Designation: D 6631 – 01

Standard Guide for Committee D01 for Conducting an Interlaboratory Study, and Determining the Precision of a Test Method[1]

This standard is issued under the fixed designation D 6631; 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 guide covers a simplified statistical procedure for planning and conducting interlaboratory evaluations of test methods.

2. Referenced Documents

2.1 *ASTM Standards:*

E 177 Practice for Use of the Terms Precision and Bias in Test Methods[2]

E 456 Terminology for Relating to Quality and Statistics[2]

E 691 Practice for Conducting an Interlaboratory to Determine the Precision of a Test Method[2]

E 1345 Practice for Reducing the Variability of Color Measurements by Use of Multiple Measurements[3]

3. Significance and Use

3.1 The purpose of an interlaboratory evaluation, as defined in this guide, is to determine the variability of results obtained in different laboratories on equivalent equipment using a prescribed test method.

3.2 The definitions of statistical terms used in this guide are contained in Terminology E 456.

4. Problem Formulation

4.1 The objective of the evaluation should be to clearly define the expected precision of the test method. Within the current limits of both the software, and the statistical protocols currently available, this effectively restricts the use of this guide to test methods which yield results that are continuous. This generally means a measured quantity, such as pH or brightness. Results that are discrete (such as counts or pass-fail), or ordered (ranked), present three special problems in the creation of a meaningful precision statement:

4.1.1 The amount of information contained in discrete and ordered data is much less than in continuous data, necessitating the collection of much more data.

4.1.2 The sensitivity (the ability to discriminate between similar samples) is much less in discrete and ordered data than continuous data.

4.1.3 Since the precision statement relies on the normal distribution, and the distributions of discrete and ordered data are usually decidedly non-normal, the normal precision statement is invalid.

4.2 Given these concerns with discrete and ordered data, a simple statement of the results obtained in these types of studies might be the most useful information for a prospective user of a test method.

5. Preliminary

5.1 Flow chart the test method.

5.2 Survey known sources of information related to the test method to establish how results are affected by variations in operating conditions, atmospheric conditions, differences between operators, etc. Select what appears to be the optimum procedure.

5.3 Provide instructions for the test method and, without comment, observe a laboratory technician perform a test according to these instructions. Revise any parts of the draft causing difficulty.

5.4 If desirable, make a comparative study with other test methods for measuring the property by using specimens with a wide range of values of the property under test (and possibly with wide ranges in other properties).

6. Preparation for Interlaboratory Study

6.1 Prepare a clear statement of the type of information required from the interlaboratory evaluation.

6.2 Based on the study made in one laboratory (Section 5), prepare a proposed master plan for the interlaboratory evaluation. Discuss the plan, in an open meeting, if possible, with other participants in the study.

6.3 Select the materials to be used in the interlaboratory evaluations so as to:

6.3.1 Cover the applicable range of the property or component to be measured, and

6.3.2 Represent as many classes of materials as feasible, to which the test method will be applied.

7. Pilot Evaluation

7.1 If the test method is new or the procedure for an old test

[1] This guide is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.20 on Quality Assurance and Statistics.

Current edition approved Feb. 10, 2001. Published April 2001.

[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

method is greatly altered, a pilot study by one laboratory involving a few materials (two or three) may reveal seriously misleading directions in the written procedure. A Box and Whisker Plot (as outlined in Practice E 1345), or a Violin Plot[4] may help to clarify this evaluation.

8. Participating Laboratory Qualification

8.1 It is important that the managers of proposed participants in an interlaboratory study be aware of the capability of those participants to run the method under study. This is not the responsibility of the originator of the study, or ASTM. A Box and Whisker Plot will also be helpful in this qualification protocol.

9. First Interlaboratory Evaluation

9.1 For the first interlaboratory evaluation use at least three materials to cover the expected range in property values and include all of the laboratories that will participate in the main interlaboratory evaluation. This evaluation will clarify the procedure, eliminate laboratories that do not comply with the procedure because of nonstandard conditions or equipment and, together with the main study, give some idea of-the time constancy of laboratory results.

10. Main Interlaboratory Evaluation

10.1 *Variables*—The major variables that can be included in an interlaboratory evaluation of a test method are: materials, laboratories, apparatus, and replicates, described as follows: (In the event that the minimums for these variables cannot be met, it is advisable that the recommendation in4.2 be considered, or that the study be abandoned).

