to estimate the short-term variability when multiple assays are conducted under repeatability conditions per time period. An estimate from the control chart data can be compared to other estimates of repeatability (within laboratory, short-term variation) if available.

7.4.2 Sample averages are examined and may provide estimates of variation caused by other factors. Such factors may include environmental effects, operator factors, reagents, or instruments.

7.5 *Systematic Procedures:*

7.5.1 Specifically designed experiments can be used to ensure all known sources of variation, such as operators (analysts), equipment, reagents, or instruments are incorporated in the general study.

7.5.2 The data generated from this program is available for additional uses, such as control charting to evaluate trends, stratification by analysis, or stratification by equipment to identify training or maintenance needs or both.

8. Specific Procedures

8.1 *Multiple Test Results Generated per Time Period:*

8.1.1 A specified number of independent test results are taken during each time period. Generally this number is 5 or

less. It is preferred that at least 25 sets of test results be obtained before developing the charts.

8.1.2 Either a range chart or a standard deviation chart is prepared. This is examined for special cause variation. If the variability appears random then an estimate of repeatability is computed. This may be done by pooling the sums of squares, using the average standard deviation, or using the average range.

NOTE 1—If the ranges or standard deviations are zero in most of the samples, then this estimate of repeatability standard deviation is suspect and probably unusable. This is usually the result of insufficient resolution of the measurement system in use or severe rounding. An estimate based on the minimum interval size should be substituted for the zeros. As a rule of thumb, consider replacing the zeros when more than about $\frac{1}{3}$ are zeros.

8.1.3 A means chart is used to examine variation among time periods. Limits on this chart permit comparison of variation between time periods using repeatability as the estimate of error.

8.1.3.1 If the control chart shows a state of statistical control then the uncertainty will be assumed approximately equivalent to the repeatability standard deviation.

8.1.3.2 In most cases it will be expected that the variability between means will show an “out of control” condition indicating that there are “special” causes of variation in addition to repeatability. The between means variation and within means repeatability estimates are then used to compute an estimate of uncertainty standard deviation.

8.1.4 Using the estimate of uncertainty standard deviation an Uncertainty Control Chart is prepared for future monitoring of the uncertainty. This chart may include control limits for means as a possible lower set of control limits along with the uncertainty control limits based on the estimate of uncertainty.

8.2 *Individual Tests:*

8.2.1 Single tests are generated at each time period. Variation among these results is evaluated.

8.2.2 In some cases, it is possible to incorporate external estimates of repeatability obtained from prior or concurrent studies.

9. Multiple Readings per Time Period

9.1 *Example 1—Absorbance of Radiochromic Dosimeters:*

9.1.1 Over a period of several days, different sets of three dosimeters were irradiated to the same nominal dose. The irradiation was conducted under standard conditions at a single irradiator facility. Possible sources of random errors could include intrinsic variation in dosimeter response and day-to-day variations in the physical environment, for example, temperature, positioning of dosimeters within the irradiator, and shielding. The data was presented in Guide 51707.

9.2 **Table 1** consists of three dosimeters irradiated and measured on a single day. Nine time periods are shown. The averages and standard deviations are computed for each time period.

9.3 Prepare a standard deviation control chart.

NOTE 2—Ranges could have been computed instead of standard deviations and a range control chart would be prepared.