



Standard Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries¹

This standard is issued under the fixed designation D4483; 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.

INTRODUCTION

The primary precision standard for ASTM test method standards is Practice E691; a generic standard that presents the fundamental statistical approach and calculation algorithms for evaluating repeatability and reproducibility precision. However, certain parts of Practice E691 are not compatible with precision as evaluated in the rubber manufacturing and carbon black industries over the past four decades. Thus a separate standard is required for precision in these two industries. This practice is being issued as a major revision of Practice D4483, which has been used for precision evaluation by Committee D11 since 1985. The basic Practice D4483 precision calculation algorithms, the same as in Practice E691, are unchanged. This new revised Practice D4483, organized to accommodate the requirements of the rubber and carbon black manufacturing industries, has three new features that provide for a more formal and structured analysis of interlaboratory test program (ITP) data.

First it addresses the overriding issues with precision evaluation over the past several decades—the frequent discovery that reproducibility for many test methods is quite poor. Experience has shown that frequently poor reproducibility is caused by only a few laboratories that differ from the remainder that give good agreement. A new procedure designated as *robust analysis* provides an improved method for detecting outliers that cause poor precision, especially poor between laboratory agreement. Second, after outlier detection the new standard provides two options; (1) outlier deletion or (2) outlier replacement. When outliers are deleted the revised standard provides a way to retain the non-outlier laboratory data. This allows for a broader database for precision calculation. The current ASTM Committee E11 computer program for calculating precision does not allow for outlier deletion in this way. Third, when exercising outlier Option 2, the standard gives a procedure for calculating special replacement values for deleted outliers in ITPs that have only a few participating laboratories. The replacement values are obtained in a way that preserves the observed data distribution of the non-outlier data. This is important since many ITPs are in the *limited number of participating laboratories* category.

1. Scope

1.1 This practice covers guidelines for evaluating precision and serves as the governing practice for interlaboratory test programs (ITP) used to evaluate precision for test methods as used in the rubber manufacturing and the carbon black industries. This practice uses the basic one way analysis of variance calculation algorithms of Practice E691. Although bias is not evaluated in this practice, it is an essential concept in understanding precision evaluation.

1.2 This practice applies to test methods that have test results expressed in terms of a quantitative continuous variable. Although exceptions may occur, it is in general limited to test methods that are fully developed and in routine use in a number of laboratories.

1.3 Two precision evaluation methods are given that are described as *robust statistical* procedures that attempt to eliminate or substantially decrease the influence of outliers. The first is a *General Precision* procedure intended for all test methods in the rubber manufacturing industry, and the second is a specific variation of the general precision procedure designated as *Special Precision*, that applies to carbon black testing. Both of these procedures use the same uniform level experimental design and the Mandel h and k statistics to review the precision database for potential outliers. However, they use

¹ This practice is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.16 on Application of Statistical Methods.

Current edition approved May 1, 2014. Published May 2014. Originally approved in 1985. Last previous edition approved in 2014 as D4483 – 14. DOI: 10.1520/D4483-14A.

slight modifications in the procedure for rejecting incompatible data values as outliers. The *Special Precision* procedure is specific as to the number of replicates per database cell or material-laboratory combination.

1.4 This practice is divided into the following sections:

	Section
Scope	1
Referenced Documents	2
Terminology	3
Significance and Use	4
Precision Evaluation—General Precision and Special Precision	5
Steps in Organizing an Interlaboratory Test Program (ITP)	6
Overview of the General Precision Analysis Procedure	7
General Precision: Analysis Step 1	8
Preliminary Graphical Data Review	8.1
Calculation of Precision for Original Database	8.2
Detection of Outliers at 5 % Significance Level Using <i>h</i> and <i>k</i> Statistics	8.3
Generation of <i>Revision 1</i> Database Using Outlier Treatment Option 1 or 2	8.4
General Precision: Analysis Step 2	9
Calculation of Precision for <i>Revision 1</i> Database	9.1
Detection of Outliers at 2 % Significance Level Using <i>h</i> and <i>k</i> Statistics	9.1
Generation of <i>Revision 2</i> Database Using Outlier Treatment Option 1 or 2	9.1.2
General Precision: Analysis Step 3	10
Calculation of Precision Using <i>Revision 2</i> Database	10.1
Special Precision Analysis—Carbon Black Testing	11
Format for Precision Table and Clause in Test Method Standards	12
Preparation of Report for Precision Analysis	13
Definitions for Selected Terms Concerned with Precision and Testing	Annex A1
Statistical Model for Interlaboratory Testing Programs	Annex A2
Calculating the <i>h</i> and <i>k</i> Consistency Statistics for Outliers	Annex A3
Spreadsheet Calculation Formulas, Table Layout, and Calculation Sequence	Annex A4
Procedure for Calculating Replacement Values of Deleted Outliers	Annex A5
Example of General Precision Evaluation—Mooney Viscosity Testing	Annex A6

1.5 Six annexes are presented; these serve as supplements to the main body of this practice. **Annex A1** and **Annex A2** are given mainly as background information that is important for a full understanding of precision evaluation. **Annex A3 – Annex A5** contain detailed instructions and procedures needed to perform the operations as called for in various parts of the practice. The use of these annexes in this capacity avoids long sections of involved instruction in the main body of this practice. This allows for a better presentation and understanding of the central concepts involved in the evaluation of precision. **Annex A6** is also important; it gives a complete example of precision evaluation that illustrates all of the procedures and options likely to be encountered in any precision evaluation, from the simple to the most complex.

1.6 *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

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D6600 Practice for Evaluating Test Sensitivity for Rubber Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 ISO Standard:³

ISO 289 Determination of Viscosity of Natural and Synthetic Rubbers by the Shearing Disk Viscometer

3. Terminology

3.1 A number of specialized terms or definitions are defined in a systematic sequential order, from simple terms to complex terms. This approach allows the simple terms to be used in the definition of the more complex terms; it generates unambiguous definitions. Thus the definitions do not appear in the usual alphabetical sequence.

3.1.1 This terminology section contains explanatory notes for many of the definitions as well as discussion on the connection between some of the terms and the various ways the terms are used in testing and precision evaluation. For special emphasis, a few terms are defined in the main text of this practice where certain precision concepts are discussed.

3.1.2 **Annex A1** is included as part of this practice with two objectives: (1) **Annex A1** presents new more comprehensive definitions drafted with substantial tutorial content, and (2) **Annex A1** presents some ancillary definitions that may promote a better understanding of precision.

3.2 Testing Terms:

3.2.1 *balanced uniform level design, n*—the plan for an interlaboratory test program for precision, where all laboratories test all the materials selected for the program and each laboratory conducts the same number of repeated tests, on each material.

3.2.2 *element, n*—the entity that is tested or observed, to evaluate a property or characteristic; it may be a single object among a group of objects (test pieces, and so forth) or an increment or portion of a mass (or volume) of a material.

3.2.2.1 *Discussion*—The generic term *element* has a number of synonyms: test piece, test specimen, portion, aliquot part, subsample, and laboratory sample.

3.2.3 *element class (or class of elements), n*—the category or descriptive name for a group of elements that have a common origin or have nominally identical properties.

3.2.3.1 *Discussion*—The term *nominally identical* implies that the elements come from a source that is as homogeneous as possible with regard to the property being measured.

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 International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

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

3.2.4.1 *Discussion*—The test method should specify that one or a number of individual measurements, determinations, or observations be made and their average or another appropriate function (median or other) be reported as the test result.

3.2.5 *testing domain, n*—the location and operational conditions under which a test is conducted; it includes a description of the element preparation (test sample or test piece), the instrument(s) used (calibration, adjustments, and settings), the selected test technicians, and the surrounding environment.

3.2.5.1 *global testing domain, n*—a domain that encompasses two or more locations or laboratories, domestic or international, typically used for producer-user testing, product acceptance, and interlaboratory test programs.

3.2.5.2 *local testing domain, n*—a domain comprised of one location or laboratory as typically used for quality control and internal development or evaluation programs.

3.3 *Material and Sampling Terms:*

3.3.1 *independent tests, n*—a set of measurements (or observations) for a defined testing domain, where, in relation to the measurement process, there is no influence of any selected measurement on any other measurement in the set.

3.3.1.1 *Discussion*—The word *independent* is used throughout this practice as an adjective to indicate the concept of independence, for samples, test pieces, and so forth, as well as tests.

3.3.2 *lot, n*—a specified mass or volume of material or number of objects; usually generated by an identifiable process, frequently with a recognized composition or property range.

3.3.2.1 *Discussion*—A lot may be generated by a common production (or other natural) process in a restricted time period and usually consists of a finite size or number. A lot may be a fractional part of a population (Interpretation 2 of population, see [Annex A1](#)). A recognized property range implies that some rough approximation is available.

3.3.3 *material, n*—a specific entity or element class to be tested; it usually exists in bulk form (solid, powder, or liquid).

3.3.3.1 *Discussion*—Material is used as a generic term to describe the *class of elements* that is tested, that is, a material may be a rubber, a rubber compound, a carbon black, a rubber chemical, and so forth. A material may or may not be homogeneous. In product testing the term material may be used to describe the *class of elements* or type of rubber products such as O-rings, hose assemblies, motor mounts, and so forth. See also [5.1.4.1](#).

3.3.4 *sample (data), n*—the number of test or observation values ($n = 1, 2, 3$, and so forth), obtained from (one or more) physical samples, by the application of a specific test (observation) method.

3.3.5 *sample (physical), n*—the number of elements or the specified mass of a material, selected according to a particular procedure, used to evaluate material, lot, or population characteristics.

3.3.5.1 *Discussion*—The term *sample* should not be used as

a synonym for *material*, see [3.3.3](#), or *target material*, see [5.1.4.1](#). Ideally several *materials* are tested in any ITP with each material being different (chemically, structurally, property wise). From each material, some number of *samples* (all nominally identical) may be taken for testing. See [3.3.4](#).

3.3.6 *test sample, n*—that part of a (physical) sample of any type taken for chemical or other analytical testing, usually with a prescribed blending or other protocol.

3.3.6.1 *Discussion*—A test sample is usually a mass or volume that is some small fractional part of a bulk material.

3.3.7 *test specimen, n*—an object (appropriately shaped and prepared) taken from a sample for physical or mechanical testing.

3.3.7.1 *Discussion*—Other terms for test specimen are: test portion, test item, and test piece (used in ISO standards).

3.4 *Statistical Terms Relating to Precision:*

3.4.1 *estimated (true or reference) mean, n*—the mean obtained on the basis of n independent replicate measurements; the greater n the better the approximation to the true or reference mean, provided there is no systematic deviation or bias.

3.4.1.1 *Discussion*—The words *mean* and *estimated mean* are frequent synonyms for *estimated (true or reference) mean*. The value for n in typical routine testing programs is of the order 1 to 10. When bias exists, the estimated (true or reference) mean so obtained estimates $[\mu + \sum B_i]$, where μ = true or reference mean and $\sum B_i$ = algebraic sum of all bias deviation terms. Therefore, if bias exists and is unknown in magnitude, the true value or μ cannot be approximated despite increased replication. See random and bias deviations in [A1.2.5](#) and [A1.2.6](#). See also [Annex A2](#).

3.4.2 *outlier, n*—a member of a set of values which is inconsistent with the other members of that set.

3.4.3 *reference value, n*—a value (usually a mean) generated by a recognized and accepted procedure that is used as a true value.

3.4.3.1 *Discussion*—Reference values are used when it is impossible or exceedingly difficult to obtain a true value. Such values are most often assigned on the basis of comprehensive testing programs sanctioned by a local or global task group, a standardization organization, or a committee devoted to domestic or international metrology.

3.4.4 *replicate, n*—one of a selected number of independent fractional parts or independent number of elements, taken from a sample; each fractional part or element is tested.

3.4.4.1 *Discussion*—The word *replicate* refers to a physical object (element). It can also be used in reference to a data set, where it refers to one of a number of independent data values.

3.4.5 *true value, n*—the measured or observed value for an element, that would be obtained for a testing domain in the absence of errors, deviations, or variations of any sort, that is, where there is no variation *system-of-causes*.

3.4.5.1 *Discussion*—The true value is also defined as the mean that would be obtained by testing all members of any population (see population in [Annex A1](#)). Typical *systems-of-causes* are the unavoidable fluctuations in the material, the

humidity, operator technique, fidelity of calibration, and so forth, in a controlled testing domain.

