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Standard Practice for ~~Determining~~ Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries[1]

This standard is issued under the fixed designation D 4483; 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 E 691; a generic standard that presents the fundamental statistical approach and calculation algorithms for evaluating repeatability and reproducibility precision. However, certain parts of Practice E 691 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 D 4483, which has been used for precision evaluation by Committee D11 since 1985. The basic Practice D 4483 precision calculation algorithms, the same as in Practice E 691, are unchanged. This new revised Practice D 4483, 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 presents guidelines for ~~preparing clear, meaningful~~ evaluating precision ~~statements for test method standards under the jurisdiction of ASTM Committee D-11 on Rubber Testing~~ and ~~for ASTM Committee D-24 on Carbon Black Testing. It explains~~ serves as the ~~potential uses~~ governing practice for ~~standard test methods and gives the requirements for~~ interlaboratory test programs ~~needed in~~ (ITP) used to evaluate precision ~~formulation, the calculation algorithms~~ for ~~precision, and the format for expressing precision.~~

~~1.2 Test~~ test methods ~~are~~ as used in ~~many ways in technology. This broad usage requires careful consideration in assessing their general precision and, where pertinent, their accuracy. Clearly outlining~~ the ~~objectives~~ rubber manufacturing and the carbon black industries. This practice uses the basic one way analysis of variance calculation algorithms of Practice E 691. 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 ~~prior to the determination~~ that have test results expressed in terms of a quantitative

[1] This practice is under the jurisdiction of ASTM Committee D~~-~~11 on Rubber and is the direct responsibility of Subcommittee D11.16 on Application of Statistical Methods.
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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 ~~essential. A critical requirement~~ 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~~v~~ designated as *Special Precision*, that ap~~m~~plies to carbon black testing. Both of ~~a standardized nomenclature system. This practice addresses~~ these procedures use the same uniform level experimental design and ~~other issues important in evaluating~~ the Mandel *h* and *k* statistics to review the precision database for potential outliers. However, t~~m~~hey use 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.

1.3~~4~~ This practice is divided into the following sections:

Example of General Precision Evaluation—Mooney Viscosity Testing

Annex A6 An Example of Precision Calculation—Mooney Viscosity

1.45 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 to 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]

D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)[3]

D 6600 Practice for Evaluating Test Sensitivity for Rubber Test Methods[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

2.2 *ISO Standard:*

ISO 5725 Precision 289 Determination of Test Methods—Determination Viscosity of Repeatability Natural and Reproducibility Synthetic Rubbers by Interlaboratory Tests[5] the Shearing Disk Viscometer

3. Terminology

3.1 *Definitions*

3.1 A number of Terms Specific specialized terms or definitions are defined in a systematic sequential order, from simple terms to complex terms. This Standard: —This section gives descriptions for approach allows the important simple terms to be used in this practice. However, Section 5 should be reviewed simultaneously with this section for a more complete understanding of the need for certain terms.

NOTE—The descriptions definition of terms are given in a logical development sequence rather than alphabetical order.

3.1.1 *accuracy, bias, precision*—to set the stage for the more specific terminology to follow, three general terms are given. Although this practice does complex terms; it generates unambiguous definitions. Thus the definitions do not address appear in the usual alphabetical ssequence.

3.1.1 This terminology section contains explanatory notes for many of accuracy or bias, these terms are presented to clearly show the difference between these two and precision.

3.1.2 *accuracy*—the degree of correspondence between an average measured value and an accepted reference or standard value for definitions as well as discussion on the material or phenomenon under test.

3.1.2.1 *Discussion*—The reference value may be established by theory, by reference to an *accepted* standard, to another test method, or in connection between some cases the average that could be obtained by applying the test method to all of the sampling units comprising a lot of the material.

3.1.3 *bias*—the difference between the average measured test result terms and the accepted reference value.

3.1.3.1 *Discussion*—High accuracy implies a small or negligible bias and when bias exists increased testing does not increase accuracy but merely enhances various ways the knowledge of the degree of bias.

3.1.4 *precision*—a measurement concept that expresses the ability to generate test results that agree with each other terms are used in absolute magnitude.

3.1.4.1 *Discussion*—The degree of agreement is normally measured inversely by the standard deviation; high testing and precision corresponds to evaluation. For special emphasis, a low (small) standard deviation.

3.1.4.2 *Discussion*—High precision may exist simultaneously with a large bias or poor accuracy.

3.2 The following specific descriptions are given for few terms that will be required to accommodate Committee D-11 and Committee D-24 test methods. The three time scales of repeatability and reproducibility discussed in 5.2 are reduced to two for

[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] *Annual Book of ASTM Standards*, Vol 09.01.

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

[5] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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

defined in the sake main text of simplification. Two preliminary terms, which define the "numbers" produced by test methods, this practice where certain precision concepts are r discusseqd.

3.1.2 Annex A1 is included as part of this practiced. T with two objectives: (*1*) Anne ax A1 pre gsents 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 *determination*element, *n*—the application of the complete test procedure to one test piece, specimen, entity that is tested or test portion observed, to produce one numerical (test) measured value to evaluate a property or characteristic; it may be a single object among a group of objects (tedst pieces, and so formth) or an average increment or portion of a mass (or volumed) of a material.

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

3.2.2 *test result*element class (or class of elements), *n*—the average (mean category or median) of descriptive name for a specified number group of d elements that have a common origin or have nominally identical properties;.

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

3.2.3 *repeatability, r*—an established value, belowtesting domain, *n*—the location and operational conditions under which the absolute difference between two "within-laboratory" a test results may be expected to lie, with is conducted; it includes a specified probability. 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.

3.2.3.1 *Discussion*—The two test results are obtained with thelocal testing domain, *same* method on nominally identical test materials under the *same* conditions (same operator, apparatus, laboratory, and specified time period), and in the absence*n*—a domain comprised of other indications the probability is 95 %. one location or laboratory as typically used for quality control and internal development or evaluation programs.

3.2.3.2 *Discussion*—The "established value" may also be called a" critical difference." 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.

3.2.4 *reproducibility, R*—an established value, below which the absolute difference between two "between-laboratory" test results may be expected to lie, withtest result, *n*—the value of a characteristic obtained by carrying out a specified probability. test method.

3.2.4.1 *Discussion*—The two test results are obtained with the *same* method on nominally identical test materials under *different* conditions (different laboratories, operators, apparatus, and in method should specify that one or a specified time period), number of individual measurements, determinations, or observations be made and their average or another appropriate function (median or other) be reported as the absence of other indications the probability is 95 %.

3.2.4.2 *Discussion*—The essential characteristic of reproducibility is the different laboratories in which the testing is conducted. test result.

3.2.5 *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 repeat-ed term)sts, on each material.

3.3 *Material and Sampling Terms:*

3.3.1 *material, n*—a repeatability estimate obtained under a short specific entity or element class to bre tested; it usually efxists in bulk forme (solid, powderi, or liquid).

3.3.1.1 *Discussion*—The time period—Material is used as a generic term to describe the *class of elements* that is tested, that is, a material may be minutes, hours, or days a rubber, a rubber compound, a carbon black, a rubber chemical, and needs to so forth. A material may or may not be homogeneous. In peroduct testifng the term material may be used f to describe the *class of elements* or type of rubber productchts such as O-rings, hose astsemblies, mothor mounts, and so forth. See also 5.1.4.1.

3.3.2 *repeatability (long-term), r*lot, *n*—a repeatability estimate obtained over specified mass or volume of material or number of objects; usually generated by an identifiable process, frequently with a long time period. recognized composition or property range.

3.3.2.1 *Discussion*—The time period—A lot may be weeks or months and needs to be specified for each test method.

3.2.6.2 *Discussion*—Events that influence long-term repeatability are the use of different operators, environmental factors (such as seasonal variations generated by a common production (or other natural) process in temperature, humidity, etc.), a restricted time period and the recalibration usually consists of a finite size or adjustment, or both, number. A lot may be a fractional part of a population (Inteqrpretation 2 of population, see Annex A1). A recognized pmroperty range implies that some rough approximation is available.

3.3.3 *reproducibility (short-term), R*—a reproducibility estimate obtained oversample (physical), *n*—the number of elements or the specified mass of a material, shelected according to a particular procedure, used to evaluate material, lot, or population characteriodstics.

3.3.3.1 *Discussion*—The time period may term *sample* should not be minutes, hours, used as a synonym for *material*, see 3.3.1,

or *target material*, see 5.1.4.1. Ideally severanl *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 specified taken for each test method. testing. See 3.3.4.

3.3.4 *reproducibility (long-term), R*—a reproducibility estimatesample (data), *n*—the number of test or observation values (*n* = 1, 2, 3, and so forth), obtained frovm (one or more) physical samples, by the application of a long period specific test (observation) method.

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

3.23.85.1 *Discussion*—The time period may be weeks—A test sample is usually a mass or volume that is some small fractional parth of a bulk material.

3.3.6 *test specimen, n*—an object (appropriately shaped and needs to be specified prepared) taken from a sample for each test method.

3.2.8.2 physical or mechanical testing.

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

3.3.7 *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 long-term reproducibility are different operators, environmental factors (such as seasonal variations of any selected measurement on any other measurement in temperature, humidity, etc.), and the recalibration or adjustment, or both, set.

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

3.4 *Statistical Terms Relating to Precision:*

3.4.1 *repeatability (Type 1)*—Type 1replicate, *r*,*n*—one of a repeatability estimate obtained in an interlaboratory program where the material(s) distributed to all laboratories is (are) in selected number of independent fractional parts or independent number of elements, taken from a prepared state ready for testing (with perhaps some minimal preparation steps required), such as Class I sample; each fractional part or II. See 5.2.1.

3.2.10 *reproducibility (Type 1)*—Type 1 element is tested.

3.4.1.1 *Discussion*—The word *R*,*replicate* refers to a reproducibility estimate obtained physical object (element). It can also be used in an interlaboratory program reference to a data set, where the material(s) distributed it refers to all laboratories is (are) in one of a prepared state ready number of independent data values.

3.4.2 *true value, n*—the measured or observed value for an element, that would be obtained for a testing (with perhaps some minimal preparation steps required), such as Class I domain in the absence of errors, deviations, or II. See 5.2.1.

3.2.11 *repeatability (Type 2)*—Type 2 variations of any sort, that is, where there is no variation *r*, a repeatability estimate*system-of-causes*.

3.4.2.1 *Discussion*—The true value is also defined as the mean that would be obtained in an interlaboratory program where some or by testing all members of any population (see population in Annex A1). Typical *systems-of-causes* are the materials(s) distributed to all laboratories require unavoidable fluctuations in temperature, humidity, operator technique, fidelity of calibration, and so forth, in a spe controlled testifng domain.

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 series of operations, exceedingly difficult to produce obtain a true value. Such values are most often assigned on the final test samples, portions, basis of comprehensive testing programs sanctioned by a local or test pieces prior to applying the test method to the material(s) global task group, a standardization organization, or item(s) under test, a committee devoted to produce one test result (value), such as Class III.

3.2.12 *reproducibility (Type 2)*—Type 2 domestic or international metrology.

3.4.4 *estimated (true or reference) mean, R,* a reproducibility estimate*n*— the mean obtained in an interlaboratory program where some or all of on the material(s) distributed to all laboratories require a specified operation or series basis of operations, to produce *n* independent replicate measurements; the final test samples, portions, or test pieces prior to applying greater *n* the test method better the approximation to the material(s) true or item(s) under test, to produce one test result (value), such as Class III.

3.2.13 *relative repeatability and reproducibility*—It reference mean, provided there is often appropriate to express repeatability and reproducibility on a relative basis, as a percent of a certain mean value. This is analogous to a coefficient of variation. Such expression is important when no systematic deviation or bias.

3.4.4.1 *Discussion*—The words *rmean and R estimated mean vary with the are frequent synonyms for estimated (true or reference) mean level of the property being measured. Relative values*. The value for *rn in typical routine testing programs is of the order 1 to 10. When bias exists, the estimated (true or reference) mean so obtained estimates [μ + Σ Bi], where μ = true or reference mean and R Σ Bi = algebraic sum of all bias deviation terms. Therefore, if bias exists and is unknown in magnitude, the true value or μ cannot be unambiguously expressed as percentages alongside the actual measured values approximated despite increased replication. See random and bias deviations in usual test result units because A1.2.5 and A1.2.6. See also Annex A2.*

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

3.5 In some test methods have" percent" as their units, for example, % Cu, % elongation. To avoid this ambiguity of the

following ~~symbols are defined by~~ definitions, the ~~use~~ term *figure* ~~*of the parentheses.*~~

~~3.2.14 *(r)*—repeatability estimate expressed as percentage~~ merit is used. A high figure of ~~the mean~~ merit is an indication of ~~the property~~ high quality or a high level of excellence or goodness for ~~which~~ the ~~estimate was obtained.~~

~~3.2.15 *(R)*—reproducibility estimate expressed as percentage~~ measurement or test domain, or both. The term *figure* ~~*of the mean merit*~~ applies to a number of ~~the property for which the estimate was obtained.~~

~~3.2.16 *acceptance difference, (duplicate determinations), AD_2*—an established value, below which the difference between two "within-laboratory"~~ test method characteristics: precision, sensitivity, bias, useful range, ruggedness and ease of operation, and rapid or automated operation.

3.5.1 *precision, ~~determinations~~n*—a ~~may be expected~~ *figure of merit* concept, it is proportional to the inverse of the dispersion of independent replicate (test or observed) values, ~~w~~ as estimated by the standard deviation, for a specified ~~probability.~~

~~3.2.16.1~~ 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 ~~*test determinations*~~*figure of merit* ~~*are obtained at*~~ *testing concepts. An additional one is test sensitivity; the* ~~*"same" time (side-by-side) with identical*~~ *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 D 6600 for more details on test* ~~*material, operators, and apparatus, and*~~ *sensitivity.*

3.5.2 *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 ~~absence~~ standard deviation; this interval should (on basis of a 95 % probabilit~~h~~y) e~~r~~ incompass duplicate i~~on~~dependent test results obtained under the ~~probability is 95 %.~~

~~3.2.16.2~~ defined local testing domain.

3.5.2.1 *Discussion*—~~If~~—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 ~~calculated difference lies (below)~~ source. Repeatability may be dependent on the ~~acceptance difference,~~ magnitude or level of the ~~two values are accepted for averaging~~ measured property and ~~the average~~ is usually reported for particular property levels or materials or element classes (that determine the ~~*test result;* if the calculated difference exceeds the acceptance difference, additional determinations are made to produce acceptable data.~~

~~3.2.17 *acceptance difference (x determinations), AD_x*—an established value, below which the maximum range (maximum value-minimum value) of a specified number of determinations (within a given laboratory)~~ level). The repeatability time period may be ~~expected to lie, with a specified probability.~~

~~3.2.17.1~~ minutes, hours, or days depending on the goals and scope of the testing.

3.5.2.2 *Discussion*—~~T~~Although repeatability as defined in 3.5.2 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 ~~determinations~~ 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 ~~at the" same" time (side-by-side) with identical test material, operators,~~ in and for one laboratory.

3.5.3 *reproducibility, R, n*—the p~~a~~recision for a defined *global t~~t~~esting domain*, obtained by way of independent tests conducted in *N* laboratories (with *n* replicabtes each) on nominally identical elements, expressed in terms of ~~other indications the probability~~ an interval or range that is ~~95 %.~~

~~3.2.17.2~~ 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.

3.5.3.1 *Discussion*—~~f~~—Each laboratory in the ~~calculated maximum range lies within~~ global domain conducts *n* repeatability tests on a material (target material), and reproducibility is evaluated based on the ~~critical range~~ mean values for the *N* laboratories for that material or ~~below~~ element class. Reproducibility may also depend on the ~~acceptance difference, all~~ level of the ~~determinations are accepted~~ measured property or on the materials tested and it is also usually reported for ~~averaging~~ 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 ~~m~~ global testing domain.

3.5.3.2 *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.

3.5.4 *relative repeagtability, (r), n*—repeatability expressed in terms of an interval (a multiple of the standard deviation) that is ~~reported as~~ 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.

3.5.5 *relative reproducibility, (R), test result; if n—* reproducibility expressed in terms of an interval (a multiple of the maximum range exceeds 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 independenta test results (on percentage basis) each obtained in different laboratories forv a defined gl;obal testing domain.

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

4. Significance and Use

4.1 Tests are conducted using established (standardized) standard test methods to generate test data. Test data that are generated used to make technical and scientific decisions for commercial processes commercial, technical, and technical operations. scientific purposes. It follows that the precision of a particular test method is an important quality characteristic or figure of the merit for a test method and also of the a decision process that involves the data. Therefore all test methods should be evaluated for precision.

4.2 Any process.

4.2 An evaluation of the precision of a test method is normally conducted with a (*1*) some selected group of typical materials or items subjected to measurement. as typically used with that method and (*2*) with a group of volunteer laboratories that have experience with the test method. The evaluation therefore represents "a snapshot an *event in time" of the precision; the results are frequently unique to the materials, the participating laboratories, and the time period of* for the evaluation. A repeat of the entire test method for these materials and laboratories. Another ITP precision evaluation at a later time with somewhat different materials and participants may not give good or exact agreement even with any previous evaluation. This characteristic of the same materials with the same laboratories at a different time, may generate precision evaluation should be clearly understood when reviewing precision data results that differ from various programs the initial ITP.

4.3 Experience as indicated in Refs **(1-4)**[5] and at various time periods.

4.3 Although elsewhere has shown that the evaluation 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 labodratories in all tests. This usual outcome for precision many ITPs is an important quality characteristic addressed in this practice by the use of the method, the resulting precision parameters (*r, R*) have to be interpreted with caution if there three-step robust analysis procedures as described in Section 7.

4.4 Caution is any thought of urged in applying them across a broad range precision results of material a particular test method to product testing especially for consumer-producer product acceptance testing. acceptance. Product acceptancetesting protocols acceptance procedures should be developed on the basis of precision data obtained in special programs that are specific to the commercial products or items and to the laboratories of the interested parties in for this type of testing.

4.4 The application of this practice is limited to test methods 1) that have test results expressed in terms of quantitative continuous variable, and 2) that are fully developed and in routine use in a number of laboratories.

5. Precision Evaluation: General Precision and Specipal Precision

5.1 Th*General Precision*—Two praecision categories are described: General Precision and Special Precision. General Precision is pr discussed first and Special Precision is described in Section 11. General Preceision evaluation follows established procedures used in the rubber mmanufacturing industry over the past four decades. The evaluation is usually conducted using a balanced uniform level design ITP with three or more madterials sengt to each of the participating laboratories with tests conducted to generate an independent *test result* , on each of two (or more) test methods. It may seem overly complex days. The ITP database is reviewed for outliers by t mhe Mandel *h* and *k* consistency statistics by the procedures in Annex A3.

5.1.1 *Opthions for Outliers*—If no outliers are found, the oceriginal database is used to developy a narrow part table of precision results. If outliers are identified, there are two options b 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 uses. Therefore, make use laboratories. Issues such as the number of those portions replicate values on each test day or the number of technicians or operacticors used tho obtain a test result, or both, which are applicable and ignore those parts that do not directly apply.

5.1.1 Although characteristic of the terminology for repeatability and reproducibility particular test, are considered on a case-by-case basis by the ITP organizing committee. Outlier treatment is given discussed in Section 3 more detail in Annex A3 and Annex A5.

5.1.2 *Types of this practice, a general discussion is repeated here.*

5.1.1.1 Repeatability refers to Test Methods—The General Precision approach has been successfully used for the ability broad range of test methods characteristic of the rubber manufacturing industry; from simple physical or chemical *samebench type laboratory tests, conducted in a few minutes (hardness and pH tests) to obtain similar (test) results under certain specified conditions.*

5.1.1.2 Reproducibility refers 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 the ability of *different* laboratories to obtain similar a final calculated test results under certain specified conditions.

5.1.1.3 If test results closely agree, then good repeatability result or good reproducibility exists.

5.1.2 The performance index. For such complex tests, any realistic precision evaluation must of a test method does not of necessity characterize a test with regard to how sensitive it is include all of the procedural steps in measuring arriving at the test result, the basic datum used in propertcision analysis, and evaluation. The procedures required for gentral precision are described in Sections 8 to measure. 10.

5.1.3 *Types of General Precision may be good simply because*—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 method result. One important test of this type is insensitive to a *performance-in-rubber* test; the basic property. A concept called "test sensitivity" has been defined evaluation of various rubbers, reinforcement fillers, or other compounding materials in statistical literature as the ratio standardized formulations. The typical stress-strain evaluation of the responsiveness a selected lot of a specified rubber will require (*1*) an appropriate sample of the test measurement rubber, (*2*) a standardized formulation and mixing operation to finite variations in the basic property in question, 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 precision testing of the measurement. This practice does not address this issue.

5.1.3 Both repeatability lot to obtain the final test results for tensile stress (modulus), elongation, and reproducibility should be determined under tensile strength properties.

5.1.4 To permit realistic or typical laboratory conditions. If extraordinary care is exercised (extremely homogeneous materials) the resulting precision evaluation for the performance-in-rubber testing it is overly optimistic. Also as ordinarily determined, repeatability necessary that all the steps in the operation be replicated, from the raw materials to the final test result. Each of these steps has both a test apparatus variability as well as a material variability. The potential component of variance and the sum of these two all variance components establishes the repeatability as normally quoted.

5.2 *Interlaboratory Distribution Scheme (Test Pieces, Specimens, overall test variance and Materials)*:

5.2.1 A key concept that must be clearly understood when contemplating interlaboratory standard deviation. To address this, two types of precision are defined. The two types are characterized by the relationgship 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 matter 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 *what is distributedtarget material to the participating laboratories. The "what", two types of precision may be classified as follows: defined:*

5.1.5.1 *Class I—FullyType 1 Precision*—A precision evaluated directly for or on, a target material; fully prepared test pieces, specimens, (or pieces or test portions), requiring portions of the target material drawn from a homogeneous source are tested, with no further processing (preparation or adjustments) other operations required prior to testing (.

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

5.2.1.2 *Class II—Intermediate prepared materials*, that require some minimal processing prior to action by testing.

5.1.6.1 *Type 2 Precision*—A precision evaluated indirectly for a target material; the test machine (example—cured rubber-sheets that must have dumbbells cut from them target material is usually combined with subsequent gaging, prior to final stress-strain testing).

5.2.1.3 *Class III—Specified (quantities of) raw a number of homogeneous ancillary materials*, that must be processed into final samples, or specimens by to form a standardized procedure (example—rubber, curatives, carbon black, oils, composite material, and antioxidants that must be mixed, processing steps taken, cured sheets prepared, dumbbell test pieces cut and gaged prior to stress-strain testing).

5.2.2 The primary purpose on samples of an interlaboratory program dictates which scheme, Class I, II, or III, this, testing is selected. If conducted and the attention property response of the target material is on evaluated.

5.1.7 The properties of the apparatus or test machine(s) in composite material are directly related to the various laboratories, how well these agree when testing the supplied test specimens, then Class I quality or perhaps Class II (both Class I and Class II being quite similar) would be selected.

5.2.2.1 However, if it is the *total operational sequence properties of a test*, such as mixing, processing, curing, die-cutting, and gaging that is of interest, then Class III would be selected. Material distribution in accordance with Class III would frequently be called for in interlaboratory precision programs where producer-user acceptance testing of raw materials is of direct importance. the target material. An example would be carbon black or synthetic rubber.

5.2.2.2 In each case (Class I, II, or III) it is necessary that example: To evaluate the distribution quality of items or materials is made from a uniform source or lot, with grade of SBR, a nominally good uniformity or homogeneity.

5.2.3 The amount sample of "within-laboratory" preparation or processing, after arrival of the circulated items or material, increases in the order Class I, II, rubber, plus curatives, filler, antioxidants, and III. Analytical chemistry and other simple physical tests often require no or very little "within-laboratory" preparation upon arrival of so forth, are mixed, cured, test portions and, therefore, make use of pieces prepared, and the resulting compound tested for specified quality properties. It is possible that a Class I distribution scheme. Conversely, what may Type 1 precision program might be called actual conducted on test pieces or quasi-performance tests portions that require more complex "within-laboratory" preparation or some minimum processing and, therefore, require or other simple operations prior to actual testing. This is, in a Class III distribution. Performance implies the

attainment of a certain minimal strict sense, an intermediate level of some critical property, tensile strength, or modulus, in precision. However, to avoid unnecessary complications, this will be designated as a standard compound for a raw material like Type 1 precision.

5.2 *Special Precision*—The carbon black or industry has adopted a synthetic rubber.

5.2.4 The type slightly revised precision evaluation procedure designated as *Special Precision*. The number of test method will often indicate the scheme replicates in each cell of interlaboratory distribution; SBR is a typical example. The" quality" of SBR may be ascertained by (*a*) certain analytical tests such uniform level design ITP is specified as fatty acid content, (*b*) certain simple physical tests, such as Mooney viscosity, or (*c*) four, two by certain performance tests, (minimum) tensile strength, modulus, or cure rate. Here categories (*a*), (*b*), and (*c*) correspond respectively to Class I, II, or III distribution schemes.

5.3 *Discussion each of Repeatability (Very Short, Short, and Long Term)*:

5.3.1 In 5.2 attention was focused on interlaboratory precision; within-laboratory precision (repeatability) is now discussed. There are at least three different viewpoints that have been expressed with regard to repeatability.

5.3.1.1 *View 1*—The smallest possible or "very short" time period is used to estimate the variation. two test technicians. The same material, apparatus, and operator is used, and repeated determinations outliers are made within reviewed by a period measured in minutes or at most within a period measured in hours.

5.3.1.2 *View 2*—A "short" time period is used for the repeated operations special procedure that produce test results. The same material and same operator (or set of operators) is employed but depends on the time period for the repeat operations is most frequently measured number of laboratories in days.

5.3.1.3 *View 3*—A "long-term" time period is used for the repeated operations that produce test results within a laboratory. This may be weeks or months. In this sense, although it may be possible to use the same material, different operators are often employed ITP and due to the long-term nature certain other changes, such as recalibration of the test apparatus, may have taken place. These changed conditions produce increased variability.

5.3.2 The time period *must be specified* as each particular test method is taken up for consideration.

5.3.3 An important added feature is the concept of "acceptance differences" for individual sets of determinations. These may be called" checking limits." Such acceptable difference values can have useful applications in analytical precision, absolute or other quickly repetitive operations, such as testing individual tensile-strength test specimens (dumbbells or rings). They permit the exclusion of outliers among the determinations.

5.3.4 It relative, is anticipated that the "acceptable difference" repeatability will be calculated expressed by a specified procedure. The procedures for determinations this Special Precision are listed in the same way that ordinary repeatability is calculated for test results. Therefore, an extra set of calculations can be performed for individual determinations to permit estimates of AD_2 or AD_x to be obtained.

5.3.5 For any given test method a task group or subcommittee will normally choose one type of repeatability and reproducibility whether short term or long term. Section 11.

6. Steps in Organizing an Interlaboratory Precision Test Program

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

6.1.1 *Organization Committee*—An organization committee or task group of qualified people and a program coordinator should be organized to conduct selected. One member of the program: committee or group should be a chairman, a statistical expert, and members well-experienced statistician familiar with the standard in question. The chairman should ensure that all instructions testing technology of the program test method as well as the content of this practice. Most ITPs are clearly communicated to all laboratories in organized on the program. A supervisor in each laboratory should be chosen.

6.2 *Type basis of a balanced uniform level design for the precision program.*

6.1.2 *Category and Type of Precision*—The task group should make the following initial decisions.

6.2.1 The— 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 obtained (Type 1 or evaluated shall be selected, see 5.1.5. Type 2).

6.2.2 The time period of 1 precision is the repeatability and reproducibility estimate; short (minutes, hours, most frequently evaluated. For some test methods such as rubber or days) polymer or long (weeks other performance-in-rubber evaluations using standard formulations, a Type 2 precision is required.

6.1.3 *Test Operator or months). Define the time period.*

6.2.3 Whether acceptance intervals are desired Technician Selection—For simple General Precision testing requiring only one operator or needed.

6.2.4 These decisions set technician, all replicate tests should be conducted by the stage for important but secondary decisions that naturally evolve from same technician unless the structure effect of the program.

6.3 *Laboratories and Materials*:

6.3.1 The number 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 sabme *operatori tesam* should be determined. The number conduct testing for all replicates again unless the effect of materials, each comprising a different level operator teams is part of the measured property, should be selected.

6.3.2 The number program.

6.1.3.1 For Special Precision testing follow the procedure of laboratories available is seldom large, using two technicians on each of two test days. See Section 11.

6.1.4 *Test Result and if the Number of Replicates*—Each test method is complex, or expensive to run, has a final value for the problem is complicated further. Therefore, the problem is finding and obtaining the cooperation of enough qualified laboratories to produce meaningful estimates of precision, rather than property under evaluation, defined as a selection from test result. A test result may be a group mean or median value of available laboratories.

6.3.3 At least ten participating laboratories are recommended. Practical considerations usually require that fewer than ten laboratories participate in the study. However, an interlaboratory study that involves fewer than six participating laboratories may not lead to reliable estimates a number of individual determinations as specified by the reproducibility of the test method.

6.3.4 The method. For the purposes of this practice, a replicate is defined as a test result. The number and type of replicate test results, *n*, within each laboratory on any material s thould be included will depend 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 precision varies over that range, the different types of materials to which the test method is applied, typically used in the difficulty (expense) industry. The selected time period shall be reported in performing the tests, and precision section of the commercial or legal need for obtaining a reliable estimate test method.

6.1.6 *Number of precision.*

6.3.5 An interlaboratory study should include at least *three* materials, and for development Target Materials—The number of broadly applicable precision statements, *five* or more target materials or classes of objects (or manufactured products) to be tested should be included. The term "materials" is used in a broad generic sense. Materials may selected. Ideally, this should be raw three or natural substances, manufactured products, etc. For each level of material, an adequate quantity (sample) of homogeneous material four with substantially different property levels. The target materials should be available for subdivision represent typical industry materials as normally used and distribution by random allocation subjected to the participating laboratories. This supply test. See 5.1.

