Designation: E2867 – 14

Standard Practice for Estimating Uncertainty of Test Results Derived from Spectrophotometry[1]

This standard is issued under the fixed designation E2867; 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 a protocol to be utilized by measurement laboratories for estimating and reporting the uncertainty of a measurement result when the result is derived from a measurand that has been obtained by spectrophotometry.

1.2 This practice is specifically limited to the reporting of uncertainty of color measurement results that are reported as color-differences in ΔE format, even though the measurement itself may be reported in other units such as percent reflectance or transmittance.

1.3 The procedures defined here are not intended to be applicable to national standardizing laboratories or transfer laboratories.

1.4 *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:*[2]

D2244 Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E284 Terminology of Appearance

2.2 *ISO Standards:*[3]

ISO 9001 Quality Management Systems—Requirements

ISO/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories

2.3 *Other Standard:*[4]

QS 9000 Quality Systems Requirements Chrysler Corporation, Ford Motor Company, General Motors Corporation

3. Terminology

3.1 *Definitions:* For definitions of terms used in this standard refer to Terminology E284.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *uncertainty, n*—a parameter associated with a measurement result or test result that reasonably characterizes the dispersion of results attributable to the particular quantity being measured of the particular characteristic being tested.

3.2.2 *instrument uncertainty conditions, n—of a measurement*, conditions wherein the measurements are made repetitively and carefully over a short timescale, without replacement of the specimen being measured in the specimen port of the instrument.

NOTE 1—Instrument uncertainty conditions always include potential specimen drift due to causes such as theromchromism, photochromism, or bleaching of the specimen. While these may be thought of as characteristics of the specimen, their effects will be picked up here under instrument uncertainty conditions.

3.2.3 *operator uncertainty conditions, n—of a measurement*, conditions wherein the measurements are made repetitively and carefully over a short timescale, with replacement of the specimen being measured by the operator completely withdrawing the specimen from the specimen port and replacing the specimen back in the specimen port prior to the ensuing measurement so that the specimen aperture samples the same location on the specimen, and the specimen has the same orientation as previous, to the best of the operator's ability to accomplish.

3.2.4 *uniformity uncertainty conditions, n—of a measurement*, conditions wherein the measurements are made repetitively and carefully over a short timescale, with replacement of the specimen being measured to an entirely new

[1] This practice is under the jurisdiction of ASTM Committee E12 on Color and Appearance and is the direct responsibility of Subcommittee E12.04 on Color and Appearance Analysis.

Current edition approved May 1, 2014. Published May 2014. Originally approved in 2013. Last previous edition approved in 2013 as E2867 – 13. DOI: 10.1520/E2867-14.

[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.

[4] Available from Automotive Industry Action Group (AIAG), 26200 Lahser Rd., Suite 200, Southfield, MI 48033, http://www.aiag.org.

location on the face of the specimen with the intent of sampling the entire surface of the specimen, or as much of the surface as is practical, by the end of the repetitive sampling run.

3.2.5 *instrument uncertainty, n*—the results of an uncertainty analysis of a measurement system made under instrument uncertainty conditions.

3.2.6 *operator uncertainty, n*—the results of an uncertainty analysis of a measurement system made under operator uncertainty conditions.

3.2.7 *uniformity uncertainty, n*—the results of an uncertainty analysis of a measurement system made under uniformity uncertainty conditions.

3.2.8 *expanded uncertainty, n*—uncertainty reported as a multiple of the standard uncertainty.

3.2.9 *measurement system, n*—the entirety of variable factors that could affect the precision, accuracy, or uncertainty of a measurement result. These include the instrument, the operator, the environmental conditions, the quality of the transfer standard, the specimen aperture size, as well as other factors.

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

3.2.11 *95 % confidence interval, n*—the 95 percentile value of an ascending-ordered distribution of differences between multiple measurement results of a derived parameter characterized by a color measurement system.

3.2.11.1 *Discussion*—This value is the cumulative distribution between zero and the stated value of the measurand that contains 95 % of all the measurement results made by this procedure.