3.5 Definitions: In some of the following definitions, the term *figure of merit* is used. A high figure of merit is an indication of high quality or a high level of excellence or goodness for the measurement or test domain, or both. The term *figure of merit* applies to a number of test method characteristics: precision, sensitivity, bias, useful range, ruggedness and ease of operation, and rapid or automated operation.

3.5.1 precision, n —a *figure of merit* concept, it is proportional to the inverse of the dispersion of independent replicate (test or observed) values, as estimated by the standard deviation, for a specified class of elements and a defined testing domain.

3.5.1.1 Discussion—The merit of a test method depends on the precision, high merit equals high precision. However, it has become customary practice to express precision in terms of the dispersion of replicate values, that is, by the standard deviation. However, this is actually a measure of imprecision; therefore, the use of the inverse of the standard deviation in this definition. Precision may be influenced by both random and bias deviations depending on the defined testing domain. There are other *figure of merit* testing concepts. An additional one is test sensitivity; the ratio of the magnitude of the measurement response for a selected property difference to the precision or accuracy of the measurement, or both. See Practice D6600 for more details on test sensitivity.

3.5.2 relative repeatability, (r), n —repeatability expressed in terms of an interval (a multiple of the standard deviation) that is a percentage of the mean level of the measured property; this interval should (on basis of a 95 % probability) encompass duplicate independent test results (on percentage basis) obtained for a defined local testing domain.

3.5.3 relative reproducibility, (R), n —reproducibility expressed in terms of an interval (a multiple of the standard deviation) that is a percentage of the mean level of the measured property; this interval should (on basis of a 95 % probability) encompass duplicate independent test results (on percentage basis) each obtained in different laboratories for a defined global testing domain.

3.5.4 repeatability, r , n —the precision for a defined *local testing domain*, obtained by way of n independent replicate tests (on nominally identical elements) expressed in terms of an interval or range that is a multiple of the standard deviation; this interval should (on basis of a 95 % probability) encompass duplicate independent test results obtained under the defined local testing domain.

3.5.4.1 Discussion—The *local testing domain* is defined as one laboratory, usually one instrument, one test technician with a *specified* replicate test time period. The words *nominally identical* imply elements drawn from a homogenous source with all reasonable effort taken to eliminate production variation within the source. Repeatability may be dependent on the magnitude or level of the measured property and is usually reported for particular property levels or materials or element

classes (that determine the level). The repeatability time period may be minutes, hours, or days depending on the goals and scope of the testing.

3.5.4.2 Discussion—Although repeatability as defined in 3.5.4 applies to a local testing domain, it can be obtained in two different ways and can be used in two different contexts. It can pertain to a common community value, obtained as an average (or pooled) value from all laboratories in an ITP among N different laboratories. This is a *global* repeatability, that applies to a *typical laboratory*, that stands as a representative of all laboratories that are part of a global testing domain. It can also pertain to the long-term or established value for a *particular laboratory* as derived from ongoing testing in that laboratory, not related to any ITP. The second use can be referred to as a local repeatability, that is, repeatability obtained in and for one laboratory.

3.5.5 reproducibility, R , n —the precision for a defined *global testing domain*, obtained by way of independent tests conducted in N laboratories (with n replicates each) on nominally identical elements, expressed in terms of an interval or range that is a multiple of the standard deviation; this interval should (on basis of a 95 % probability) encompass duplicate test results, each obtained in different laboratories for a defined global testing domain.

3.5.5.1 Discussion—Each laboratory in the global domain conducts n repeatability tests on a material (target material), and reproducibility is evaluated based on the mean values for the N laboratories for that material or element class. Reproducibility may also depend on the level of the measured property or on the materials tested and it is also usually reported for particular levels or materials. Reproducibility usually does not have the dual interpretation or use as previously discussed for repeatability, since it is a *group characteristic* that only applies across a number of laboratories in a global testing domain.

3.5.5.2 Discussion—It is appropriate to also express precision on a relative basis, as a percent of a certain mean value. This is analogous to a coefficient of variation. A relative expression may be important when the precision varies with the level of the property being measured. Frequently the relative precision is reasonably constant when so expressed. To avoid any confusion with measured properties that are expressed in percentages, for example, % copper, % elongation, and so forth, relative precision is expressed using parentheses that enclose the symbols for repeatability and reproducibility.

3.6 Additional terms concerning certain types of precision will be defined in 5.1. Better understanding can be gained by giving these definitions, which relate to the nature of the material to be tested, in that section.

4. Significance and Use

4.1 Tests are conducted using standard test methods to generate test data that are used to make decisions for commercial, technical, and scientific purposes. It follows that the precision of a particular test method is an important quality characteristic or figure of merit for a test method and a decision process.

4.2 An evaluation of the precision of a test method is normally conducted with (1) some selected group of materials as typically used with that method and (2) with a group of volunteer laboratories that have experience with the test method. The evaluation represents an *event in time* for the test method for these materials and laboratories. Another ITP precision evaluation with somewhat different materials or even with the same materials with the same laboratories at a different time, may generate precision results that differ from the initial ITP.

4.3 Experience as indicated in Refs (1-4)⁴ and elsewhere has shown that the poor reproducibility among the laboratories of a typical ITP is almost always due to interlaboratory bias. Certain laboratories are always low or high compared to a reference as well as other laboratories in all tests. This usual outcome for many ITPs is addressed in this practice by the use of the three-step robust analysis procedures as described in Section 7.

4.4 Caution is urged in applying precision results of a particular test method to product testing for consumer-producer product acceptance. Product acceptance procedures should be developed on the basis of precision data obtained in special programs that are specific to the commercial products and to the laboratories of the interested parties for this type of testing.

5. Precision Evaluation: General Precision and Special Precision

5.1 *General Precision*—Two precision categories are described: General Precision and Special Precision. General Precision is discussed first and Special Precision is described in Section 11. General Precision evaluation follows established procedures used in the rubber manufacturing industry over the past four decades. The evaluation is usually conducted using a balanced uniform level design ITP with three or more materials sent to each of the participating laboratories with tests conducted to generate an independent *test result*, on each of two (or more) test days. The ITP database is reviewed for outliers by the Mandel *h* and *k* consistency statistics by the procedures in Annex A3.

5.1.1 *Options for Outliers*—If no outliers are found, the original database is used to develop a table of precision results. If outliers are identified, there are two options for outlier treatment; Option 1, outlier deletion, is the first choice. Option 2, outlier replacement, is chosen for an ITP with a minimum (approximately six) number of laboratories. Issues such as the number of replicate values on each test day or the number of technicians or operators used to obtain a test result, or both, which are characteristic of the particular test, are considered on a case-by-case basis by the ITP organizing committee. Outlier treatment is discussed in more detail in Annex A3 and Annex A5.

5.1.2 *Types of Test Methods*—The General Precision approach has been successfully used for the broad range of test methods characteristic of the rubber manufacturing industry;

from simple physical or chemical *bench type* tests, conducted in a few minutes (hardness and pH tests) to a complex multistep test method, such as an aging test. Such a test requires preliminary property measurement, a substantial aging period (days) followed by aged property measurement to obtain a final calculated test result or performance index. For such complex tests, any realistic precision evaluation must of necessity include all of the procedural steps in arriving at the test result, the basic datum used in precision analysis, and evaluation. The procedures required for general precision are described in Sections 8 – 10.

5.1.3 *Types of General Precision*—In addition to the General Precision aging tests as previously cited, other tests also require a more complex total sequence of operations to generate a final test result. One important test of this type is a *performance-in-rubber* test; the evaluation of various rubbers, reinforcement fillers, or other compounding materials in standardized formulations. The typical stress-strain evaluation of a selected lot of a specified rubber will require (1) an appropriate sample of the rubber, (2) a standardized formulation and mixing operation to prepare a compound using standard compounding materials, (3) processing of this compound to prepare cured or vulcanized molded sheets at a selected time and temperature, (4) cutting and gaging of dumbbell (or other) test pieces, and (5) the testing of the lot to obtain the final test results for tensile stress (modulus), elongation, and tensile strength properties.

5.1.4 To permit realistic precision evaluation for the performance-in-rubber testing it is necessary that all the steps in the operation be replicated, from the raw materials to the final test result. Each of these steps has a potential component of variance and the sum of all variance components establishes the overall test variance and standard deviation. To address this, two types of precision are defined. The two types are characterized by the relationship between the material (or element class) tested and the material directly evaluated for precision. To explain this, it is necessary to introduce and define a new term, *target material*.

5.1.4.1 *target material, n*—the material (or class of elements) that is the primary focus of attention for a precision evaluation program; however, it may not be tested in its usual or ordinary physical state.

5.1.5 Using the term *target material*, two types of precision may be defined:

5.1.5.1 *Type 1 Precision*—A precision evaluated directly for or on, a target material; fully prepared test pieces or test portions of the target material drawn from a homogeneous source are tested, with no processing or other operations required prior to testing.

5.1.5.1.1 *Discussion*—An example is a lot comprised of died-out, gaged dumbbells for stress-strain testing.

5.1.5.2 *Type 2 Precision*—A precision evaluated indirectly for a target material; the target material is usually combined with a number of homogeneous ancillary materials to form a composite material, and on samples of this, testing is conducted and the property response of the target material is evaluated.

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

5.1.6 The properties of the composite material are directly related to the quality or properties of the target material. An example: To evaluate the quality of a grade of SBR, a sample of the rubber, plus curatives, filler, antioxidants, and so forth, are mixed, cured, test pieces prepared, and the resulting compound tested for specified quality properties. It is possible that a Type 1 precision program might be conducted on test pieces or portions that require some minimum processing or other simple operations prior to actual testing. This is, in a strict sense, an intermediate level of precision. However, to avoid unnecessary complications, this will be designated as a Type 1 precision.

5.2 *Special Precision*—The carbon black industry has adopted a slightly revised precision evaluation procedure designated as *Special Precision*. The number of replicates in each cell of a uniform level design ITP is specified as four, two by each of two test technicians. The outliers are reviewed by a special procedure that depends on the number of laboratories in the ITP and the precision, absolute or relative, is expressed by a specified procedure. The procedures for this Special Precision are listed in Section 11.

6. Steps in Organizing an Interlaboratory Test Program

6.1 The steps required to organize an ITP, with a discussion for each procedural step, are as follows:

6.1.1 *Organization Committee*—An organization committee or task group and a program coordinator should be selected. One member of the committee or group should be a statistician familiar with the testing technology of the test method as well as the content of this practice. Most ITPs are organized on the basis of a balanced uniform level design for the precision program.

6.1.2 *Category and Type of Precision*—For all programs except for carbon black testing, a General Precision ITP is organized. For carbon black testing a Special Precision ITP is organized. The type of precision to be evaluated shall be selected, see 5.1.5. Type 1 precision is the most frequently evaluated. For some test methods such as rubber or polymer or other performance-in-rubber evaluations using standard formulations, a Type 2 precision is required.

6.1.3 *Test Operator or Technician Selection*—For simple General Precision testing requiring only one operator or technician, all replicate tests should be conducted by the same technician unless the effect of different technicians is part of any program. For more complex tests where several operators or technicians are required to perform a sequence of different steps to arrive at a test result, the same *operator team* should conduct testing for all replicates again unless the effect of different operator teams is part of the program.

6.1.3.1 For Special Precision testing follow the procedure of using two technicians on each of two test days. See Section 11.

6.1.4 *Test Result and Number of Replicates*—Each test method has a final value for the property under evaluation, defined as a test result. A test result may be a mean or median value of a number of individual determinations as specified by the test method. For the purposes of this practice, a replicate is defined as a test result. The number of replicate test results, n , within each laboratory on any material should be specified. In

most ITPs this is two. For some tests, three or four replicates, as in Special Precision, may be selected. All analysis is conducted on test results.

6.1.5 *Time Period for Repeatability*—The time period between replicate tests within any laboratory should be selected. This time period is usually one of days, in the range from 1 to 7 days. For special tests (long aging periods) replicate tests may require a longer time span. For other special testing operations, shorter time periods (minutes, hours) may be selected. The primary consideration is how the test method is typically used in the industry. The selected time period shall be reported in the precision section of the test method.

6.1.6 *Number of Target Materials*—The number of target materials or classes of objects (or manufactured products) to be tested should be selected. Ideally, this should be three or four with substantially different property levels. The target materials should represent typical industry materials as normally used and subjected to test. See 5.1.