6.1.7 *Preparation of Homogeneous Target Materials*—A hompogeneous lot of each of the target materials should include a be prepared, with sufficient reserve of 50 % beyond quantity, so that retests can be made if needed. If the requirements material allows for possible later use in retesting in one or more laboratories. When the material(s) a blending operation to ensure homogeneity, this should be tested done. If blending is (are) not homogeneous, it is important to prepare the samples in the manner prescribed by the test method, preferably starting with one batch of commercial material for each level. Some modifications may possible, special procedures should be necessary conducted to ensure that obtain the amount of most homogeneous material available (or collection of elements) that is sufficient to cover the experiment and keep a stock in reserve.

6.3.6 At each level, *p*, separate containers (the number possible by way of closely monitored laboratory or other preparation operations). Documentation should be used where there is 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 material deteriorating when test method are required for the container has once been opened. In final database (after outlier treatment) in the case of unstable materials, special instructions on storage and treatment should be prescribed.

6.4 *Actual Organization of ITP. For the Tests*— The interlaboratory more important industry test plan is as shown methods, 12 to 18 laboratories should participate. If six or more laboratories are not in Table 1, a table that indicates the final database, an analysis can be conducted with fewer laboratories, but the estimates of precision, especialsly reproducibility, are seriously compromised and replicates. With *q* levels only represent very rough estimates.

6.1.9 *Packaging and n replicates, 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 among the *p* total laboratories has prior to carry out *qn* tests. A decision is necessary (for each test method) as need to whether a "replicate" is to be a "determination" or a" test result" as defined in this practice. specified. The performance shipment of these tests all materials should be organized and coordinated with the operators instructed test schedule (discussed as follows:

6.4.1 All *qn* tests should be performed by one and follows) so that all materials are available for the same operator or operator set, using the same equipment throughout.

6.4.2 Each group of *n* tests belonging to one level must be carried out under repeatability conditions, in scheduled test dates.

6.1.10 *Testing Instructions*—Although all ITPs are usually conducted for a specified interval of time.

6.4.3 It is essential standard test method that a group includes the complete set of instructions for the test, some supplemendtal instructions are requirepd. One important supplementabil instyructiond is tihe schedule for the testing. All tests should be performed independently as if they were *n* tests on different materials.

6.4.4 The number of replicates, *n*, must be specified. Each replicate may be *one* test result or *one* determination in accordance

TABLE 1 OPrecigsion Progral Tem—Basic Dat Resultsa[A]

LMateverial– (*j*) ==> –Laboratory (*i*)	1	2	3	j4	...	*q*
1 1 2 2 i 3 4 5 ... *yijk* ... *p* *p*			*Yijk*	–		*yi1*

[A] The following notation is used:

(a) Laboratories, there are *p* as a total

$L_i (i = 1, 2 \ldots p)$

(*b*) Materials or levels, there are *q* as a total

$m_j (j = 1, 2 \ldots q)$

(*c*) Replicates, there are *n* as a total in each cell or $L_i m_j$ combination. There are normally an equal number of *n* values (usually 2) in each cell.

(*d*) y_{ijk} is a single test result value.

Example—Cell (*i, j*) contains n_{ij} results y_{ijk} ($k = 1, 2, \ldots n_{ij}$).

Notation used:

Laboratories, a total of *p*, *L* (*i*) = 1, 2, 3, ... *p*

Materials or Levels, a total of *q*, *m* (*j*) = 1, 2, 3, ..., *q*

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

Yijk = a single test result value; where *k* = 1, 2, ... *n* (*ij*); see cell (23) of table for example

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

with specified days, and all participating laboratories should conduct the requirements of the test method. Normally, *n* is two. A larger number may be as specified if necessary.

6.4.5 In on-line statistical process control situations, a single determination is often considered a test result, particularly if the precision of a duplicate determination by the test result does not show cost effective improvement over that method. The schedule should allow for adequate material delivery time. Any special modifications of a single determination the test result. This can method should be h clearly described as well as spfuecial infstructions as to operators or technicians (one, two, or more) versus replicate testing. If an ITP is to be conducted for many users of a test method. It is method at this planning stage that the decision has some intermediate development level, it is essential to be made whether or not to have give all participating laboratories instructions for conducting the precision statement present both test method as well as all the precision of a single determination test result required ITP instructions.

6.1.11 *ITP Test Data Report*—A *test report data form* should be prepared by the ITP coordinator and a duplicate determination test result. If the decision is made copy sent to run duplicate determinations, the minimum testing required for each test material consists of two sets of duplicate determinations conducted on each of two different days.

6.5 *Instructions to Operators*:

6.5.1 The operators should receive no instructions other than those contained in participating laboratory along with the test method; these materials and instructions. This form should suffice.

6.5.2 Prior contain locations to testing, report the operators should be asked to comment on following: the standard name of the laboratory; the test dates as actually used; and state whether for each target material tested, the instructions contained in it are sufficiently clear.

6.5.3 All participating laboratories should report their test value (test result) for each replicate test (day), reported if possible to one more significant figure than is customary or prescribed in this practice.

6.6 *Reporting the Test Results*—Each laboratory supervisor normally used (that is, do not truncate). The test report form should write also ask for a full report on description of the tests containing the following particulars:

6.6.1 The final test results (avoid transcription and typing errors).

6.6.2 The original individual observations and determination values from which the final results were derived. This is required if" acceptable difference" parameters (AD_2 equipment or AD_x) are to be calculated.

6.6.3 The date(s) on which the samples were received and the date(s) and time(s) on which they were tested.

6.6.4 Comments and information machines used (model number, condition), comments about irregularities or disturbances that may have occurred during any unintended deviations from the test.

6.6.5 Information about the equipment used, standard test procedure and disclosure of any mishaps or other relevant information.

6.7 The results pertinent information. The completed test report should be reported using returned to the format given in Table 1. ITP coordinator.

7. Overview of General Precision Analysis C Proncedure

7.1 *Analysis Operation Sequence*—This section gives a quick overview of the procedures required for Interlaboratory Test Data

7.1 The the analysis of interlaboratory data to evaluate test method precision is conducted as the ITP database and provides the user with a "one-way" analysis better appreciation of v the complete analysis process. Some background on outliers is also presenected in this section for a better appreciation of this topic. The General Prevcision procedure may require as many as three analysis operations or m overall steps. The actual number wiall be determined by the test program. Annex A1 gives uniformity of the basic statistical model for such an analysis. This annex should 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 reviewed to become familiar with required depending on the potential sources extent of variation outliers in the database b. Annex A4 contaings investructigons for all threde analysis operations and also gives the details on how to better appreciate layout the resquired tables and their interlinking that enables the automatic recalculation of the final precision calculations. This annex also gives the basic expressions for 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 *Outliers*—Outliers are test result and derived test result values, that deviate so much from the bulkPreliminary 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 (for a certain level) that they are considered to be irreconcilable with remainder review is conducted after cell averages and cell standard deviations (or cell ranges) have been calculated. Part of this review is the data. Although some care must be exercised in handling outliers, experience has shown that a certain small fraction generation of special plots of cell averages and cell standard deviations or cell ranges versus laboratoriy number. These plots, as described in any interlaboratory test program may produce 8.1.3, will clearly show potential outlier values. The most frequent causes are either testing blunders or inadequate control over internal testing conditions (poor test procedures, test machine maintenance, calibration). The outlier problem values.

7.1.2 *Analysis Step 1*—The original database is analyzedd to generate valuess for repeatability and reproducibility for each material (or target material) and the b 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* values as developed in Practice E 691. See Annex A2 statistics and Annex A3 for background on the development of these precision results for each material. The calculated *h* and *k* statistics and the rationale for the 95 % confidence level used for outlier rejection.

7.2 *Preliminary Analysis*—A preliminary analysis of the database consists of the following two initial steps:

7.2.1 Tabulate the data in the format as given in Table 1. In this table the number of laboratories is designated by *p*, the number of materials (levels) by *q*, and the number of test result replicates by *n*. The table contains *pq*" cells," each cell containing *n* replicates for the usual condition of an equal number of replicates per cell. In most interlaboratory test programs for precision, $n = 2$.

7.2.2 Inspect the data for any unusual results detectable by simple review. If any unusual data values are discovered make a note and proceed as described as follows.

7.3 *Full Analysis*—The full analysis of the precision data is normally conducted in two parts. Part 1 is an analysis of all of the data as reported by all participating laboratories. This analysis as described below, will generate additional tables that are used compared to identify any outliers in the database. If no outliers are found, the required precision parameters are calculated from the (original) database.

7.3.1 *Outlier Rejection*—If outliers are present, outlier rejection techniques are used to eliminate the indicated data values. After the outliers are removed and replaced by data values in accordance with 7.5 (handling outlier and missing values), a Part 2 reanalysis is conducted on the adjusted database. This Part 2 analysis yields the precision parameters that are used to prepare the precision section of the standard.

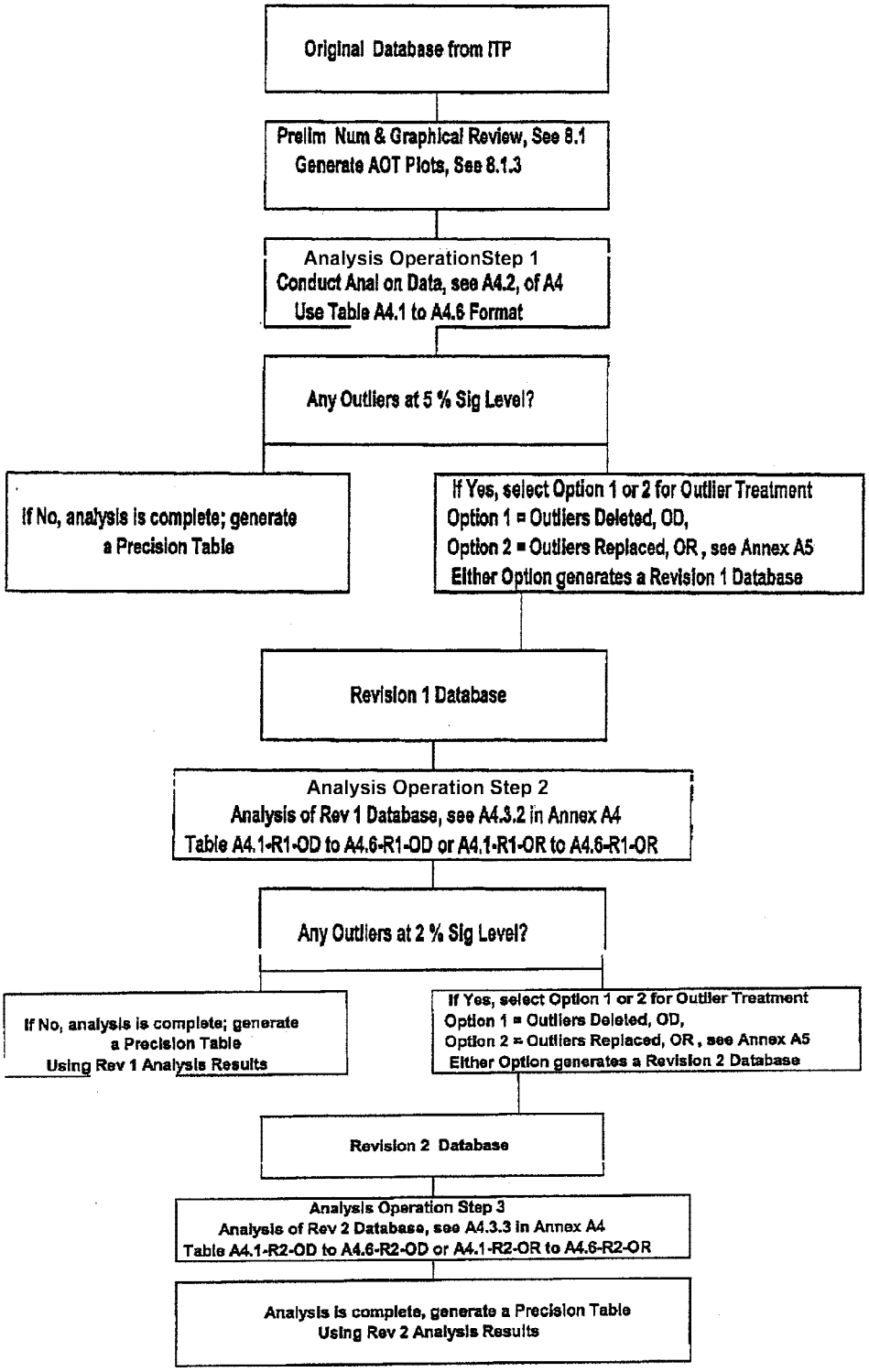
7.4 *Part 1 Analysis*—Conduct a Part 1 analysis in accordance with Practice E 691 calculation algorithms (these are given in Section 8 and Annex A2 and Annex A3 of this practice) on the data as it exists in a Table 1 format. This is done using either (1) the Practice E 691 computer (software) program,[6] or (2) typical spreadsheet calculation procedures. Four main steps in accordance with 7.4.1 to 7.4.4 are required. The Practice E 691 computer program performs all four steps and generates the required tables in addition to subsequent calculations for 5 % significance level critical *r h and R.k values to determine if there are any significant outlier values. If spreadsheet calculations there are performed, separate table generation steps may be required as follows:*

7.4.1 Calculate none, the *average* of each cell in analysis is complete and the Table 1 layout values found for repeatability and reproducibility abre ulsed to generate a table of precision results for the averages as in Table test method. If there are any significant outliers, Analysis Step 2. For this standard "average" refers to arithmetic mean.

7.4.2 Calculate is required.

7.1.3 *Analysis Step 2*—If there are any outliers at the *standard deviation* for each cell in 5 % significance level, the layout as shown in Table 1. (See Note 1.) Tabulate the calculated standard deviations as in Table 3.

7.4.3 Calculate the *h-value* for each cell in the Table outlying values are either (*1*) deleted using Option 1 layout. or (*2*) replaced



NOTE—Refer to Example Precision Calculations in Annex A6 for tables with data.

FIG. 1 Decision Tree Diagram for ITP Data Analysis

using Option 2. See Annex ~~A2~~ 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 ~~calculation~~ repeatability and ~~other details. Prepare~~ reproducibility, designated as *R1* precision values. This analysis produces a ~~table~~ new set of calculated *h*~~-values in the same format as Table 2, if a spreadsheet calculation is used.~~

TABLE 3 2 Cell VaPreciasionce Progr Standard Dm—Cell Avierationges[A]

NOTE 1—Uniform-Level Experiment

tMateverial– (*j*) ==> –Laboratory (*i*)	1	2	j3	4	...	*q*
1 1 2 2 i 3 4 5 ... p *p*			avg *Yijk* s*ij*			

[A] Symbols are defined as follows:
s_{ij} = cell standard deviation.
Notation sed:
Laboratories, a total of *p*, *L* (*i*) = 1, 2, 3, ... *p*
Materials or Levels, a total of *q*, *m* (*j*) = 1, 2, 3, ..., *q*
Replicates, a total of *n* per cell; a cell = each combination of *L* (*i*) *m* (*j*); normally *n* = 2
avg *Yijk* = average of cell (ij) for *n* test results

TABLE 2 3 Precision Program—Cell Av Std Derviagetions[A]

tMateverial– (*j*) ==> –Laboratory (*i*)	1	2	3	j4	...	*q*
1 1 2 2 i 3 4 5 ... p *p*			SD*Yijk*	$\bar{y}_{ij}$		

[A] $\bar{y}_{ij}$ = cell average.
Notation used:
Laboratories, a total of *p*, *L* (*i*) = 1, 2, 3, ... *p*
Materials or Levels, a total of *q*, *m* (*j*) = 1, 2, 3, ..., *q*
Replicates, a total of *n* per cell; a cell = each combination of *L* (*i*) *m* (*j*); normally *n* = 2
SD*Yijk* = standard deviation of cell (*ij*) for *n* test results

7.4.4 Calculate the *k-value* for each cell in the Table 1 layout. See Annex A3 for calculation and other details. Prepare a table of *k*-values in the same format as Table 3, 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 spreadsheet calculation 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—MAlthough 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 Generalg 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 a selected 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.

7.3 *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 E 691. 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.

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*, the number of values in the calculation, replicates, each test result replicate is designated as a divisor for the sum of squares in the calculation of a standard deviation. Y_{ijk} value. The divisor should be (most frequently used design has two replicates per cell or $n - 1$). If = 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 used, correct prepared by calculating, for all cells, the spreadsheet standard deviations by multiplying them by [deviation for the $n/(n - 1)]^{1/2}$.

7.4.5 *Review 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.*

8.1.3 *Graphical Review of Calculations*—Review 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 laboratory number 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, 9, and 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, OR, and OD appended in 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, 9, and 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 a. The correspondk-ing values in accordance with Table A4.2 are used as the procedures in arguments for Table A4.3. Refer to Annex A2 and Annex A3. Reject any A3 for cell averages, (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 are significant at both tables be generated in the 95 % (*p analysis.*

8.2.4 Table A4.5 is used to calculate = 0.05) confidence level. Reject any cell standard deviations, (*k*-values), that are significant at the 95 % ($p = 0.05$) confidence level.

7.4.6 If no-values for each cell averages or cell standard deviations in the database. The corresponding values in Table A4.4S are rejected, used as the Part 2 analysis is not required and the calculations arguments to calculate *k*-values in Table A4.5. Refer to Annex A3 for S_r, S_R,*k*-value calculations.

8.2.5 Table A4.6 is used to calculate the precision parameters, *r*, *R*, (*r*), and (*R*) may be made in accordance with Section 8.

7.5 *Blank or Missing CellR*). Values—If any outlier rejections are made, or if there are missing data in the original database, the problem of blank cells in the Table 1 format must be addressed. The recommended method to replace any blank cells is the use of a special or average value for the missing cell value in accordance with the instructions as given in the next section.

7.5.1 *Cell Replacement for Practice E 691 Computer Analysis*—If the Practice E 691 computer analysis is used, the blank cell replacement values must be inserted into the database in the Table 1 format and a reanalysis conducted. The Practice E 691

computer program is not structured to accommodate 'blank' data cells. The T_1, *replacement* T_2, *test result values must be inserted into any* T_4 *and cell*n *and so that*p *are required to calculate both the recalculated average or the original average*r *and recalculated standard deviation (variance) if both are observed for that level or the original standard deviation, are preserved or unchanged by the addition of the replacement values.*

7.5.1.1 The *recalculated average*, is*R*. See the (new) average calculated after removing the cell average outlier value(s) from the indicated cell(s).

7.5.1.2 The *recalculated standard deviation (and variance)*, is the standard deviation (variance) calculated after removing the cell standard deviation outlier value(s) from the indicated cell(s). The technique for cell value replacement under the stipulations imbedded calculation algorithms, 1 to 5, in accordance with 7.5.1, is described in 7.5.2 for spreadsheet analysis Table A4.6 and also in Annex A7.

7.5.2 *Cell Replacement A4 for Spreadsheet Analysis*—If a spreadsheet analysis is used a number the details on these calculations.

8.3 *Detection of intermediate tables will be needed in Outliers at the spreadsheet in addition to the five tables as specified in 7.4. In addition to Table 1, Table 2, 5 % Significance Level Using h and Table 3 and the k Statistics*—The calculated values for *h*-value in Table A4.3 and the calculated values of *k*-value tables, the following tables in Table A4.5 are reviewed for potential outlier values.

8.3.1 If the Part 1 analysis are recommended—Cell Average Deviation, *d*, and Cell Standard Deviation Squared (that is, Variance). The cell average deviation table is used in the construction of the Table A4.3 *h*-value table. The for any cell standard deviation squared table is used 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 calculation of Table A4.5 *k*-value for any cell equals or exceeds the pooled 5 % significance level critical S_r and*k*-value as given in the operation to replace blank Table A3.1, that particular cell standard deviation values.

7.5.2.1 In Annex A7 value is declared as an example outlier.

8.3.3 If outliers are detected, a summary of precision analysis the outliers detected is given for presented in the spreadsheet approach. This annex illustrates how form of a number sub-table at the bottom of supplementary spreadsheet calculations are made. Refer to this annex Table A4.6 showing the laboratory numbers that had 5 % significance outliers for additional details on cell replacement operations both *h* and *k* for some general comments on each material. See Table for an example. When outliers are present, a revised database is generated by the use of either Option 1, outlier problem in precision analysis.

7.5.3 If more than one deletion, or Option 2, outlier of a given type (cell average replacement, as described in 8.4.

8.3.4 If there are no outliers for either cell averages or cell standard deviation)s, the precision analysis is rejected for a particular laboratory complete and if the cell resulting values for other materials in general appear to be out-of-line (although not officially rejected) with results of the other laboratories, serious consideration should be given to totally eliminating the laboratory from the database for analysis.

7.6 *Part 2 Analysis*—After all blank cells have been replaced with appropriate averages after (1) any outlier rejection operations, or (2) missing cell values have been allowed for, the adjusted database shall be subjected to a Part 2 analysis. From this second analysis, calculate S_r, S_R, *r*, *R*, (*r*), and (*R*) in accordance with Section 8.

7.7 *Preparation of Research Report for Precision Evaluation*—All precision evaluation programs shall may be well documented by the preparation of used to prepare a research report that shall be placed on file at ASTM Headquarters. This report shall contain important information concerning the interlaboratory program as follows:

7.7.1 Test method designation,

7.7.2 Number and identification of participating laboratories,

7.7.3 Materials used, identification or formulations, or both,

7.7.4 Type of precision evaluated; time period of precision (hours, days, weeks),

7.7.5 Dates of test program,

7.7.6 Basic (raw) data obtained, in Table 1 format,

7.7.7 Calculations performed table for evaluating precision parameters, including method used for outlier rejection and method used for replacing missing values,

7.7.8 Results of precision calculations in Table 4 format, and

7.7.9 Any unusual outcome of the test program.

8. Calculating the Precision Parameters

8.1 Although Annex A1 gives substantial background and discussion on the repeatability variance and standard deviation, on the between-laboratory variance method.

8.4 *Generation of cell averages and on the reproducibility variance and standard deviation, the basic calculation algorithms for these parameters R1 Database Using Outlier Option 1 or 2*—If outliers are given in this section. The calculations apply to each material.

8.1.1 *Repeatability Variance, Standard Deviation*—For any material, detected, the repeatability variance designated by $(S)^2_r$ database is calculated in accordance with Eq 1.

TABLE 4 Example—ASTM XXXX—Type 1—Precision[A] (Measured Properl Daty = XXXX in MPa)

N FORMATE 1—I fAD$_2$orAD$_x$ is de Each Matermined, the results may be given in a tabl—Spe sc similar to Table 4.

NOTE 2— Pooled or average valuecis fiorn: Call tarbuon Blatck Ted parameters may be given if appropriate.

NOTE 3—p = xx, q = 4, n = 2.g

Material	Mean Level, (MPa)	Within Laboratories[B]			Between Laboratories[B]		
		Material (s_i)					
r	(r)	S_R	R	(R)			
Date	Test Result 1	Test Result 2	R	(R) Operator or Technician			
A	XX	X	X	X	X	X	X
Day 1	xxx	xxx	X	X	X	X	Xxxxxx
B	XX	X	X	X	X	X	X
Day 2	xxx	xxx	X	X	X	X	X
C	XX	X	X	X	X	X	X
D	XX	X	X	X	X	X	X
Pooled or Average Values	XX	X	X	X	X	X	X
Pooled or Average Values	XX	X	X	X	X	X	Xxxxxx

[A] The time period for precision is days.

[B] Symbols are defined as follows:

s_r = within-laboratory standard deviation.

r = repeatability (in measurement units).

(r) = repeatability (in percent).

If actual measurement units are %, these values represent percent relative, such as, percent of a percent.

S_R = standard deviation for total between-laboratory variability.

R = reproducibility (in measurement units).

(R) = reproducibility (in percent).

$$(S)^2_r = \Sigma\,(1\ to\ p)(Si)^2/p \quad (1)$$

where:

$(Si)^2$ = cell variance for Laboratory i, and

p = total number of laboratories.

The repeatability standard deviation is given in Eq 2.

$$S_r = [\Sigma\,(1\ to\ p)(Si)^2/p]^{1/2} \quad (2)$$

8.1.2 *Between-Laboratory Variance*—A derived intermediate parameter revised using either Option 1 or 2. The revision procedure is the term called the 'between-laboratory' variance, designated by $(S^2)_L$. This is evaluated from the variance of the 'cell averages,' (laboratory averages for any level), designated by $(S^2)\bar{x}$, and the repeatability variance.

$$(S^2)_L = (S^2)\bar{x} - (S)^2_r/n \quad (3)$$

The term $(S^2)_L$ is used described in the calculation of the reproducibility variance and standard deviation in accordance with 8.1.3. It can also be used as an indicator of the inherent variation between laboratories without the influence of the within-laboratory variation. Experience has shown, however, that the within-laboratory variation A4.3.

8.4.1 Option 1 is substantially smaller than between-laboratory variation. In certain circumstances $(S^2)_L$ may calculate to less than zero; if this occurs $(S^2)_L$ 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.

8.1.3 *Reproducibility Variance, Standard Deviation*—The (total) variance among all the values for a given material is defined as the reproducibility variance, in accordance with Eq 4.

$$(S)^2_R = (S^2)_L + (S)^2_r \quad (4)$$

Substituting for $(S^2)_L$ produces Eq 5.

$$(S)^2_R = (S^2)\bar{x} - (S)^2_r/n + (S)^2_r \quad (5)$$

Simplifying and taking the square root produces Eq 6.

$$(S)_R = [(S^2)\bar{x} + (S)^2_r(n-1)/n]^{1/2} \quad (6)$$

8.2 The calculations for the above parameters are provided as part deletion of the output of the Practice E 691 computer

~~software program. For spreadsheet analysis the usual spreadsheet calculation procedures may be used as well as specific calculations set up in the form of macro commands. Annex A6 also contains computational formulas that may prove to be beneficial for spreadsheet precision calculations. This annex contains the formula for unequal numbers of~~ *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 operations of Analysis Step 3.

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.

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

	Material 1		Material 2		Material *q*	
Laboratory Number	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

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

NOTE—Measured Property = xxxxxx, in xx.

		Within Laboratories			Between Laboratories			
Material	Mean Level	*Sr*	*r*	(*r*)	*SR*	*R*	(*R*)	No. Laboratories[A]
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. 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 ~~per cell.~~

~~8.3 Annex A4 describes~~ 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 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* (crit) and *k* (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.

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*) ~~or~~), and (*R*)~~, and~~) using the ~~mean level~~*M*.

~~8.4 Annex A5 is addressed to carbon black testing. It describes a special treatment of within-cell test values (test results) and their review for data consistency~~ 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 ~~behavior. It also specifies a special procedure for selecting~~ rejection following 11.3.1. Plot the ~~mode~~ values of ~~precision parameter expression, either absolute~~ *R* and (*R*) versus *M* or ~~relative,~~ Y_{AV}, the mean value for ~~both reproducibility and repeatability.~~

~~8.5 Annex A7 previously discussed, is an example of a typical precision evaluation~~ material measured property, for ~~Mooney viscosity. All calculations are included~~ all materials in ~~this example.~~

9. ~~Format~~ the ITP. Perform a least squares regression for ~~Precision~~ both relationships, and ~~Bias Section (Clause) of Standard~~

~~9.1 The results of~~ record the ~~formal analysis shall be contained in a specific section or clause~~ coefficient of ~~the test method entitled "Precision~~ determination, designated as C_d, for each parameter *R* and ~~Bias."~~

~~9.2 *Introductory Subclauses*—The precision and bias section shall begin with two paragraphs giving important details on the interlaboratory program.~~

~~9.2.1 A statement citing that Practice D 4483 is the reference document~~ (*R*).

11.4.1 Select for the ~~precision section.~~

~~9.2.2 A caveat statement on the general applicability~~ mode of ~~the~~ precision ~~results, in accordance with 9.2.2.1.~~

~~9.2.2.1 The precision results in this precision and bias section give an estimate of~~ expression, the ~~precision of this test method~~ parameter, *R* or (*R*), with the ~~materials (rubbers, etc.) used in the particular interlaboratory program as described below. The precision parameters should not be used~~ lowest value for ~~acceptance or rejection testing~~ C_d. This establishes which of ~~any group~~ the two modes of ~~materials without documentation that they are applicable~~ expression has the least relationship to ~~those particular materials and~~ the ~~specific testing protocols~~ level of the ~~test method.~~

~~9.3 A second subclause shall consist of one~~ measured property or ~~more paragraphs that give details on~~ inversely which parameter is the ~~interlaboratory program followed by one or more tables~~ most independent of ~~results of~~ the ~~precision testing. The introductory paragraphs should answer the following questions:~~

~~9.3.1 What type precision was estimated, Type 1~~ measurement level. This lowest C_d or ~~Type 2?~~

~~9.3.2 What~~ most independent parameter is ~~the time period for repeatability, reproducibility—short term (define), long term (define)?~~

~~9.3.3 What is~~ to be used to prepare a ~~test result? How many determinations? Average (mean) or median?~~

~~9.3.4 How many laboratories participated (~~ *p*~~)?~~

~~9.3.5 How many materials (~~*q*~~)?~~

~~9.3.6 How many replicates (~~*n*~~)? What is a replicate?~~

~~9.3.7 At what time was~~ final precision table in the ~~interlaboratory program conducted (month, year)?~~

~~9.3.8 Are there any unusual results that~~ format as indicated by Table 6. The selected mode of expression applies to both repeatability and reproducibility. Follow the ~~reader should be aware of?~~

~~9.3.9 How do~~ rules for expressing General Precision as outlined in Section 12 using, where appropriate, the designation Special Precision. The columns [*r* and *R* ~~vary as the mean level of the measured property varies? Can these variations be described by a simple mathematical relationship (linear, log, etc.)? See the Annexes.~~

~~9.4 *Table of Precision Parameters*— A table with the general format such as Table 4 should be prepared. This includes the following information:~~

~~9.4.1 ASTM test method designation and year of issue,~~

~~9.4.2 Type of precision; time period used for~~ or (*r*) and (*R*,

~~9.4.3 Measured property,~~

9.4.4 Materials,)] 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 measurement, and

9.4.5 *r* 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*), and for completeness of record the within and between laboratory standard deviation, s_r and S_R.