4. Summary of Practice

4.1 This practice establishes a protocol for measurement laboratories to assess the uncertainty of their measurement system from test specimens or from control samples of materials similar in both first-surface characteristics and color to those being measured and reported.

4.2 Where control samples are used, the process will be to establish control samples representative of the type of materials to be measured. Control samples will be processed to assess the various uncertainty components of measurement results, the results retained in a control chart, and the rolling average of the uncertainty components of the control samples used as a surrogate for assessing the uncertainty of a similar specimen.

4.3 Some of the components of uncertainty for color measurement result are instrument uncertainty, operator uncertainty, and uniformity (of the specimen) uncertainty.

5. Significance and Use

5.1 Many competent measurement laboratories comply with accepted quality system requirements such as ISO 9001, QS 9000, or ISO 17025. When using standard test methods, the measurement results should agree with those from other similar laboratories within the combined uncertainty limits of the laboratories' measurement systems. It is for this reason that quality system requirements demand that a statement of the uncertainty of the test results accompany every test result.

5.2 Preparation of uncertainty estimates is a requirement for laboratory certification under ISO 17025. This practice describes the procedures by which such uncertainty estimates may be calculated.

6. Concepts in Reporting Uncertainty of Test Results

6.1 A commonly cited definition (**1, 2**)[5] paraphrased to form a single citation 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 uncertainty as an attribute of an individual test result, not as a property defining statistical variation of test results.

6.2 The methodology for classification of uncertainty types has been classified as Type A and Type B as discussed in references (**2**) and (**3**). Type A estimates of uncertainty include estimates based upon knowledge of the statistical character of the measurement results, or estimates based upon statistical analysis of replicate measurement results. The latter may include results from control sample monitoring programs, or proficiency testing. Type B estimates of uncertainty include estimates from calibration certificates and manufacturer's specifications. Type A are evaluated by statistical methods and Type B by non-statistical methods.

6.3 The goal of reporting uncertainty is to account for all potential causes contributing to uncertainty in the measurement result. Uncertainty for a single measurement result is then

$$(s_1^2 + s_2^2 + \ ... + \ s_n^2)^{1/2}$$

where s_1 is the estimate of the uncertainty of the first factor contributing to variance, s_2 the second, and so on, through all n components of variance.

6.4 Uncertainty in this practice shall be reported as the 95% confidence interval of the largest component of all the components of uncertainty assessed.

6.5 The minimum components contributing to variance shall be the instrument uncertainty, the operator uncertainty, the uniformity uncertainty, and the uncertainty of the traceability scheme.

7. Procedure

7.1 Measure the test specimen a minimum of 20 times, and preferably as many as 30 times, under instrument uncertainty conditions. Make all measurements in compliance with the manufacturer's recommendations including prior standardization of the instrument using a white tile, a black tile, or light trap, and a grey tile, if required.

7.2 There will be n^* $(n - 1) / 2$ possible color-differences between the n measurement results taken two-at-a-time in all possible combinations.

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

7.3 Calculate the absolute value of the color-differences between each of these combinations and retain the results in a list. Calculate these color-differences in accordance with a color-difference equation chosen from Practice D2244.

7.4 Sort the list in ascending order. The member of the sorted list whose index is *Int* [0.95 * n * (n-1) / 2] contains the value of the 95 % confidence interval of the instrument uncertainty s_1. The symbol *Int* means the integer value of the expression in brackets.

7.5 Measure the test specimen a minimum of 20 times, and preferably as many as 30 times, under operator uncertainty conditions. Follow the operations of 7.1 – 7.3 using this data set to calculate the 95 % confidence interval of operator uncertainty s_2.

7.6 Measure the test specimen a minimum of 20 times, and preferably as many as 30 times, under uniformity uncertainty conditions. Follow the operations of 7.1 – 7.3 using this data set to calculate the 95 % confidence interval of uniformity uncertainty s_3.