6.1.7 *Preparation of Homogeneous Target Materials*—A homogeneous lot of each of the target materials should be prepared, with sufficient reserve quantity, so that retests can be made if needed. If the material allows for a blending operation to ensure homogeneity, this should be done. If blending is not possible, special procedures should be conducted to obtain the most homogeneous material (or collection of elements) that is possible by way of closely monitored laboratory or other preparation operations. Documentation should be provided to ascertain the homogeneity. If any ancillary materials are required as for Type 2 precision, these lots should be either standard reference materials or special documented homogeneous lots.

6.1.8 *Number of Laboratories*—For a reliable estimate of precision, at least six laboratories skilled in the test method are required for the final database (after outlier treatment) in the ITP. For the more important industry test methods, 12 to 18 laboratories should participate. If six or more laboratories are not in the final database, an analysis can be conducted with fewer laboratories but the estimates of precision, especially reproducibility, are seriously compromised and only represent very rough estimates.

6.1.9 *Packaging and Delivery of Materials*—All the materials required for any ITP should be appropriately packaged to prevent any change with time or storage in the properties to be measured. Appropriate storage conditions in each participating laboratory prior to test need to be specified. The shipment of all materials should be coordinated with the test schedule (discussed as follows) so that all materials are available for the scheduled test dates.

6.1.10 *Testing Instructions*—Although all ITPs are usually conducted for a standard test method that includes the complete set of instructions for the test, some supplemental instructions are required. One important supplemental instruction is the schedule for the testing. All tests should be performed on specified days, and all participating laboratories should conduct the test as specified by the test method. The schedule should allow for adequate material delivery time. Any special modifications of the test method should be clearly described as well as special instructions as to operator responsibilities.

two, or more) versus replicate testing. If an ITP is to be conducted for a test method at some intermediate development level, it is essential to give all participating laboratories instructions for conducting the test method as well as all the required ITP instructions.

6.1.11 *ITP Test Data Report*—A test report data form should be prepared by the ITP coordinator and a copy sent to each participating laboratory along with the test materials and instructions. This form should contain locations to report the following: the name of the laboratory; the test dates as actually used; and for each target material tested, the test value (test result) for each replicate test (day), reported if possible to one more significant figure than is normally used (that is, do not truncate). The test report form should also ask for a description of the test equipment or machines used (model number, condition), comments about any unintended deviations from the standard test procedure and disclosure of any mishaps or other pertinent information. The completed test report should be returned to the ITP coordinator.

7. Overview of General Precision Analysis Procedure

7.1 *Analysis Operation Sequence*—This section gives a quick overview of the procedures required for the analysis of the ITP database and provides the user with a better appreciation of the complete analysis process. Some background on outliers is also presented in this section for a better appreciation of this topic. The General Precision procedure may require as many as three analysis operations or overall steps. The actual number will be determined by the uniformity of the data in the database. If there are no outliers, only Analysis Step 1 is used. If outliers are present, Analysis Steps 2 and 3 may be required depending on the extent of outliers in the database. Annex A4 contains instructions for all three analysis operations and also gives the details on how to layout the required tables and their interlinking that enables the automatic recalculation of the final precision parameters, r and R , when outliers are deleted or replacement values are substituted into the basic data Table 1 format. Fig. 1 is a decision tree or flow chart diagram that outlines the steps in the complete analysis process.

7.1.1 *Preliminary Data Review*—A quick numerical review of any database is important to gain a first impression of the

results of any ITP. This preliminary data review is conducted after cell averages and cell standard deviations (or cell ranges) have been calculated. Part of this review is the generation of special plots of cell averages and cell standard deviations or cell ranges versus laboratory number. These plots, as described in 8.1.3, will clearly show potential outlier values.

7.1.2 *Analysis Step 1*—The original database is analyzed to generate values for repeatability and reproducibility for each material (or target material) and the h and k statistics calculated. See Annex A3. Annex A4 gives the instructions for generating six tables that yield values for the h and k statistics and the precision results for each material. The calculated h and k values are compared to the 5 % significance level critical h and k values to determine if there are any significant outlier values. If there are none, the analysis is complete and the values found for repeatability and reproducibility are used to generate a table of precision results for the test method. If there are any significant outliers, Analysis Step 2 is required.

7.1.3 *Analysis Step 2*—If there are any outliers at the 5 % significance level, the outlying values are either (1) deleted using Option 1 or (2) replaced using Option 2. See Annex A3, Annex A5, and 5.1.1. On the basis of either option, the resulting revised database, designated as Revision 1 or $R1$, is analyzed to generate new values for repeatability and reproducibility, designated as $R1$ precision values. This analysis produces a new set of calculated h and k values that are compared to 2 % significance level critical h and k values to determine if there any significant outlier values at this level. If there are none, the analysis is complete and the values found for repeatability and reproducibility are used to generate a table of $R1$ precision results for the test method. If there are any significant outliers, Analysis Step 3 is required.

7.1.4 *Analysis Step 3*—If any of the $R1$ calculated h and k values exceed the 2 % significance level critical h and k values, the outlying values are either (1) deleted using Option 1 or (2) replaced using Option 2. On the basis of either option, the resulting $R2$ database is analyzed to generate new values for repeatability and reproducibility, designated as $R2$ precision values. This completes the analysis sequence, and the values found for repeatability and reproducibility for each material are used to prepare a table of precision results for the test method.

NOTE 1—Although complete analysis algorithms using spreadsheet procedures are given in this practice, a special computer program has been developed by ASTM Committee E11 to calculate repeatability and reproducibility equivalent to this practice, and the software for this is available from ASTM. See Ref (5). However, the ASTM program is not able to accommodate databases that have blank cells. See 8.1 and Annex A4 for more details on calculation procedures.

7.1.5 The General Precision part of this practice does not address the issue of attempting to fit a relationship; r , R , (r) or (R) versus the property (level) for any ITP for two reasons. First, most ITPs do not have a sufficient number of materials to produce any meaningful functionality of precision versus material level; the degrees of freedom for any obtained fit are small. Second, experience has shown that even when there are several materials in an ITP, a good fitting linear or other relationship is not obtained. It should be remembered that any ITP is an event in time that gives an indication of the general level of precision for three or four materials in the test

TABLE 1 Precision Program—Basic Data^A

Material (j) ==> Laboratory (i)	1	2	3	4	...	q
1						
2			Y_{ijk}			
3						
4						
5						
...						
p						

^A Table layout for uniform level ITP.

Notation used:

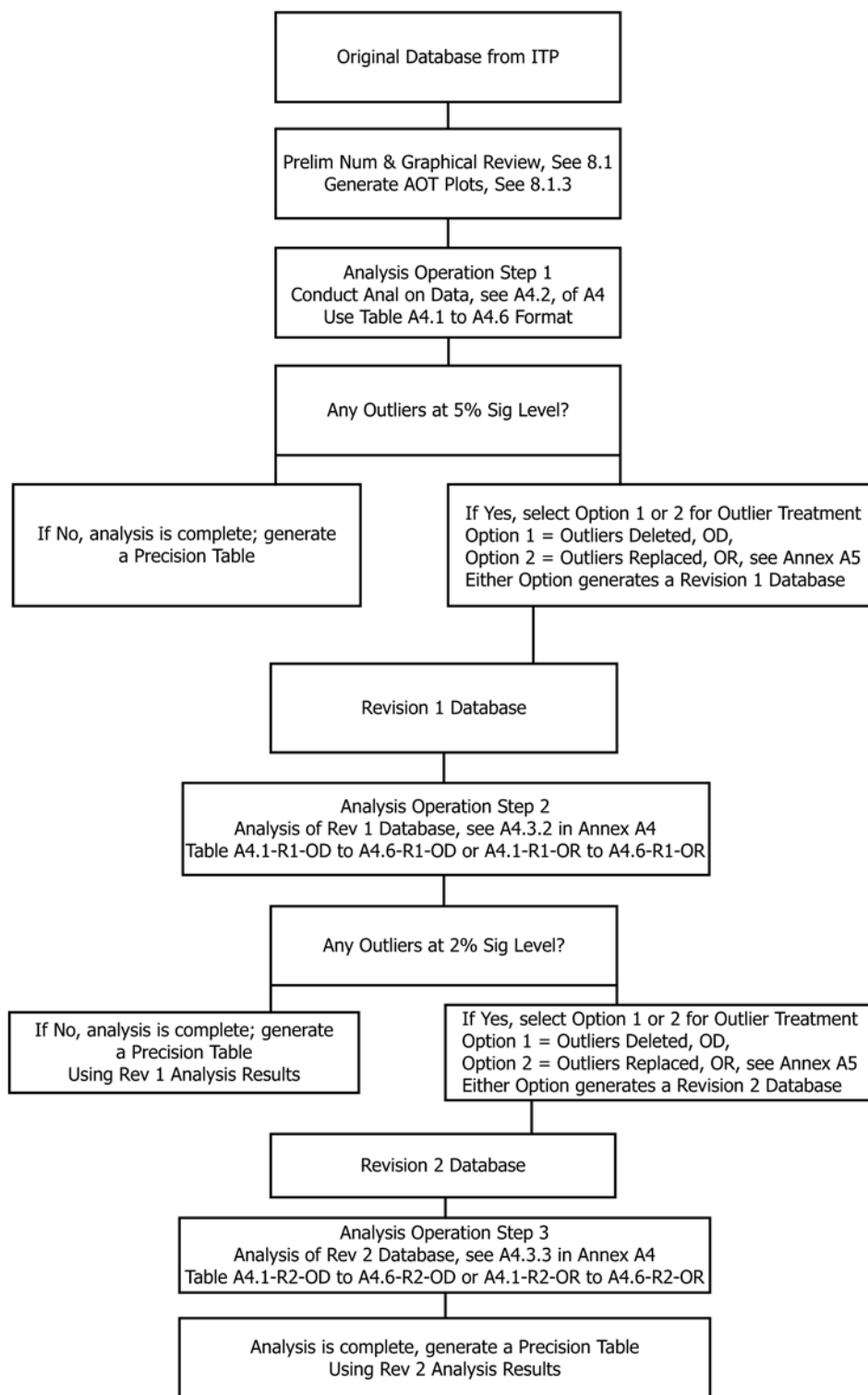
Laboratories, a total of p , $L(i) = 1, 2, 3, \dots, p$

Materials or Levels, a total of q , $m(j) = 1, 2, 3, \dots, q$

Replicates, a total of n per cell; a cell = each combination of $L(i)$ $m(j)$; normally $n = 2$

Y_{ijk} = a single test result value; where $k = 1, 2, \dots, n(j)$; see cell (23) of table for example

Cells (i, j); each cell contains n test result values



NOTE 1—Refer to Example Precision Calculations in [Annex A6](#) for tables with data.

FIG. 1 Decision Tree Diagram for ITP Data Analysis

number of laboratories. With some occasional exceptions, the precision found is usually quite different for each material with no detectable pattern or functionality.

7.2 [Annex A2](#) gives a statistical model that demonstrates the influence of both random and bias components of variation

inherent in any precision evaluation. Section [A2.5](#) gives the derivation of the expressions for repeatability and reproducibility in terms of the between laboratory and within laboratory variance and illustrates how both of these are related to random and bias components of variance.

7.2.1 The term S_L^2 is used in the calculation of the reproducibility variance and standard deviation in accordance with A2.5.1. Experience has shown, however, that the within-laboratory variation is substantially smaller than between-laboratory variation. In certain circumstances S_L^2 may calculate to less than zero; if this occurs, S_L^2 is set equal to zero. This less than zero situation may occur when there is substantial within cell variation of such a nature that when laboratory cell averages are calculated, they agree quite well. The analyst is cautioned to check the final (that is, after all outlier treatments, if any, have been completed) calculations to see if there are any less than zero values for S_L^2 and replace the less than zero value(s) with zero before calculating and reporting the final precision values.

7.3 *Background on Outliers*—The recognition and removal of the incompatible test values in any precision database is a subject with some controversy. If true outliers are not removed and their magnitude is substantial, seriously inflated values may be obtained for both precision parameters. This can result from only a few of the participating laboratories. However, caution must be exercised to ensure that high (or low) magnitude but bona fide values, not be deleted. If such values are removed, the precision estimates will be too optimistic. The procedures as presented in this practice attempt to find a middle ground position, designated as a *robust analysis*. Although objective, probability-based techniques are used to declare incompatible values as outliers, all outlier rejection operations have a substantial conditional character and require some input and experience from the analyst.