9.5 *Pooled Values for* Table 4 *Format*—If pooled or average values, or both, for the precision parameters set up, in the format of Table 4 are desired, use the following procedure.

9.5.1 *Average*—The average applies to the column of mean level values only. The (arithmetic) average is calculated in the normal manner.

9.5.2 S_r *and* S_R— For these two parameters, the pooled values are the square root of the mean variance of each column (of standard deviation values).

9.5.3 *r and R*—These parameters are equal to their respective standard deviations multiplied by 2.83 (standard deviation times a constant) and therefore are to be pooled by the same procedure as for S_r and S_R.

9.5.4 *(r) and (R)*—There are two options for calculating the pooled values for these two relative (percent) precision parameters.

9.5.4.1 *Option 1*—For each row of the table, these parameters are also equal to a standard deviation times a constant. But the constant [2.83 × (1/mean level value) × 100] changes for each row of the table. Therefore one pooling method is to obtain the square root of the mean value of each row value squared, in the measurement units, (*r8) column and 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.*

912.51.4.2 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—The alternative pooling method outlier replacement is chosen, the number of laboratories that 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*) by dividing), 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* by 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 mean level precision. It may have very little meaningful value (bottom of Column 1 mean level value) and multiplying by 100.

9.5.5 Experience shows that the two options do not give exact agreement. The recommended method or applicability.

12.1.5 When there is Option 2. The option adopted is not really very critical; a substantial difference in precision among the materials, the use of a pooled value is simply may give a *general indicator* of overall precision and minor differences are false impression of no substantial consequence.

9.6 *Significant Figures in Precision Table*—Computer calculations frequently generate several figures or decimal places after the decimal point. All overall precision. It would be better to direct the values placed in user to select a material from the precision table should be rounded that is closest in mean value to a specific material under consideration to determine the number expected precision instead of figures after using the decimal point that pooled value. Ultimately, it is realistic from the standpoint responsibility of those conducting the measurement capability 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 very frequently only discussed in Annex A2. The one or two decimal places not counting any leading zeros for values smaller than unity. The relative 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 parameters (*r*) ITP was conducted in accordance with Practice D 4483 (the latest revision year designation), and (*R*), the year the ITP was conducted. A statement that the reader should be given refer to only one figure after the decimal point Practice D 4483 for values below 100 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 no figures (whole numbers) acceptance or rejection testing for values above 100.

9.7 *Statements for Precision*:

9.7.1 Typical statements for any group of materials or products without documentation that the results of the precision section evaluation actually apply to the products or clause materials tested.

12.2.3 A statement giving (*1*) category of a standard shall be listed in accordance with one the precision, that is, General

Precision or Special Precision (Carbon Black), (*2*) the type of ~~two styles, either 9.7.1.1 and 9.7.1.2~~ precision, Type 1 or ~~9.7.2.1 and 9.7.2.2.~~

~~9.7.1.1 The~~ Type 2, (*3*) the number, ~~*difference*~~*p, of laboratories participating in the ITP, (4) the number,* ~~*between two single test results*~~*q, and description of the materials (or* ~~*determinations) found on identical test material under*~~ *target materials) used, (5) the* ~~*repeatability conditions prescribed*~~ *number of within-laboratory replicates, n, (6) the time span for the repeatability or within-laboratory replicates, (hours, days), (7) the definition of a* ~~*particular*~~ *test* ~~*will exceed the*~~ *result (average, median of* ~~*repeatability on an average*~~*x number of* ~~*not more than once in 20 cases in*~~ *determinations or individual measurements), (8) the option chosen form outlier treatment, deletion, or replacement, and* ~~*correct operation*~~ *(9) any unusual features of the* ~~*method.*~~

~~9.7.1.2 The *difference* between two single and independent test~~ ITP.

12.2.4 A table of precision results ~~found by two operators working under~~ as set forth in 12.1 should be part of the ~~prescribed reproducibility conditions~~ clause. Ensure that the table (inserted into the test method standard in ~~different~~ Table 6 format) gives the final number of laboratories that remained after outlier deletion or replacement. Some comments on ~~identical test material will exceed~~ the ~~*reproducibility* on an average~~ outcome of ~~not more than once in 20 cases in~~ the ~~n~~ results should be given.

12.2.5 Generic statements on repeatability and ~~correct operation~~ reproducibility shall be part of the ~~method.~~

~~9.7.1.3 These two statements apply to either a particular mean level in a~~ precision ~~table (see Table 4) or to an overall~~ clause using the recommended text as set forth as follows. A 95 % confidence level ~~common~~ (or $p = 0.05$) applies to ~~a standard or table, which is designated as a 'pooled' value, that is, a special average value (see 9.5). The statement should make it clear which type of precision value is addressed, individual mean levels in a table or a pooled value.~~

~~9.7.2 Alternatively,~~ these statements ~~of~~ where Table xx designates the ~~following form may be prepared for use in~~ final table as inserted into the ~~Precision clause of any~~ test method.

~~9.7.2.1~~

12.2.5.1 *Repeatability*—The repeatability, or local domain precision, of this test ~~*xxxx* has~~ method has been established ~~as *xxxx*. Two single test results (or determinations) that differ~~ by ~~more than *xxxx* (expressed~~ the values found in ~~appropriate terms) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.~~

~~NOTE 2—Appropriate action may be an investigation~~ Table xx, for each of the ~~test method procedure or apparatus for faulty operation or~~ materials as listed in the ~~declaration of a significant difference~~ table. If calculated, pooled repeatability values are also listed in the ~~two materials, samples, etc., which generated the two test results.~~

~~9.7.2.2 *Reproducibility*—The reproducibility of test *xxxx* has been established as *xxxx*.~~ table. Two single test results ~~(or determinations) produced in separate laboratories~~ (obtained by the proper use of this practice) that differ by more than ~~*xxxx* (expressed in appropriate terms) must be considered as suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical/commerical actions be taken.~~

~~9.7.2.3 These two statements apply to either a particular mean level in a precision table (see Table 4) or to an overall level common to a standard or table, which is designated as a 'pooled' value, that is, a special average value (see 9.5). The statement should make it clear which type of precision value is addressed, individual mean levels in a table or a pooled value.~~

~~9.7.2.4 Repeatability and reproducibility expressed as a percentage of the mean level,~~ (tabulated values for *r*~~)~~, in measurement units, and if listed, ~~(R), have equivalent application statements as above for *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*~~)~~ ~~statements,~~), will be very large. For these circumstances omit the ~~d~~ relative expressions of precision from a Table 6 format.

12.4 *Spe~~n~~cial Pre~~-~~b*cision Sec*t~~w~~*ion or Clause*—The expression for Special Precision should in general follow the ~~two test results is expressed~~ 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 ~~an arithmetic mean (average)~~ 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 ~~two test results.~~

~~9.7.3 *Bias Statement*—For most test methods bias cannot~~ precision evaluation shall be ~~determined. In that case, the following statement~~ prepared for any ITP. This is ~~recommended:~~

~~9.7.3.1 *Bias*—In test method terminology, bias is~~ a full comprehensive report of all ITP details, not the ~~difference between an average test value~~ report that each participating laboratory prepares and returns as part of the ~~reference (true) test property value. Reference values do not exist for this test method since~~ ITP. This full report should contain information on the ~~value or level~~ details of the ~~test property is exclusively defined by~~ organization and execution of the ~~test method. Bias, therefore, cannot be determined.~~

~~9.7.3.2 For those test methods where bias can be determined, a statement~~ 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 ~~its magnitude should be included.~~

~~9.8 *Modification ITP laboratory number,*~~

13.1.5 Number and description of ~~Precision Table Format—If~~ materials or target materials, *q*,

13.1.6 Definition of a test result, number of replicates, *n*, and time span for ~~certain technical reasons,~~ repeatability,

13.1.7 Information on technicians conducting the ~~precision table format as specified above is considered inappropriate for testing,~~ any ~~particular test method standard, a modified format may be used. If a modified format~~ special details,

13.1.8 Details on preparation of materials, how homogeneity is ~~a~~ 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 ~~section, clearly documenting the need~~ results, comments on outcome, and

13.1.13 Draft of precision section for the ~~modified format and explaining the modifications made.~~

~~10.~~ test method.

14.

15. Keywords

~~10.1 accuracy;~~

15.1 general precision; interlaboratory ~~study;~~ test program; ITP; precision; repeatability; reproducibility; ~~statistics~~ special precision

ANNEXES

(Mandatory Information)

~~**A1. STATISTICAL MODEL**~~

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 ~~Model:~~ 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 F*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 = or > 4) will have an approximate 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 A1.2.1. 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.

~~$y = M + \Sigma d(j)$~~ (A1.1)

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

μj

where:

M y_i = value obtained for a measurement when all deviations, d(j), are zero, that is, the ideal outcome of a measurement, and measurement value, at time (*i*), using specified equipment and operators, at one laboratory or location (among a total of *p* laboratories),

$\Sigma d(j)$ = (algebraic) sum of (j) individual deviations or measurement perturbations, generated by whatever "system-of-causes"

μ_o = constant term (mean value), that exists for dictates the measurement system.

A1.1.2 The term *M* is expressed in practice, for any measurement system, as the average general magnitude of all *y*-values in the overall measurement program; it is also called measured parameter for the level of the property. (The particular test,

μ_j

=

constant term *M* is used in this Annex in place of μ, frequently defined as the true value). A more useful format is obtained when Eq A1.1 is expressed as an expanded model in Eq A1.2, where Σd ((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_i, 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.

$$y = M + B_i + B_m + B_L + B_g + e_b(l) + e_b(s) + e_w(l) + e_w(s) + e(g) \quad \text{(A1.2)}$$

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

BMBOP BGEB EW

where:

B_{iL} = inherent biasbias deviation term unique to one laboratory or systematic deviation, characteristic of the design of the measurement system; it exists under all measurement conditions, local domain,

B_{mM} = bias (systematic deviation) contributed by the measuring machine; it is deviation term unique to a particular the specific instrument or machine,

B_{tOP} = bias contributed by the laboratory; it is deviation term unique to conditions in a particular laboratory, the operator(s) conducting the test,

B_{g_G} = general (generic)generic bias of a "to be specified" nature (certain measurement systems may require more than one such term), deviation term; to account for other bias factors,

$e_b(l)$ E_B = between- laboratory (global domain) random deviation of long-term nature, that is, over a period of several weeks or months, term, and

$e_b(s)$ E_W = between-laboratorywithin 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 short-term nature, 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 period number of days, subcomponents that represent typical sources of variation between laboratories.

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

where:

$e_{w(l)}$ = within-laboratory randomrandom deviation of term attributable to a long-term nature (weeks, months), laboratory or location,

E_L

$e_{w(s)}$ = within-laboratory randomrandom deviation in the use of a short-term nature (days), and the specific instrument or machine,

E_M

$e_{(g)}$ = general (generic) randomrandom deviation of a "to be specified" nature (certain measurement systems may require more than one such term). inherent in the operator's technique, and

E_{OP}

E_G = generic random deviation term; to account for other random factors.

A1.1.3 In a perfect measurement world all biases and

The within laboratory random deviations of Eq A1.2 would deviation term, E_W, may also be zero. In the real world of measurement, these terms take on certain values and the sum of their collective values acts as a perturbation 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 MB_G *value for each measurement. Both or* E_G *testing perturbations, may be bias and random components due to temperature, long-term time period (time of the actual value year), and so forth.*

A2.3.2 $\mu_o + \mu_j$ *Terms*—In the variance absence of each bias or random deviations of these terms are important when considering testing and precision programs. Tests to determine the significance any kind, a number of materials or object classes would have individual terms usually involve a statistical comparison measured test values given by the sum of the variances attributed to two terms, $\mu_o + \mu_j$. The term μ_o uniquely characterizes the terms.

A1.2 *The (B) general magnitude of the measured parameter. Each material or Bias Terms:*

A1.2.1 The object class would be characterized by the value of μ_j, which would produce a varying value for the *(B)* terms is dependent on sum $[\mu_o + \mu_j]$ across the measurement system number of materials or object classes in the system-of-causes, for test program and the generation of sum would be the biases. The *(B)* terms *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 model may be either fixed materials or variable as well as plus or minus, depending on object classes produced by the measurement system under consideration. process that generates them. These usually unknown number of bias and random variations are designated by $\Sigma(b) + \Sigma(e)$. For any system, 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 (*B 1) terms are typically a non-random finite distribution special test programs to discover and therefore eliminate the values for causes or (2) a particular bias term will not documented correction procedure that eliminates the bias. For most precision ITPs, special care is required to ensure some minimal level of necessity sum variation in the lots of materials selected for the program, that is, to z make them as homogeneous as possible. Any residual production veariance adds to the population constituting measurement variance or basic precision as evaluated by the system. ITP.*

A2.3.4 *Measurement Bias terms that are fixed under one system of causes Terms*—Bias deviations may be variable under another different system of causes and vice-versa.

A1.2.2 The inherent bias divided into two classes: *B locali is characteristic of the overall design of the machine or apparatus. This type of global. A local bias is frequently of importance in chemical tests for a fixed offset that applies to certain constituents whose theoretical content can be calculated, specific conditions within a larger testing domain, for example, percent chlorine in sodium chloride. A given a single test d machinev 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 be low or high due in comparison to unique design features.*

A1.2.3 One other laboratories or instruments.

A2.3.4.1 When the domain consists of a larege number of machines or laboratories, the local bias terms; may be *Bvariable g, (plus or minus) deviations unique to each of these machines or laboratories and the distribution may be included either random with a zero mean in the model to allow for any (non-inherent) 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 systematic deviation not attributable to test machines a generic condition that is common within the domain or laboratories.*

A1.2.4 The (*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 *B system-of-causesm may be variable under another system-of-causes and vice versa. As an example, consider the bias terms* B_L *and* $B\text{-}L_M$ *which apply to most types of testing. As an example, for 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,* BL_L, *would be fixed but* Bm_M *would be variable, each machine potentially having a unique value. For a measurement system domain consisting of a number of typical laboratories, each with one machine, both* $B_{m\ B_L}$ *and* $B\text{-}L_M$ *would be variable for the multilaboratory measurement system domain, but of course both* Bm_L *and* BL_M *would be fixed or constant for the system-of-causes in each laboratory.*

A1.3 laboratory. One or more generic bias terms, *The (e)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:*

A1.3.1 The (*e*) Terms—These terms arep 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 a variance equal operators, in addition to var(*e*). the special case where another source, a generic source, is an important component. The distribution of the (*e*) these terms is assumed to be approximately normal but in practice it is usually sufficient if the distribution is unimodal. The (random) value of each of the (*e*) terms 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 (*e*) random terms is may be greatly diminished or eliminated depending on the sampling and replication plan, since in the long run each terms averages out to zero (or approximately zero) and the *mean* y_i v is essentially unperturbed.

A2.3.6 *New Term, M($_j$)*—With highly replicated testing programs (both production and test measurement replication) the average values obtained in any program are estimates of the value of a new combined term as given as follows:

$$M(j) = [\mu_o + \Sigma(b) + \Sigma(B)] + \mu_j \quad \text{(A2.4)}$$

and *M* (*j*) is perturbed by the mean value for the material or class of objects tested, for one laboratory or location, *j*, for the specific equipment and operators used during the existing time period. It contains bias components or potential bias components for all of these conditions. If all biases are fixed for any given program, the three terms in the bracket can be considered as a constant, and the *average test value* varies across the number of materials or object classes because of the varying value of μ_j. If the biases vary across the system, then both μ_j and the biases influence the average value for any candidate test and material.

A2.4 Evaluating Process and Measurement Variance

A2.4.1 Eq A2.1 may be used to illustrate how the variance of individual measurements, y_i, may be related to the terms or components of the equation. Recall that μ_o and μ_j are constants, $\Sigma(b)$ and $\Sigma(e)$ refer to the sum of bias and random components, respectively, for the production process, and $\Sigma(B)$ terms only. This long run zero-average character stands in contrast and $\Sigma(E)$ refer to the behavior sum of bias and random components, respectively, for the fixed (*B*) terms where an increased number test measurement operation. The magnitude of measurements increases the knowledge (accuracy) of individual components are ordinarily not known and the actual (*B*) value.

A1.3.2 To make equation can be simplified by combining the model building as accurate as possible as in the case bias and random components for both sources where $\Sigma(b, e)$ = sum of bias and random components for the *production process* and $\Sigma(B, E)$ = sum of bias terms, one or more generic and random deviation terms, components for the *measurement procedure*.

$$y_i = \mu_o + \mu_j + \Sigma(b,e) + \Sigma(B,E) \quad \text{(A2.5)}$$

The variance of any individual measurement y_i, designated by $s^2(g)$, may be included iny_i) is:

$$s^2(y_i) = [\Sigma \text{ Var } (b,e)] + [\Sigma \text{ Var } (B,E)] \quad \text{(A2.6)}$$

where:

[Σ Var *(b,e)*] = variance, that is the model to account sum of individual bias and random variances, for any potential source the *production* process, and

[Σ Var *(B,E)*] = variance, that is the sum of special individual bias and random deviations not attributable to variances, for the general or common 'within' or 'between' laboratory categories.

A1.4 *R measurement* procedure.

Eq A2.6 can be written in simplified format as:

$$s^2(y_i) = s^2(tot) = s^2(p) + s^2(m) \quad \text{(A2.7)}$$

where:

$s^2(tot)$ = total variance among the (B) materials or object classes in a test program,

$s^2(p)$ = variance due to the production process, and

$s^2(m)$ = variance due to the measurement operation.

A2.5 Relating the Bias and Random Terms to Measurement Precision:

A12.45.1 *Between Laboratory Variation* —The expanded series of (*B*) terms in Eq A12.2 gives insight into the potential individual sources of measurement bias between laboratories. However in any testing domain. However, to express the between laboratory test results in relation to the (*B*) terms, it is convenient to use a collective (or total *B*) term designated as (*B*)Total, (*Tot*), which is the (algebraic) sum of all (*B*) terms. The variance of (*B*)Total *B* (*Tot*) is the between-laboratory bias variance. The total between-laboratory variance is When the sum results of an ITP for precision are analyzed, the total between-laboratory bias variance and the between-laboratory random variance, e_b, (either long or short) and is given by Eq A1.3.

~~$\mathrm{Var}[(B)\mathrm{Total}] + Var[e_b] = (\sigma^2)_L$~~

~~= between−laboratory variance (A1.3)~~

~~The between-laboratory variance does not include the random within-laboratory variation. The value of $(\sigma^2)_L$ (for any material), is estimated in accordance with Eq A1.4, from the between-laboratory variance of cell averages, $(S^2)\bar{x}$,~~ diminished by the adjusted ~~value of $(S^2)_r$, the pooled within-cell variance. See Section 8.~~

~~$(\sigma^2)_L = (S^2)\bar{x} - (S^2)_r/n = (S)^2_L$ (A1.4)~~

~~The normal pooled within-cell variance, $(S^2)_r$, is adjusted or divided by n, the number of test values per cell, to put both of the variances in the equation on the same basis, that is, averages of n values.~~

~~A1.4.2 In Eq A1.4 and those to follow, population statistics are represented by Greek letter symbols and the estimates of the statistics are represented by English letter symbols. In Eq A1.4 the estimate $(S)^2_L$, is equated to the population statistic $(\sigma^2)_L$.~~

~~A1.5 *Relating the (e) Terms to Measured Precision*—The expanded series~~ sum of ~~random (e) terms gives insight into~~ the ~~individual sources of random deviations (or errors) that perturb~~ between-laboratory bias variance plus the ~~*M* value. However as in the case of the (*B*) terms, for any specific precision program with a defined time period for repeat tests, it is easier~~ total between-laboratory random variance due to ~~relate the test results to precision evaluation by selecting one $e_{\bar{b}E_B}$ *and one e w term, that is, commonly either a (l) long or (s) short time period; other time periods may be specified if needed.*~~

~~A1.5.1 *Within-Laboratory (e) Term Evaluation*:~~

~~A1.5.1.1 Within a single laboratory, repeated testing on a given material generates a series of values for~~ terms, designated as ~~$e_{\bar{w}E_B}(l)$ or~~Tot). ~~$e_{\bar{w}E_B}(sTot)$ depending on~~ is defined as the ~~time scale for measurements. From the series~~ sum of ~~such repeat measurements~~ the ~~simplest expression of within-laboratory~~ variance of all random ~~$e_{\bar{w}E_B}$ *is given by Eq A1.5. For simplicity the (l) and (s) notations will be dropped and e w alone used with the assumption that either time span can be used in the developed relationships.*~~

~~$\mathrm{Var}[e_w] = (\sigma)^2 = \text{simple within−laboratory variance}$ (A1.5)~~

~~This applies to a particular laboratory and to a particular material.~~

~~A1.5.1.2 It is the general practice in precision analysis to assume that $(\sigma)^2_w$ will be approximately equal from laboratory to laboratory for any well-established and standardized test method and on this basis the individual cell estimates of $(\sigma)^2_w$ can be pooled for any material to obtain a collective value representing all laboratories. However the skill and internal control procedures used in conducting test measurements varies among even well-experienced laboratories and this will be reflected in the pooled $(\sigma)^2_w$ variance for any given material.~~

~~A1.5.1.3 This varying testing skill situation can be addressed by use of the generic term $e_w(g)$. Thus a more realistic estimate of within-laboratory variance for any given laboratory is~~ terms as expressed ~~by~~ in Eq ~~A1.6, a variance specific to a given laboratory.~~ A2.2. Thus:

~~$\mathrm{Var}[e_w] + \mathrm{Var}[ew(g)] = (\sigma)^2_w$~~

$= \text{specific within−laboratory variance}$ (A2.8)

$$\mathrm{Var}[B(Tot)] + \mathrm{Var}[E_B(Tot)] = S^2_L \quad \text{(A2.8)}$$

where:

~~$Var[e_w]$~~ S^2_L = ~~basic within-laboratory~~between-laboratory variance, ~~a variance that is characteristic of routine use of the test method, that is, uniform over all laboratories, and~~ with S^2_L evaluated for an ITP as given by Eq A2.9.

$$S^2_L = S^2(Y_i) - (S^2_r/n) \quad \text{(A2.9)}$$

where:

~~$Var[e_w(g)]$~~ $S^2(Y_i)$ = ~~an added within-laboratory variance (component) specific~~variance among the cell averages across all laboratories, with Y_i defined as cell average for any laboratory, i, and

S^2_r = within cell variance pooled across all laboratories, adjusted or ~~unique~~ divided by n, the number of values per cell, to ~~a particular laboratory; it is approximately zero for good laboratories.~~ put both variances on an equivalent basis of mean values (averages of n).

~~The simple variance of A1.5.2.1 has been redefined as a basic variance. The specific within-laboratory variance defined~~ As indicated by Eq ~~A1.6, which contains two components, may also be called the specific repeatability variance, $(\sigma)^2$~~ A2.9, ~~S^2_r, *unique to*~~ *is a special derived variance that does not include the random within-laboratory variation.*

A2.5.2 *Within-Laboratory Variation* —Within any ~~one~~ laboratory, repeated testing (for a defined test domain) on a given material or at a given level generates a series of measurement values and a series of values for E_W. The within laboratory variance, S^2_W, is given by Eq ~~A1.7.~~ A2.10:

~~$\mathrm{Var}[e_w] + \mathrm{Var}[e_w(g)] = (\sigma)^2_r$~~

= specific repeatability variance (A2.10)

$$\mathrm{Var}[E_W] = S^2_W \quad \text{(A2.10)}$$

~~W~~For a standardized test method, it is general practice in precision evaluation and analysis to assume that S^2_W will be approximately equal for all laboratories. On this basis, the individual ~~repeatability variances~~ values for S^2_W (one for each ~~l~~laboratory for esach material) may be pooled ~~the relationship is expressed by Eq A1.8, where the estimated value, (S)~~ to obtain a collective or global value representative of all laboratories. Therefore, for each material or level, S^2r_W, is a universal value characteristic of all laboratories in the ITP and by assumption, all laboratories likely to use the test method. However, experience has shown that the skill and the internal control practices used in conducting tests varies even among well-experienced laboratories.

A2.5.3 This varying testing skill and general laboratory competence can be addressed by the ~~eq~~ use of a generic within laboratory term, E_{WG}, where the double subscript denotes a within laboratory generic random deviation component. Using this, a more well-defined within laboratory variance is:

~~Pooled $(\sigma)^2_r = (S)^2_r$ = repeatability variance~~

$$\mathrm{Var}[E_W] + \mathrm{Var}[E_{WG}] = S^2_W \text{ (sp)} \quad \text{(A2.11)}$$

~~Since in typical interlaboratory programs there is usually only 1 degree of freedom (DF) estimate of $(\sigma)^2_r$ for each laboratory and material, the pooled (~~

where ~~S)~~ ^2r_W (sp), the specific within laboratory variance, is equal to the ~~parameter~~ sum of ~~direct importance.~~

~~A1.5.2 *Between-Laboratory (e) Term Evaluation*—The term e_b, either long or short time span, represents random variations between (among) a group of laboratories that measure a common material and as such e_b is one component of the overall~~ universal within laboratory ~~variation. Interlaboratory~~ test ~~programs do not ordinarily provide a direct estimate~~ variance characteristic of the test, ~~e_{bE_W} *in the same sense that*~~, and another variance component unique to a particular laboratory. The variance associated with ~~$e_{wE_{WG}}$~~ is ~~evaluated.~~

~~A1.6~~ essentially zero for good well-controlled laboratories. Allowing for the potential existence of ~~*Combined (B) and (e) Term Between-Laboratory Evaluation*~~E_{WG} ~~*—The total variation*~~ *terms among between-laboratory test results (for any material) which is defined as laboratories, the reproducibility repeatability variance,* ~~(σ)~~ S^2R_r, ~~*is the sum of four sources or components of variance, for any selected time period, as given*~~ *defined by Eq ~~A1.9.~~ A2.12:*

~~$\mathrm{Var}[(B)\ \mathrm{Total}] + \mathrm{Var}[e_b] + \mathrm{Var}[e_w(g)] + \mathrm{Var}[e_w] = (\sigma)^2_R$~~

(A2.12)

$$\mathrm{Var}[E_W] + \mathrm{Var}[E_{WG}] = S^2_r \quad \text{(A2.12)}$$

where S^2_r is a pooled value across all laboratories for any material or level, each individual laboratory value having $(n - 1)$ degrees of freedom where n = number of replicates tested.

A2.5.4 *Combined Between and Within Laboratory Variation*—The total combined variation for between and within laboratory test results for any selected time period, defined as the reproducibility variance and designated as S^2_R, is the sum of four potential sources of variation.

$$\mathrm{Var}[B(Tot)] + \mathrm{Var}[E_B(Tot)] + \mathrm{Var}[E_W] + \mathrm{Var}[E_{WG}] = S^2_R \quad \text{(A2.13)}$$

The estimate of this variance, ~~(S^2 S)~~ ^{2}~~R~~*R*, is equal to the total ~~variance or mean square,~~ variation among all ~~the~~ values for each material (or level) in the ~~interlaboratory program.~~ ITP. Recall that ~~(B)Total~~ (*Tot*) represents a number of potential ~~separate~~ sources of ~~bias as given in Eq A1.2.~~ between laboratory bias. Interlaboratory testing experience has ~~shown~~ demonstrated that the left to right order of the variance terms in Eq ~~A1.9 (left to right),~~ A2.13 is the approximate order of magnitude of these terms.

~~A1.7 *Relationship Between (B)*~~

A2.5.5 *Defining Repeatability and ~~(e) Terms~~ Reproducibility*—Repeatability and ~~Precision Parameters r and R:~~

~~A1.7.1 Repeatability,~~ reproducibility are each equal to a range or interval that is a special multiple of the respective standard deviation. The repeatability, designated as *r*, is ~~defined by Eq A1.10 in terms of the estimated statistic rather than the population statistic.~~ given by

~~repeatability = $r = \phi\,(2)^{1/2}\,S_r$~~ (A2.14)

$$\text{repeatability} = r = \phi\,(2)^{1/2}\,S_r \quad \text{(A2.14)}$$

~~A1.7.2 Reproducibility,~~

and reproducibility, designated as *R*, is ~~defined by Eq A1.11 on the same basis.~~ given as:

~~reproducibility = *R*~~

$$= \phi\,(2)^{1/2}\,S_R \quad \text{(A2.15)}$$

reproducibility = $R = \phi\ (2)^{1/2}\ S_R$ (A2.15)

A1.7.3 The coefficient

The term $(2)^{1/2}$ is derived from the fact that required since r and R are equal to are defined as the maximum difference between two (single) test results that can be expected on the basis of a chance or random occurrence alone at the 5 % probability level or 95 % confidence level. The variance of the difference $(x_1 - x_2)$ for two values taken at random from a population is equal to the sum of the variances for values (of x) taken one at a time from the same population. Since there are two x values, the sum of the variances is simply the variance of x values times two and the square root places this term on a standard deviation basis.

A2.5.5.1 Thus $[(2)^{1/2}S_R]$ is the standard deviation of differences. The factor ϕ depends on both the total degrees of freedom (number of test results available) in the estimation for either of the variances $(\sigma)^2_r$ standard deviations and $(\sigma)^2_R$ and on the shape of the distributions of the variable bias terms and the (*c*) *E* terms. The normal assumptions for these terms are (1) unimodal distributions, (2) (*1*) the distributions are unimodal, (*2*) the number of test results not too small is sufficient (approximately 20), and (*3*) a confidence probability level (of $p = 0.05$) = 0.05 or confidence level of 95 % is chosen. Under these assumptions, the value of ϕ is similar to a *t*- value or approximately 2.0, and therefore Eq A1.10 the simplified expressions for r and Eq A1.11 may be rewritten as R are:

repeatability = $r = 2.83\ S_r$ (A2.16)

reproducibility = $R = 2.83\ S_R$ (A2.17)

A2. PRACTICE E691 CALCULATIONS FOR 'CELL AVERAGE' OUTLIERS:

A3. CALCULATING THE *h* VALUES

A2.1 *General Background*—Practice E 691 was originally introduced in 1979 as the basic document for performing precision analysis for all ASTM test method standards. It was most recently revised in 1987. The fundamental calculation algorithms for *r* and *R* used in Practice E 691 are the same as found in Practice D 4483 (1989 and current version), in ISO TR 9272 used by ISO TC45 and in the generic ISO standard, ISO 5725.