7.7 Sort the uncertainties obtained from Sections 7.1 – 7.6 s_1, s_2, s_3 in ascending order with the smallest of the three in s_1 and the next larger in s_2, and so forth. Let

$$s'_1 = s_1 \quad (1)$$

$$s'_2 = (s_2^2 - s_1^2)^{1/2} \quad (2)$$

$$s'_3 = (s_3^2 - s_2^2)^{1/2} \quad (3)$$

The value of s_2 used in Eq 3 is the original experimentally assessed value, not that value which results from the calculation of Eq 2 which is s'_2. This isolates the uncertainties, each of which has been until now included in each of the measured uncertainties, into a separate uncertainty contribution attributable to each subsequent type of uncertainty considered. However, consult the cautionary remarks in Appendix X1 to this Practice at X2.1 – X2.4.

7.8 Calculate the combined uncertainty

$$U = (s_1'^2 + s_2'^2 + s_3'^2)^{1/2} \quad (4)$$

where the elements s'_1, s'_2, and s'_3 are the uncertainties from 7.4.

The combined uncertainty may be expressed in the form '*mean value* ± *U* to a 95 % confidence interval.' The value of *U* shall be expressed to no greater number of significant digits than are expressed in the mean, and shall be rounded in accordance with Practice E29. All calculations should be carried out with the full precision of the machine employed at all times and rounded only when the final values, seen in the worked example in the table in X1.2.1, are calculated.

7.9 In some infrequently occurring instances, the length of time required to make a measurement may make it impossible to make as many as 30 measurements. Under those circumstances it is permissible to reduce the number of measurements made to a smaller number providing that it can be demonstrated that the results are sufficient to the intended purpose.

8. The Substitution of Control Samples

8.1 Under circumstances where it would prove a hardship, or is infeasible to utilize the test specimen for this determination, it is permissible to substitute control samples of a like material for the material being considered in the test result.

8.2 Values quoted using control samples shall be rolling averages of the last four determinations of the uncertainty by the operations of 7.1 – 7.8 using the same control sample in each of the four determinations. Each of the four determinations must be demonstrated to be 'in-control' by maintenance of control charts of the uncertainty determinations.

8.3 The control samples should be chosen to be as alike the material being reported as possible and special attention should be paid to the matter of uniformity uncertainty when control samples are chosen. Uniformity uncertainty is the most likely component to be discrepant between a control sample and a test specimen.

8.4 The first surface of control samples involved in measurements of reflection properties should be as nearly identical as possible to the first surface of the test sample.

8.5 Reports of uncertainty where a control sample is substituted for a test sample shall state the facts of the substitution and identify the control sample utilized.

9. Reporting Statement

9.1 *Form of the Reporting Statement*—In reporting uncertainty, a statement such as the following may be useful: "The uncertainty of the value reported was found to be X.XX (here report the uncertainty value.) This value was determined using XX (here report the number of measurements made in the assessment of uncertainty; ninety, for instance) measurements categorizing the instrument, operator and uniformity uncertainty. This value is the combined expanded standard uncertainty in color difference units according to the (here name the color-difference equation used) equation calculated from D65 and the CIE 10° Observer, which defines the uncertainty of the measured value to a confidence level of 95 percent."

9.2 The following measurement parameters shall be defined and specified in the uncertainty expression: (*1*) the identity of the measured sample being reported, (*2*) the color difference equation used in the uncertainty assessment, (*3*) the values of *l* and *c*, only for DE_{CMC}, (*4*) the Illuminant–Observer combination used, (*5*) the number of measurements in each of the uncertainty component assessments, (*6*) the level expressed as a percentage to which the coverage factor raises the confidence.

10. Applications of Uncertainty

10.1 The uncertainty results can be used by those who want to assess the reliability of a measured value. Without an uncertainty indication, measurement results cannot be compared, either among themselves or to a reference value given in a specification or standard. The uncertainty value may be further used to decide whether there is a difference between results from different laboratories.