7.4 *Outlier Appearance Patterns*—Outliers frequently occur in one of two general appearance patterns: (1) *None or Infrequent*—There are no outliers or there are only a few outliers; one or two for every 20 data cells in a Table 1 format or (2) *Extensive*—Outliers occur in greater numbers, three, four, or more for every 20 data cells and frequently in several of the cells for any laboratory. When outliers are extensive they may frequently be of substantial magnitude. There are of course some intermediate cases between these two extremes.

7.5 *Rationale 1 for Outlier Rejection*—There are two points of view on what significance level should be adopted for outlier rejection. The extremely conservative approach maintains that outliers should rarely be eliminated in any ITP. This is based in part on the concept that in the preliminary stages of test method development, outlier rejection will lead to an overly optimistic impression of the quality of the test method. This approach usually adopts a probability significance level of 0.5 % ($p = 0.005$), for outlier rejection. This approach has some limited merit for the initial stages of development for any test method especially when only a few laboratories participate in an ITP. This significance level is specified by Practice E691. However, this approach has some serious limitations as described as follows.

7.6 *Rationale 2 for Outlier Rejection*—For well-established test methods and any group of laboratories, experience has taught that there is a distribution of skill and testing competence, from poor to good. This capability range argues for a more realistic approach to the outlier issue; the use of a

5 % significance level, $p = 0.05$ (or a 95 % confidence level) for the declaration of incompatible values as outliers. This is the usual level for most statistical significance tests and will in general reject the results of laboratories that have poor quality control for internal testing and are in need of improved operating procedures.

7.6.1 Allowing a few *poor* laboratories to inflate the evaluated precision gives a false negative impression of the true precision defined by laboratories with good control of testing operations. The precision of the *good* laboratories (the majority of those participating) should be the benchmark for industry-wide precision level for any test method. The use of the robust General and Special Precision procedures to identify these poor quality control laboratories can lead to a general industry-wide improvement for any test method provided that feedback is employed to encourage the poor performing laboratories to improve testing operations.

7.7 *Sequential Review of Outliers*—Experience in outlier review at the 5 % significance level raises the issue of a subsequent review of the database once the 5 % outliers are deleted. To properly frame this operation, recall that the h and k statistics represent ratios of either individual cell averages or cell standard deviations to the *across all laboratory* standard deviation for each parameter. The influence of any outlier extends to both the outlier value itself (the numerator for h and k), as well as the standard deviation for all laboratories (the denominator for h and k).

7.7.1 The removal of 5 % significance outliers will generate a second (or Revision 1) database with substantially reduced *across all laboratories* or denominator standard deviation for either the h or k statistics, or both. When outliers are deleted the resulting revised database is one that might have been obtained had the outlying laboratories not volunteered for the ITP. The question now presents itself: Can this *R1* database be reviewed again for h and k outliers using the newly calculated *across all laboratory* h and k standard deviations.

7.7.2 For any ITP that contains six or more original laboratories, the answer to this question is yes, and the second or revised database should be reviewed for any potential outliers. However, to guard against the generation of an excessively optimistic precision, the significance level for this second review should be more rigorous than for the initial review and should be conducted at the 2 % significance level. For any ITP that contains less than 6 laboratories, the decision to conduct a second review is left to the judgment of the analyst.

7.8 *Special Case Circumstances for Outliers*—In the analysis of larger databases (12 or more laboratories) it may happen (infrequently) that there are three or more suspicious or potential h and/or k outliers in the database for Step 2. When this happens the calculated h or k values for the three or more laboratories may be close to the critical values but usually do not exceed them. If there is a fairly large difference between these calculated h or k values (for the suspicious three or more outlier values) and the remaining h or k values that constitute the bulk of the laboratories that give good agreement, it is recommended that a 5 % significance level be used for the Step 2 procedure in place of the 2 % significance level.

experience (2004) in the analysis of such databases has shown that this exception to the standard Step 2 procedure will eliminate those laboratories that do not have good control over their testing operations. This prevents the results of such laboratories from inflating the precision estimate of the ‘in good agreement’ laboratories (the majority of the total number) that should constitute the benchmark for the industry for the test method in question.

8. General Precision: Analysis Step 1

8.1 Preliminary Numerical and Graphical Data Review—Prior to the detailed calculations of Analysis Step 1, it is important to review the data by a graphical technique that gives insight into the uniformity of the database. The most frequently used precision evaluation is a uniform level design; all laboratories test the same number of replicates and test all materials. **Table 1** indicates the layout for this uniform level design and gives the format for tabulating the basic data. There are a total of p laboratories and a total of q materials or element classes and a total of pq cells in the table. Each cell of the table, which constitutes a laboratory-material combination, contains n replicates, each test result replicate is designated as a Y_{ijk} value. The most frequently used design has two replicates per cell or $n = 2$.

8.1.1 Calculating Cell Averages, Cell Ranges, or Standard Deviations—A table in the format of **Table 2** is prepared by calculating the average of the n replicates per cell as given in **Table 1**. After cell averages have been calculated they should be reviewed for any apparent outlier values as described in **8.1.3** and these noted for evaluation as given in the formal Step 1 outlier rejection procedure as described in **8.3** and **8.4**. See also **Annex A3**.

8.1.2 A table in the format of **Table 3** is prepared by calculating, for all cells, the standard deviation for the n replicates per cell. Alternatively, cell ranges, denoted by w , the absolute difference between the maximum and minimum values in each cell, may be calculated. Both the cell ranges and the cell standard deviations should also be reviewed for any apparent outlier values and these noted for evaluation as given in the formal Step 1 outlier rejection procedure as described in **8.3** and **8.4**. See **Annex A3**.

TABLE 2 Precision Program—Cell Averages^A

Material (j) ==> Laboratory (i)	1	2	3	4	...	q
1						
2			avg Y_{ijk}			
3						
4						
5						
...						
p						

^A Table layout for uniform level ITP.

Notation used:
Laboratories, a total of p , $L(i) = 1, 2, 3, \dots, p$
Materials or Levels, a total of q , $m(j) = 1, 2, 3, \dots, q$
Replicates, a total of n per cell; a cell = each combination of $L(i)$ $m(j)$; normally $n = 2$
avg Y_{ijk} = average of cell (ij) for n test results

TABLE 3 Precision Program—Cell Std Deviations^A

Material (j) ==> Laboratory (i)	1	2	3	4	...	q
1						
2			SD Y_{ijk}			
3						
4						
5						
...						
p						

^A Table layout for uniform level ITP.

Notation used:
Laboratories, a total of p , $L(i) = 1, 2, 3, \dots, p$
Materials or Levels, a total of q , $m(j) = 1, 2, 3, \dots, q$
Replicates, a total of n per cell; a cell = each combination of $L(i)$ $m(j)$; normally $n = 2$
SD Y_{ijk} = standard deviation of cell (ij) for n test results

8.1.3 Graphical Review of Cell Values—The general distribution of the data to disclose any potential outliers, is reviewed with special plots of the cell averages and the cell ranges or standard deviations, using a typical spreadsheet program. Prepare two new tables, one for cell averages, and one for cell ranges. Cell ranges are used here because they facilitate certain calculation options that will be employed later in treating outliers, that is, either deletion or replacement. However, cell standard deviations may be used. For the cell average table and for the first material, generate two columns in the table; the first column contains the laboratory number, 1 to N , and the second column contains the corresponding cell average. Repeat this two-column *laboratory number-cell average* sequence for all materials. Prepare a table for cell ranges in the same manner as for cell averages with the *laboratory number-cell range* dual column scheme.

8.1.3.1 Using the prepared tables, for each laboratory-material pair of columns, sort the cell averages (or cell ranges) in ascending order (across all laboratories) retaining the laboratory number with the cell value in the sorting operation. For each parameter (cell average or cell range), plot the parameter value versus the laboratory number in ascending parameter value order, using a line plot procedure. This is designated as an *ascending order trend* or AOT plot.

8.1.3.2 For an ITP with no outliers, the cell average plot is typically a positive slope straight line with some reasonable degree of point scatter. If any outliers are present, they will be at the opposite ends of the plot, and will show substantial departure from the straight line of the central data point region. The cell range plot may contain more curvature from the low end (which may contain zero values) toward the central point region, but it will also clearly show the outliers at the high value end of the plot. Ascending order plots will be used in the operation to replace outlier values with *replacement values* as outlined in **Annex A5**.

8.2 Calculation of Precision for Original Database—Comprehensive specific instructions for this are given in **Annex A4**.