A2.2 Practice E 691 differs however from all of these other standards in the way it addresses outliers or potential outliers. The other standards evaluate potential outliers on the basis of (1) Cochran's test for within-cell variances, across all laboratories for each material, and (2) Dixon's test for within-cell averages, across all laboratories for each material. Practice D 4483 in its 1989 version allowed for the use of an alternative test for within-cell averages, a procedure called Tiejten-Moore's test (see discussion in A2.4). The Practice E 691 approach makes use of two new parameters called" consistency statistics," designated by the symbols ***h*** *AND* ***k*** *DATA CONSISTENCY STATISTICS*

A3.1 General Background

A3.1.1 The test results of a typical ITP when placed in a Table 2 and Table 3 format may well contain cell values that appear to be outliers. It is necessary to review the data and make a decision on how to treat these outliers. This should identify any one, two, or more potential outliers that have substantial deviations from the mean for a particular material in the database. Outlier treatment consists of rejection of all identified outliers using one of two options. Option 1 is the deletion of the outliers to generate a reduced size database. Option 2 is the replacement of the outliers by a procedure that maintains the character of the distribution of the non-outlier data.

A3.1.2 Some outlier rejection techniques use the difference between the outermost value and the adjacent value as the basis for rejection. This works well as long as potential outliers do not occur as pairs with minimal pair separation, but substantial separation from the nearest value in the database. Frequently, when this occurs, the rejection techniques fail to identify the outermost value(s) and the rejection iteration process stops.

A3.1.3 Both the General and the Special Precision sections of this practice use two particular parameters, called *consistency statistics*, to reject potential outliers, the *h* and *k*. The general philosophy of the Practice E 691 approach will be described in this annex values as well as the calculation algorithms for the developed by J. Mandel and used in Practice E 691. The *h*-value table. Calculation procedures and some additional discussion specific statistic is a parameter used to review the between-laboratory cell averages for potential outliers, and the *k*-values will be given in Annex A3.

A2.3 *Defining statistic is a parameter used to review the h Statistic*—The between-laboratory consistency statistic, cell standard deviations for potential outliers.

A3.2 Defining and Calculating the *h* Statistic

A3.2.1 *h-value*—The between-laboratory *cell average* consistency statistic, *h*, is calculated using the cell averages for all laboratories and is defined as follows for each material or *q* : level in the ITP.

$h = d/(S)\bar{x}$ (A2.1)

$$h = d / S(Y_{AV}) \quad \text{(A3.1)}$$

YAV (i) YAVS(YAV)

where:

d = [$\bar{y}_i - \bar{Y}$], Y $_{AV}$ (i) − Y$_{AV}$,
$\bar{y}_i Y_{AV}$ = cell average (being tested),individual cell average, for any laboratory (*i*),
(i)
Y_{AV} = average of *all* cells, for any material, and
(S)x = standard deviation of cell averages for any material or *q* level across all laboratories.
S(Y$_{AV}$)

A2.3.1 The

The *h*-value is the ratio of the deviation, *d*, of the each individual laboratory cell average for any laboratory *i*, from the overall cell average of for all laboratories, divided by the standard deviation among all the cell averages across all the laboratories. The special parameter *h*-value may be considered as a standardized variate (or *z*-function) with a mean of zero and a standard deviation of *l*.

A2.3.2 Large zero. Large *h*-values (+ or −) (plus or minus) indicate considerable substantial discrepancy from the overall zero average on the basis in multiples of a multiple of the cited *S* (Y_{AV}) standard deviation. Practice E 691 calculates deviation.

A3.2.2 *Calculating Critical h-values* —After an *h*-value is calculated for each laboratory for all materials, in distinction to the other precision standards that restrict their attention and calculation to suspiciously large within-cell standard deviations or to suspiciously small or large, within-cell averages for each material. The Practice E 691 procedure generates two additional tables that material, the values are analyzed for significantly high reviewed to determine if any of the calculated *h* (-values exceed a certain critical value. If a calculated *k* values; see Annex A3) indicating laboratories that are not consistent with the remainder (bulk) of the laboratories.

A2.4 *Benefits of the General Practice E 691 Outlier Approach*—The Practice E 691 technique of using *h* (and *k*) values is superior to the technique used by both Cochran's and Dixon's tests that uses the difference between the most extreme value (small or large in the case of Dixon's) and the value next in magnitude, as the basis for-value exceeds a test of significance for rejection of the most extreme value as an outlier. For situations where two extreme values lie close to each other and together they depart significantly from the remainder of the values, both the Cochran's and Dixon's tests fail to show the departure of the two values from the remainder of the non-suspect values. This was one of the advantages of the Tiejten and Moore test discussed above, since it looks at any number of suspicious values at the same time and avoids the masking effect of two (or more) outliers lying close to each other.

A2.5 critical *Decision on Significant h-values*—Practice E 691 takes an overly conservative approach on the issue of what is to be declared as a significant *h*-value (or-value, designated as *k*-value); it uses a 99.5 % confidence level to make this decision. This philosophy is based in part on a customary view held by statisticians, that outliers should rarely be eliminated from any interlaboratory test program (ITP). This view is based in large part on the supposition that the ITP is being done*h* (crit), at a preliminary stage in the development of a test method and that rejecting the outliers gives a false impression of the quality some selected probability or capability of significance level, the method. This view has merit for the initial phases of development for any new method and has some justification for an ITP with only a few laboratories and a few materials since it *h*-value in question is often difficult considered to decide if outliers for any laboratory are indeed different from the other laboratories.

A2.5.1 For well-established test methods however, the existence of a gradation of skill represent an outlier and general testing competency in any large group of laboratories, argues for a modified approach to the outlier issue. For precision evaluation of established test methods with a reasonably large number of participating laboratories with several materials, there is justification to reject outliers value for a particular laboratory on the basis of the more typical and universally used 95 % confidence level rather than a 99.5 % level. This is the approach as adopted in this practice.

A2.5.2 The 95 % confidence level approach will in general, reject the results of laboratories cell that have poor internal testing control and are in need of improved operating procedures. Allowing these "poor" laboratories to inflate generated the precision results (obtained if their results are not rejected) gives a false indication of the merit or inherent quality of any test method as used by those laboratories that take the time and effort to conduct testing with proper internal control. *h*-value is identified for outlier treatment. The precision value of the group of "good" laboratories (usually the majority of participating laboratories) should be the benchmark of test quality for any test method.

A2.6 *Calculating Critical h-values*—The critical value for *h*, *h* (crit),(crit) depends on the number of laboratories in the ITP and at for any-c probability or signifidecance level, it may be calculated in accordance with the following equation: by:

$$h(\text{crit}) = (p - 1)\, t / \{[p\,(t^2 + p - 2)]\}^{1/2} \quad \text{(A3.2)}$$

$$h\,(\text{crit}) = (p - 1)\, t / [p\,(t^2 + p - 2)]^{1/2} \quad \text{(A3.2)}$$

where:

p = number of laboratories in ~~ITP, and~~ the ITP,

t = ~~S~~student's *t* at selected ~~confidence~~ significance level, with ~~DF = (~~ df = (*p* ~~— 2) (a two-tailed t value).~~ − 2), a 2-tailed value, and

df = degrees of freedom.

~~A2.7 *Table of Critical h-values*—Table A2.1 gives calculated *h*-values, *h* (crit), at the 95 % confidence level (*p* = 0.05). These are the values as specified for the analysis of precision evaluation in accordance with this practice. If for well justified reasons another confidence level is desired for precision evaluation, it should be noted as a footnote in the precision table, the value of the alternative confidence level should be given~~

A3.3 Defining and Calculating the ~~reason for its adoption.~~

A3. PRACTICE E691 CALCULATION FOR CELL STANDARD DEVIATION OUTLIERS: *k*-VALUES

A3.1 The within-laboratory consistency statistic, designated as a ***k*** *Statistic*

A3.3.1 *k-value*—The *cell standard deviation* consistency statistic, *k*, is an indicator of how the within-laboratory variability (individual individual) cell standard deviation, under repeatability conditions) deviation for any selected laboratory, compares to the overall or pooled standard deviation. This comparison is done on a material overall (or level) by material basis. Values substantially greater than one indicate greater within-laboratory variation (for that cell) compared to the average for pooled across all laboratories.

A3.2 laboratories) *cell standard deviation*. The usual approach to tests of significance for variability statistics, is the use of an the *F*-ratio, a ratio of two variances. Therefore for the basic derivation of However, the *k*-value and the development is expressed as a ratio of tables of significant or critical two standard deviations since it is easier to comprehend this ratio when reviewing data. The *k*-values, the variance-value is used rather than standard deviation.

A3.3 The *k*-value is expressed developed as a ratio of two standard deviations because the ratio of standard deviations is easier to comprehend in reviewing data. The units for standard deviation are the same as the units of measurement for the test.

A3.4 In follows.

A3.3.2 In the usual *F*-ratio approach, the significance of any o in-dividual cell-variance compared to the pooled variance of all the cells (for any material) excluding the one cell being tested is given by Eq A3.1 by:

$$F = (S)^2 / [(\Sigma(Si)^2 / (p - 1)] \quad \text{(A3.1)}$$

$$F = S^2_{(i)} / [\Sigma\, S^2_{(p-i)} / (p - 1)] \quad \text{(A3.3)}$$

Σ S2(p − i) p

where:

(S)$S^2_{(i)}$ = cell variance being tested for potential significance, laboratory (*i*),
Σ(*Si*)Σ $S^2_{(p-i)}$ = sum of cell variances other than one being tested, variances, excluding cell (*i*), and
p = the number of laboratories in the ITP.

The within-laboratory consistency statistic, *k*, as calculated in the Practice E 691 computer program or as it should be calculated for a spreadsheet analysis,-value is defined for any selected cell by Eq A3.24 and is calculated for each material by:

$$k = (S) / (S)_r \quad \text{(A3.2)}$$

$$k = S(i) / S_r \quad \text{(A3.4)}$$

Sr

where:

(S) S(i) = cell standard deviation of the cell being tested, for laboratory (*i*), and
(S)S_r = repeatabilitypooled cell standard deviation (for any selected material) (this (across all laboratories), this is the pooled value over all laboratories). initially calculated repeatability standard deviation (see Eq A3.5).

A3.53.3 *Calculating Critical k-values* —For purposes of calculating critical *k*-values to evaluate potential significance for any selected cell,-values, designated as *k* (crit), the following development is presented. The repeatability variance is given by Eq A3.35:

$$(S)^2_r = [\Sigma(Si)^2 + (S)^2] / p \quad \text{(A3.3)}$$

$$S^2_r = [\Sigma\, S^2_{(p-i)} + S^2_{(i)}] / p \quad \text{(A3.5)}$$

Combining Eq A3.13, Eq A3.24, and Eq A3.35 gives Eq A3.46:

$$k = \{[[[p / (1 + (p - 1) / F)]]]\}^{1/2} \quad \text{(A3.6)}$$

$$k = \{[p / (1 + (p - 1)) / F]\}^{1/2} \quad \text{(A3.6)}$$

The degrees of freedom (DF) freedom, df, for *F* in Eq A3.46 are (*n* − 1) for the numerator and (*p* − 1)(*n* − 1) for the denominator.

A3.6 denominator, where *n* = number of replicates per cell. Eq A3.46 may be used to calculate critical *k*-values, *k* (crit),(crit) for any values of *p* and *n*, at any a selected confidence level, significance level by reference to the applicable critical *F* value at the indicated DF values. Table A3.1 gives critical *k*-values at the 95 % confidence level (*p* = 0.05) df for various numbers of

laboratories, for $n = 2$ numerator and 3, cell replicate values.

A4. ESTABLISHING A FUNCTIONAL RELATIONSHIP BETWEEN r (OR R) and M

A4.1 A functional relation between r (or R) and M may or may not exist. The reasoning and computational procedures presented as follows may apply to r, R, (r), and (R). They are presented for r only. Only three types denominator.

A3.4 Identification of relationships will be considered:

A proportionality relation:

$$r = \nu M \tag{A4.1}$$

A linear relation:

$$r = u + \nu M \tag{A4.2}$$

A logarithmic relation:

$$\log r = c - d \log M \tag{A4.3}$$

or its equivalent:

$$r = CM^{d} \tag{A4.4}$$

A4.2 Eq A4.3 and also A4.4 when $d > 0$ (general case) will then lead to $r = 0$ for $M = 0$, which may seem unacceptable from an experimental point of view. Frequently, Outliers Using the values of Critical M encountered in practice will have a lower limit larger than zero such that these equations can be used without introducing serious systematic errors.

A4.2.1 For $u = 0$ and $d = 1$, Eq A4.2 and Eq A4.3 will be identical to Eq A4.1, and when u lies near zero or d, or both, lies near unity. Two or all three of these equations may yield practically equivalent fits. In that case, Eq A4.1 should be preferred because it involves only one parameter and, therefore, permits a simple statement.

A4.2.2 If, in a plot of r_j against M_j, or log r_j against log M_j, the set of points is found to lie reasonably close to a straight line, a line drawn by hand may provide a satisfactory solution, but if for some reason a numerical method of fitting is preferred, the procedure of Eq A4.3 is recommended.

A4.3 The fitting of a straight line is complicated by the fact that both M and r are estimated. Since the slope, ν, is usually small, of the order of 1 or less, errors in M have little influence and the errors in r predominate. The purpose is to derive values of r for given values of M; therefore, a regression of r on M is appropriate. This should be a weighted regression because the standard error or r is proportional to the value of r. With weights W_j for r_j, the computational formulas are as follows:

$$S_1 = \sum_j W_j, S_2 = \sum_j W_j M_j, S_3 = \sum_j W_j M_j^{2}, \tag{A4.5}$$

$$S_4 = \sum_j W_j r_j, \text{ and } S_s = \sum_j W_j M_j r_j \tag{A4.6}$$

Then, for Eq A.1

$$\nu = S_5 / S_3 \tag{A4.7}$$

and for Eq A4.2,

$$u = \frac{S_3 S_4 - S_2 S_5}{S_1 S_3 - S_2^{2}} \tag{A4.8}$$

$$\nu = \frac{S_1 S_5 - S_2 S_4}{S_1 S_3 - S_2^{2}} \tag{A4.9}$$

A4.4 The weights, W, must be proportional to r^{-2}, but the values of r_j are subject to errors; the same will hold for the weights. To correct for these and reduce the errors in the final equation, the following iterative procedure is recommended:

A4.4.1 Writing r_{oj} for the original values of r obtained by one of the calculation procedures, apply the above equations for u or ν with weights:

$$W_{oj} = r_{oj}^{-2} (j = 1,2, \ldots q) \tag{A4.10}$$

which results in equations

$$r_{1j} = v_1 M_j \text{ or } r_1 = u_1 + v_1 M_j \quad \text{(A4.11)}$$

From these are computed adjusted values of r_j,

$$r_{1j} = v_1 M_j \text{ or } r_{1j} = u_1 + v_1 M_j \; (j = 1,2, \ldots q) \quad \text{(A4.12)}$$

and the computations are then repeated with the adjusted weights $W_{1j} = r_{1j}^{-2}$ giving

$$r_2 = v_2 M \text{ or } r_2 = u_2 + v_2 M \quad \text{(A4.13)}$$

A4.4.2 The step from W_{oj} to W_{1j} is effective in eliminating gross errors in the weights, and the equations r_2 should be considered as the final result.

A4.5 The standard error of log r is approximately proportional to $V(r)$, the coefficient of variation of r. Since the standard error of r is proportional to the value of r, the standard error of log r will be independent of r and an unweighted regression of log r on log M is appropriate when Eq A4.3 is considered.

A4.5.1 For Eq A4.3 the computational formulas are as follows:

$$S_1 = \sum_j \log M_j, \qquad S_2 = \sum_j (\log M_j)^2, \quad \text{(A4.14)}$$

$$S_3 = \sum_j \log r_j, \qquad S_4 = \sum_j (\log M_j)(\log r_j). \quad \text{(A4.15)}$$

and

$$c = \frac{S_2 S_3 - S_1 S_4}{q S_2 - S_1^2} \quad \text{(A4.16)}$$

$$d = \frac{q S_4 - S_1 S_3}{q S_2 - S_1^2} \quad \text{(A4.17)}$$

A5. PROCEDURE FOR CARBON BLACK PRECISION EVALUATION

A5.1 *Introduction*—The evaluation of precision for the test methods of Committee D-24 on Carbon Black shall be conducted in accordance with the procedure outlined in this annex. This procedure differs from the requirements as set forth in the main test of this practice. Each cell of the basic precision format table (Table 1 of this practice) contains four values as described as follows. The cell averages and cell standard deviations are used to examine outlier characteristics of the interlaboratory database by means of a protocol that differs from the basic Practice D 4483 protocol. Additionally, special calculations are made in this annex to select the mode of precision expression (absolute or relative) that is most free of influence by the level of the measured property. This special annex procedure is used so that (*1*) all carbon black test method precision programs are conducted in the same manner, and (*2*) precision results can be compared across the tests normally employed in the carbon black manufacturing industry.

A5.2 *Terminology*—The terminology used for D-24 precision sections shall be in harmony with the terminology as used in Practice D 4483. The word 'sample' shall not be used in place of the word 'material' when discussing the number of labs, materials, days and replicates for any ITP. Samples in the context of Practice D 4483 are representative portions (or pieces) of a material scheduled for testing that are sent out to each laboratory in the ITP.

A5.3 *Materials Selected and Data Collection*—The number of materials (carbon blacks) for the precision program shall be selected based on the recommendations of Section 6. For the operations as described in this annex it is recommended that at least five materials be selected. This number of materials provides for four degrees of freedom in evaluating the significance of the coefficient of determination as described in A5.5. Tests on the selected 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. A test result is the average or median of a number of individual determinations (measurements) as specified by the method. Record all values as indicated in Table A5.1 for each material and laboratory. Each set of four values in the Table A5.1 arrangement, constitutes one cell of the final data tabulation of the entire interlaboratory test program when all the data are arranged in the basic Practice D 4483 Table 1 format. All testing shall be conducted on the same test machine or apparatus.

A5.4 *Table A5.1 Data Review and Calculations:*

A5.4.1 For each material and each laboratory calculate the average, designated as the cell average and the standard deviation, designated as the cell standard deviation, of the four values as listed in Table A5.1 format. These two statistics (cell average, cell standard deviation) are used to review the laboratories for internal testing consistency (outlier behavior) on a material-by-material basis. Although both of these statistics contain two undifferentiated components of variation, that is, between tests-between days

and between tests within a day, each statistic serves as a useful index for the internal consistency (outlier) comparison.

A5.4.2 *Reviewing the Cell Averages*—Arrange the data for all laboratories and materials in the format of Table A5.2. For each material calculate ***h*****-***values for the column of cell averages as specified by the procedures outlined in Annex A2. Also in accordance with the procedures of Annex A2, calculate the 95 % confidence level critical h-value, h (crit).*

A5.4.3 *Reviewing the Cell Standard Deviations*—For each material calculate the *k*-value for the column of cell standard deviations as specified by Annex A3. Also calculate for each material the 95 % confidence level critical *k*-value, *k* (crit), in accordance with Annex A3.

A5.4.4 The determination of outlier laboratories is done independently for average, using the *h*- and *h* (crit) values and standard deviation, using***k*** Values

A3.4.1 When all the *k*-*h* *and k (crit) values. Outlier laboratories are determined* *values have been calculated using Eq A3.1 and Eq A3.4 respectively, and tabulated for any database generated by comparing a particular ITP, they are reviewed to determine if any of the calculated h-h or and k-value tok values exceed the critical h and (crit) or k (crit) value, respectively. The absolute value of values.*

A3.4.2 Table A3.1 gives the calculated 2 % and 5 % significance level (or *h*-value is used for this comparison. Laboratories are rejected in order from highest to lowest absolute calculated*p* = 0.02, *h*- *p = 0.05) critical values for both k-value exceeding the h and (crit) or k (crit) value, respectively,, for each material, until:*

(a) various numbers of laboratories, all outliers have been rejected *p* = 3 to 30, and the number of remaining laboratories is twenty, or greater, OR

(b) cell replicates, only twenty un-rejected laboratories remain, including some within the lower range of *h*-*n = 2, 3, or k-values exceeding 4. This is used for the h (crit) or k (crit) value, respectively.*

If twenty or fewer laboratories participate in the study, reject only one laboratory two-step procedure for each material for average or standard deviation. If no laboratories exceed *h* (crit), retain all average data. If no laboratories exceed *k* (crit), retain all standard deviation data.

A5.4.5 After reviewing the review of data as specified in A5.4.2 to A5.4.4, the issue of blank cells (missing values) in the basic Practice D 4483 Table 1 format needs to be addressed. Refer to 7.5 and 7.6 database for pothential outliers as well as Section described in Sections 8 for the precision calcuations.

A5.5 *Relationship Between Reproducibility Precision Parameters and M:*

A5.5.1 This section gives the necessary instructions 9.

A4. SPREADSHEET CALCULATION FORMULAS FOR PRECISION PARAMETERS, RECOMMENDED SPREADSHEET TABLE LAYOUT AND DATA CALCULATION SEQUENCE

A4.1 Calculation Formulas

A4.1.1 When a dedicated computer program is not available to select calculate precision, the type of repeatability and reproducibility may be calculated using typical spreadsheet procedures and algorithms. The final precision parameter; either the absolute, *R*, expressed in measurement units calculations involve a series of sums or the relative (*R*) expressed totals. The calculation formulas are given in p this section. In A4.2 a recommended spreadsheet, table layout is presented that facilitates the calculations. A4.3 gives some recommendations for setting up the most general expression of precision. General expression of precision table sequence and conducting the analysis. Fig. 1 is a defcision tree diasgram that mode of expression that has the least dependence gives guidance on the measured property level, *M*, the average material value over all laboratories.

A5.5.2 Calculate the precision parameters as specified in Section 8, on the Table A5.2 database remaining after applying the procedures sequence of 7.5 for missing values. Plot the values of steps. Recall that *Rp,* and *(R) versus M. Perform a least squares regression for each = number of laboratories in the two parameters, R* and *(R), and record the coefficient of determination, designated ITP.*

NOTE A4.1—The calculations were set up for this practice as CD, for each parameter.

A5.5.3 Select for the mode of precision expression, the parameter with the lowest CD, annex using Lotus 123. It is assumed that is, either *R* for absolute expression or (*R*) for percent expression. This establishes which any spreadsheet program can be used, however some of the two modes of expression is to particular algorithms may be u slightly different than indicated in preparing a table of precision parameters in the precision section of the test method standard. If this annex.

A4.1.2 *Uniform Level ITP Design, R n*has the lowest CD, use the absolute mode; if *(R) has the lowest CD, use the relative mode. The mode of expression selected applies to both the reproducibility and repeatability parameters of the table of precision results.*

A5.5.4 Allowing for the decision on precision parameter selection made = 2—All laboratories in A5.5.3, follow the instructions as set forth in Section 9 for general guidance in preparing the final table(s) of precision results and the accompanying precision statements for the ITP test method standard. In preparing these statements, it should be made clear whether the precision applies to individual mean levels in a table or to pooled values.

A6. SPREADSHEET CALCULATION FORMULAS FOR PRECISION PARAMETERS

A6.1 all materials; each material has *With n = 2n = 2 replicates per cell:*

cell and the summations are over all laboratories.

$$T_1 = \Sigma \bar{y}_i \quad \text{(A6.1)}$$

$$T_1 = \Sigma\, Y_{AV} \quad \text{(A4.1)}$$

where:
Y_{AV} = cell average for laboratory (*i*).

$$T_2 = \Sigma(\bar{y})^2 \quad \text{(A4.2)}$$

$$T_2 = \Sigma\, (Y_{AV})^2 \quad \text{(A4.2)}$$

$$T_3 = \Sigma(W_i)^2 \quad \text{(A4.3)}$$

$$T_3 = \Sigma\, (w)^2 \quad \text{(A4.3)}$$

where:
w = range of cell values, laboratory (*i*).
(for $n = 2$ only)

$$T_4 = \Sigma(S_i)^2 \quad \text{(A4.4)}$$

$$T_4 = \Sigma\, (S)^2 \quad \text{(A4.4)}$$

NOTE A6.1—Use

where:
S = cell standard deviation, laboratory (*i*).
For the calculations as outlined as follows use either T_3 or T_4.

$$S_r^{2} = T_3^{2p} = \frac{T_4}{p} \quad \text{(A6.5)}$$

$$S^2_r = T_3 / 2p = T_4 / p \quad \text{(A4.5)}$$

$$S_L^{2} = \{[p\,T_2 - T_1^{2}p(p-1) - \left|\frac{S_r^2}{2}\right| \quad \text{(A6.6)}$$

$$S^2_L = \{[p\,T_2 - T_1^{2}p(p-1) - \left|\frac{S_r^2}{2}\right| \quad \text{(A4.6)}$$

$$S_R^{2} = S_L^{2} + S_r^{2} \quad \text{(A6.7)}$$

$$M = T_1 / p \quad \text{(A6.8)}$$

$$M = T - (T_1)^2 / p\,(p-1) - [S^2_r / 2] \quad \text{(A6.8)}$$

$$r = 2.83\sqrt{s_r} \quad \text{(A6.9)}$$

$$S^2_R = S^2_L + S^2_r \quad \text{(A4.7)}$$

$M_{AV} = T_1 / p$, material average for all laboratories (A4.8)

$$r = 2.83\,(S^2 \quad \text{(A4.9)}$$

$$r = 2.83\,(S^2_r)^{1/2} = \text{repeatability} \quad \text{(A4.9)}$$

$$R = 2.83\sqrt{S_R^2} \quad \text{(A6.10)}$$

$$R = 2.83\,(S^2_R)^{1/2} = \text{reproducibility} \quad \text{(A4.10)}$$

NOTE A6.2—If

A4.1.3 For any ITP with $s_L^{\,2}$ is negative, substitute $s_L^{\,2} = 0$ in Eq A6.7.

NOTE A6.3—Symbols used:

$\bar{y}_i$ or $\bar{y}$ = average cell (test result) value,
W_i = range*n* equal to more than two but with a constant number of cell-*y* values (for replications for each material-laboratory combination, the computation equations are identical to Eq A4.1-A4.10 with the following exceptions: (*1*) the value of $n = 2$ only),
S_i = cell standard deviation,
M = average of all *y* values (for each level), and
p = number of laboratories.

See Section 8 for other symbols used.

A6.2 *With n > 2 (a constant value over all cells)*—The computational equations are identical to A6.1 except that the value of n is used in place of 2 in the denominator of the second last term of Eq A6.6. The value of A4.6, and (*2*) sTr_3 *is not calculated, the value for* S^2_r *is obtained by means of the* T_4/p *expression of in Eq A6.5.*

A6.3 *With A4.5.*

A4.1.4 For any ITP with an unequal numbers of n replicates per cell:

$$T_5 = \Sigma n_{\bar{y}_i} \quad \text{(A4.11)}$$

$$T_5 = \Sigma\,[n_i\,(Y_{AV})_i],\ n_i = \text{number of replicates in cell } i \quad \text{(A4.11)}$$

$$T_6 = \Sigma n_i (\bar{y}_i)^2\ T_7 = \Sigma n_i \quad \text{(A6.13)}$$

$$T_6 = \Sigma\,(n_i)\,(Y_{AV})^2_i \quad \text{(A4.12)}$$

$$T_7 = \Sigma\,(n_i) \quad \text{(A4.13)}$$

$$T_8 = \Sigma(n_i)^2 \quad \text{(A4.14)}$$

$$T_8 = \Sigma\,(n_i)^2 \quad \text{(A4.14)}$$

$$T_9 = \Sigma(n_{r-1})(S_i)) \quad \text{(A4.15)}$$

$$T_9 = \Sigma\,(n_i - 1)\,(S^2_i) \quad \text{(A4.15)}$$

where:
S^2_i = variance for cell *i*.

$$S^2 \quad \text{(A4.16)}$$

$$S^2_r = T_9 / (T_7 - p\,) \quad \text{(A4.16)}$$

$$S_r^{2} = \{+[T_9](T_7 - p) \quad \text{(A6.16)}$$

$$S^2_L = \{ \mid [T_9(T_7 - p)$$ (A4.17)

$$S_L{}^2 = \left(\frac{T_6 T_7 - T_5{}^2}{T_7(p-1)} - S_r{}^2 \right) (T_7(p-1)] \; T_7{}^2 - T_8$$ (A6.17)

$$S_L{}^2 = (_{T_6 T_7 - (T_5)^2}] / [T_7(p-1)] \; - S^2_r \{[T_7(p-1)] / [(T_7)^2 - T_8]\}$$ (A6.17)

$$S_R{}^2 = S_L{}^2 + S_r{}^2$$ (A6.18)

$$S^2_R = S^2_L + S^2{}_r$$ (A4.18)

$$M_{AV} = T_5 / T_7$$ (A4.19)

Calculate *M*, *r*, and *R* as in accordance with A6.1 using:

$$M = \frac{T_5}{T_7}$$ (A6.19)

A7. AN EXAMPLE OF PRECISION CALCULATIONS—MOONEY VISCOSITY TESTING

A7.1 *Introduction*—The calculations illustrated in this Mooney viscosity example are performed using the spreadsheet analysis technique rather than the Practice E 691 computer analysis. This approach can better demonstrate the operations required Eq A4.9 and A4.10.

A4.2 Table Layout for the various analysis steps. The data in this example, which were obtained in an interlaboratory test program (ITP) in 1982, are the same as used for the example in the previous version Spreadsheet Calculations

A4.2.1 *Table Organization*—This section contains a listing of this practice, that is, Practice D 4483 – 89. Although all the precision calculation algorithms have not changed for this current version tables required with a brief description of Practice D 4483, the outlier rejection technique has changed, that is, it is conducted by means of linking between the Practice E 691 tables to permit all calculations to be automatically performed to give the values for *r* *h*value and *k*-value analysis. This is in contrast to the previous Practice D 4483 – 89 technique of using the Dixon's Outlier test for cell averages*R*, once all tables have been set up and the Cochran's Maximum Variance test for cell variances (standard deviations).