10.2 The uncertainty value is also necessary when comparing results to allowable values, for example, tolerance limits or allowable (legal) specifications. To make a correct pass-fail decision one needs an uncertainty value which, together with the measured value, is in the range of the total tolerable limit.

10.3 In the provided example found in Appendix X1, the data reports the range of the total combined uncertainty as 0.41 total color difference units. Therefore, the range of uncertainty is greater than, or equal to, zero and less than, or equal to, 0.41 total color difference units, ΔE^*_{ab}.

11. Keywords

11.1 control samples; spectrophotometry; test results; uncertainty; uncertainty budget

APPENDIXES

(Nonmandatory Information)

X1. A WORKED EXAMPLE OF THE UNCERTAINTY CALCULATIONS

X1.1 Underlying Data

X1.1.1 The underlying data may be found on the ASTM Committee E12 web site and downloaded by any user. The data is contained in three files. The first file named INSTRUMENT UNCERTAINTY.csv contains 20 measurements of a white optically-brightened paper made under instrument uncertainty conditions. The second, named OPERATOR UNCERTAINTY.csv, contains 20 measurements of the same sample made under operator uncertainty conditions. The third file, named UNIFORMITY UNCERTAINTY.csv, contains 20 measurements made under uniformity uncertainty conditions.

X1.2 Intermediate Values

X1.2.1 Because the resulting intermediate values are complex, many, and are subject to machine representation variation, a limited set of results to the first four digits of precision will be given for this example both before and after their separation from each other by Eq 1-3 in 7.7 of this document. The three calculated values from this example, for D65-1964 Observer CIELAB ΔE^*_{ab} are

	Consolidated	Unconsolidated
Instrument Uncertainty	0.0513	0.0513
Operator Uncertainty	0.0529	0.0129
Uniformity Uncertainty	0.4091	0.4057

X1.3 Results

X1.3.1 The following table provides the uncertainty calculated from these example data.

Instrument Uncertainty	0.0513
Operator Uncertainty	0.0129
Uniformity Uncertainty	0.4057
Total Measurement Uncertainty	0.41 ΔE^*_{ab} D65–1964 10° Observer

X2. ON THE CALCULATIONS

X2.1 The four equations in 7.7 and 7.8 imply the consolidation of the components of uncertainty by addition under quadrature. The addition of 95 % confidence intervals obtained from standard deviations of normal distributions definitely add under quadrature to form 95 % confidence intervals of the composite. However, the addition of 95 % confidence intervals of non-normal distributions under quadrature may not obtain an exact 95 % confidence interval of the composite.

X2.2 At the same time, one desires to deconsolidate the components of uncertainty to determine which are largest, and therefore the most likely subjects for reduction of uncertainty. Accordingly, ASTM has adopted the 95 % confidence interval of the largest consolidated component as the definition of uncertainty, and allowed the deconsolidation under quadrature. The user must exercise caution that the assumptions employed in doing so are, at least, mostly met. These assumptions include that the distributions of differences in L*, a*, and b* are normal, independent, and have identical standard deviations, or nearly so.

X2.3 That these assumptions are made dictates another limitation of the method. That is, the method adopted is limited to quantification of color-differences expressed in ΔE format. The determination of uncertainty of data sets of other derivative parameters, such as whiteness index or others, will have to wait the determination that the distribution of those differences are closely enough aligned with the chi distribution that the deconsolidation under quadrature is effective.

X2.4 Accordingly, while the total 95 % confidence interval of the largest consolidated component is entirely valid, users should view with caution the values obtained by deconsolidation under quadrature. They may serve as a guideline as to where further improvements in reduction of uncertainty may be made, but they should likely not be reported or promulgated beyond the laboratory deriving them.

REFERENCES

(1) International Organization for Standardization, International Vocabulary of Basic and General Terms in Metrology, Geneva, Switzerland, 1993 (VIM).

(2) JCGM 1000:2008 Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement, Paris, France, JCGM 2008 (GUM).

(3) Taylor, B. N., and Kuyatt, C. E., NIST Technical Note 1297, Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results, 1994.