NOTE 2—In Sections 8 – 10 Tables A4.1 to A4.6 are discussed; these are tables that the analyst will prepare in a computer spreadsheet according to the instructions as outlined in **Annex A4**. There are no actual (printed) Tables A4.1 to A4.6 (with the appended letter designations) in the standard. The table letter designations R1, R2, R3, R4, R5, R6, R7, R8, R9, R10, R11, R12, R13, R14, R15, R16, R17, R18, R19, R20, R21, R22, R23, R24, R25, R26, R27, R28, R29, R30, R31, R32, R33, R34, R35, R36, R37, R38, R39, R40, R41, R42, R43, R44, R45, R46, R47, R48, R49, R50, R51, R52, R53, R54, R55, R56, R57, R58, R59, R60, R61, R62, R63, R64, R65, R66, R67, R68, R69, R70, R71, R72, R73, R74, R75, R76, R77, R78, R79, R80, R81, R82, R83, R84, R85, R86, R87, R88, R89, R90, R91, R92, R93, R94, R95, R96, R97, R98, R99, R100, R101, R102, R103, R104, R105, R106, R107, R108, R109, R110, R111, R112, R113, R114, R115, R116, R117, R118, R119, R120, R121, R122, R123, R124, R125, R126, R127, R128, R129, R130, R131, R132, R133, R134, R135, R136, R137, R138, R139, R140, R141, R142, R143, R144, R145, R146, R147, R148, R149, R150, R151, R152, R153, R154, R155, R156, R157, R158, R159, R160, R161, R162, R163, R164, R165, R166, R167, R168, R169, R170, R171, R172, R173, R174, R175, R176, R177, R178, R179, R180, R181, R182, R183, R184, R185, R186, R187, R188, R189, R190, R191, R192, R193, R194, R195, R196, R197, R198, R199, R200, R201, R202, R203, R204, R205, R206, R207, R208, R209, R210, R211, R212, R213, R214, R215, R216, R217, R218, R219, R220, R221, R222, R223, R224, R225, R226, R227, R228, R229, R230, R231, R232, R233, R234, R235, R236, R237, R238, R239, R240, R241, R242, R243, R244, R245, R246, R247, R248, R249, R250, R251, R252, R253, R254, R255, R256, R257, R258, R259, R260, R261, R262, R263, R264, R265, R266, R267, R268, R269, R270, R271, R272, R273, R274, R275, R276, R277, R278, R279, R280, R281, R282, R283, R284, R285, R286, R287, R288, R289, R290, R291, R292, R293, R294, R295, R296, R297, R298, R299, R300, R301, R302, R303, R304, R305, R306, R307, R308, R309, R310, R311, R312, R313, R314, R315, R316, R317, R318, R319, R320, R321, R322, R323, R324, R325, R326, R327, R328, R329, R330, R331, R332, R333, R334, R335, R336, R337, R338, R339, R340, R341, R342, R343, R344, R345, R346, R347, R348, R349, R350, R351, R352, R353, R354, R355, R356, R357, R358, R359, R360, R361, R362, R363, R364, R365, R366, R367, R368, R369, R370, R371, R372, R373, R374, R375, R376, R377, R378, R379, R380, R381, R382, R383, R384, R385, R386, R387, R388, R389, R390, R391, R392, R393, R394, R395, R396, R397, R398, R399, R400, R401, R402, R403, R404, R405, R406, R407, R408, R409, R410, R411, R412, R413, R414, R415, R416, R417, R418, R419, R420, R421, R422, R423, R424, R425, R426, R427, R428, R429, R430, R431, R432, R433, R434, R435, R436, R437, R438, R439, R440, R441, R442, R443, R444, R445, R446, R447, R448, R449, R450, R451, R452, R453, R454, R455, R456, R457, R458, R459, R460, R461, R462, R463, R464, R465, R466, R467, R468, R469, R470, R471, R472, R473, R474, R475, R476, R477, R478, R479, R480, R481, R482, R483, R484, R485, R486, R487, R488, R489, R490, R491, R492, R493, R494, R495, R496, R497, R498, R499, R500, R501, R502, R503, R504, R505, R506, R507, R508, R509, R510, R511, R512, R513, R514, R515, R516, R517, R518, R519, R520, R521, R522, R523, R524, R525, R526, R527, R528, R529, R530, R531, R532, R533, R534, R535, R536, R537, R538, R539, R540, R541, R542, R543, R544, R545, R546, R547, R548, R549, R550, R551, R552, R553, R554, R555, R556, R557, R558, R559, R560, R561, R562, R563, R564, R565, R566, R567, R568, R569, R570, R571, R572, R573, R574, R575, R576, R577, R578, R579, R580, R581, R582, R583, R584, R585, R586, R587, R588, R589, R590, R591, R592, R593, R594, R595, R596, R597, R598, R599, R600, R601, R602, R603, R604, R605, R606, R607, R608, R609, R610, R611, R612, R613, R614, R615, R616, R617, R618, R619, R620, R621, R622, R623, R624, R625, R626, R627, R628, R629, R630, R631, R632, R633, R634, R635, R636, R637, R638, R639, R640, R641, R642, R643, R644, R645, R646, R647, R648, R649, R650, R651, R652, R653, R654, R655, R656, R657, R658, R659, R660, R661, R662, R663, R664, R665, R666, R667, R668, R669, R670, R671, R672, R673, R674, R675, R676, R677, R678, R679, R680, R681, R682, R683, R684, R685, R686, R687, R688, R689, R690, R691, R692, R693, R694, R695, R696, R697, R698, R699, R700, R701, R702, R703, R704, R705, R706, R707, R708, R709, R710, R711, R712, R713, R714, R715, R716, R717, R718, R719, R720, R721, R722, R723, R724, R725, R726, R727, R728, R729, R730, R731, R732, R733, R734, R735, R736, R737, R738, R739, R740, R741, R742, R743, R744, R745, R746, R747, R748, R749, R750, R751, R752, R753, R754, R755, R756, R757, R758, R759, R760, R761, R762, R763, R764, R765, R766, R767, R768, R769, R770, R771, R772, R773, R774, R775, R776, R777, R778, R779, R780, R781, R782, R783, R784, R785, R786, R787, R788, R789, R790, R791, R792, R793, R794, R795, R796, R797, R798, R799, R800, R801, R802, R803, R804, R805, R806, R807, R808, R809, R810, R811, R812, R813, R814, R815, R816, R817, R818, R819, R820, R821, R822, R823, R824, R825, R826, R827, R828, R829, R830, R831, R832, R833, R834, R835, R836, R837, R838, R839, R840, R841, R842, R843, R844, R845, R846, R847, R848, R849, R850, R851, R852, R853, R854, R855, R856, R857, R858, R859, R860, R861, R862, R863, R864, R865, R866, R867, R868, R869, R870, R871, R872, R873, R874, R875, R876, R877, R878, R879, R880, R881, R882, R883, R884, R885, R886, R887, R888, R889, R890, R891, R892, R893, R894, R895, R896, R897, R898, R899, R900, R901, R902, R903, R904, R905, R906, R907, R908, R909, R910, R911, R912, R913, R914, R915, R916, R917, R918, R919, R920, R921, R922, R923, R924, R925, R926, R927, R928, R929, R930, R931, R932, R933, R934, R935, R936, R937, R938, R939, R940, R941, R942, R943, R944, R945, R946, R947, R948, R949, R950, R951, R952, R953, R954, R955, R956, R957, R958, R959, R960, R961, R962, R963, R964, R965, R966, R967, R968, R969, R970, R971, R972, R973, R974, R975, R976, R977, R978, R979, R980, R981, R982, R983, R984, R985, R986, R987, R988, R989, R990, R991, R992, R993, R994, R995, R996, R997, R998, R999, R1000, R1001, R1002, R1003, R1004, R1005, R1006, R1007, R1008, R1009, R1010, R1011, R1012, R1013, R1014, R1015, R1016, R1017, R1018, R1019, R1020, R1021, R1022, R1023, R1024, R1025, R1026, R1027, R1028, R1029, R1030, R1031, R1032, R1033, R1034, R1035, R1036, R1037, R1038, R1039, R1040, R1041, R1042, R1043, R1044, R1045, R1046, R1047, R1048, R1049, R1050, R1051, R1052, R1053, R1054, R1055, R1056, R1057, R1058, R1059, R1060, R1061, R1062, R1063, R1064, R1065, R1066, R1067, R1068, R1069, R1070, R1071, R1072, R1073, R1074, R1075, R1076, R1077, R1078, R1079, R1080, R1081, R1082, R1083, R1084, R1085, R1086, R1087, R1088, R1089, R1090, R1091, R1092, R1093, R1094, R1095, R1096, R1097, R1098, R1099, R1100, R1101, R1102, R1103, R1104, R1105, R1106, R1107, R1108, R1109, R1110, R1111, R1112, R1113, R1114, R1115, R1116, R1117, R1118, R1119, R1120, R1121, R1122, R1123, R1124, R1125, R1126, R1127, R1128, R1129, R1130, R1131, R1132, R1133, R1134, R1135, R1136, R1137, R1138, R1139, R1140, R1141, R1142, R1143, R1144, R1145, R1146, R1147, R1148, R1149, R1150, R1151, R1152, R1153, R1154, R1155, R1156, R1157, R1158, R1159, R1160, R1161, R1162, R1163, R1164, R1165, R1166, R1167, R1168, R1169, R1170, R1171, R1172, R1173, R1174, R1175, R1176, R1177, R1178, R1179, R1180, R1181, R1182, R1183, R1184, R1185, R1186, R1187, R1188, R1189, R1190, R1191, R1192, R1193, R1194, R1195, R1196, R1197, R1198, R1199, R1200, R1201, R1202, R1203, R1204, R1205, R1206, R1207, R1208, R1209, R1210, R1211, R1212, R1213, R1214, R1215, R1216, R1217, R1218, R1219, R1220, R1221, R1222, R1223, R1224, R1225, R1226, R1227, R1228, R1229, R1230, R1231, R1232, R1233, R1234, R1235, R1236, R1237, R1238, R1239, R1240, R1241, R1242, R1243, R1244, R1245, R1246, R1247, R1248, R1249, R1250, R1251, R1252, R1253, R1254, R1255, R1256, R1257, R1258, R1259, R1260, R1261, R1262, R1263, R1264, R1265, R1266, R1267, R1268, R1269, R1270, R1271, R1272, R1273, R1274, R1275, R1276, R1277, R1278, R1279, R1280, R1281, R1282, R1283, R1284, R1285, R1286, R1287, R1288, R1289, R1290, R1291, R1292, R1293, R1294, R1295, R1296, R1297, R1298, R1299, R1300, R1301, R1302, R1303, R1304, R1305, R1306, R1307, R1308, R1309, R1310, R1311, R1312, R1313, R1314, R1315, R1316, R1317, R1318, R1319, R1320, R1321, R1322, R1323, R1324, R1325, R1326, R1327, R1328, R1329, R1330, R1331, R1332, R1333, R1334, R1335, R1336, R1337, R1338, R1339, R1340, R1341, R1342, R1343, R1344, R1345, R1346, R1347, R1348, R1349, R1350, R1351, R1352, R1353, R1354, R1355, R1356, R1357, R1358, R1359, R1360, R1361, R1362, R1363, R1364, R1365, R1366, R1367, R1368, R1369, R1370, R1371, R1372, R1373, R1374, R1375, R1376, R1377, R1378, R1379, R1380, R1381, R1382, R1383, R1384, R1385, R1386, R1387, R1388, R1389, R1390, R1391, R1392, R1393, R1394, R1395, R1396, R1397, R1398, R1399, R1400, R1401, R1402, R1403, R1404, R1405, R1406, R1407, R1408, R1409, R1410, R1411, R1412, R1413, R1414, R1415, R1416, R1417, R1418, R1419, R1420, R1421, R1422, R1423, R1424, R1425, R1426, R1427, R1428, R1429, R1430, R1431, R1432, R1433, R1434, R1435, R1436, R1437, R1438, R1439, R1440, R1441, R1442, R1443, R1444, R1445, R1446, R1447, R1448, R1449, R1450, R1451, R1452, R1453, R1454, R1455, R1456, R1457, R1458, R1459, R1460, R1461, R1462, R1463, R1464, R1465, R1466, R1467, R1468, R1469, R1470, R1471, R1472, R1473, R1474, R1475, R1476, R1477, R1478, R1479, R1480, R1481, R1482, R1483, R1484, R1485, R1486, R1487, R1488, R1489, R1490, R1491, R1492, R1493, R1494, R1495, R1496, R1497, R1498, R1499, R1500, R1501, R1502, R1503, R1504, R1505, R1506, R1507, R1508, R1509, R1510, R1511, R1512, R1513, R1514, R1515, R1516, R1517, R1518, R1519, R1520, R1521, R1522, R1523, R1524, R1525, R1526, R1527, R1528, R1529, R1530, R1531, R1532, R1533, R1534, R1535, R1536, R1537, R1538, R1539, R1540, R1541, R1542, R1543, R1544, R1545, R1546, R1547, R1548, R1549, R1550, R1551, R1552, R1553, R1554, R1555, R1556, R1557, R1558, R1559, R1560, R1561, R1562, R1563, R1564, R1565, R1566, R1567, R1568, R1569, R1570, R1571, R1572, R1573, R1574, R1575, R1576, R1577, R1578, R1579, R1580, R1581, R1582, R1583, R1584, R1585, R1586, R1587, R1588, R1589, R1590, R1591, R1592, R1593, R1594, R1595, R1596, R1597, R1598, R1599, R1600, R1601, R1602, R1603, R1604, R1605, R1606, R1607, R1608, R1609, R1610, R1611, R1612, R1613, R1614, R1615, R1616, R1617, R1618, R1619, R1620, R1621, R1622, R1623, R1624, R1625, R1626, R1627, R1628, R1629, R1630, R1631, R1632, R1633, R1634, R1635, R1636, R1637, R1638, R1639, R1640, R1641, R1642, R1643, R1644, R1645, R1646, R1647, R1648, R1649, R1650, R1651, R1652, R1653, R1654, R1655, R1656, R1657, R1658, R1659, R1660, R1661, R1662, R1663, R1664, R1665, R1666, R1667, R1668, R1669, R1670, R1671, R1672, R1673, R1674, R1675, R1676, R1677, R1678, R1679, R1680, R1681, R1682, R1683, R1684, R1685, R1686, R1687, R1688, R1689, R1690, R1691, R1692, R1693, R1694, R1695, R1696, R1697, R1698, R1699, R1700, R1701, R1702, R1703, R1704, R1705, R1706, R1707, R1708, R1709, R1710, R1711, R1712, R1713, R1714, R1715, R1716, R1717, R1718, R1719, R1720, R1721, R1722, R1723, R1724, R1725, R1726, R1727, R1728, R1729, R1730, R1731, R1732, R1733, R1734, R1735, R1736, R1737, R1738, R1739, R1740, R1741, R1742, R1743, R1744, R1745, R1746, R1747, R1748, R1749, R1750, R1751, R1752, R1753, R1754, R1755, R1756, R1757, R1758, R1759, R1760, R1761, R1762, R

pairs to the usual ASTM table identification numbers help to make the tables self-identifying. Their use improves comprehension both in table generation and in reviewing the tables during analysis. The use of these appended designations is further explained and discussed in Sections 8 – 10. See also A4.2.2 and A4.3 in Annex A4.

The test result values for the original database are entered into a table, designated as Table A4.1. This tabular format is also described as Table 1 in the main body of the standard. However, to preserve continuity between Annex A4 and the instructions of 8.2, the table identification terminology of Annex A4 will be used.

8.2.1 The next step is to set up a tabular format designated as Table A4.2 for cell averages and cell averages squared. The corresponding values in Table A4.1 are the argument values for Table A4.2.

8.2.2 Table A4.3 is generated next, cell average deviations, denoted by d and the calculated h -values. The corresponding values in Table A4.2 are used as the arguments for Table A4.3. Refer to Annex A3 for cell deviation d and h -value calculations.

8.2.3 Table A4.4R for cell ranges and cell ranges squared and Table A4.4S for cell standard deviations and cell variances (standard deviations squared) both address the same issue; the within cell variation. It is recommended that both tables be generated in the analysis.