A7.2 *Details on the Precision ITP*—The Mooney viscosity measurements were made in accordance with Test Methods D 1646. basic table of data has been generated. The ITP was conducted layout is for seven different materials (rubbers) as illustrated in Table A7.1, which also lists the conditions of test. On each of two separate test days, one week apart, the Mooney viscosity of each of the materials was measured one time; therefore a *test result* is a single determination. In the nomenclature of a Table 1 format (see 7.2), $p = 11$, $q = 7$ and uniform level design with $n = 2$. The precision evaluated was a Type description is directed mainly to Analysis Step 1. If outliers are found for Step 1, although there were some preliminary mill-massing steps then the calculation operations of Step 2 and perhaps Step 3 will be required. For a full understanding of these two additional steps, it is necessary to completely review the precision evaluation example in Annex A6, which gives instructions for each rubber, these additional calculations.

A4.2.2 For this annex, the tables will be identified as c Table A4.1, Table A4.2, and so forth. Each of these is set up for a specific calculation. However, to avoid having blank tables (with the appropriate format as discussed in this annex) added to the (1982) Section 7 specifications length of Test Method D 1646, prior the standard, the reader is referred to viscosity measurement.

A7.3 The basic or raw Annex A6. Annex A6 contains each table as discussed in Annex A4, filled in with data frobm tahe Mooney viscosity precision example. Therefore, when the set up for Table A4.1 format is discussed in this annex, refer to the ITP corresponding table in Annex A6, which is Table A6.1 which gives both the table format and actual data. Starting with Table A4.1, the numerous calculations on these data are presented in a series of tables differ from the format of Tables 2 and 3 in the main body of this annex. The primary tables, starting with Table A7.2, are indicated by practice, in the use of a double or side-by-side data display format. This double table number after the annex designation, A7. Tables that are derived from setup permits a primary table, are indicated by quick view of the datab and calculated parameters as data is entered and processed.

A4.2.3 There are potentially three analysis operation steps for any ITP. The number of steps actually required depends on the primary table with an appended letter designation to distinguish quality or uniformity of data in the database. If outliers are found, then a second and perhaps a thivrd analysis step will be required. Each of these analysis operations should be conducted on a separate *sheet* or secondary table from tabbed page of the primary table. Thus tables with computer spreadsheet program. This

facilitates the same root number but with different letter designations, that is, a, b, etc. analysis and avoids confusion. If outliers are directly related found for any analysis operation, there are two options to each other.

A7.4 *Preliminary Analysis Data Review*—Table A7.2 lists continue with the Day 1–Day 2 data analysis.

A4.2.3.1 *Outlier Option 1: Removal by Cell Deletion*—The simplest option for outliers is the seven materials and deletion of the e outlievr from the database as expressed in l a Table A4.1 format. See A4.3.2 fori more details on this. T

A4.2.3.2 *Outlier Option 2: Cell Replacement Values for Outliers*—If this option is given chosen, cell replacement values are calculated by the procedures as described in Annex A5. This option involves more work but it may be the required Table 1 format only option for a limited ITP database with a small number of 7 laboratories.

A4.2.4 The three potential analysis steps are described in Sections 8-10. 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 bottom extent of outliers in the database. The table are given description outlined as follows is for Analysis Step 1, the day averages, first set of calculations for any ITP, (see Section 8), prior to the 2-Day averages, possible rejection of any incompatible values as outliers.

A4.2.4.1 The word *cell* is used in two different contexts; it is the between-laboratory standard deviation intersection of each day a row with a column in a computer spreadsheet, and the pooled between-laboratory standard deviation over both day columns. Although these are not specified it is also, for any ITP, the combination of a laboratory and a material as in Table 1 format, they are easy in the main body of this practice. The word cell will be italicized when it refers to a computer spreadsheet. In many cases there is a dual usage or meaning, a Table 1 cell is also a spreadsheet cell.

A4.2.4.2 Although described as follows, a Table A4.1 may contain blank table *cells*. All table cells that have data must contain the number of replicate values characteristic of the design of the ITP. For most General Precision ITP, $n = 2$ and each cell must contain both values. The original database generated in some ITPs may be useful one where one or more laboratories report only one value for a particular material, that is, they did not fully participate and only supplied partial data. The partial data review.

A7.5 for such a laboratory cannot be used since the spreadsheet program as set up in this annex requires that all Table A4.1 *Full Analysis—Part 1:cells*

A7.5.1 *Part 1: Cell Averages*—The data (for Analysis Step 1, 2, or 3) be uniform, that is, have the required number of replicates or no values at all.

Table Number and Name	Table Description
Table A4.1 Basic Data from ITP	This is the basic Table A7.2 areused to construct Table A7.3, a table of a Table A4.1 format, with actual data entered.
Table A4.1 Basic Data from ITP	This is the basic Table 1 format (as discussed in the main body of this practice); Rows = Laboratories; Columns in Replicate 1, 2 format = materials. Two spreadsheet columns are required for each material. Each (double column) ITP cell contains two test results. In generating all tables beyond Table A4.1, preserve the same row-column identification for laboratories and materials. Remember, go to Annex A6, Table A6.1, for an example of a Table A4.1 format, with actual data entered.
Table A4.2 Cell Averages, Averages Squared	This is a dual table, cell averages by using the usual spreadsheet calculation operations. See Note A7.1. At thebottom of the cell average table, three parameters are calculated for each material: the material cell average (average of all cell averages); the cell average standard deviation; and cell average variance, that is, (*S*) $\bar{x}$ and (*S*) $\bar{x}_2$, given in the table by the symbols STD and VAR.
Table A4.2 Cell Averages, Averages Squared	This is a dual table, cell averages in left side and cell averages squared in the right side, each side preserving the laboratory-material row versus column format of Table A4.1. Totals are calculated for each material column; cell average totals = T_1, cell average squared totals = T_2. Also calculate for the left section, the grand cell average (all laboratories), the variance, and standard deviation of the cell averages (across all laboratories) . Note—Do not truncate the significant figures for any total in any of these tables. Retain 4 significant digits for all calculations.

NOTE A7.1—The spreadsheet calculations were carried out with the @Avg, @Stds, @Vars, and the @Sum functions as called in addition to other typical spreadsheet cell calculation procedures.[6]

A7.5.2 Using the material cell average (of each material), the cell deviation table was calculated by subtract average from the individual cell average for each laboratory on a material-by-material basis (see Table A7.4 cell deviations, a table of these tables. Retain 4 significant digits for all calculations.

Table Number and Name	Table Description
~~Table A4.3 Cell Average Deviations, *h-values* was calculated by dividing each cell deviation by the applicable material cell average standard deviation. This operation yields Table A7.5. The critical *h* values~~	~~A dual table, *h* (crit), is obtained from Table A2.1 (in Annex A2); for eleven laboratories *h* (crit) is 1.81. See Annex A2 for *h*-value analysis~~
	~~A7.5.3 Reviewing Table A7.5 for observed *h*-values that exceed (crit) indicates that there are seven critica~~
	~~3-Material 4, Laboratory 8-Material 2, Laboratory 10-Material 1, Laboratory 10-Material 5, Laboratory 11-~~
	~~11-Material 6,-values.~~
Table A4.3 Cell Average Deviations, *d* and *h*-values	A dual table, cell deviations *d*, *d* = cell (*i*) − (all cell avg); in the left section and cell *h*-values in the right section. Review the cell *h*-values cant at the 5 % level in some manner appropriate for the spreadsheet being used, such as making the value bold and italic, shaded, or for calculation of *h*-values.
~~Table A4.4R Cell Ranges and Laboratory 11-Material 7. Laboratories 10 Ranges Squared~~	~~A dual table, cell ranges on left and 11 do not agree well with the overall 'average' viscosity values.~~
	~~A7.5.4 *Part 1: Cell Standard Deviations*—A table of standard deviations was generated by applying th~~
	~~deviation calculation function to the Day 1–Day 2 values of Table A7.2. This operation yields Table A7.6~~
	~~table the variance cell squared totals T_3 for each material.~~
Table A4.4R Cell Ranges and Ranges Squared	A dual table, cell ranges on left and cell ranges squared on the right. For each left-hand-side cell, the cell range may be obtained from Ta spreadsheet function (such as @IF or ABS) to convert those negative difference values to positive values for the cells of Table A4.4R. I age range for each material. Calculate the cell squared totals T_3 for each material.
~~Table A4.4S Cell Standard Deviations and Variances~~	~~A dual table, with cell standard deviation are given as pooled values (over the eleven cell values for each material). Table A7.7 is generate~~
	~~value of Table A7.6 to give a table of cell standard deviations squared, that is, variances. At the bottom of Table A7.7 the pooled cell va~~
	~~The square root of this is used next to generate Table A7.8, a table of *k values* obtained by dividing each *individual* material cell standa~~
	~~material cell standard deviation.~~
	~~A7.5.5 Table A7.7 is not required to calculate a table of T_4.~~
Table A4.4S Cell Standard Deviations and Variances	A dual table, with cell standard deviations on the left and cell variances on the right. It is convenient to calculate the pooled variance for e viations. Place these at the bottom of each left-hand-side column. Calculate the total for the cell variances; place these values at botton ances on the right side. Total of cell variances for each material = T_4.
~~Table A4.5 Cell *k*-values as described in A7.5.4 because Table A7.6 has the necessary information to calculate the~~	~~A single table, cell *k*-values, that is, individual cell standard deviations and the pooled 'cell standard deviation' for each material. It is given because it will be needed (in modified format) in the process of replacing the to-be-rejected outlier values as described in A7.6.~~
	~~A7.5.6 Reference to Annex A3, Table A3.1, for p = 11 and n = 2, yields a critical *k*-value, *k* (crit), of 1.~~
	~~A7.8 indicates that there are five observed *k*-values that exceed *k* (crit); Laboratory 2-Material 1, Lab~~
	~~Laboratory 6-Material 6, Laboratory 6-Material 7, and Laboratory 11-Material 3. Laboratory 6 demonstrate~~
	~~repeat the viscosity measurements on the indicated Day 1–Day 2 basis.~~
	~~A7.5.7 Although value bold and italic.~~
Table A4.5 Cell *k*-values	A single table, cell *k*-values. See Annex A3 for calculation of *k*-values. For each *k*-value that equals or exceeds the 5 % significance level the value bold and italic.
~~Table A4.6 Calculations for Precision~~	~~A table giving thenextste p is not strictly required for an analysis of precision, it is included in this example to illustrate the difference in the~~
	~~sion parameters *Sr* and *SR*, calculated (1) on the original database (no outliers rejected), and (2) on the adjusted database after all outl~~
	~~placed with the special average values. Table A7.9, Part A, lists the values of the primary calculated variances (Sr^2 and $(S)\ x^2$ for each~~
	~~calculate the intermediate parameter $(S2)'$ and Calculation 5 evaluates *R*.~~
Table A4.6 Calculations for Precision	A table giving the sequence of calculations for precision. The calculations are performed for each material separately, thus a column is rec sert values for T_1, T_2, and either T_3 or T_4, by means of spreadsheet linking to the appropriate preceding tables. Calculation 1 is a calcu T_3 or T_4. Calculation 2 evaluates $(S_L)^2$ using T_1 and T_2. Calculation 3 is a calculation of $(S_R)^2$, using $(S_L)^2$ and $(Sr)^2$. Calculation 4 evalu evaluates *R*.
	~~At the calculations that yield the final parameters *Sr* and *SR*. Refer to Section 8 for the governing equations. Step 1 and if used, Step 2 o~~
	~~and 2 % significance levels. This sub-table indicates the outlying laboratories for both *h* and *k*.~~
	At the bottom of Table A4.6, material means (averages) are given as well as the standard deviations *Sr* and *SR*. Also listed is a sub-table Step 2 or 3 outlier review at the 5 % and 2 % significance levels. This sub-table indicates the outlying laboratories for both *h* and *k*.
	~~Note: The original database pooled values are: *Sr* = 0.82 and *SR* = 2.44.~~
	~~A7.6 *Rejection and Replacement of (Spreadsheet) Outlier Values:*~~
	~~A7.6.1 *Cell Average Replacement*—The rejected cell averages have been indicated in A7.5.3. Table A7.~~
	~~by replacing the rejected cell averages by special cell averages that preserve the *recalculated cell average* an~~
	~~*standard deviation. of Table A4.6. For a fill-in operation, the values in Table A4.6 must be inserted manu~~
	Note: The values for *n* and *p* in Table A4.6 can either be active or be a fill-in format. The value of *n* will be 2, but *p* will vary depending on laboratories deleted for either *h* or *k* values. For active *p* values, a count function should be performed for the cell values in Table A4.5- A4.3.1, for each material. This counts the number of laboratories after both *h* and *k* deletions. The result enters the appropriate c operation, the values in Table A4.6 must be inserted manually.

A4.2.5 *Setting up the Spreadsheet* ~~This is done—~~Begin on Sheet 1 of a ~~material-by-material basis.~~ spreadsheet program. This will be used for Analysis Step 1. The ~~recalculated cell average~~ first set of calculations is for the original database. For any subsequent analysis operations with a complete set of recalculations after outliers are removed from the database or outliers replaced, one or more additional computer program sheets will be used. Calculations are facilitated if each table occupies a single screen area, using the *page down* command to go to the next table. Refer to Annex A6 for more details on Steps 2 and 3.

A4.2.5.1 *Link Table A4.2 to A4.1*—For Laboratory 1 and Material 1, use the appropriated spreadsheet average function (such as an @function or AVERAGE) to calculate the average for *Cell* 1 in Table A4.2, using the corresponding two adjacent (spreadsheet) *cells* on Row 1 of Table A4.1, for Laboratory 1 and Material 1, as the argument spreadsheet range. Repeat for all ~~cells (for that material) omitting~~ table *cells*. After this is completed, calculate the ~~outlier~~ cell average squared value.s for all *cells*

on the right side of Table A4.2 by the appropriate spreadsheet squared function algorithm using the left-hand-side *cell* averages.

A4.2.5.2 *Link Table A4.3 to A4.2*—For Material 1, using the appropriate spreadsheet algorithm, subtract from each laboratory *cell* average ~~can be easily obtained~~ in ~~spreadsheet calculations~~ the left-hand-side of Table A4.2 the overall *cell* average. This gives *d*. Divide each calculated *d* by ~~erasing~~ the standard deviation of all *cell* averages to give the calculated *h*-value. Repeat for ~~c~~ all materials. The calculation output for *h*-values is entered into the corresponding (row-column) *cell* in ~~a table, producing a null or missing cell value (but not a zero or 0.0 value).~~

~~A7.6.2 The location~~ the right-hand-side section of Table A4.3.

A4.2.5.3 *Link Table A4.4 to Table A4.1* —For Laboratory 1 and Material 1, calculate the ~~rejected outliers are indicated~~ standard deviation for *Cell* 1 in Table ~~A7.10~~ A4.4, by means of the appropriate spreadsheet function for standard deviation, using the corresponding two adjacent *cells* on Row 1 of Table A4. ~~E~~1 (Laboratory 1 and Material 1), as the spreadsheet argument range. Repeat for all materials or *cells*. Ensure that the divisor for standard deviation calculation is ($n - 1$), not n, where n = number of values for standard deviation calculation for each material. In spreadsheet terminology, this is often designated as a ~~re~~ *sample standard deviation* calculation. Using the appropriate algorithm, square each *cell* standard deviation value; the result is ~~equal to~~ entered into the ~~recalculated material cell average. Compare~~ corresponding *cell* on the ~~recalculated values of Table A7.10 with~~ variance or the ~~original database values~~ right side of Table ~~A7.3.~~

~~A7.6.3 *Cell Standard Deviation (Variance) Replacement*—The rejected cell standard deviation values have been indicated in A7.5.6.~~ A4.4.

A4.2.5.4 *Link Table ~~A7.11 has been generated by replacing the rejected~~ A4.5 to A4.4S*—For Material 1, divide each individual (within) cell standard ~~deviations squared~~ deviation, by the ~~special~~ pooled value for (within) cell standard deviations ~~squared, that preserve~~ (this is the square root of the pooled ~~recalculated cell standard deviations squared, designated by~~ or mean variance) to obtain ~~*Sr*~~*k-values. Repeat for all materials. The k-values are entered into the corresponding cells in Table A4.5.*

A4.2.5.5 *Link Table A4.6 to Tables A4.2, A4.4S, or A4.4R, or Combination Thereof*—For Material 1, use the appropriate spreadsheet function or algorithm to bring the totals T_1, T_2~~. Again~~, T_3, or T_4, or combination thereof, into Table A4.6. Repeat this for all materials. The source for each total should be the total at the bottom of each of the appropriate columns in Tables A4.2, A4.4S, or A4.4R. For Calculation 1 in Table A4.6, use the formula given in the table to calculate each of the parameters for ~~rejected outliers and their replacements are indicated by~~ all materials in the ~~underlines. Each underlined value equals~~ ITP. The formula should use the ~~pooled~~ active values for ~~*Sr*~~*n and p as well as values for that material as brought in from Tables A4.2, A4.4S, or A4.4R. When Calculation 5 of Table A4.6 is completed, the entry of values for T_1, T_2, T_3, or T_4, or combination thereof, along with values for ~~that material. Compare Tables A7.11~~ p and ~~A7.7, from which it is generated in the spreadsheet, by the recalculation process as described above.~~*

~~A7.6.4 The Table A7.10~~ *n* (by means of their linkages to preceding tables) will produce an immediate result for all intermediate and ~~Table A7.11 recalculations as described~~ final precision calculations in ~~A7.6.1 to A7.6.3 provide~~ the ~~new values for a recalculation~~ table.

A4.3 Sequence ~~of~~ *Sr* ~~and~~ *SR* ~~on~~ Database Calculations for Precision

A4.3.1 *Outliers in Analysis Step 1 (Sheet 1)*—As previously noted, the ~~adjusted (outliers removed) database, using the spreadsheet~~ Step 1 analysis ~~technique. However, since~~ operation or set of calculations should be performed on Sheet 1 of the ~~Practice E 691~~ computer spreadsheet program. If any incompatible values are declared as outliers at the 5 % significance level, ~~t~~the database shall be revised according to 8.4 to either delete outliers for any ~~selected automatic rejection technique~~, laboratory or to insert replacements into the ~~issue of replacing~~ database for those *cells* that contain outliers. If any outliers ~~in a Practice E 691 analysis must be addressed as given in~~ are found, it is necessary to conduct Analysis Step 2 (Sheet 2) on the ~~next section.~~

~~A7.7 *Rejection and Replacement*~~*R1 database. The calculations for analysis of ~~(Practice E 691) Outlier Values:~~ the R1*

~~A7.7.1 The rejection~~ database are facilitated by copying all of ~~Practice E 691 analysis outliers is~~ the ~~same as for~~ executed Tables A4.1 to A4.6 on Sheet 1, onto corresponding locations in Sheet 2 of the spreadsheet, with all programmed ~~c~~calculations active, that is, not as values or copying Sheet 1 and renaming it as Sheet 2. These tables on Sheet 2 are now designated as (*1*) Tables A4.1-R1-OR to Table A4.6-R1-OR for replaced outliers or (*2*) Tables A4.1-R1-OD to Table A4.6-R1-OD for deleted outliers.

A4.3.2 *Outliers in Analysis Step 2 (Sheet 2): Option 1 Outlier Deletion*—All deletion operations can be facilitated by marking on a printed out Table A4.1, all table *cells* that have significant *h*~~-values~~ and *k*~~-values as generated by~~ values. To delete data, simply delete from Table A4.1 all the ~~Practice E 691~~ *cells* that have a 5 % significance level *h* or *k* value; that is, delete both values in each ITP design cell, which occupy two spreadsheet cells. When this is done, the typical spreadsheet program ~~are reviewed with~~ will give some *ERROR* indication at several calculation *cell* locations in Tables A4.2-R1-OD to Table A4.6-R1-OD. (*ERROR* is used generically in the ~~cr~~ following text to indicate the specific spreadsheet error flag.) This is due to the deletion of one or more argument values ~~evaluated~~ in Table A4.1-R1-OD and some subsequent tables as well.

A4.3.2.1 *Correcting the ERROR Cells —ERROR* notations will appear in two general locations (*1*) in columns as data entries that come from tables above them in the sequence of tables, that is, values used to calculate parame~~n~~ters for a particular column such as averages, standard deviations, and ~~marked. Although 7.5.1 provides~~ so forth, and (*2*) at the bottom of columns where averages, standard deviations, and so forth were previously located. To correct the tables, start with the first table that contains a spreadsheet cell that has an *ERROR* notation, and delete the *ERROR cell* that is a data entry, not an *ERROR cell* at the base of a

TABLE A76.110 (3-R1-OR): Cell Standa Averdage Deviations Sq d and h-values: AOT Replarced—ment for 5 % Outliers Removed

Laboratory Number	1	2	3	4
1				
2				
3				
4				
5				
6				
6				
8				
9				
10				
11				
Sum (= *T4*)3.482				
h = d / S (Yav)3.482				
(*Sr*)^2				
(*S; wher*)^2				
Sr				
e d = avg Cell i – avg All Cells, Sr				

Laboratory Number	1	2	3	4
1	0.5000.0000.5000			
2	0.3170.3200.7200			
3	0.0000.6050.0450			
4	0.5000.0000.0000			
5	0.4050.1250.2450			
6	1.1250.1100.5000			
6	1.1250.1100.5000			
8	0.2450.0050.5000			
9	0.0800.0000.2450			
10	0.1250.0000.5000			
11	0.1800.0200.3380			
Sum (= *T4*)3.482	1.2053.7180.6303			
h = d / S (Yav)3.482	1.2053.7180.6303			
(*Sr*)^2	0.3170.1100.3380			
(*S; wher*)^2	0.3170.1100.3380			
Sr	0.5630.3310.5810			
e d = avg Cell i – avg All Cells, Sr	0.5630.3310.5810			

column. Correcting the data entry value or *cell* will automatically correct the *ERROR* (calculated value) at the base of the column.

A4.3.2.2 The use of a spreadsheet *delete* operation for any *ERROR cell* will make the *cell* in question blank. Continue this for all tables until all *ERROR* indications are removed and replaced by blank values, not zeros. This will produce correct calculations for all parameters. Also remove from all tables any zero *cell* values that are generated by the deletions from any of the preceding tables. If they are not removed, the bottom of the table column calculations will be in error. For Option 1, outlier values, an example

deletion, the revised precision parameters will automatically be calculated and appear in Table A4.6-R1-OD of Sheet 2, after all *ERROR* entries are removed.

A4.3.3 *Outliers in Analysis Step 2 (Sheet 2): Option 2 Outlier Replacement*—When this option is chosen, replacement values are inserted into the *cacleuls* that contain outliers. Insert into the experimental design cells of Table A4.1 (individual) *cell* data replacement values or DRVs, as evaluated in Annex A5. These will be h in celpls that have a significant *h* or *k* value. Correct any possible *ERROR* occurrences, if they appear, as described in A4.3.2.1 and A4.3.2.2. For Option 2, insertion of DRVs, the revised precision parameters will automatically be calculated and appear in Table A4.6-R1-OR of Sheet 2.

A4.3.4 *Outliers in Analysis Step 3 (Sheet 3)*—The precision values for (Sheet 2) *R1* analysis are accepted as final if there are no outliers at the 2 % significance level.

A4.3.4.1 If any outliers are found at the 2 % significance level, the procedure as previously cited (for 5 % significance) is followed to either do a P Option 1 deletion of all outliers to generate a *R2* OD database or select Option 2 and calculate replacement values. When these are inserted into the *R1* OR database, a *R2* OR database is generated.

A4.3.4.2 If outliers are found, copy the executed Tables A4.1-R1-OR to A4.6-R1-OR or Tables A4.1-R1-OD to A4.6-R1-OD, of spreadsheet Sheet 2 to spreadsheet Sheet 3 with active values as above or copy Sheet 2 and rename as Sheet 3. These *R2* tables, when completed as indicated as follows, will be designated as Table A4.1-R2-OR to Table A4.6-R2-OR or the corresponding Table A4.1-R2-OD to Table A4.6-R2-OD. The purpose of a Sheet 3 analysis (to obtain is to delete or replace the 2 % significance outliers and thereby generate final *R2* precision parameters after values.

A4.3.4.3 Once outlier rejection), values have been deleted from any *cell* or DRVs have been calculated (using Annex A5) and inserted into the Day 1–Day 2 appropriate *cells* of Table A4.1-R2-OR or A4.1-R2-OD in Sheet 3, the new precision values will appear in Sheet 3 Table A4.6-R2-OR or Table A4.6-R2-OD after any *ERROR* indications are removed. These Sheet 3 Table A4.6-R2-OR or Table A4.6-R2-OD values are the final precision parameters, *r* and *R* for the ITP.

A4.3.5 *Precision Result Rounding* —The final precision results as given in Table A4.6, Table A4.6-R1, or Table A4.6-R2 (with either outlier option) are transferred into a Table 1 6 format (Table A7.2 in (see 12.1) for insertion into the test method. When this annex example) must is done, the final precision parameters should be replaced. Thus rounded to the number of significant digits or figures that are technically attainable in e usual practice with the test method, with perhaps one more significant figure than normall (y employed.

A5. PROCEDURE FOR CALCULATING REPLACEMENT VALUES FOR DELETED OUTLIERS

A5.1 Introduction

A5.1.1 If outliers abre found in Analysis Step 1 format), at the 5 % significance level, there are two options. Option 1 is to delete the outliers and thereby generate a revised or *R1* database. Option 2 is to replace the outliers in a way that essentially preserves the distribution of the non-outlier data as described in more detail in A5.2. This annex provides the algorithms to address the replacement process when outliers are found at either the 5 % or 2 % significance level.

A5.1.2 Outlier Option 2 (replacement) is usually the choice when outliers are found with a small database with a limited number of laboratories (approximately six or less). Replacing outlier values, rather than deleting them, preserves the size of the database. The procedure to calculate replacement values however must be one that is *consistent with the observed data distribution* in the database. The replacement procedure as described in this annex fulfills this objective. The procedure consists of the evaluation or calculation of two types of replacements.

A5.2 The Replacement Procedure

A5.2.1 The replacement procedure (for b either Step 1 or 2) is one that replaces outliers with realistic values. The inyitial operation evaluates replacement values for each outlier *cell average* and any replacement cell each outlier *cell standard deviation or variance.*

A7.7.2 Table A7.12. The first type of replacement is designated as a table derived from Table A7.2 on the basis parameter replacement value, or PRV. There are two possible types of PRVs described as follows that might be inserted into the 7.5.1 criteria. It has database. Although only one is selected, both are described in order to demonstrate the merit of the selected second type of replacement.

A5.2.2 *Distribution Mean Parameter Replacement*—The first possible approach for a PRV is to insert into the database a value equal to the distribution or actual database mean for all cell average and outlier values for any material. There are two types of distribution means (*1*) for cell averages or (*2*) for cell standard deviations replaced with the special averages. or cell ranges. The replaced values word *mean* applies to both. If only one PRV is being considered and there are ten or more laboratories, this will ndot substantially change the nature of the distribyution. However, if two types or more outliers are being replaced and the number of underline as indicated in laboratories is much less than ten, this may narrow the table footnotes. The technique distribution and therefore give a falsely optimistic standard deviation for (*1*) the final precision results (if no further outliers are found) or (*2*) for denomingator standard deviation for the *h* or *k* statistics, or both, thant will be demonstrated by referring to Material 1. used for outlier review at the 2 % significance level. For this material there reason, this type of replacement is not chosen.

TABLE A76.12 (1): Mooney Viscosity: Inte Original Baboratory Testic Data—Ou from tlihers Replaced[A] ITP

Laboratory Number	1	2	
1	46.0	47.0	51
2	48.2	49.0[B]	49
3	46.9	46.9	48
4	47.0	46.0	51
5	45.6	46.5	50
6	48.5	47.0	50
8	48.2	48.9	50
9	46.0	46.4	50
10	46.7	47.2	51
11	46.0	45.4	50
1 Day Average[D]	46.8	47.0	50.
2 Day Average		46.9	
B-Lab Std[E]	1.03	1.11	0.7
Pooled B-Lab Std		1.07	

[A] Tabulated data—Mooney viscosity units, ML 1 + 4. Outliers replaced with either 'cell average' or mean 'cell variance' (Standard Deviation).
[B] Cell standard deviation replacement = _____.
[C] Cell average replacement = |_____.
[D] First column each material = Day 1 Test result; Second column = Day 2.
[E] B-Lab Std = Between-laboratory standard deviation.

A5.2.3 *Ascending Order Trend (AOT) Parameter Replacement*—The alternative approach for a PRV is to use a value that substantially preserves the observed distribution as illustrated by the ascending order trend plots as discussed in 8.1.3. This is designated as an ascending order trend or AOT replacement or PRV for a cell average replaced and mean. Each AOT replacement or PRV is in essence a predicted value; one cell standard deviation replaced.

A7.7.3 *Cell Standard Deviation that would be expected for the laboratory in question, absent the unexpected perturbation that generated the outlier illustrated by the off-the-line behavior in the AOT plot. This AOT replacement does not narrow the observed distribution in the same sense as a distribution mean value replacement.*

A5.2.4 *Outlier Replacement (n = 2) Categories* —There are two different categories for outlier replacements, *parameter*—Laboratory 2 has an replacements or PRVs as previously discussed, and a *data replacement value* or DRV. After PRVs have been determined for all outlier cell averages and cell standard deviation. Two values must be inserted in this cell, deviations (or ranges), the next step is the calculation of DRVs for each cell of Table A4.1 format that have (1) contained a cell variance equal *parameter* outlier.

A5.2.4.1 The *DRVs* are required to 0.317 (see insert into a Table A7.11), and (2) an average A4.1 data format (to generate a Table A4.1-R1-OR) to permit a recalculation of 48.6 (see the revised precision values based on the new *R1* database. See Annex A4 and the Table A7.10). The technique A4.1 to do this A4.6 series. Once the initial basic data Table A4.1 is reavised to generate a Tablye A4.1-R1-OR, all the succeeding tables, A4.2-R1-OR to A4.6-R1-OR, are recaiglculated by the autfomatic calculation prwocess as described in Annex A4. The procedures as described (for this Annex A5) are for uniform level designs with two cell values or *n* = 2 (2 replicates). Two = 2. The procedures may be slightly amended for *n* = 3 situations. The precision example in Annex A6 on Mooney viscosity testing illustrates the entire AOT replacement process and the operations described in this annex as well as Annex A3 and Annex A4.