10.1.1 *Materials*—A minimum of six materials differing in the property or component to be measured and covering the useful scales of the process are needed to evaluate the sensitivity of a test method.

10.1.2 *Laboratories*—In the evaluation of a test method, an absolute minimum of three laboratories or locales must be used, but at least five are strongly recommended.

10.1.3 *Apparatus*—If different types of apparatus (or different procedures) are to be included in the study, an absolute minimum of three laboratories is required for each type. At least five are needed to obtain a reasonable estimate of reproducibility. (It must be emphasized that the word "different" in this section, could apply to multiple manufacturers, or models of equipment.) However, different equipment, using different techniques or protocols for measurement, for example., rotational and efflux cup viscometers, must NOT be used in the same study.

10.1.4 *Replicates*—Each evaluation must at least be run in duplicate.

10.2 *Intralaboratory Precision* is usually ascertained by having repeats made over the shortest possible time, preferably the same day. If considered desirable, a completely unbiased estimate of intralaboratory error can be obtained by use of "blind" repetition. In this procedure two samples of at least one, and preferably more, of the test materials are distributed as separate materials. If all the materials are duplicated, the total amount of work can be kept the same by not repeating the test at another time. However, elimination of the time variable might alert cooperators to the use of blind repetition which, in any event, is not readily applicable when all the test materials differ markedly in properties.

10.3 For analytical test methods, the precision of which is usually very good, two repeats each in duplicate are often sufficient. However, owing to the much more variable nature of measurements of the physical or application test properties, the repeats should be increased, unless it has been decided to use a test method mainly for ranking a series of materials. For most test methods of this type, three repeats should be used, but even when the precision is quite poor the suggested maximum is four. If the intralaboratory variability is high, the number of replications should also be increased.

10.4 In order to obtain sufficient interlaboratory degrees of freedom so that the estimated interlaboratory precision will not appear poorer than it really is, the number of laboratories must increase as the number of test materials decreases.

10.5 *Instructions to Participant*—Use the master plan agreed upon by the evaluation group after careful discussion. This plan should include instructions on the following:

10.5.1 Care of round-robin specimens and what to do in case of loss of specimens or results (missing results can be ignored only if a sufficient number of participating laboratories and materials are included),

10.5.2 Adjustment and calibration of the test apparatus,

10.5.3 Order of testing the specimens,

10.5.4 Recording results on the test form,

10.5.5 Detailed test procedure, to include:

10.5.5.1 Scope,

10.5.5.2 Test method,

10.5.5.3 Other instructions relevant to use of test method or operation, as for example, replication and standardization,

10.5.5.4 Dates for performance of tests,

10.5.5.5 Instructions about personnel,

10.5.5.6 Instructions on compilation, calculation, and reporting,

10.5.5.7 Standard report form for results and conditions,

10.5.5.8 Instructions on return of reports and, if applicable, materials (including address), and

10.5.5.9 Closing date.

10.6 *Allocation of Specimens*—If appropriate, specimens may be selected from several locales, but should be coordinated from one place. Prepare from each material enough specimens to provide the required test material for the participating laboratories and a sufficient number of additional specimens for replacement of lost or spoiled specimens. Label each specimen by means of a code symbol and identify the specimens on a separate key sheet for future reference. Completely randomize the specimens of a particular material before dividing them into groups to be distributed among the laboratories. Where necessary, the same specimens may be sent in turn to each participating laboratory.

10.7 *Results Form*—Supply each laboratory with results forms to ensure that all results and pertinent information are

[4] Hintze, J. L., and Nelson, R. D., "Violin Plots: A Box Plot-Density Trace Synergism", *The American Statistician*, May 1998, pp. 181-184.

reported in a uniform manner. In addition to space for measurement results, the form should provide space for such information as: relative humidity, temperature, instrument type, deviations from the specified procedure, unusual observations, and constructive comments, as required.

10.8 *Final Report*—After all participating laboratories report their results, and the results are analyzed statistically, according to Practice E 691, or a method that yields equivalent results, a research report shall be generated giving such information as: the raw data, the observations from 10.7, and the statistical analysis results. In addition, a precision statement (and if appropriate, a bias statement), shall be created, according to Practice E 177.

11. Keywords

11.1 bias; interlaboratory study; precision