8.2.4 Table A4.5 is used to calculate k -values for each cell in the database. The corresponding values in Table A4.4S are used as the arguments to calculate k -values in Table A4.5. Refer to Annex A3 for k -value calculations.

8.2.5 Table A4.6 is used to calculate the precision parameters, r , R , (r) , and (R) . Values for T_1 , T_2 , T_4 and n and p are required to calculate r and R . See the imbedded calculation algorithms, 1 to 5, in Table A4.6 and also Annex A4 for the details on these calculations.

8.3 *Detection of Outliers at the 5 % Significance Level Using h and k Statistics*—The calculated values for h in Table A4.3 and the calculated values of k in Table A4.5 are reviewed for potential outlier values.

8.3.1 If the Table A4.3 h -value for any cell equals or exceeds the 5 % significance level critical h -value as given in Table A3.1, that particular cell value is declared as an outlier.

8.3.2 If the Table A4.5 k -value for any cell equals or exceeds the 5 % significance level critical k -value as given in Table A3.1, that particular cell value is declared as an outlier.

8.3.3 If outliers are detected, a summary of the outliers detected is presented in the form of a sub-table at the bottom of Table A4.6 showing the laboratory numbers that had 5 % significance outliers for both h and k for each material. See Table in Annex A6 for an example. When outliers are present, a revised database is generated by the use of either Option 1, outlier deletion, or Option 2, outlier replacement, as described in 8.4.

8.3.4 If there are no outliers for either cell averages or cell standard deviations, the precision analysis is complete and the resulting values for r , R , (r) , and (R) may be used to prepare a precision table for the test method.

8.4 *Generation of R1 Database Using Outlier Option 1 or 2*—If outliers are detected, the database is revised using either Option 1 or 2. The revision procedure is described in A4.3.

8.4.1 Option 1 is the deletion of the n cell values in Table A4.1 that are indicated as outliers and the correction of ERR indications in certain cells in Tables A4.2 to A4.6 that result from the deletion process as described in A4.3. The deletion applies to both cell averages as indicated by equal or greater than 5 % critical h -values and to cell standard deviations as indicated by equal or greater than 5 % critical k -values. Once all ERR corrections have been made the database is designated as a R1 database. Each R1 table designation contains the appended symbols, R1-OD, outliers deleted. This revised OD database will be reviewed again for outliers now at the more critical 2 % significance level as described in Analysis Step 2.

8.4.2 Option 2 is the replacement of the n cell values in Table A4.1 that are indicated as outliers. The replacement applies to both cell averages and to cell standard deviations as indicated by greater than 5 % critical values. For either the h or k values, the replacement is a two sequence, one- or two-stage process. All of the details for this are described fully in Annex A5. Once replacements have been generated by the Annex A5 procedure, they are inserted into the database, replacing the outlier values, to produce a R1 database using the table identification symbol, R1-OR, outliers replaced. This revised OR database will be reviewed again for outliers now at the more critical 2 % significance level as described in Analysis Step 2.

8.5 *R1 Database Tables*—A second set of tables in the format of A4.1 to A4.6 is prepared for the Step 2 analysis. As previously noted, this second set should be (1) tables designated as A4.1-R1-OD to A4.6-R1-OD for the selection of outlier Option 1, or (2) tables designated as A4.1-R1-OR to A4.6-R1-OR for Option 2 outlier replacement. Once the deletions or the replacements have been made, according to the instructions in Annex A4, the new set of precision values will appear in Table A4.6-R1-OD or Table A4.6-R1-OR depending on the option chosen.

9. General Precision: Analysis Step 2

9.1 *Detection of Outliers at the 2 % Significance Level Using h and k Statistics*—The calculated values for h in Table A4.3-R1-OD or Table A4.3-R1-OR and the calculated values of k in Table A4.5-R1-OD or A4.5-R1-OR are reviewed for potential outlier values at the 2 % significance level. The calculated h and k values must be greater than the 2 % significance level for outliers to be rejected. For each of these tables, a sub-table is generated at the bottom of either table to summarize the results of the h and k comparisons of calculated values versus critical values. See Annex A6 for an example. If outliers are detected, the database is revised using either Outlier Option 1 or 2. The revision procedure is described in A4.3.

9.1.1 Option 1 is the deletion of the n cell values in Table A4.1-R1-OD that are indicated as outliers and the correction, as previously noted, of ERR indications in certain cells in Tables A4.2-R1-OD to A4.6-R1-OD that result from the deletion process. Once all ERR corrections have been made the database is designated as a R2-OD database. This revised OD database will be used for the operation of the test method.

9.1.2 Option 2 is the replacement of the n cell values in Table A4.1-R1-OR that are indicated as outliers. The replacement applies to both cell averages as indicated by greater than 2 % critical values for either h or k . The replacement is a two sequence, one- or two-stage process. All of the details for this are described fully in [Annex A5](#). Once replacements have been generated by the [Annex A5](#) procedure, they are inserted into the database to produce a R2-OR database. This revised OR database will be used for the operations of Analysis Step 3.

10. General Precision: Analysis Step 3

10.1 *Final Precision Results*—Although the [Fig. 1](#) decision tree diagram or flow sheet implies that Analysis Step 3 involves an analysis operation, the analysis has already been conducted automatically with the outlier treatment as described in Step 2. Step 3 is really a review of the precision results that have been obtained previously from the R2 database. The automatic calculation procedure of the interlinked Tables A4.1 to A4.6 produces the new precision results once either outlier Option 1 (deletion) or Option 2 (replacement) have been selected and the deletion or replacement operations completed.

10.1.1 Analysis Step 3 is the end of the precision calculations when outliers have been found at both the 5 % and 2 % significance levels. The results for either Table A4.6-R2-OD or Table A4.6-R2-OR are used to generate a Precision Table for the test method under review. Refer to Section 13 on the appropriate format for a precision table, see [Table 6](#), and the appropriate text for the precision clause or section.

11. Special Precision Analysis—Carbon Black Testing

11.1 *Background*—The evaluation of test methods for the carbon black manufacturing industry shall be conducted by the procedures as described in this section for the typical uniform level experimental design. These procedures differ from the requirements as set forth in the General Precision procedure as follows: (1) the number of replicates in each cell of the [Table 1](#) format is specified as four, (2) the cell averages and cell standard deviations are reviewed for potential outliers by a procedure that differs from that as specified for General Precision in terms of the potential number of outliers deleted, see [11.3.1](#), and (3) special calculations are conducted to select the mode of precision expression for reproducibility (absolute or relative) that is most free of influence of the magnitude of the measured property on the reported precision value. Note also that in reviewing discordant data values as potential outliers, only the 5 % significance level h and k values in [Table A3.1](#) are used to reject outliers.

11.1.1 The terminology as set forth in Section 3, as well as the terminology in [Annex A1](#) shall apply to the procedures for this special precision. Frequently in the carbon black industry and elsewhere, the word *sample* is used as a synonym for the

TABLE 5 Format for Interlaboratory Data—Special Precision: Carbon Black Testing

Laboratory Number	Material 1		Material 2		Material q	
	Cell Avg	Cell Std Deviation	Cell Avg	Cell Std Deviation	Cell Avg	Cell Std Deviation
1	xx	xx	xx	xx	xx	xx
2	xx	xx	xx	xx	xx	xx
...	xx	xx	xx	xx	xx	xx
p	xx	xx	xx	xx	xx	xx

word *material* in the discussion of interlaboratory testing, that is, a grade of carbon black used in an ITP is frequently referred to as a *sample*. This can be a source of confusion and is not consistent with the terminology of this practice. To avoid confusion, the terms *material* or *target material*, or both, shall be used for what is tested (for example, a series of different grades of carbon black), in the process of organizing, reporting, and discussing interlaboratory test programs and the precision parameters as calculated from such programs.

11.2 *Materials Selected, Initial Data Recording*—The number of materials (or target materials), which will normally be different grades of carbon black, shall be selected as recommended in [6.1.6](#). It is recommended that at least five materials be selected for any ITP. This number of materials provides for at least four degrees of freedom in evaluating the coefficient of determination as described in [11.4](#).

11.2.1 Tests on the selected materials (or target materials), shall be conducted in accordance with the specified test method to produce two test results on each of two separate *test* days for a total of four test results. All testing shall be conducted on the same test machine or apparatus. A test result is the median or average of the number of determinations as specified by the test method. For each material, the data values are recorded in an initial data format as indicated in [Table 4](#). Each set of four values constitutes one cell of the general data tabulation as specified in the General Precision [Table 1](#) format. However for carbon black testing, a different final data tabulation is used as given by [Table 5](#), a format that contains results for all materials in the ITP, as obtained from calculations. See [11.3](#) on the data for each material in the [Table 4](#) format.

11.3 *Data Review and Calculations*—After a series of tables in [Table 4](#) format are prepared, one for each material and each laboratory, the next step is to use the data of each table to calculate a cell average and a cell standard deviation for each material-laboratory combination or cell. The results of these calculations are recorded in [Table 5](#) format. On a material by material basis, the cell averages of [Table 5](#) are reviewed for any potential outliers using the h statistic and the cell standard deviations are reviewed for any potential outliers using the k statistic. Outliers are determined on the basis of a 5 % significance level for $h(\text{crit})$ and $k(\text{crit})$. Although both the cell average and the cell standard deviation of [Table 5](#) each contain two undifferentiated components of variation, between tests-between days and between tests-within days, the h and k statistic procedure serves a useful purpose to detect any potential outliers on these special cell values.

TABLE 4 Initial Data Format for Each Material—Special Precision: Carbon Black Testing

Date	Material (i)		Operator or Technician
	Test Result 1	Test Result 2	
Day 1	xxx	xxx	xxxxx
Day 2	xxx	xxx	xxxxx

TABLE 6 Example of Precision Table Organization—Type 1: Precision for ASTM XXXXX

NOTE 1—Measured Property = xxxxxx, in xx.

Material	Mean Level	Within Laboratories			Between Laboratories			No. Laboratories ^A
		<i>Sr</i>	<i>r</i>	(<i>r</i>)	<i>SR</i>	<i>R</i>	(<i>R</i>)	
A								
B								
C								
D								
Pooled or Average Values								

^A List number of laboratories in final database, also list the Option chosen; if Option 2, indicate with number of laboratories in parentheses.

Notation used:

Sr = within-laboratory standard deviation (in measurement units)

r = repeatability (in measurement units)

(*r*) = repeatability (in percent of mean level)

SR = between-laboratory standard deviation (for total between laboratory variation in measurement units)

R = reproducibility (in measurement units)

(*R*) = reproducibility (in percent of mean level)

See text of Precision Clause for discussion of precision results of this table

11.3.1 The review process for carbon black ITP testing is based on the premise that a substantial number of laboratories participate in the ITP, some number greater than 20. For each material in the **Table 5** format, calculate the *h*-value and *k*-value for each cell (or laboratory) by the procedure as specified in **Annex A3**. A value for *h*(crit) and *k*(crit) at the 5 % significance level is selected from **Table A3.1**. The calculated *h*-values and *k*-values are reviewed to determine if any are greater than *h*(crit) or *k*(crit). The rejection process is conducted on the basis of the following rules.

11.3.1.1 If there are no calculated *h* -values or *k*-values greater than *h*(crit) or *k*(crit), all cell averages or standard deviations, or both, are retained.

11.3.1.2 If there is only one *h*-value or *k*-value greater than *h*(crit) or *k*(crit), reject the cell average or standard deviation.

11.3.1.3 If more than one *h*-value is greater than *h*(crit) or more than one *k*-value is greater than *k*(crit), the rejection process proceeds as follows:

(1) If there are 20 or fewer laboratories in the ITP, reject only one cell average or cell standard deviation per material, with the greatest (absolute value) calculated *h* or *k* value.

(2) If there are greater than 20 laboratories in the ITP and there are several *h*-values or *k*-values, or both, greater than the respective *h*(crit) and *k*(crit), reject cell averages or cell standard deviations, or both, starting with the highest (absolute value) calculated *h* and *k* values and proceeding downward, until the number of remaining laboratories is 20, or all the *h* values greater than *h*(crit) or *k* values greater than *k*(crit) have been rejected, and use this as the database for precision evaluation.

11.3.2 If any outliers are rejected following the rules of **11.3.1**, the resulting database with outlier data deleted is designated as an *R1* database. Conduct a second precision analysis on the *R1* database to generate the final table of precision parameters to be used in the operations as described in **11.4**.