A5.3 *PRVs for Outliers at 5 % Significance Level*—Outlier values at the 5 % significance level shall be replaced using the AOT replacement procedure as described in A5.3.1-A5.3.3. These procedures apply in principle to any of three databases: the original database, the *R1* database, or the *R2* database. The *R1* and *R2* databases will potentially contain PRVs as determined by a previous outlier replacement process.

A5.3.1 *PRVs: Cell Average Outliers* —For each material, visually fit a (least squares type) straight line through the central data point region of the cell average AOT plot and extend the line to both extreme ends of the plot. Alternatively, a linear regression may be used to fit the straight line, however, do not include in the data set any questionable outlier end points. For the outlier values (low or high end of plot), determine the difference between the outlier value (plotted point) and that ~~have~~ point on the extended line at the x-axis~~p~~ location of the laboratory in question. Add or subtract this difference to the outlier value to produce a new value that is *on the fitted line* at that *x*-axis location. For each outlier, this *on the line* value is the cell average ~~(48.6)~~ PRV for that laboratory.

A5.3.2 *PRVs: Cell Range Outliers* —For each material, visually fit a straight line through the central value point region of the cell range AOT plot and extend the line to the high value end of the plot. Repeat the procedure as cited in A5.3.1 to evaluate a new value on the fitted line. For each outlier, this *on the line* value is the cell range PRV for that laboratory.

A5.3.3 *PRVs: Cell Standard Deviation Outliers*—If cell standard deviations were calculated initially rather than cell ranges, evaluate a standard deviation PRV using the same procedure as described for cell range outliers in A5.3.2. For ITP designs that have $n = 2$, the replacement cell standard deviation (SDev) can be converted to a cell range, w, ~~equivalent to a variance of 0.317 or a standard deviation of 0.563. For data pairs, the range,~~ by using: w, ~~is related to~~ = (Sdev) $(2)^{1/2}$. In the ~~standard deviation of~~ following equations, a value for the ~~two values, (*Si*), by Eq A7.1.~~

~~$$w = (2)^{1/2}\,(Si) \qquad \text{(A7.1)}$$~~

~~In general, the data pair~~ range is required for calculating DRVs.

NOTE A5.1—The equations to ~~be inserted~~ calculate DRVs using PRVs for ranges as given in ~~any cell, may~~ A5.4 can be ~~calculated by Eq A7.2 and Eq A7.3, with (Avg), being the average~~ altered for ~~the cell.~~

~~$$\text{Data Value 1} = (\text{Avg}) - (w/2) \qquad \text{(A7.2)}$$~~

~~$$\text{Data Value 2} = (\text{Avg}) + (w/2) \qquad \text{(A7.3)}$$~~

~~For this cell therefore, a~~ use with standard ~~deviation of 0.563 equals a range of $1.41 \times 0.563 = 0.794$ and rounding 0.794 to 0.80 the two values are; $48.6 - 0.40 = 48.2$ and $48.6 + 0.40 = 49.0$. This procedure is repeated on a cell-by-cell basis until all outlier cell standard~~ deviations ~~have been replaced.~~

~~A7.7.4 *Cell Average Replacement (n = 2)*—Laboratory 10 has an outlier cell average~~ rather than ranges. For ITP where $n = 2$, substitute the value ~~(for Material 1) that must be replaced. The two replacement values must (1) be equal to~~ for the ~~recalculated material average of 46.9 (see Table A7.10), and (2) have a~~ range ~~equivalent to the standard deviation of that *particular cell*, since that cell was not a cell standard deviation outlier. The cell standard deviation is 0.354 and $w = 1.41 \times 0.354 = 0.50$. Therefore~~, that is, (SDev)*1.414, into the equations.

A5.4 *DRVs for Outliers at 5 % Significance Level*—After PRVs have been determined for all outlier cell averages and cell standard deviations (or ranges) at the 5 % significance level, the next step is the calculation of DRVs for insertion into a Table A4.1 format. For the DRV process, procedures are used that maintain the values not declared as outliers at their observed values in the database. As an example, when only a replacement cell average is required, (that is, the cell range is not an outlier), the actual or existing cell range shall not be changed by the replacement. Also, when only a replacement cell range is required, the existing cell average shall be maintained. There are four possible combinations of PRVs that require DRVs. The procedures for these are described in A5.4.1-A5.4.4.

A5.4.1 *Cell Average Outlier with Non-Outlier Cell Range*—For the two DRVs for a cell average outlier, add one half and subtract one half of the original or existing cell range, ECR, to and from the PRV (cell average), as obtained in A5.3.1, using Eq A5.1 and A5.2. This gives two cell values, *DRV*1 and *DRV*2 that yield the replacement cell average. Insert the replacement values into the Table A4.1 format database.

$$DRV1 = PRV(\text{cell average}) + ECR/2 \qquad \text{(A5.1)}$$

$$DRV2 = PRV(\text{cell average}) - ECR/2 \qquad \text{(A5.2)}$$

To avoid the confusion of excessive notation, all DRVs (each of four categories) are ~~46.9 − 0.25 = 46.65 = 46.7~~ identified as *DRV*1 and ~~46.9 + 0.25 = 47.15 = 47.2.~~ *DRV* 2.

A5.4.2 *Cell Average Outlier with Cell Range Outlier*—For the two DRVs for this situation, add one half and subtract one half of the AOT plot evaluated PRV(cell range), as obtained in A5.3.2, to and from the PRV(cell average) as obtained in A5.3.1, using Eq A5.3 and A5.4. This gives the two new cell data values, *DRV*1 and *DRV*2, that yield the replacement cell average and the replacement cell range. Insert the DRVs into the Table A4.1 format database.

$$DRV1 = PRV(\text{cell average}) + PRV(\text{cell range})/2 \qquad \text{(A5.3)}$$

$$DRV1 = PRV(\text{cell average}) - PRV(\text{cell range})/2 \qquad \text{(A5.4)}$$

A5.4.3 *Cell Range Outlier with Non-Outlier Cell Average*—For the two DRVs required for this situation, add one half and subtract one half of the AOT evaluated PRV(cell range) as obtained in A5.3.2, to and from the original or existing cell average, ECA, using Eq A5.5 and A5.6. This gives the two new cell data values, *DRV*1 and *DRV*2, that yield the original cell average and the replacement cell range. Insert these into the Table A4.1 format database.

$$DRV1 = ECA + PRV(\text{cell range}) / 2 \quad \text{(A5.5)}$$

$$DRV2 = ECA - PRV(\text{cell range}) / 2 \quad \text{(A5.6)}$$

A5.4.4 *Cell Range Outlier with Cell Average Outlier*—Follow the same procedure as in A5.4.2. This gives two cell data values with the replacement cell average and the replacement cell range. Insert these into the Table A4.1 format database.

A5.5 *PRVs for Outliers at 2 % Significance Level*—For an Analysis Step 2 review of the revised or *R1* database, follow the instructions of A5.5 and A5.6 that apply to a ~~cell-by-cell basis always~~ significance level of 2 %.

A5.5.1 *PRVs: Cell Average Outliers* —For each material, replot the cell average data to give a new AOT plot, using the revised data of Table A4.1-R1-OR. The data in the Table A4.1-R1-OR format will have new replacement values for all 5 % significance outliers. Follow the procedure as described in A5.3.1 to determine the PRV *cell average* for outliers at the 2 % significance level.

A5.5.2 *PRVs: Cell Range Outliers* —For each material, replot the cell ~~standard deviation~~ range data in an AOT plot, using the revised data of Table A4.1-R1-OR. Follow the procedure as described in A5.3.2 to ~~calculate~~ determine the PRV *cell range ~~used~~* for outliers at the ~~dual value calculation.~~

~~A7.7.5 *Cell 2 % significance level.*~~

A5.5.3 *PRVs: Cell Standard Deviation Outliers*—If cell standard deviations were calculated initially rather than cell ranges, calculate a replacement standard deviation using the cell range procedure as described in A5.5.2. As previously noted, for ITP designs with $n = 2$, the replacement cell standard deviation (SDev) can be converted to a cell range, w, by using: $w = (\text{Sdev})\ (2)^{1/2}$.

A5.6 *DRVs for Outliers at 2 % Significance Level*—After PRVs have been determined for all outlier cell averages and ~~Average Replacement (n > 2)~~

~~If there~~ cell standard deviations (or ranges), at the 2 % significance level, the next operation is the calculation of DRVs for Table A4.1 format. These are ~~m~~ required to generate a Table A4.1-R2-OR format~~h~~, to permit a recalculation of t~~w~~he revised precision values (repeatability and reproducibility) based on the new *R2* databas~~p~~e. See Annex A4. Just as for the 5 % significance level calculations, there are four possible combinations of parameter outliers that require data replacements for a~~y~~ *R2* database. For A5.6.1 to A5.6.4, the outliers are at the 2 % significance levels and the database being considered for revision is the *R1* database. After 2 % significance level outliers have been replaced (both PRVs and DRVs) for a *R1* database, it becomes a *R2* database and is used to calculate the final or terminal values for repeatability and reproducibility. Refer to the flow sheet diagram in Fig. 1.

A5.6.1 For the four outlier combination categories as discussed in A5.4.1-A5.4.4, repeat the calculations for DRVs based on evaluated PRVs using AOT plots of the *R1* database. Use the equations as cited in these sections.

A6. AN EXAMPLE OF GENERAL PRECISION EVALUATION—MOONEY VISCOSITY TESTING

A6.1 Introduction

A6.1.1 This annex presents a detailed example of the Three-Step Analysis General Precision evaluation with ~~two inserted cell values by~~ emphasis on how outliers are detected and how the original database is revised to obtain robust precision estimates that are free of outlier effec~~h~~ts. All precision calculations are given, starting with a basic Table 1(or equivalent Table A4.1) format, using the calculation formulas and other operations in the series of tables as described in ~~A7.7.3 and A7.7.4. This replacement however unbalances~~ Annex A4. Most of the table in this annex use a two part identification system; first a sequential table number starting with Table ~~1 format database, producing unequal replicates among~~ A6.1 and a second identification set of symbols in parenthesis that indicate the ~~cells.~~ purpose of the table. The ~~analysis~~ sequential number is required for computer preparation of the standard and the second identification symbol set permits a better comprehension of the context and use of each of the tables. There is a connection between the tables of Annex A4 and of the tables of this ~~type~~ annex in terms of ~~database may be conducted by way~~ their context and use. This second set of symbols inside the pare~~qu~~nthesis indicates this connection between the two annexes. Therefore the first Table A6.1 (1) of ~~A6.3~~ this annex is equivalent to Table A4.1 in Annex ~~A6.~~

~~A7.7.6 *Comparing A4, and the Outlier Adjusted Databases: Practice E 691 versus Cochran Test* —~~The previous version second Table A6.2 (2) is equivalent to Table A4.2 of ~~Practice D 4483 (1989) made use~~ Annex A4, and so forth for all tables with identification symbols (3), (4R), (4S), (5) and (6). Each of ~~Cochran's max variance test~~ the tables in the sequence (1) to (6) performs a unique function in the calculati~~m~~on operation. There are four final tables in this annex that are not part of the Annex A4 - Annex A6 connection and do not use this two part identification system, i.e.. Tables A6.36 to A6.39. Note that Annex A4 does not have this two part table identifying system since in this standard ~~d~~ no Annex A4 tables have been generated. The Annex A4 table designation ~~(~~s are specified for ~~v~~ the user of the standard to employ in setting up a spreadsheet for any actual analysis ope~~)~~ration.

A6.1.2 Two outlier treatment options may be chosen after outliers are detected. Option 1 is the deletion of all outliers and the calculation of precision results on the revised and reduced database. Option 2 is the replacement of outliers with AOT replacements (PRV, DRV) and the calculation of precision results on the revised database. For purposes of illustration, both of these options are given in this example. An additional feature is illustrated, the use of technical judgment by the statistical analyst to override the outcome of a particular objective outlier rejection procedure. The reasons for this are cited.

A6.1.3 The ITP for Mooney Viscosity Testing was conducted in 19892 using the version Table A8.3 , shows of the ASTM standard for Mooney viscosity testing, Test Methods D 1646, that only two cells had significant outliers existed at that time. Test Methods D 1646 is equivalent to ISO 289. Four materials (rubbers) were used and nine laboratories participated in the 95 % confidence level; Laboratory 2-Material ITP. The rubbers, identified as Materials 1 to 4, and Laboratory 11-Material 3. The Practice E 691 some of the details of the testing are described as follows.

k-

Material Number	Material Description	Test Conditions
1	SBR1712 (37.5 oil ext)	ML 1+4 at 100°C
2	IIR (Butyl) NIST SRM 388	ML 1+8 at 100°C
3	SBR1712 BMB (37.5, 65 N339)	ML 1+4 at 100°C
4	NR (natural rubber)	ML 1+4 at 100°C

NIST = National Institute of Standards and Technology, the new name for the National Bureau of Standards
SRM = Standard Reference Material as developed by NIST
BMB = Blal + 65 of carbon black N339
BMB = Black Masterbatch, 37.5 Oil + 65 of carbon black N339

A6.1.4 Samples of each of the four materials were sent out to the nine participating laboratories, and viscosity tests were conducted on two separate days one week apart. A test result is one determination (measurement) of Mooney viscosity at the same confidence level eliminated five cells; Laboratory 2-Material indicated time and temperature. Therefore for this ITP, $p = 9$, $q = 4$ and $n = 2$. A Type 1 precision was evaluated with one additional operation just prior to testing; Materials 1, L 3, abond 4, were mill-massed as specified in Section 7 of the 1982 versysion of Test Methods D 16-46. Material 2, Laboratory 6-Material 6, Laboratory 6-Material 7, the IIR, an SRM, was not mill-massed since this was not specified in Test Methods D 1646 for this reference material.

A6.1.5 *Organization of the Mooney Example Precision Evaluation*—The ordinary practice to evaluate precision for any given ITP, is to use the sequence of steps as outlined in Fig. 1 and Laboratory 11-Material 3. discussed in the overview Section 7. The C detailed instructions are in Sections 8-10. If outliers are found for Step 1, one of the two outlier options is selected and the analysis proceeds to Step 2 and on to Step 3 if needed based on this decision, see again Fig. 1. However to better illustrate precision evaluation in this example, calculations are given for both outly mier optionss. Although outliedr replacement is Option 2, the poor performance calculations for this option will be demonstrated first as Part 1. After that, the simpler Option 1 approach of Laboratory 6.

A7.7.7 *Comparing outlier deletion will be demonstrated as Part 2. The preliminary data and graphical review, given in A6.2.1, is not repeated for the Part 2 outlier deletion option.*

A6.2 Part 1: Outlier Adjusted Database: Practice E 691 *versus Dixon's Test* —The previous version Replacement—Analysis Step 1

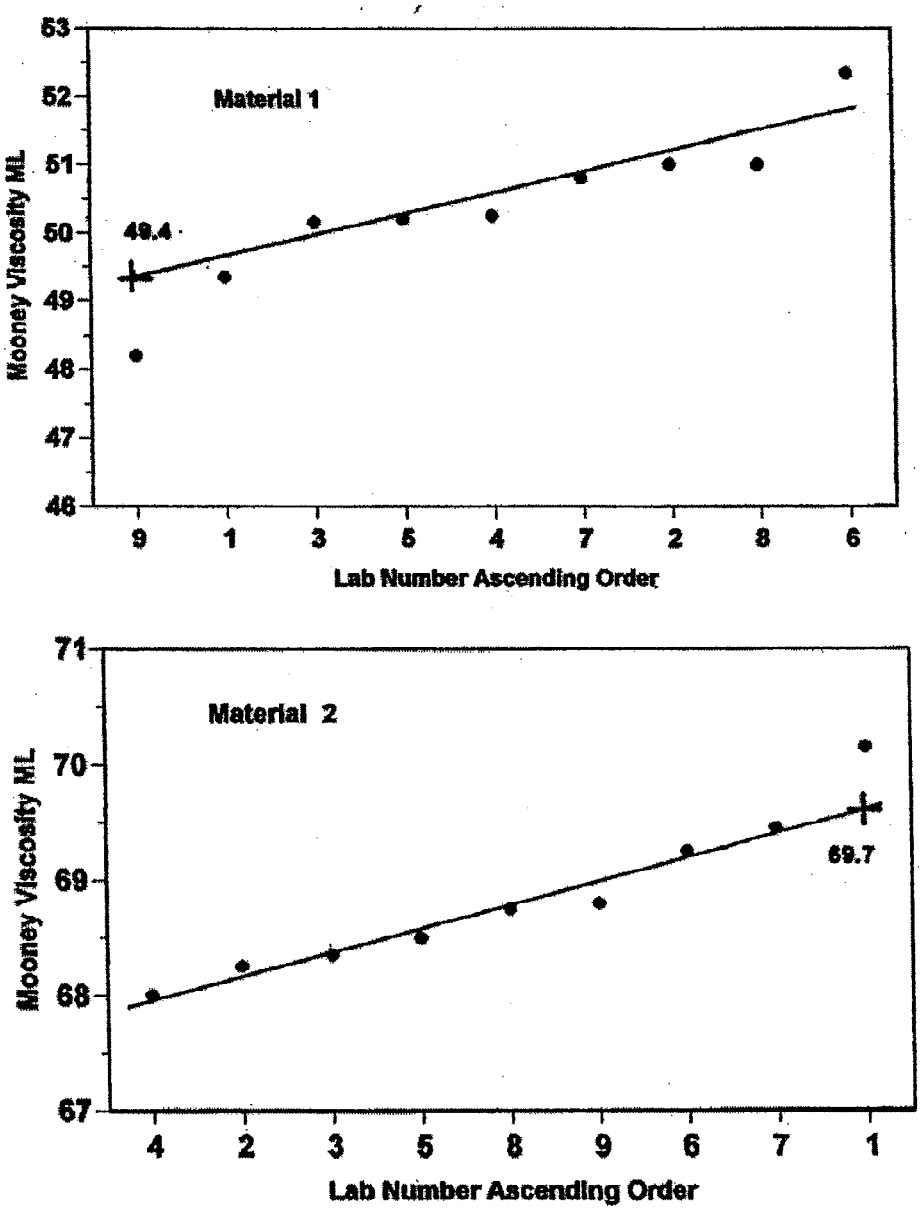
A6.2.1 *Preliminary Review*—Table A6.1, as set up in Sheet 1 of Practice D 4483 made use the computer spreadsheet program (see Annex A4), is a tabulation of Dixon's Test the original data in a format as specified in 8.1.1 and 8.1.2. Although it is not necessary for cell average outlier analysis. Reference the analysis steps to follow, it is informative to obtain averages and standard deviations of all columns in the table and the results for these calculations are illustrated.

A6.2.1.1 The next operation is to generate Tables 2 and 3. To avoid unnecessary redundant tables, the basic Table A8.4 2 and 3 data tabulation is combined with other tabulations and calculations in a dual-table format. This dual-table format is required for the full analysis and is fully described in Annex A4. Therefore, the Table 2 format is given in the left side of Table A6.2 and the 1989 version Table 3 data tabulation format is given in the left side of Practice D 4483, shows Table A6.4S, for within cell standard deviations or in Table A6.4R, for within cell ranges.

A6.2.1.2 The graphical examination of the ITP data is conducted using Figs. A6.1-A6.4 and Fig. A6.5. Fig. A6.1 illustrates plots of *cell average* Mooney viscosity versus laboratory number in ascending viscosity order for Materials 1 and 2 and Fig. A6.2 illustrates similar plots for Materials 3 and 4. These plots serve a dual purpose: an initial review of the original data and a second operation to calculate the Outlier Option 2 AOT replacement values for outliers as described in A5.2.2 in Annex A5.

A6.2.1.3 Fig. A6.1 indicates that only there may be two cell averages were rejected at potential outliers for Material 1, one low outlier for Laboratory 9 and perhaps a high outlier for Laboratory 6. These deviate from the 95 % confidence level; central region linear trend line. This line will be used in the AOT replacement operation to be conducted later. For Material 2, one high potential outlier for Laboratory 10-Material 1 is indicated. In Fig. A6.2, Material 3 has one low potential outlier for Laboratory 9 and Material 4 has two potential outliers, low for Laboratory 9 with a less likely high value for Laboratory 8.

A6.2.1.4 Similar plots for cell ranges in Figs. A6.3 and A6.4 are slightly different than the cell average plots. There are no low-end outliers. All low values indicate good agreement, and as a result, these plots have a low-end curvilinear nature prior to the central linear region. This is ignored in drawing the trend lines. Material 1 has two potential high-end cell range outliers for



NOTE—With linear trend line and PRV indicated.

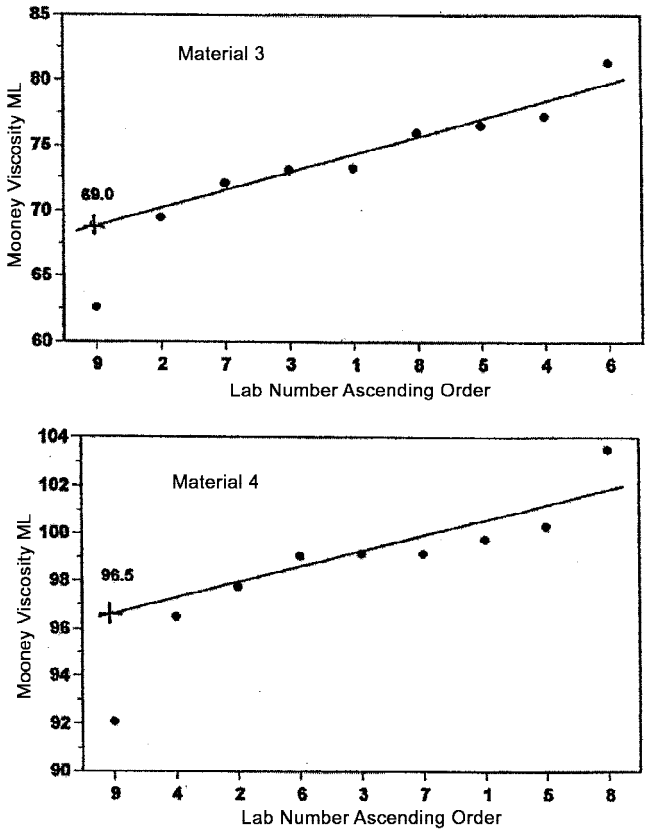
FIG. A6.1 AOT Plots—Original Cell Averages for Materials 1 and 2

Laboratories 1 and 4 . Material ~~7~~ 2 has no potential outliers. Materials 3 and 4 in Fig. A6.4 both have potential outliers for Laboratory 4 and perhaps one for Laboratory 9. The plots give an overall impression of the degree of data uniformity for each of the four materials. The other features will be discussed later.

A6.2.2 *Precision Calculations and Outlier Review for Original Database*—The basic Ste ~~E~~p 1 Analysis operation begins by calculating the precision values for *r* and *R* for the original database. The initial calculation for *r* and *R* using the procedures as set forth in Annex A4, establishes a foundation for comparisons of the reduction in these two parameters as outliers are deleted. The next operation is an examination of the database to detect any potential outliers at the 5 % significance level. Both of these operations will be conducted in parallel and described as each table in the sequence Table A6~~9~~.1(1) to Table A6.6(7) is reviewed.

A6.2.2.1 Table A6.2(2), set up in the dual format for all four materials, has cell averages on the left and cell averages squared on the right. Two totals, T_1 for *cell averages* and T_2 for *cell averages squared* (as required for final precision analysis, see Table A6.6(7)), are obtained for each column or material in the table. Also indicated are results for the overall cell average, variance, and standard deviation for individual cell averages for all nine laboratories.

A6.2.2.2 Table A6.3(3) contains the *cell average* deviations, *d*, on the left and the cell *h* ~~-value analysis eliminated seven cell averages at~~-values on the ~~-same confidence level; Laboratory 10-Material 1, Laboratory 8-Material 2, Laboratory 11-Material 2,~~



NOTE—With linear trend line and PRV indicated.

FIG. A6.2 AOT Plots—Original Cell Average for Materials 3 and 4

~~Laboratory 3-Material 4, Laboratory 10-Material 5, Laboratory 11-Material 6, and Laboratory 11-Material 7. The poor performance~~ right, where for each material:

$$d = (Y_{AV}(i) - Y_{AV}) \quad \text{(A6.1)}$$

$$h = d / S_{(YAV)} \quad \text{(A6.2)}$$

where:

$Y_{AV}(i)$ = cell (i) average,
Y_{AV} = average of all cell averages, and
$S_{(YAV)}$ = standard deviation of ~~Laboratory 11 was missed by the Dixon's Test as well as the very marginal performance of Laboratory 10.~~

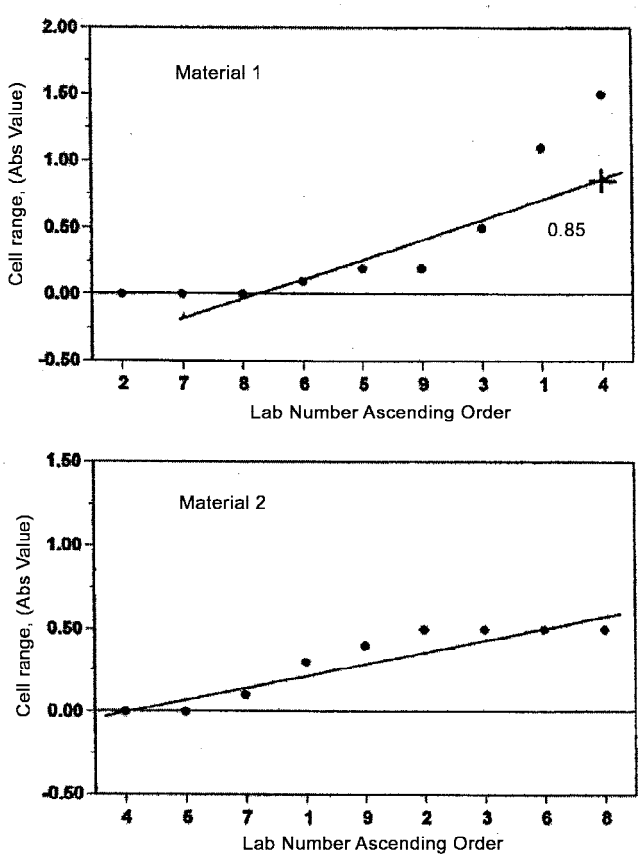
~~A7.8~~ cell averages, see Annex A3.

The values for ~~*Full Analysis—Part 2:*~~ Y_{AV}

~~A7.8.1 Using the Table A7.12 adjusted database (outliers replaced)~~ and ~~the Practice E 691 computer program, the Part 2 analysis may be conducted. For the spreadsheet precision analysis, Table A7.10 and Table A7.11~~ $S_{(YAV)}$, descriptively indicated, are ~~used to perform~~ found at the ~~calculations as indicated in Part B~~ bottom of ~~Table A7.9.~~

~~A7.8.2 The results of~~ the ~~precision calculations are given in the standard Practice D 4483 format in~~ left section of Table ~~A7.13. One material stands out with very poor between-laboratory precision, Material 6-SBR (BMB). This is a carbon black filled black masterbatch material. Testing programs conducted subsequent to~~ A6.3(3). Below the ~~date~~ right side of ~~this ITP have shown that one important source of~~ the ~~poor between-laboratory precision~~ table, is ~~the viscosity variation introduced by the mill-massing operation~~ an inset sub-table that ~~was part of~~ gives the ~~preliminary treatment of all~~ h (crit) at the ~~Mooney test specimens. (The black masterbatch material is sensitive to this mill-massing). The other rubbers of this ITP are clear rubbers and are not as sensitive to this operation. Specimen preparation options have been recently introduced into Test Method D 1646 to avoid some of these problems. At~~ 5 % significance level for the ~~bottom~~ indicated number of ~~Table A7.13, between-laboratory pooled values have been calculated~~ laboratories, that ~~omit Material 6; these pooled values are more representative of clear rubbers.~~

~~A7.8.3 All of the values in precision Table A7.13 are representative of some average or typical laboratory operation. As a rough approximation, three grade levels of testing skill and degree of internal test control contribute to the~~ is, ~~*collective results p*~~of the



NOTE—With dashed linear trend line and replacement values indicated.

FIG. A6.3 AOT Plots—Original Cell Ranges for Materials 1 and 2

~~table—Good, Intermediate, and Poor. Although some of the poor results have been removed from the database by the Practice E 691~~ = 9. Critical values for both ~~*h*~~ *h and k ~~analysis, certain marginal data~~ are ~~still part~~ given in Table A3.1 of Annex A3. The calculated column h-values (for each material) that equal or exceed the ~~adjusted database.~~*

~~A7.8.4 Fig. A7.1 illustrates plots~~ critical value 1.78, have a bold-italic indication. There are four cells with significant *h*-values: Laboratory 1, Material 2, and Laboratory 9, Materials 1, 3 and 4.

A6.2.2.3 Table A6.4(4R) and A6.5(4S) indicate the dispersion (variation) for the day-1 versus day-2 test results. Actually only one of these two tables is absolutely needed, but both have been generated for this example. Table A6.4R contains the *within cell* ranges on the left and the cell ranges squared on the right. For each material, the *cell range* squared total, T_3, is given. Cell ranges for an ITP program with $n = 2$ may be converted into standard deviations by; SDev = $w / (2)^{1/2}$, where w is the range. Table A6.5(4S) is next, it has *within cell* standard deviations on the left and variances (standard deviations squared) on the right. On the right side, the total of all variances, T_4, as well as the pooled or average variance is given for each material.

A6.2.2.4 The analysis for outliers for cell standard deviations is conducted by means of Table A6.6(5), the tabulation of the *k*-values for all cells for each material is generated using:

$$k = S(i) / S_r \quad \text{(A6.3)}$$

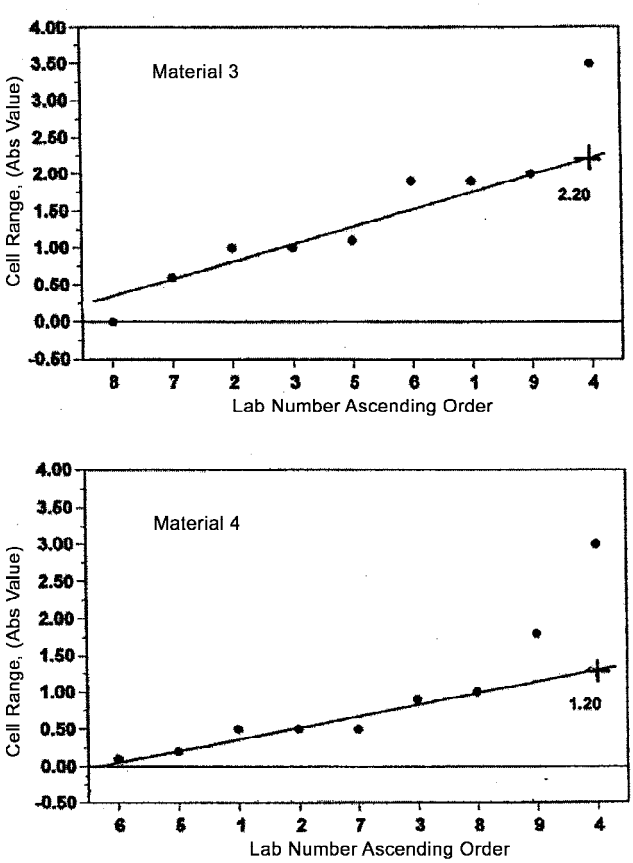
where:

$S(i)$ = cell standard deviation for Laboratory *i*, and

S_r = pooled cell standard deviation (across all laboratories), see Annex A3.

The pooled standard deviations (square root of pooled or average variance) are given at the bottom of both Table A6.5(4S) and Table A6.6(5). Part of Table A6.6(5) is an inset sub-table that gives *k* (crit) at the 5 % significance level for $p = 9$ and $n = 2$. There are three calculated *k*-values equal to or above the critical value of 1.90, Materials 1, 3 and 4 for Laboratory 4. These cells have a bold italic indication.

A6.2.2.5 This completes Analysis Step 1. Before proceeding to Step 2 it is informative to consult Table A6.7(6), the precision results for the original database. The *r* values span the interval from 0.74 to 3.43 and the *R* values from 1.97 to 15.15. If no outliers had been detected in the Step 1 analysis, this table would constitute the end of the analysis and the values as they appear in Table A6.7(6) would be used to prepare a final table of precision results for entry into the test method. In addition to the five internal calculations of Table A6.7(6) to give the final values for *r* and *R* ~~versus average Mooney viscosity~~, the table also gives, at the



NOTE—With linear trend line and PRV indicated.

FIG. A6.4 AOT Plots—Cell Ranges for Materials 3 and 4

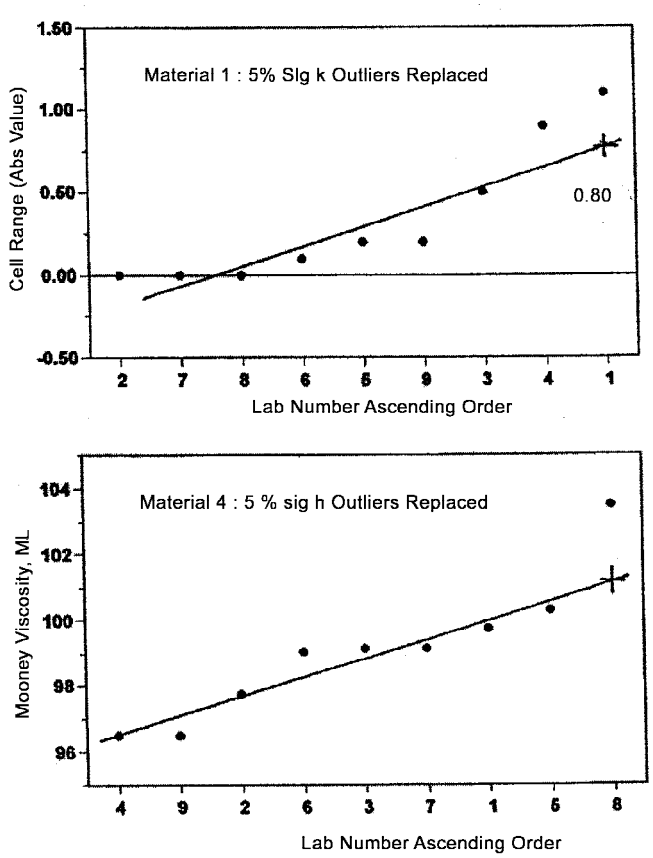
bottom, the mean value for each material as well as the repeatability standard deviation *Sr* and ~~Fig. A7.2 is a similar plot of~~ the reproducibility standard deviation *SR* and values for (*r*) and (*R*~~) versus Mooney viscosity. Visually fitted regres-sion lines have been drawn as indicated ignoring~~), the relative precision in percent of the mean for ~~M~~ each material ~~6~~. The results of the Step 1 outlier analysis for the *h* and *k* statistics are given in a sub-table at the bottom of Table A6.7(6). The Step 1 outlier analysis has indicated a number of outliers at the 5 % significance level. The presence of these outliers calls for a Step 2 analysis operation on a revised ITP database.

A6.3 Part 1: Outlier Replacement - Analysis Step 2

A6.3.1 *Outlier Treatment*—The Step 2 analysis process is twofold: (*1*) it generates a revised database on which the second round of calculations is conducted to obtain revised values for *r* and *R*, and other parameters, using the procedures as set forth in Annex A4, and (*2*) the revised database is examined to detect any potential outliers at the 2 % significance level.

A6.3.1.1 The Step 2 analysis is started with the calculations for Option 2 replacements for the 5 % significance outliers as detected in Step 1. In preparation for this, a second set of spreadsheet tables is generated. To make comparisons and table identification easier Step 1 vs Step 2, (and also Step 3) the table designations for Step 2 retain the (second symbol set) use of (1) to (6) with two added symbols within the parenthesis. First , the Revision 1 database symbol R1 is added and Table A6.1 (1) in Step 1 becomes Table A6.8 (1-R1) in Step 2. The second addition for Option 2 tables is the symbol, OR , where OR designates "outliers replaced". Thus to complete the identification, Table A6.8 (1-R1) becomes Table A6.8 (1-R1-OR) for Step 2, Option 2. Recall that Step 1 is conducted on the original database. This same system of additional symbols is employed for the Step 3 group of tables. In Step 3 the Revision 2 database symbol R1 is replaced with R2, thus Step 2 Table A6.8 (1-R1-OR) becomes Table A6.15 (1-R2-OR) in Step 3. There are a total of 21 tables for the three steps of the OR analysis. The same procedure is applied to the 14 tables in the "outlier deleted" or OD analysis. For the OD analysis it is not necessary to duplicate the first seven tables of the original database.

A6.3.2 *Step 2 Analysis: Replacement of 5 % Significance Outliers*—To implement Outlier Option 2, AOT replacement values must be obtained for the outliers discovered in the Step 1 analysis. Refer to Annex A5 for the AOT procedure. Basically two operations need to be performed; evaluate PRVs and then calculate DRVs for both cell averages and to cell standard deviations or ranges. Once this has been done calculation of the new set of precision values for the *R1* database can be conducted.



NOTE—With linear trend line and PRV indicated.

FIG. A6.5 AOT Plots—Revised (R1) Database for Materials 1 and 4

A6.3.2.1 *PRVs for Cell Averages*—This operation for *cell averages* is conducted, using the procedure of Annex A5 in conjunction with Figs. A6.1-A6.4 and Fig. A6.5. In Fig. A6.1 the value for Laboratory 9 was declared as an outlier in the Step 1 analysis. The PRV of 49.4 for Laboratory 9, Material 1, indicated by a cross symbol, was obtained by the A5.3.1 procedure. The cell average PRV of 69.7 for Material 2 was obtained for Laboratory 1, using the same procedure. In Fig. A6.2, the cell average PRVs (69.0, 96.5) for Lab 9 for both materials were calculated in the same manner. In Fig. A6.3, the cell range PRV for Laboratory 4 is evaluated as 0.85. In Fig. A6.4 the cell range PRVs of 2.20 and 1.20 were obtained for Laboratory 4 for Materials 3 and 4, respectively, using the same procedure. The PRVs for cell averages are tabulated as Item 1 in Part A of Table A6.36, and the PRVs for cell ranges are tabulated as Item 2 in Part A of Table A6.36.

A6.3.2.2 *DRVs*—The next operation is to convert these cell PRVs into cell DRVs using the procedures of A5.4. The cell DRVs are required for entry into a Table A6.1(1) format to generate a new Table A6.8(1-R1-OR).

(1) DRVs for Cell Average—There are two types of cell average DRVs as outlined in A5.4. For this example, all cell average DRVs are the first type as described in A5.4.1, that is, for *Cell Average Outlier with Non-Outlier Cell Range* . The cells scheduled for replacement do not have accompanying cell range outliers. The DRVs for this first type can be calculated using the PRV (for cell averages) obtained in A6.3.2.1, and the existing cell range for that cell, using Eq A5.1 and A5.2 in A5.4.1. The data entries in Item 3 Part B of Table A6.36 were obtained using these two equations with PRVs (cell average) in Part A and the cell ranges that exist for the four cells in question (these are listed in parentheses next to the replacement averages in Part A). The calculated *cell average* DRVs are shown in Item 3 of Part B of Table A6.36.

(2)DRVs for Cell Range—The cell range PRVs, as listed in Item 2 of Part A in Table A6.36, need to be converted to cell range DRVs. All three of these are of the third type, that is, *Cell Range Outlier with Non-Outlier Cell Average,* see A5.4.3. The conversion from PRV to DRVs (duplicate data values) is achieved for any selected cell, using (*1*) the PRV range obtained in A6.3.2.1, and (*2*) the existing cell average for that cell and Eq A5.5 and A5.6. The results of these calculations are shown in Item 4 of Part B of Table A6.36.

A6.3.3 *Step 2 Analysis: Precision for Revised Database with Outlier Replacements*—Once the outlier replacements have been calculated and tabulated in Table A6.36, the revised database can be reanalyzed. This begins with Table A6.8(1-R1-OR). The DRVs of Table A6.36 are substituted for the individual cell outlier values in Table A6.8(1-R1-OR); these are indicated with italics. Once the replacement values for all cells have been entered into Table A6.8(1-R1-OR), the *R1* precision results appear in Table A6.14(6-R1-OR).

TABLE A76.13 Precision Parameters for Test Method D 1646—Mooney Viscosity (Type4R-R1 Precision-OR)A

Nor: CE 1—
Sr =ll Repeatability, standard dgeviation,r = Repeatability (measurement units),
(r) = Rgepeatability (relative basis, percent),
SR = Reprodqucibility, standard deviation,
d: AOT R = Reproducibility (measurcement units), and
(R) = Repr fodr 5 % Oucibility (relative basis, percent).s
Material

Sr	r (r) SR R (R)
1. SBR1500	46.90.561.583.381.0
2. SBR1712	50.40.330.931.850.6
3. EPDM	68.00.581.642.411.6
4. BUTYL (IIR388)	68.70.240.680.990.4
5. SBR BLEND	68.70.601.702.470.8
6. SBR (BMB)	75.10.872.463.283.1
7. NR	99.40.832.352.361.8
Average	68.2
Pooled Values[C]	0.611.732.541.6
Pooled Values[C] Excluding Material 6	1.1

Pooled Values[C] Excluding Material 6	1.1

[A] Short-term, days, with *p* = 11, q = 7, and n = 2, and outliers in database removed.
[B] In Mooney torque units.
[C] Option 2 for (*r*), (*R*).

A6.3.3.1 Table A6.14(6-R1-OR) indicates that the repeatability *r* has been reduced, values now span the interval 0.76 to 2.92 and *R*) lines. There is a very mild dependence of spans the interval 1.76 to 11.27. On an overall or pooled basis the repeatability

has been improved for *r* by a reduction factor of 0.88 (that is, 12 % less for *r*) and the reproducibility for *R* has been improved by a reduction factor of 0.76 (24 % less for *R*) using the *R1* database generated by the outlier replacement procedure.

A6.3.4 *Step 2 Analysis: Detection and Replacement of 2 % Significance Outliers*—Once DRVs for the 5 % outliers are entered into the Table A6.8(1-R1-OR), the calculation operations for all subsequent tables follow automatically. Critical values for *h* and *k* at the 2 % significance level are obtained from Table A3.1. Table A6.10(3-R1-OR) shows a cell average outlier for Material 4 in Laboratory 8. The calculated *h*-value of 2.07 exceeds the critical *h*-value of 2.00. Table A6.13(5-R1-OR) indicates that the cell range for Material 1 in Laboratory 1, is an outlier with a calculated *k*-value of 2.15 exceeding the 2 % critical value 2.09.

A6.3.4.1 The final action required for a Step 2 analysis is the replacement of the data values found to be outliers at the 2 % significance level. Fig. A6.5 illustrates AOT plots for Material 1 with the range value of 0.80 indicated as the replacement of outlier value 1.10 for Laboratory 1. Also shown is the plot for Material 4 with the cell average replacement value of 101.2 for the outlier 103.5 for Laboratory 8. The two PRVs, 0.80 and 101.2, need to be converted into DRVs. The cell range PRV of 0.80 is converted to DRVs using A5.4.3 and the cell average PRV of 101.2 is converted to DRVs using A5.4.1, as described in A6.3.2.1 and A6.3.2.2. These replacement values are shown in Table A6.10(3-R1-OR) in bold italic font.

A6.4 Part 1: Outlier Replacement—Analysis Step 3

When the DRVs for the two 2 % significance outlier values in the Step 2 analysis are inserted into Table A6.8(1-R1-OR), a new Table A6.15(1-R2-OR) is generated, an *R2* database. Refer to the sequence, Table A6.15(1-R2-OR) to Table A6.21(6-R2-OR); the last table gives the *R2* and final Option 2 precision for repeatability and reproducibility. Comments on the improved precision or reduction in *r* and *R* ~~on viscosity with however~~ will be postponed until the Option 1 analysis is conducted in Part 2.

A6.5 Part 2: General Precision Analysis—Option 1: Outlier Deletion

A substantial ~~scatter~~ portion of the work for Part 2–Option 1 has already been done in Part 1.Tables A6.1(1)-A6.6(5) and Table A6.36, and the two sub-tables at the bottom of Table A6.21(6-R2-OR) all indicate the values that have been declared as *h* and *k* outliers in the Part 1 analysis. If Option 1, outlier deletion, had been an initial analysis decision or a decision after Step 1, the preliminary review of section A6.2.1 and the precision calculations and outlier review of the original database as described in section A6.2.2 would be the first operation for a Part 2 analysis. These constitute Part 2–Step 1 and do not need to be repeated here.

A6.6 Part 2: Outlier Deletion—Analysis Step 2

A6.6.1 *Deletion of 5 % Significance Outliers*—Since all outliers have been detected in Part 1, the deletion process is all that is required for this Part 2 analysis. However in the ordinary analysis of an ITP, if Option 1 is chosen as an initial decision, the outlier detection steps for both the 5 % and 2 % significance outliers would be required prior to the action now described.

A6.6.1.1 Table A6.22(1-R1-OD) shows the results of the deletion process on the original database Table A6.1(1), to generate the *R1* database. The tabulated values that have been declared significant, at the 5 % level for *h* and *k* outliers, have been deleted. Tables A6.23(2-R1-OD) to A6.28(6-R1-OD) are also shown with the blank cells at the locations indicated by the deleted 5 % outliers. In the spreadsheet analysis, all of the blank cells in this series of tables will initially have an *ERROR* indication. As explained in Annex A5, each cell *ERROR* value must be deleted to produce a blank cell. The final precision results are given in Table A6.28(6-R1-OD). Comparing the results of the outlier replacement Option 2 with the outlier deletion Option 1, indicates that Option 1 in general gives smaller values for both *r* and *R*. A more detailed discussion of the two options will be conducted in section A6.8.

A6.6.2 *Deletion of 2 % Significance Outliers*—The next operation is the deletion of cell values that have been declared as outliers at the 2 % significance level. Note at the bottom of Table A6.25(6-R1-OD) that two values are indicated; the cell average for Material 4 for Laboratory 8 and cell range (or standard deviation) for Material 1 for Laboratory 1. The ~~relative (percent) expression~~ case of Material 1–Laboratory 1 requires some special consideration by the analyst. Refer to A6.25(4R-R1-OD). If the Laboratory 1 range of 1.10 is deleted, we are left with six range values much smaller than 1.10, three of which are zero.

A6.6.2.1 Although it is possible to get perfect agreement for two Mooney viscosity measurements one week apart in three of the laboratories, this occurrence must be viewed with some caution. Most technicians know when a special test or ITP is being conducted and they know that good agreement is the goal. A temptation exists to make the results look good. The analyst's judgment in this instance is that the pooled standard deviation (pooled range) would be unrealistically low if the Laboratory 1 value of 1.10 were to be deleted. Therefore, a decision is made to override the objective analysis outcome and not delete the 1.10.

A6.6.2.2 In the Part 1 analysis, the Laboratory 1 range of 1.10 for Material 1 was removed, but it was replaced by a value of 0.80. This is different than an outright deletion that removes a laboratory from the list of participants for any material. The deletion of only the Material 4 Laboratory 8 value from the *R1* database, yields Table A6.29(1-R2-OD). This table represents the *R2* database.

A6.6.3 *Alternative Option for Special Case Outlier Treatment*—The decision to retain the Material 1–Laboratory 1 range of 1.10, brings up a possibility for consideration; the combined use of Option 1 and Option 2 for outlier treatment. In the case of the Part 2 Step 2 analysis, it is possible for the analyst to use the Option 2 AOT replacement of 0.80 for this Laboratory range value, rather than deleting it. This is an alternative option that may be used. It is a judgment call by the analyst.

A6.7 Part 2: Outlier Deletion—Analysis Step 3

A6.7.1 The final precision results for Part 2–Option 1 are given in Table A6.35(6-R2-OD). Comparing the results of the outlier replacement Option 2 with the outlier deletion Option 1, Table A6.21(6-R2-OR) versus Table A6.35(6-R2-OD), indicates that Option 1 in general gives smaller values for both *r*) and (*R*.

A6.8 Discussion of Precision Results: Option 1 versus Option 2

A6.8.1 *Option 1 (Deletion) versus Option 2 (AOT Replacement)*—The comparison of the two options is illustrated in Table A6.37, and in Table A6.38 reduction factors for *r* and *R* are given. Both tables may be summarized as follows.

A6.8.1.1 For repeatability, the two Options are essentially equal for Materials 1 and 2. However, for Material 3 and especially Material 4, the Option 1 outlier deletion procedure gives increased reductions or substantially improved repeatability. The pooled values give a reduction factor of 0.65 for Option 1 deletion versus a reduction factor of 0.78 for Option 2 replacement; an overall 20 % advantage for Option 1.

A6.8.1.2 For reproducibility, the two Options are essentially equal for Material 1 and 3, but the Option 1 (deletion) gives improvement for Material 2 and substantial improvement for Material 4. The pooled values give a reduction factor of 0.64 for Option 1 deletion versus a reduction factor of 0.70 for Option 2 replacement; an overall 9 % advantage for Option 1.

A6.8.2 *Precision versus the Four Materials*—The precision performance among the four materials for the Option 1 (deletion) procedure is indicated in Table A6.37. These results have been inserted into the standard Table 6 format summary of precision as described in Section 12. The precision in this format for the Mooney viscosity example is given in Table A6.39 that lists all the precision parameters and also the final number of laboratories in the ITP database after deletion of all outliers.

A6.8.2.1 Materials 1, 2, and 4 give repeatability values, *r*, that are roughly equal, 0.92, 0.76, and 1.03 respectively. These three *r* values differ substantially as a group, from those obtained for the original database: 1.29, 3.43, and 2.54 respectively for Materials 1, 2, and 4. The outlier removal operation has reduced the *r* parameter and gives an indication that all three are very nearly equal. In a technical sense this is not surprising since Materials 1, 2, and 4 are non-pigmented or clear rubbers, and they should respond to the measurement process in a similar manner within the confines of a single laboratory.

A6.8.2.2 Material 3 is an SBR black masterbatch (SBR-BMB) with 65 phr of N339 carbon black. Note that the repeatability for Material 3 is substantially poorer (higher *r*) compared to the other three by a factor of 2.7 on an overall basis. Reasons for this lack of precision are discussed in A6.8.3.

A6.8.2.3 The Option 1 (deletion) reproducibility, *R*, for Materials 1 and 4 is essentially equal (2.71 and 2.50) while Material 2 has the lowest *R* at 1.49. Again Material 3 is very high, $R = 10.84$; roughly by a factor of 5 compared to the other three materials on a overall basis. This is about twice the repeatability comparative precision factor of 2.7. For Materials 1 to 4, the Option 1 reproducibility is substantially improved (lower *R*) compared to the original database *R* values of 3.37, 1.97, 15.15, and 8.84 respectively. Note the considerable differences for the original database *R* values among Materials 1, 2, and 4 compared to the much more nearly equal values (Materials 1, 2, 4) as previously noted.

A6.8.2.4 The roughly equal reproducibility, *R*, for Materials 1 and 4 (SBR and NR) is again a reasonably expected outcome; similar test response in a between laboratory sense for these two un-pigmented rubbers. Material 2 (butyl, reference rubber) is produced to have high uniformity (good homogeneity bale to bale); it is used as a reference rubber to check the operation of Mooney viscometers. This uniformity undoubtedly accounts for part of its good performance. Also this rubber was not subjected to the mill-massing operation.

A6.8.3 *SBR-BMB Precision*—The very poor performance for Material 3, the SBR-BMB, was the subject of further investigation when this ITP was conducted. Subsequent laboratory work showed that the problem was attributed to the procedure used to mill-mass the rubber prior to conducting the Mooney test. In the mill-massing procedure, the mill temperature, the mill nip (opening) and the time on the mill were not sufficiently well-specified and controlled. Both factors were found to play a very important role in the amount of rubber breakdown. Variation in this prior mill massing operation was the source of the poor precision; variable breakdown leads to variable viscosity.

A6.8.3.1 The breakdown for the SBR-BMB was a combination of (*1*) rupture of rubber-carbon black intermolecular bonding and (*2*) ordinary chain rupture. The clear mill-massed rubbers, SBR 1712 and NR, also suffered some chain rupture, but the existence of the additional greater magnitude breakdown mechanism for the SBR-BMB made it much more susceptible to mill-massing variations and produced the poor precision. Test Methods D 1646 and ISO 289 were subsequently revised to eliminate the mill massing operation for BMB rubbers.

A6.8.3.2 Due to the poor precision (high *r* and *R*) for the SBR-BMB, this material was not included in the pooled value calculations in Table A6.39. Pooling is recommended only when the precision values are reasonably close or vary in some known way for all materials in any ITP.

A6.8.4 *Final Observations*—The 3 Step Analysis outlier removal operation using the *h* and *k* statistics, Step 1 at the 5 % significance level and Step 2 at the 2 % significance level on the revised database, has given improved repeatability and reproducibility, compared to the original database. Option 1 (deletion) yields nearly equal *r* and *R* parameters for all three un-pigmented rubbers. A good analysis outcome can be obtained using either Option 1 or Option 2, but Option 1 involves less computation and it yields better precision. Option 1 is the preferred choice when there are nine or more laboratories in any ITP.

A6.8.4.1 The 3 Step Option 1 Analysis has in essence isolated a *core group* of laboratories that have good control of Mooney

viscosity testing. Table A6.29(1-R2-OD) indicates that Laboratories 4 and 8 each had three outliers deleted. These two laboratories have poor control over testing and are in need of improvement. Laboratory 1 also is in need of some remedial efforts, it had two outliers, one of which was not deleted in Option 1 as previously cited. Laboratory 8 had one outlier, and it may need some attention to testing procedures. The *core group* of five laboratories (2, 3, 5, 6, and 7) had good control over their testing domain. For Materials 1, 2, and 4, the relative repeatability (*r*) was 1.8, 1.1, and 1.0 % and the relative reproducibility (*R*) was 5.4, 2.2, and 2.5 % respectively. The precision attained by this *core group* should be the benchmark for Mooney viscosity testing in the rubber manufacturing industry.

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(**3**) Veith, A. G., "Precision in Polymer Testing: An Important World-Wide Issue," *Polymer Testing*, Vol 7, 1987, pp. 239-267.

(**4**) Veith, A. G., "A New Approach to Evaluating Inter-Laboratory Testing Precision," *Polymer Testing*, Vol 12, 1993, pp. 113-184.

(**5**) ASTM Customer Service, 100 Barr Harbor Dr, W. Conshohocken, PA 19428, USA; Phone: 610-832-9585, Fax: 610-832-9555, web site: www.astm.org.

TABLE A76.32 (2): Cell Averages—Moo and Avey Viragescos Squared: Original Datya

Laboratory Number	1	2	3	4
1	46.5	51.0	67.5	
2	48.6	49.6	69.0	
3	46.9	49.4	68.0	
4	46.5	51.0	66.0	
5	46.1	50.2	65.5	
6	47.8	50.3	66.5	
7	46.3	50.2	68.3	
8	48.6	52.4	69.5	
9	46.2	50.8	69.4	
10	42.3	51.0	70.5	
11	45.7	48.2	68.4	
AVG (Average)	46.48	50.35	68.03	
STD (Standard Deviation)[A]	1.71	1.08	1.57	
VAR (Variance)[B]	2.939	1.173	2.450	

VAR (Variance)[B] 2.939 1.173 2.450

[A]Standard Deviation = $(S)\bar{x}$.
[B]Variance = $(S)\bar{x}^2$.

TABLE A76.43 (3): Cell Average═ Deviations, d and h-values: Original Data

Laboratory Number	1	2	3	4
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
Aver age (*d*)				
AverBold and)				
Stan dard Dev i ation (d)1.71				
S Italic = significant values				

h = d / S (Yav); where d = avg Cell i – avg All Cells, S (Yav) = std dev of Cell avgs.71

Laboratory Number	1	2	3	4
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
Aver age (*d*)				
AverBold and)				
Stan dard Dev i ation (d)1.71				
S Italic = significant values				

h = d / S (Yav); where d = avg Cell i – avg All Cells, S (Yav) = std dev of Cell avgs.71

1	0.02	0.65	0.53
2	2.12	0.75	0.97
3	0.42	1.00	0.08
4	0.02	0.65	2.03
5	0.43	0.20	2.58
6	1.27	0.10	1.53
7	0.23	0.15	0.22
8	2.07	2.00	1.47
9	0.28	0.45	1.32
10	4.23	0.65	2.47
11	0.78	2.15	0.32
Aver age (*d*)	0.00	0.00	0.00
AverBold and)	0.00	0.00	0.00
Stan dard Dev i ation (d)1.71	1.08	1.57	0.63
S Italic = significant values	1.08	1.57	0.63

TABLE A76.54 (4R): Cellh=V Ratıngesᴬ and Ranges Squared: Original Data

Laboratory Number	1	2	3	4
1				

Laboratory Number	1	2	3	4
1				

T3 = Sum Cell 'Ranges Squared'

Calculation algorithm for any ITP cell Range, with duplicates in cells, cxx and dxx;

1			
1	0.01	0.60	0.34
2	1.24	0.69	0.62
3	0.25	0.93	0.05
4	0.01	0.60	1.29
5	0.25	0.19	1.64
6	0.74	0.09	0.97
7	0.13	0.14	0.14
8	1.21	1.85[B]	0.94
9	0.16	0.42	0.84
10	2.47[B]	0.60	1.57
11	0.46	1.99[B]	0.20

[A] 95 % Confidence Level *h*(crit) = 1.81.
[B] Significant *h* Value.

TABLE A7.6.5 (4s): Cell Standard Deviations and Variances: Original Data

Laboratory Number	1	2	3	4
1		0.707	0.000	0.707
2		2.546	0.566	0.849
3		0.000	0.778	0.212
4		0.707	0.000	0.000
5		0.636	0.354	0.495
6		1.061	1.061	0.707
7		0.071	0.141	0.354
8		0.495	0.071	0.707
9		0.283	0.000	0.495
10		0.354	0.000	0.707
11		0.424	0.141	2.333
Pooled Variance		0.877	0.202	0.802
Pooled Standard Deviation		0.936	0.449	0.896

Pooled Standard Deviation		0.936	0.449	0.896

TABLE A76.76 (5): Cell Standard De k-viations Sqluared

NOTE 1—*T4 = Sum(Ss: Ori)²; [(Sqi) ² = (Si)Squnared].*
NOL DATE 2—*(Sr)² = T4/p = T4*/11.a
Laboratory Number

1

2 3 4 5

1

2 3 4 5

1	0.5000.0000.5000.00
2	6.4800.3200.7200.00
3	0.0000.6050.0450.04
4	0.5000.0000.0000.12
5	0.4050.1250.2450.12
6	1.1251.1250.5000.00
7	0.0050.0200.1250.00
8	0.2450.0050.5000.12
9	0.0800.0000.2450.00
10	0.1250.0000.5000.12
11	0.1800.0205.4450.09
SUM(= *T4*)9.645	2.2208.8250.6303.92
Bold and italic = significant values	2.2208.8250.6303.92
k = S(i)9.645	
(Sr)2	0.8770.2020.8020.05
(/ Sr; where S(i)2	0.8770.2020.8020.05

TABLE A76.87 C(6): Moonell k-y Viscosity: Calculations for PrecisAion–Original Data

Laboratory Number	1	2	3	4	
1	0.76	0.00	0.79	0	
2	2.72[B]	1.26	0.95	0	
3	0.00	1.73	0.24	0	
4	0.76	0.00	0.00	1	
5	0.68	0.79	0.55	1	
6	1.13	2.36[B]	0.79	0	
8	0.53	0.16	0.79	1	
9	0.30	0.00	0.55	0	
10	0.38	0.00	0.79	1	
11	0.45	0.31	2.60[B]	1	

TABLE A76.98 Pr(1-R1-OR): Mooney Visicon Psity: AOT Replaracementer C Valculaes (Italionsc) for Each Ma 5 % Outlierials

Part A—All Data Values Included:

Material	$(Sr)^2$	$(S)\bar{x}^2$	$[(Sr)^2]/2$
2	0.202	1.173	0.101
3	0.802	2.450	0.401
4	0.057	0.397	0.029
5	0.357	0.975	0.178
6	1.245	23.647	0.623
7	1.039	7.829	0.519
Pooled Values	0.654		

Part B—Outliers Removed:

Material	$(Sr)^2$	$(S)\bar{x}^2$	$[(Sr)^2]/2$
1	0.317	0.973	0.158
2	0.109	0.310	0.055
3	0.338	2.450	0.169
4	0.057	0.197	0.029
5	0.357	0.604	0.178
6	0.758	9.534	0.379
7	0.692	2.964	0.346
Pooled Values	0.376		
Pooled Values Excluding Material 6			

TABLE A76.109 (2-R1-OR): Cell Averages—Outli, Aver Valge Squaresd: AOT Replacements fovr 5 % Outliedrs

Laboratory Number	1	2	3
1		46.5	51.0
2		48.6	49.6
3		46.9	49.4
4		46.5	51.0
5		46.1	50.2
6		47.8	50.3
7		46.3	50.2
8		48.6	50.4
9		46.2	50.8
10		46.9	51.0
11		45.7	50.4
Standard Deviation[A]		0.986	0.55
Variance[B]		0.973	0.31

[A] Standard Deviation = $(S)\,\bar{x}$.
[B] Variance = $(S)\,\bar{x}^2$.