11.4 *Expressing the Evaluated Precision for Carbon Black Testing*—Calculate the precision parameters *r*, *R*, (*r*), and (*R*) using the formulas as specified in **A4.1**. The calculations shall be on the original database if there are no outliers, or on the *R1* database after any potential outlier rejection following **11.3.1**.

Plot the values of *R* and (*R*) versus *M* or *Y_{AV}*, the mean value for material measured property, for all materials in the ITP. Perform a least squares regression for both relationships, and record the coefficient of determination, designated as *C_d*, for each parameter *R* and (*R*).

11.4.1 Select for the mode of precision expression, the parameter, *R* or (*R*), with the lowest value for *C_d*. This establishes which of the two modes of expression has the least relationship to the level of the measured property or inversely which parameter is the most independent of the measurement level. This lowest *C_d* or most independent parameter is to be used to prepare a final precision table in the format as indicated by **Table 6**. The selected mode of expression applies to both repeatability and reproducibility. Follow the rules for expressing General Precision as outlined in Section **12** using, where appropriate, the designation Special Precision. The columns [*r* and *R* or (*r*) and (*R*)] for the parameter with the highest *C_d* may be omitted from the format of **Table 6**.

12. Format for Precision Table and Section or Clause in Test Method Standards

12.1 *General Precision Table*—Precision is expressed in summary form in a **Table 6** format. Each summary precision table should have a heading to indicate: (1) use of General Precision or Special (Carbon Black) Precision, (2) the type of precision (Type 1 or Type 2), see **5.1.3 – 5.1.5**, and (3) the measured property and its measurement units.

12.1.1 For each material tested, the following shall be recorded: (1) the material identification, (2) the mean level of the measured property, (3) the repeatability standard deviation, *Sr*, (4) the repeatability, *r*, (in measurement units), (5) the relative repeatability, (*r*), in percent of the mean level, (6) the reproducibility standard deviation, *SR*, (7) the reproducibility, *R*, in measurement units, (8) the relative reproducibility, (*R*), in percent of the mean level, and (9) the number of laboratories in the final database as used to evaluate precision.

12.1.2 If there are no outliers, the value for item (9) in **12.1.1** is the number of laboratories for the original database. If outliers are found and Option 1 deletion is used, the number will be less than the number for the original database. If Option 2 outlier replacement is chosen, the number of laboratories

did not have outliers replaced, should be indicated in this column with a parentheses around the number. Explain this with a footnote to the table.

12.1.3 If the mean value of a measured parameter for any material is very close to zero, the relative precision, (r) and (R), will be very large. For these circumstances omit the relative expressions of precision from a [Table 6](#) format. The precision table should also contain, as footnotes, an explanation of the table symbols used.

12.1.4 The calculation of pooled or average values is recommended only if the values for r and R are roughly equal for all materials. When there is a substantial difference in precision among several materials, caution should be exercised in the interpretation of a pooled or average precision. It may have very little meaningful value or applicability.

12.1.5 When there is a substantial difference in precision among the materials, the use of a pooled value may give a false impression of the overall precision. It would be better to direct the user to select a material from the table that is closest in mean value to a specific material under consideration to determine the expected precision instead of using the pooled value. Ultimately, it is the responsibility of those conducting the ITP to determine what constitutes a substantial difference among materials and the reporting of a pooled value.

12.2 *General Precision Section or Clause*—The results of the precision evaluation should be displayed in a section or clause in the test method standard entitled “Precision and Bias.” The concept of bias is discussed in [Annex A2](#). The one or more paragraphs or sub-clauses should contain information on the following issues concerning the ITP and the evaluated precision.

12.2.1 A statement that the precision ITP was conducted in accordance with Practice D4483 (the latest revision year designation), and the year the ITP was conducted. A statement that the reader should refer to Practice D4483 for terminology and other details on the precision evaluation.

12.2.2 A caveat statement that the precision as evaluated by the ITP may not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of the precision evaluation actually apply to the products or materials tested.

12.2.3 A statement giving (1) category of the precision, that is, General Precision or Special Precision (Carbon Black), (2) the type of precision, Type 1 or Type 2, (3) the number, p , of laboratories participating in the ITP, (4) the number, q , and description of the materials (or target materials) used, (5) the number of within-laboratory replicates, n , (6) the time span for the repeatability or within-laboratory replicates, (hours, days), (7) the definition of a test result (average, median of x number of determinations or individual measurements), (8) the option chosen for outlier treatment, deletion, or replacement, and (9) any unusual features of the ITP.

12.2.4 A table of precision results as set forth in [12.1](#) should be part of the clause. Ensure that the table (inserted into the test method standard in [Table 6](#) format) gives the final number of laboratories that remained after outlier deletion or replacement. Some comments on the outcome of the results should be given.

12.2.5 Generic statements on repeatability and reproducibility shall be part of the precision clause using the recommended text as set forth as follows. A95 % confidence level (or $p = 0.05$) applies to these statements where Table xx designates the final table as inserted into the test method.

12.2.5.1 *Repeatability*—The repeatability, or local domain precision, of this test method has been established by the values found in Table xx, for each of the materials as listed in the table. If calculated, pooled repeatability values are also listed in the table. Two single test results (obtained by the proper use of this practice) that differ by more than the tabulated values for r , in measurement units, and if listed, (r), in percent, shall be considered as suspect, that is, to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.

12.2.5.2 *Reproducibility*—The reproducibility, or global domain precision, of this test method has been established by the values found in Table xx, for each of the materials as listed in the table. If calculated, pooled reproducibility values are also listed in the table. Two single test results obtained in different laboratories (by the proper use of this practice) that differ by more than the tabulated values for R , in measurement units, and if listed, (R), in percent, shall be considered as suspect, that is, to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.

12.2.6 Bias is defined in [A1.2.5](#) in terms of *bias deviation*, a deviation for a measured value from a true or reference value. Bias is not addressed in this practice, since for essentially all the test methods that will be evaluated for precision, the evaluation of bias is not possible because no reference or true value exists or may be determined. For all such test methods, a statement should be included as the last item in the precision clause, stating that bias is not determined. Using the word bias as a synonym for bias deviation, the suggested statement text is as follows.

12.2.6.1 *Bias*—Bias is the difference between a test value and a reference or true value. Reference values do not exist for this test method, therefore bias cannot be determined.

12.3 *Special Precision Table*—The Special Precision table shall conform to the rules for General Precision.

12.3.1 If the mean value of a measured parameter for any material is very close to zero, the relative precision, (r) and (R), will be very large. For these circumstances omit the relative expressions of precision from a [Table 6](#) format.

12.4 *Special Precision Section or Clause*—The expression for Special Precision should in general follow the rules for General Precision ([12.2.1 – 12.2.5](#)) including the recommended text in [12.2.5.1](#) and [12.2.5.2](#) taking into account the differing repeatability and reproducibility procedures as set forth in [Tables 4 and 5](#). State if there are substantial reasons for a differing mode of expression.

13. Report for Precision Evaluation ITP

13.1 A full report of the precision evaluation shall be prepared for any ITP. This is a full comprehensive report of all ITP details, not the report that each participating laboratory prepares and returns as part of the ITP. The report should

contain information on the details of the organization and execution of the program as follows:

13.1.1 Identify the organization committee, where located, coordinator, and date of ITP,

13.1.2 Category of precision, General Precision, or Special Precision,

13.1.3 Type of precision, Type 1 or Type 2,

13.1.4 Number of laboratories, p , and their names without connection to ITP laboratory number,

13.1.5 Number and description of materials or target materials, q ,

13.1.6 Definition of a test result, number of replicates, n , and time span for repeatability,

13.1.7 Information on technicians conducting the testing, any special details,

13.1.8 Details on preparation of materials, how homogeneity is documented,

13.1.9 Details on packaging and delivery of materials to all ITP participants,

13.1.10 Copies of all ITP Data Reports from each participating laboratory,

13.1.11 ITP analysis report, with all tables as designated in **Annex A4**, full description of all analysis steps, options chosen for outlier rejection, and other required comments,

13.1.12 Table of precision results, comments on outcome, and

13.1.13 Draft of precision section for the test method.

14. Keywords

14.1 general precision; interlaboratory test program; ITP; precision; repeatability; reproducibility; special precision

ANNEXES

(Mandatory Information)

A1. DEFINITIONS FOR SELECTED TERMS CONCERNED WITH PRECISION AND TESTING

A1.1 General Background

A1.1.1 This annex gives comprehensive definitions drafted to contain substantial information content with emphasis on basic concepts. Some ancillary definitions are also given that may promote a better understanding of precision. The word *uncertainty* is used in some of the following definitions in a sense that implies the typical everyday meaning, that is, *a sense of doubt*. The more specific statistical or measurement term *uncertainty* is defined in **A1.2.8.1**. The definitions as presented in Section 3 of this practice (Terminology) should be understood in using this annex.

A1.2 Basic Statistical Definitions

A1.2.1 *variation, n*—the existence of deviations (differences) among measured element values for repeated independent tests (observations) for a particular class of elements; generated by perturbations produced by one or more *system-of-causes*.

A1.2.1.1 *Discussion*—Deviations are produced by some group of factors or causes, acting within a certain domain that jointly influence the independent measurement or observation output. This is called a *variation system-of-causes*. Typical *system-of-causes* are the unavoidable fluctuations in temperature, humidity, operator technique, fidelity of calibration, and so forth, in a controlled testing domain.

A1.2.1.2 *production variation, n*—variation in properties due to one or more deviation *system-of-causes* that are (1) inherent in the process that generates a particular material or class of elements, or (2) inherent in the storage or conditioning prior to testing, or both, after such generating processes are complete.

A1.2.1.3 *measurement variation, n*—variation due to one or more deviation *system-of-causes* inherent in the operation of

instruments or machines that evaluate certain properties for a material or class of elements, in a defined testing domain.

A1.2.2 *distribution, n*—the characteristic dispersion (scattering) pattern of independent element values generated by one or more variation *system-of-causes*; defined by the range (maximum to minimum) and the ordering of the element values based on their frequency of occurrence.

A1.2.2.1 *Discussion*—In a graphical sense, ordering is related to the number (or frequency) of element values in any small range (or point) along the element value axis. The independent values may be arranged along this axis in one of three general patterns; (1) a unimodal or symmetrical dispersion around a highest frequency central value with a decreased frequency of occurrence the greater their plus and minus difference from the central value (2) dispersed in a uniform frequency across a value range, or (3) asymmetrically dispersed above and below a central or other special value. The concept of a distribution usually applies to data values rather than physical elements although it may apply to both. Both production and measurement variation may contribute to the total variation. A distribution may be characterized by a mathematical equation called the probability density function that describes the frequency of occurrence of any value, with parameters that define the location and shape of the distribution.

A1.2.3 *normal distribution, n*—a distribution that is symmetric (unimodal) and bell-shaped; it may be defined by a unique probability density function that contains two parameters; the central value or mean and the standard deviation.

A1.2.3.1 *Discussion*—Most of the data obtained from testing, with certain exceptions, will have a unimodal distribution that is normal or approximates a normal distribution. The means of n values ($n = \text{or} > 4$) will have a normal

distribution even when the source or individual value distribution ($n = 1$) is not normal.

A1.2.4 population, n —the distribution (collection) of independently distributed elements that constitute the totality for a defined system; it may refer to any one of the following: (1) one or several elements, (2) a finite but large number of elements, or (3) a hypothetical infinite number of elements.

A1.2.4.1 Discussion—The preceding definition is for a physical population or a collection of elements. An additional understanding is data population, the collection of all data values produced by testing (or observing) the physical population (or parts thereof). All three population interpretations imply that the elements are generated by some identifiable process and have a rough approximation available for a property range. Testing programs, defined by the testing domain and the sampling program, may vary from a very limited focus of attention, Interpretation 1, to a broad focus of attention, Interpretation 3.

A1.2.5 random deviation, n —a difference (plus or minus) between an independently measured or observed value and a known (or estimated) mean or an accepted reference value; the differences vary in magnitude, usually have a normal (unimodal) distribution, and for a long run series of replicates in a stable domain, the sum and mean of the differences is zero.

A1.2.5.1 Discussion—Increased replication reduces the random uncertainty of a mean (but not the total uncertainty which may contain a bias component, see bias deviation definition as follows) and provides a more reliable estimate of the true or reference mean property. The definition of *long run* depends on the goal of the testing. For routine testing, the number of replicates, n , may be of the order of 10. For critical testing, n may be two or three times this value. For an intermediate number of replications, the mean of the random deviations may be reduced to a small value that may be considered to be zero, depending on the scope of the testing.

A1.2.6 bias deviation, n —a constant difference (plus or minus), absent any random deviations, between an independently measured or observed element value and the true or accepted reference value for a defined domain.

A1.2.6.1 Discussion—A bias deviation is a systematic or offset difference produced by some system perturbation. For some domains the offset affects all measurements equally; for others the offset may vary with the magnitude of the measured value. When a reference value is known, the bias deviation may be evaluated by eliminating (or reducing to a negligible value) the effect of random variation by a long-run series of measurements. When the test domain is altered, the magnitude (and less likely the sign) of the bias deviation may change. Any system may have more than one source for bias, and bias

deviations, unlike random deviations, do not sum to zero. The word bias is frequently used as a synonym for bias deviation.

A1.2.7 Although accuracy and trueness are not evaluated in this practice, their definitions are given here to provide additional background for a better understanding of their relationship to precision. In some of the definitions to follow, the term *figure of merit* is used. A high figure of merit is an indication of high quality or a high level of goodness of the measurement system for a given parameter of the system.

A1.2.7.1 accuracy, n —a test characteristic proportional to the inverse of the difference between an individual test value and the true or reference mean value for some class of elements.

A1.2.7.2 Discussion—When the absolute difference is small the inverse is large or high and the testing is said to have *high accuracy*. The observed difference is influenced by both random and bias deviations when both types of deviations exist.

A1.2.7.3 trueness, n —a test characteristic proportional to the inverse of the difference between the long-run estimated mean (for high n) and the true or reference mean value for some class of elements.

A1.2.7.4 Discussion—Since the estimated mean is a long-run (high n) estimate, the random deviations sum to approximately zero and the influence of random deviations is substantially reduced or eliminated. The observed difference is influenced by the sum of the bias terms only. Thus trueness is a testing concept that is intended to evaluate bias.

A1.2.8 As previously noted, the concept of uncertainty needs some attention. The definition given as follows is a definition that attempts to capture the general nature of the concept. As the definition and discussion indicate, uncertainty is local, and precision is global. It has been defined equivalently, but using different words, by a number of organizations addressing this concept.

A1.2.8.1 uncertainty, n —a test characteristic for a local domain; it is the magnitude of the difference between the measured (observed) element value and an accepted reference value and includes both random and bias deviations.

A1.2.8.2 Discussion—The word Uncertainty is capitalized in the use as defined in [A1.2.8.1](#) to distinguish it from the ordinary use of the word. As indicated, *goodness* or *merit* and uncertainty (doubt about the measurement), are inversely related. Uncertainty is a characteristic of a local testing domain; each local domain for any defined test, may have a different uncertainty value. Precision (both repeatability and reproducibility) is a characteristic of a global testing domain; the precision values obtained in any ITP are intended for universal application, that is, to a number of laboratories as a group.

A2. STATISTICAL MODEL FOR INTERLABORATORY TESTING PROGRAMS

A2.1 Introduction

A2.1.1 Although this practice does not address the evaluation of bias or accuracy, it is important that the influence of bias in interlaboratory testing be well understood. This annex provides some background on the influence of random and bias deviations by the use of a statistical model for interlaboratory testing.

A2.1.2 In the real world, all measurements are perturbed by a *system-of-causes* that produces test deviations or error. Typical cause systems are fluctuations in atmospheric pressure, temperature, humidity, attention of test operators to the details of a test, and so forth. There are two general *deviation or variation categories* for any specified domain. These are defined by the character and source of *deviations* that perturb the testing or observed values compared to what would be obtained under ideal conditions. Two major categories of variation are:

A2.1.2.1 *Production Variation*—Variation in properties due to one or more deviation *system-of-causes* that are inherent in the process that generates a particular material or class of elements or inherent in the storage or conditioning (prior to testing), or both, after such generating processes are complete.

A2.1.2.2 *Measurement Variation*—Variation due to one or more deviation *system-of-causes* inherent in the operation of instruments that evaluate certain properties for a material or class of elements, in a defined testing domain.

A2.1.3 Within each category, deviations may be of two different types, (1) random, plus and minus differences about some central (true) value or (2) bias or systematic differences. Both types may occur in either category. The domain of the testing program determines the system-of-causes. These *cause systems* can vary from simple to complex. The production process is broadly defined; it can be (1) the ordinary operation of a manufacturing facility, (2) a naturally occurring and ongoing process, or (3) some smaller scale processing that generates a material or class of objects for testing. The discussion applies to both objects and materials.

A2.1.4 Objects may be discrete manufactured items or test pieces generated by a particular preparation process. Materials may be tested in a direct manner, such as the tensile stress or modulus of a polymer or in an indirect manner, such as the quality of a carbon black or other additive in a standard formulation by a performance-in-rubber test. When performance-in-rubber testing is conducted, the designation *target material* is used for the material, since a composite containing the target material is tested, not the material itself. This composite testing may involve objects or test specimens for the measurement process. These testing concepts, target material, and Type 1 and Type 2 precision are defined and discussed in 5.1.3 – 5.1.5 of this practice.

A2.2 General Model

A2.2.1 For any testing domain, each measurement, y_i , can be represented as a linear additive combination of fixed or

variable (mathematical) terms as indicated by Eq A2.1. Each of these terms is an individual deviation or component of variation and the sum of all component deviations is equal to the total variation observed in the individual measurement. It is assumed that all participants test a selected number of classes of objects or different materials drawn from a common lot, employ the same type of apparatus, use skilled operators, and conduct testing according to a test method standard, in one or more typical laboratories or test locations.

$$y_i = \mu_o + \mu_j + \Sigma(b) + \Sigma(e) + \Sigma(B) + \Sigma(E) \quad (\text{A2.1})$$

where:

- y_i = measurement value, at time (i), using specified equipment and operators, at one laboratory or location (among a total of p laboratories),
- μ_o = constant term (mean value), that dictates the general magnitude of the measured parameter for the particular test,
- μ_j = constant term (mean value), unique to material or object class (j),
- $\Sigma(b)$ = (algebraic) sum of the number of component *bias deviations* in the *process* that produced material or object class (j),
- $\Sigma(e)$ = (algebraic) sum of the number of component *random deviations* in the *process* that produced material or object class (j),
- $\Sigma(B)$ = (algebraic) sum of the number of component *bias deviations*, for measurement (i), generated by the *measurement system*, and
- $\Sigma(E)$ = (algebraic) sum of the number of component *random deviations*, for measurement (i), generated by the *measurement system*.

A2.2.2 An alternative approach is to use a single μ term, that is, μ_r , in place of the two terms $\mu_o + \mu_j$, where both of the characteristics defined by μ_o and μ_j are contained in the single term. Eq A2.1 indicates that there are three groups that contribute to the value of y_i , (1) constant terms (population mean values), (2) bias deviations, and (3) random deviations.

A2.3 Specific Model Format

A2.3.1 A more useful format is obtained when Eq A2.1 is expressed in the format of Eq A2.2 where the generic summations are replaced by a series of typical individual terms or components appropriate to interlaboratory testing on a number of different object classes or materials, over a particular time period.

$$y_i = \mu_o + \mu_j + \Sigma(b) + \Sigma(e) + B_L + B_M + B_{Op} + B_G + E_B + E_w \quad (\text{A2.2})$$

where:

- B_L = bias deviation term unique to one laboratory or local domain,
- B_M = bias deviation term unique to the specific instrument or machine,

- B_{OP} = bias deviation term unique to the operator(s) conducting the test,
 B_G = generic bias deviation term; to account for other bias factors,
 E_B = between laboratory (global domain) random deviation term, and
 E_W = within laboratory (local domain) random deviation term.

The B_L term is exclusively a between laboratory bias, the terms B_M , B_{OP} , and B_G may be either between laboratory or within laboratory components depending on the scope of the testing, that is, whether these components are part of the chosen within laboratory repeatability testing. The between laboratory random deviation term, E_B , is usually the sum of a number of subcomponents that represent typical sources of variation between laboratories.

$$E_B = E_L + E_M + E_{OP} + E_G \quad (\text{A2.3})$$

where:

- E_L = random deviation term attributable to a laboratory or location,
 E_M = random deviation in the use of the specific instrument or machine,
 E_{OP} = random deviation inherent in the operator's technique, and
 E_G = generic random deviation term; to account for other random factors.

The within laboratory random deviation term, E_W , may also be the sum of a number of subcomponents due to varying operator(s) technique, different instruments or machines of a given design, if such factors are part of the testing domain, in addition to the time period for repeatability measurements. Typical B_G or E_G testing perturbations, may be bias and random components due to temperature, long-term time period (time of the year), and so forth.

A2.3.2 $\mu_o + \mu_j$ Terms—In the absence of bias or random deviations of any kind, a number of materials or object classes would have individual measured test values given by the sum of the two terms, $\mu_o + \mu_j$. The term μ_o uniquely characterizes the general magnitude of the measured parameter. Each material or object class would be characterized by the value of μ_j , which would produce a varying value for the sum $[\mu_o + \mu_j]$ across the number of materials or object classes in the test program and the sum would be the *true* or unperturbed test value.

A2.3.3 Production Terms $\Sigma(b) + \Sigma(e)$ —There will always be some bias and random variation in the materials or object classes produced by the process that generates them. These usually unknown number of bias and random variations are designated by $\Sigma(b) + \Sigma(e)$. For testing in general, appropriate sampling and replication plans will reduce the random components to some selected level. However, increased sampling and replication does not reduce bias components; such action merely enhances the fidelity of the evaluated magnitude of these effects, if reference materials are available. Reducing or removing bias requires (1) special test programs to discover and eliminate the causes or (2) a documented correction procedure that eliminates the bias. For most precision ITPs,

special care is required to ensure some minimal level of variation in the lots of materials selected for the program, that is, to make them as homogeneous as possible. Any residual production variance adds to the measurement variance or basic precision as evaluated by the ITP.

A2.3.4 Measurement Bias Terms—Bias deviations may be divided into two classes: *local* or *global*. A local bias is a fixed offset that applies to certain specific conditions within a larger testing domain, for example, a single test machine or laboratory among many machines or laboratories. Such biases are the principle component of between laboratory differences, that is, one laboratory or test instrument is always low or high in comparison to other laboratories or instruments.

A2.3.4.1 When the domain consists of a large number of machines or laboratories, the local biases may be *variable* (plus or minus) deviations unique to each of these machines or laboratories and the distribution may be either random with a zero mean in the long run or a nonrandom finite distribution with a nonzero mean. A global bias is either (1) a fixed offset that applies across the whole testing domain and is unique to a generic condition that is common within the domain or (2) an inherent deviation in a particular design of a test apparatus. Although more than one global bias may exist, global biases usually are not considered to have a distributional character.

A2.3.4.2 Bias terms that are fixed under one *system-of-causes* may be variable under another *system-of-causes* and vice versa. As an example, consider the bias terms B_L and B_M which apply to most types of testing. For a *particular laboratory* (with one test machine) both of these bias terms would be constant or fixed. For a *number of test machines*, all of the same design in a given laboratory, B_L would be fixed but B_M would be variable, each machine potentially having a unique value. For a domain consisting of a *number of typical laboratories*, each with one machine, both B_L and B_M would be variable for the domain, but both B_L and B_M would be fixed or constant for the *system-of-causes* in each laboratory. One or more generic bias terms, B_G , may be present in any test domain. These represent unique bias effects not attributable to test machines, operators, or laboratories.

A2.3.5 Measurement Random Terms—These terms are deviations or components that are frequently called *error*. Random deviations are plus or minus values that have an expected mean of zero over the long run. As indicated in [Eq A2.2](#) there are three potential sources of random variations: laboratories, test machines, and operators, in addition to the special case where another source, a generic source, is an important component. The distribution of these terms is assumed to be approximately normal but in practice it is usually sufficient if the distribution is unimodal. The value of each random term influences the measured y_i value on an individual measurement basis. However, in the long run, when y_i values are averaged over a substantial number of measurements, the influence of the random terms may be greatly diminished or eliminated depending on the sampling and replication plan, since in the long run each term averages out to zero (or approximately zero) and the *mean* y_i is essentially unperturbed.

A2.3.6 New Term, $M(j)$ —With highly replicated testing programs (both production and test measurement programs)