TABLE A6.12 (4S-R1-OR): Cell Standard Deviations and Variances: AOT Replacement for 5 % Outliers

	Cell Std Deviations					Cell Variances				
	Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
	1	0.778	0.283	1.344	0.354	1	0.6050	0.0800	1.8050	0.1250
	2	0.000	0.354	0.707	0.354	2	0.0000	0.1250	0.5000	0.1250
	3	0.354	0.354	0.707	0.636	3	0.1250	0.1250	0.5000	0.4050
	4	0.636	0.000	1.556	0.849	4	0.4050	0.0000	2.4200	0.7200
	5	0.141	0.000	0.778	0.141	5	0.0200	0.0000	0.6050	0.0200
	6	0.071	0.354	1.344	0.071	6	0.0050	0.1250	1.8050	0.0050
	7	0.000	0.071	0.424	0.354	7	0.0000	0.0050	0.1800	0.1250
	8	0.000	0.354	0.000	0.707	8	0.0000	0.1250	0.0000	0.5000
	9	0.141	0.283	1.414	0.849	9	0.0200	0.0800	2.0000	0.7200
Pooled SDev		0.362	0.272	1.044	0.552	T4 =	1.18000	0.66500	9.81500	2.74500
						Pooled Variance	0.1311	0.0739	1.0906	0.3050

TABLE A6.13 (5-R1-OR): k-values: AOT Replacement for 5 % Outliers

Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	***2.15***	1.04	1.29	0.64
2	0.00	1.30	0.68	0.64
3	0.98	1.30	0.68	1.15
4	1.76	0.00	1.49	1.54
5	0.39	0.00	0.74	0.26
6	0.20	1.30	1.29	0.13
7	0.00	0.26	0.41	0.64
8	0.00	1.30	0.00	1.28
9	0.39	1.04	1.35	1.54
Pooled SDev	0.362	0.272	1.044	0.552
k(crit) 2% Sig Level at n=2 , indicated p ;				
p =	9	9	9	9
k(crit) =	2.09	2.09	2.09	2.09
Lab#>k(crit)	1	none	none	none

Bold and italic = significant values
k = S(i) / Sr; where S(i) = indiv cell std dev, Sr = pooled all lab std dev

TABLE A6.14 (6-R1-OR): Mooney Viscosity Calculations for Precision AOT Replacements for 5 % Outliers

	ITP for : n =	2	2	2	2
	p =	9	9	9	9
		Matl 1	Matl 2	Matl 3	Matl 4
	T1 =	454.500	619.150	668.150	891.650
	T2 =	22959.070	42596.943	49727.848	88374.313
	T4 =	1.18000	0.66500	9.81500	2.74500
Calc 1	(Sr)^2 = T4 / p =	0.1311	0.0739	1.0906	0.3050
	(SL)^2 = { [p T2 - (T1)^2] / p (p - 1) } - [(Sr)^2 / 2]				
Calc 2	(SL)^2 =	0.7869	0.3208	15.0965	4.4182
	(SR)^2 = (SL)^2 + (Sr)^2				
Calc 3	(SR)^2 =	0.9181	0.3947	16.1870	4.7232
	r = 2.8 [(Sr)^2]^0.5 = Repeatability				
Calc 4	r =	1.014	0.761	2.924	1.546
	R = 2.8 [(SR)^2]^0.5 = Reproducibility				
Calc 5	R =	2.68	1.76	11.27	6.09
	Material Averages	50.50	68.79	74.24	99.07
Standard Deviation, Sr =		0.362	0.272	1.044	0.552
Standard Deviation, SR =		0.958	0.628	4.023	2.173
	Relative Precision:	Matl 1	Matl 2	Matl 3	Matl 4
	(r) ==>	2.01	1.11	3.94	1.56
	(R) ==>	5.31	2.56	15.17	6.14
	Step 1: Outliers at 5% Significance Level for Materials 1 to 4				
		Matl 1	Matl 2	Matl 3	Matl 4
	For h : Lab #	9	1	9	9
	For k : Lab #	4	none	4	4
	Step 2 : Outliers at 2% Significance Level for Materials 1 to 4				
		Matl 1	Matl 2	Matl 3	Matl 4
	For h: Lab #	none	none	none	8
	For k : Lab #	1(a)	none	none	none

Note: Cell values for Lab 1 Material 1 not deleted for 2 % Sig k-value. See Annex A6 for discussion.

TABLE A6.15 (1-R2-OR): Mooney Viscosity: AOT Replacement Values (italic) for 2 % Outliers

Lab #	Material 1		Material 2		Material 3		Material 4	
	Day1	Day2	Day1	Day2	Day1	Day2	Day1	Day2
1	***49.0***	***49.8***	***69.6***	***70.0***	72.3	74.2	100.0	99.5
2	51.0	51.0	68.0	68.5	69.0	70.0	97.5	98.0
3	50.4	49.9	68.1	68.6	72.6	73.6	98.7	99.6
4	***49.8***	***50.7***	68.0	68.0	***76.2***	***78.4***	**95.9**	**97.1**
5	50.3	50.1	68.5	68.5	76.0	77.1	100.2	100.4
6	52.4	52.3	69.5	69.0	80.4	82.3	99.0	99.1
7	50.8	50.8	69.5	69.4	71.8	72.4	98.9	99.4
8	51.0	51.0	69.0	68.5	76.0	76.0	**100.7**	**101.7**
9	***49.3***	***49.5***	69.0	68.6	***68.0***	***70.0***	**95.9**	**97.1**
Day Avg	50.44	50.57	68.80	68.79	73.59	74.89	98.53	99.10
2 Day Avg		50.51		68.79		74.24		98.82
Bet Lab SDev	1.02	0.85	0.67	0.59	3.92	4.02	1.77	1.51
Pooled Bet Lab SDev		0.94		0.63		3.97		1.64

Replaced Values = Bold, Italic

TABLE A6.16 (2-R2-OR): Cell Average, Average Squared: AOT Replacement for 2 % Outliers

	Cell Averages					Cell Averages Squared			
	Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
	1	49.40	69.80	73.25	99.75	1	2440.36	4872.04	5365.56	9950.06
	2	51.00	68.25	69.50	97.75	2	2601.00	4658.06	4830.25	9555.06
	3	50.15	68.35	73.10	99.15	3	2515.02	4671.72	5343.61	9830.72
	4	50.25	68.00	77.30	96.50	4	2525.06	4624.00	5975.29	9312.25
	5	50.20	68.50	76.55	100.30	5	2520.04	4692.25	5859.90	10060.09
	6	52.35	69.25	81.35	99.05	6	2740.52	4795.56	6617.82	9810.90
	7	50.80	69.45	72.10	99.15	7	2580.64	4823.30	5198.41	9830.72
	8	51.00	68.75	76.00	101.20	8	2601.00	4726.56	5776.00	10241.44
	9	49.40	68.80	69.00	96.50	9	2440.36	4733.44	4761.00	9312.25
	T1 =	454.550	619.150	668.150	889.350	**T2 =**	22964.008	42596.943	49727.848	87903.503
	Cell Avg	50.51	68.79	74.24	98.82					
Var Cell Avg		0.8384	0.3578	15.6417	2.6125					
SDev Cell Avg		0.916	0.598	3.955	1.616					

Note: variance cell avgs = S^2(Yav)

TABLE A6.17 (3-R2-OR): Cell Average Deviation d and h-values: AOT Replacement for 2 % Outliers

	Cell Deviations , d					Cell h-values				
	Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
	1	-1.11	1.01	-0.99	0.93	1	-1.21	1.68	-0.25	0.58
	2	0.49	-0.54	-4.74	-1.07	2	0.54	-0.91	-1.20	-0.66
	3	-0.36	-0.44	-1.14	0.33	3	-0.39	-0.74	-0.29	0.21
	4	-0.26	-0.79	3.06	-2.32	4	-0.28	-1.33	0.77	-1.43
	5	-0.31	-0.29	2.31	1.48	5	-0.33	-0.49	0.58	0.92
	6	1.84	0.46	7.11	0.23	6	2.01	0.76	1.80	0.14
	7	0.29	0.66	-2.14	0.33	7	0.32	1.10	-0.54	0.21
	8	0.49	-0.04	1.76	2.38	8	0.54	-0.07	0.45	1.47
	9	-1.11	0.01	-5.24	-2.32	9	-1.21	0.01	-1.32	-1.43
						h(crit) 2%Sig Level at indicated p				
All Lab Cell Avg		50.51	68.79	74.24	98.82	p =	9	9	9	9
SDev Cell Avg		0.916	0.598	3.955	1.616	h(crit)	2.00	2.00	2.00	2.00
						Lab#>h(crit)	NA	NA	NA	NA

Bold and italic = significant values
h = d / S (Yav); where d = avg Cell i – avg All Cells, S (Yav) = std dev of Cell avgs

TABLE A6.18 (4R-R2-OR): Cell Range, Range Squared: AOT Replacement for 2 % Outliers

Cell Ranges					Cell Ranges Squared				
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	0.800	0.400	1.900	0.500	1	0.640	0.160	3.610	0.250
2	0.000	0.500	1.000	0.500	2	0.000	0.250	1.000	0.250
3	0.500	0.500	1.000	0.900	3	0.250	0.250	1.000	0.810
4	0.900	0.000	2.200	1.200	4	0.810	0.000	4.840	1.440
5	0.200	0.000	1.100	0.200	5	0.040	0.000	1.210	0.040
6	0.100	0.500	1.900	0.100	6	0.010	0.250	3.610	0.010
7	0.000	0.100	0.600	0.500	7	0.000	0.010	0.360	0.250
8	0.000	0.500	0.000	1.000	8	0.000	0.250	0.000	1.000
9	0.200	0.400	2.000	1.200	9	0.040	0.160	4.000	1.440
Avg Range	0.300	0.322	1.300	0.678	T3 =	1.7900	1.3300	19.6300	5.4900

T3 = Sum Cell 'Ranges Squared'

TABLE A6.19 (4S-R2-OR): Cell Standard Deviation and Variances: AOT Replacement for 2 % Outliers

	Cell Std Deviations					Cell Variances			
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	0.566	0.283	1.344	0.354	1	0.3200	0.0800	1.8050	0.1250
2	0.000	0.354	0.707	0.354	2	0.0000	0.1250	0.5000	0.1250
3	0.354	0.354	0.707	0.636	3	0.1250	0.1250	0.5000	0.4050
4	0.636	0.000	1.556	0.849	4	0.4050	0.0000	2.4200	0.7200
5	0.141	0.000	0.778	0.141	5	0.0200	0.0000	0.6050	0.0200
6	0.071	0.354	1.344	0.071	6	0.0050	0.1250	1.8050	0.0050
7	0.000	0.071	0.424	0.354	7	0.0000	0.0050	0.1800	0.1250
8	0.000	0.354	0.000	0.707	8	0.0000	0.1250	0.0000	0.5000
9	0.141	0.283	1.414	0.849	9	0.0200	0.0800	2.0000	0.7200
Pooled SDev	0.315	0.272	1.044	0.552	T4 =	0.89500	0.66500	9.81500	2.74500
					Pooled Variance	0.0994	0.0739	1.0906	0.3050

TABLE A6.20 (5-R2-OR): k-values: AOT Replacement for 2 % Outliers

Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	1.79	1.04	1.29	0.64
2	0.00	1.30	0.68	0.64
3	1.12	1.30	0.68	1.15
4	2.02	0.00	1.49	1.54
5	0.45	0.00	0.74	0.26
6	0.22	1.30	1.29	0.13
7	0.00	0.26	0.41	0.64
8	0.00	1.30	0.00	1.28
9	0.45	1.04	1.35	1.54
Pooled SDev	0.315	0.272	1.044	0.552
k(crit) 2% Sig Level at n=2 , indicated p ;				
p =	9	9	9	9
k(crit) =	2.09	2.09	2.09	2.09
Lab#>k(crit)	NA	NA	NA	NA

Bold and italic = significant values
k = S(i) / Sr; where S(i) = individual cell standard deviation, Sr = pooled all lab standard deviation

TABLE A6.21 (6-R2-OR): Mooney Viscosity Calculations for Precision AOT Replacements for 5 % and 2 % Outliers: Final Precision

		ITP for : n =	2	2	2	2
		p =	9	9	9	9
			Matl 1	Matl 2	Matl 3	Matl 4
		T1 =	454.550	619.150	668.150	889.350
		T2 =	22964.008	42596.943	49727.848	87903.503
		T4 =	0.89500	0.66500	9.81500	2.74500
Calc 1	(Sr)^2	= T4 / p =	0.0994	0.0739	1.0906	0.3050
	(SL)^2	= { [p T2 - (T1)^2] / p (p - 1) } - [(Sr)^2 / 2]				
Calc 2		(SL)^2 =	0.7887	0.3208	15.0965	2.4600
	(SR)^2	= (SL)^2 + (Sr)^2				
Calc 3		(SR)^2 =	0.8881	0.3947	16.1870	2.7650
	r	= 2.8 [(Sr)^2]^0.5 = Repeatability				
Calc 4		r =	0.883	0.761	2.924	1.546
	R	= 2.8 [(SR)^2]^0.5 = Reproducibility				
Calc 5		R =	2.64	1.76	11.27	4.66
Material Averages			50.51	68.79	74.24	98.82
Standard Deviation, Sr =			0.315	0.272	1.044	0.552
Standard Deviation, SR =			0.942	0.628	4.023	1.663
Relative Precision:			Matl 1	Matl 2	Matl 3	Matl 4
		(r) ==>	1.75	1.11	3.94	1.56
		(R) ==>	5.22	2.56	15.17	4.71
Step 1:	Outliers at 5% Significance Level for Materials 1 to 4					
			Matl 1	Matl 2	Matl 3	Matl 4
For h :	Lab #		9	1	9	9
For k :	Lab #		4	none	4	4
Step 2 :	Outliers at 2% Significance Level for Materials 1 to 4					
			Matl 1	Matl 2	Matl 3	Matl 4
For h:	Lab #		none	none	none	8
For k :	Lab #		1	none	none	none

TABLE A6.22 (1-R1-OD): Mooney Viscosity–Revised Data: Outliers 5 % Sig Outliers Removed

	Material 1		Material 2		Material 3		Material 4	
Lab #	Day1	Day2	Day1	Day2	Day1	Day2	Day1	Day2
1	48.8	49.9			72.3	74.2	100.0	99.5
2	51.0	51.0	68.0	68.5	69.0	70.0	97.5	98.0
3	50.4	49.9	68.1	68.6	72.6	73.6	98.7	99.6
4			68.0	68.0				
5	50.3	50.1	68.5	68.5	76.0	77.1	100.2	100.4
6	52.4	52.3	69.5	69.0	80.4	82.3	99.0	99.1
7	50.8	50.8	69.5	69.4	71.8	72.4	98.9	99.4
8	51.0	51.0	69.0	68.5	76.0	76.0	104.0	103.0
9			69.0	68.6				
Day Avg	50.67	50.71	68.70	68.64	74.01	75.09	99.76	99.86
2 Day Avg		50.69		68.67		74.55		99.81
Bet Lab SDev	1.08	0.86	0.64	0.41	3.73	3.94	2.07	1.56
Pooled Bet Lab SDev		0.97		0.54		3.84		1.83

TABLE A6.23 (2-R1-OD): Cell Averages and Averages Sqaured: 5 % Outliers Removed

	Cell Averages				Cell Averages Squared				
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	49.35		73.25	99.75	1	2435.42		5365.56	9950.06
2	51.00	68.25	69.50	97.75	2	2601.00	4658.06	4830.25	9555.06
3	50.15	68.35	73.10	99.15	3	2515.02	4671.72	5343.61	9830.72
4		68.00			4		4624.00		
5	50.20	68.50	76.55	100.30	5	2520.04	4692.25	5859.90	10060.09
6	52.35	69.25	81.35	99.05	6	2740.52	4795.56	6617.82	9810.90
7	50.80	69.45	72.10	99.15	7	2580.64	4823.30	5198.41	9830.72
8	51.00	68.75	76.00	103.50	8	2601.00	4726.56	5776.00	10712.25
9		68.80			9		4733.44		
T1 =	354.850	549.350	521.850	698.650	T2 =	17993.648	37724.903	38991.558	69749.813
Cell Avg	50.69	68.67	74.55	99.81					
Var Cell Avg	0.8812	0.2464	14.6067	3.2587					
SDev Cell Avg	0.939	0.496	3.822	1.805					

Note: variance cell avg = S^2 (Yav)

TABLE A6.24 (3-R1-OD): Cell Average Deviation, d and h-values: 5 % Outliers Removed

Cell Deviations , d					Cell h-values				
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	-1.34		-1.30	-0.06	1	-1.43		-0.34	-0.03
2	0.31	-0.42	-5.05	-2.06	2	0.33	-0.84	-1.32	-1.14
3	-0.54	-0.32	-1.45	-0.66	3	-0.58	-0.64	-0.38	-0.36
4		-0.67			4		-1.35		
5	-0.49	-0.17	2.00	0.49	5	-0.53	-0.34	0.52	0.27
6	1.66	0.58	6.80	-0.76	6	1.77	1.17	1.78	-0.42
7	0.11	0.78	-2.45	-0.66	7	0.11	1.57	-0.64	-0.36
8	0.31	0.08	1.45	3.69	8	0.33	0.16	0.38	***2.05***
9		0.13			9		0.26		
					h(crit) 2%Sig Level at indicated p				
All Lab Cell Avg	50.37	68.83	73.52	98.58	p =	7	8	7	7
SDev Cell Avg	0.939	0.496	3.822	1.805	h(crit)	1.89	1.95	1.89	1.89
					Lab#>h(crit)	none	none	none	8

Bold and italic = significant values
h = d/S (Yav); where d = avg Cell i – avg All Cells, S (Yav) = std dev of Cell avgs

TABLE A6.25 (4R-R1-OD): Cell Range, Range Squared: 5 % Outliers Removed

Cell Ranges					Cell Ranges Squared				
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	1.100		1.900	0.500	1	1.210		3.610	0.250
2	0.000	0.500	1.000	0.500	2	0.000	0.250	1.000	0.250
3	0.500	0.500	1.000	0.900	3	0.250	0.250	1.000	0.810
4		0.000			4	0.000	0.000	0.000	0.000
5	0.200	0.000	1.100	0.200	5	0.040	0.000	1.210	0.040
6	0.100	0.500	1.900	0.100	6	0.010	0.250	3.610	0.010
7	0.000	0.100	0.600	0.500	7	0.000	0.010	0.360	0.250
8	0.000	0.500	0.000	1.000	8	0.000	0.250	0.000	1.000
9		0.400			9		0.160		
Avg Range	0.271	0.313	1.071	0.529	T3 =	1.5100	1.1700	10.7900	2.6100

T3 = Sum Cell 'Ranges Squared'

TABLE A6.26 (4S-R1-OD): Cell Standard Deviation and Variance: 5 % Sig Outliers Removed

	Cell Std Deviations					Cell Variances				
	Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
	1	0.778		1.344	0.354	1	0.6050		1.8050	0.1250
	2	0.000	0.354	0.707	0.354	2	0.0000	0.1250	0.5000	0.1250
	3	0.354	0.354	0.707	0.636	3	0.1250	0.1250	0.5000	0.4050
	4		0.000			4		0.0000		
	5	0.141	0.000	0.778	0.141	5	0.0200	0.0000	0.6050	0.0200
	6	0.071	0.354	1.344	0.071	6	0.0050	0.1250	1.8050	0.0050
	7	0.000	0.071	0.424	0.354	7	0.0000	0.0050	0.1800	0.1250
	8	0.000	0.354	0.000	0.707	8	0.0000	0.1250	0.0000	0.5000
	9		0.283			9		0.0800		
Pooled SDev		0.328	0.270	0.878	0.432	T4 =	0.75500	0.58500	5.39500	1.30500
					Pooled Variance		0.1079	0.0731	0.7707	0.1864

TABLE A6.27 (5-R1-OD): k-values: 5 % Sig Outliers Removed

Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	***2.37***		1.53	0.82
2	0.00	1.31	0.81	0.82
3	1.08	1.31	0.81	1.47
4		0.00		
5	0.43	0.00	0.89	0.33
6	0.22	1.31	1.53	0.16
7	0.00	0.26	0.48	0.82
8	0.00	1.31	0.00	1.64
9		1.05		
Pooled SDev	0.328	0.270	0.878	0.432
k(crit) 2% Sig Level at n=2 , indicated p ;				
p =	7	8	7	7
k(crit) =	1.90	1.90	1.90	1.90
Lab#>k(crit)	1	none	none	none

Bold and italic = significant values
k = S(i) / Sr; where S(i) = indiv cell std dev, Sr = pooled all lab std dev

TABLE A6.28 (6-R1-OD): Mooney Viscosity Calculations for Precision Outliers 5 % Sig Level Removed

		ITP for n =	2	2	2	2
		p =	7	8	7	7
			Matl 1	Matl 2	Matl 3	Matl 4
		T1 =	354.850	549.350	521.850	698.650
		T2 =	17993.648	37724.903	38991.558	69749.813
		T4 =	0.75500	0.58500	5.39500	1.30500
Calc 1	(Sr)^2	= T4 / p =	0.1079	0.0731	0.7707	0.1864
	(SL)^2	= { [p T2 - (T1)^2] / p (p - 1) } - [(Sr)^2 / 2]				
Calc 2		(SL)^2 =	0.8273	0.2098	14.2213	3.1655
	(SR)^2 = (SL)^2 + (Sr)^2					
Calc 3		(SR)^2 =	0.9351	0.2829	14.9920	3.3519
	r = 2.8 [(Sr)^2]^0.5 = Repeatability					
Calc 4		r =	0.920	0.757	2.458	1.209
	R = 2.8 [(SR)^2]^0.5 = Reproducibility					
Calc 5		R =	2.71	1.49	10.84	5.13
	Material Averages		50.69	68.67	74.55	99.81
Standard Deviation, Sr =			0.328	0.270	0.878	0.432
Standard Deviation, SR =			0.967	0.532	3.872	1.831
	Relative Precision:		Matl 1	Matl 2	Matl 3	Matl 4
		(r) ==>	1.81	1.10	3.30	1.21
		(R) ==>	5.34	2.17	14.54	5.14
	Step 1:	Outliers at 5% Significance Level for Materials 1 to 4				
			Matl 1	Matl 2	Matl 3	Matl 4
	For h :	Lab #	9	1	9	9
	For k :	Lab #	4	none	4	4
	Step 2 :	Outliers at 2% Significance Level for Materials 1 to 4				
			Matl 1	Matl 2	Matl 3	Matl 4
	For h:	Lab #	none	none	none	8
	For k :	Lab #	1(a)	none	none	none

(a) Note: Cell values for Lab 1 Material 1 not deleted for 2 % significant k-value. See Annex A6 for discussion.

TABLE A6.29 (1-R2-OD): Mooney Viscosity Revised Data: 2 % Sig Outliers Removed

	Material 1		Material 2		Material 3		Material 4	
Lab #	Day1	Day2	Day1	Day2	Day1	Day2	Day1	Day2
1	48.8	49.9			72.3	74.2	100.0	99.5
2	51.0	51.0	68.0	68.5	69.0	70.0	97.5	98.0
3	50.4	49.9	68.1	68.6	72.6	73.6	98.7	99.6
4			68.0	68.0				
5	50.3	50.1	68.5	68.5	76.0	77.1	100.2	100.4
6	52.4	52.3	69.5	69.0	80.4	82.3	99.0	99.1
7	50.8	50.8	69.5	69.4	71.8	72.4	98.9	99.4
8	51.0	51.0	69.0	68.5	76.0	76.0		
9			69.0	68.6				
Day Avg	50.67	50.71	68.70	68.64	74.01	75.09	99.05	99.33
2 Day Avg		50.69		68.67		74.55		99.19
Bet Lab SDev	1.08	0.86	0.64	0.41	3.73	3.94	0.98	0.78
Pooled Bet Lab SDev		0.97		0.54		3.84		0.89

Note: Lab 1 Material 1, 2 % significant k-value outlier not removed. See Annex A6 for discussion.

TABLE A6.30 (2-R2-OD): Cell Averages and Averages Squared: 2 % Outliers Removed

Cell Averages					Cell Averages Squared				
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	49.35		73.25	99.75	1	2435.42	0.00	5365.56	9950.06
2	51.00	68.25	69.50	97.75	2	2601.00	4658.06	4830.25	9555.06
3	50.15	68.35	73.10	99.15	3	2515.02	4671.72	5343.61	9830.72
4		68.00			4		4624.00		
5	50.20	68.50	76.55	100.30	5	2520.04	4692.25	5859.90	10060.09
6	52.35	69.25	81.35	99.05	6	2740.52	4795.56	6617.82	9810.90
7	50.80	69.45	72.10	99.15	7	2580.64	4823.30	5198.41	9830.72
8	51.00	68.75	76.00		8	2601.00	4726.56	5776.00	
9		68.80			9		4733.44		
T1 =	354.850	549.350	521.850	595.150	T2 =	17993.648	37724.903	38991.558	59037.563
Cell Avg	50.69	68.67	74.55	99.19					
Var Cell Avg	0.8812	0.2464	14.6067	0.7284					
SDev Cell Avg	0.939	0.496	3.822	0.853					

Note: variance cell avg = S^2(Yav)

TABLE A6.31 (3-R2-OD): Cell Average Deviations, d and h-values: 2 % Sig Outliers Removed

Cell Deviations , d					Cell h-values				
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	-1.34		-1.30	0.56	1	-1.43		-0.34	0.65
2	0.31	-0.42	-5.05	-1.44	2	0.33	-0.84	-1.32	-1.69
3	-0.54	-0.32	-1.45	-0.04	3	-0.58	-0.64	-0.38	-0.05
4		-0.67			4		-1.35		
5	-0.49	-0.17	2.00	1.11	5	-0.53	-0.34	0.52	1.30
6	1.66	0.58	6.80	-0.14	6	1.77	1.17	1.78	-0.17
7	0.11	0.78	-2.45	-0.04	7	0.11	1.57	-0.64	-0.05
8	0.31	0.08	1.45		8	0.33	0.16	0.38	
9		0.13			9		0.26		
					h(crit) 2%Sig Level at indicated p				
All Lab Cell Avg	50.69	68.67	74.55	99.19	p =	7	8	7	6
SDev Cell Avg	0.939	0.496	3.822	0.853	h(crit)	1.89	1.95	1.89	1.80
					Lab#>h(crit)	NA	NA	NA	NA

Bold and italic = significant values
h = d / S (Yav); where d = avg Cell i – avg All Cells, S (Yav) = std dev of Cell avgs

TABLE A6.32 (4R-R2-OD): Cell Ranges and Ranges Squared: 2 % Outliers Removed

Cell Ranges					Cell Ranges Squared				
Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	1.100		1.900	0.500	1	1.210		3.610	0.250
2	0.000	0.500	1.000	0.500	2	0.000	0.250	1.000	0.250
3	0.500	0.500	1.000	0.900	3	0.250	0.250	1.000	0.810
4		0.000			4		0.000		
5	0.200	0.000	1.100	0.200	5	0.040	0.000	1.210	0.040
6	0.100	0.500	1.900	0.100	6	0.010	0.250	3.610	0.010
7	0.000	0.100	0.600	0.500	7	0.000	0.010	0.360	0.250
8	0.000	0.500	0.000		8	0.000	0.250	0.000	
9		0.400			9		0.160		
Avg Range	0.271	0.313	1.071	0.450	T3 =	1.5100	1.1700	10.7900	1.6100

T3 = Sum Cell 'Ranges Squared'

TABLE A6.33 (4S-R2-OD): Cell Standard Deviations and Variances: 2 % Outliers Removed

	Cell Std Deviations					Cell Variances				
	Lab #	Matl 1	Matl 2	Matl 3	Matl 4	Lab #	Matl 1	Matl 2	Matl 3	Matl 4
	1	0.778		1.344	0.354	1	0.6050		1.8050	0.1250
	2	0.000	0.354	0.707	0.354	2	0.0000	0.1250	0.5000	0.1250
	3	0.354	0.354	0.707	0.636	3	0.1250	0.1250	0.5000	0.4050
	4		0.000			4		0.0000		
	5	0.141	0.000	0.778	0.141	5	0.0200	0.0000	0.6050	0.0200
	6	0.071	0.354	1.344	0.071	6	0.0050	0.1250	1.8050	0.0050
	7	0.000	0.071	0.424	0.354	7	0.0000	0.0050	0.1800	0.1250
	8	0.000	0.354	0.000		8	0.0000	0.1250	0.0000	
	9		0.283			9		0.0800		
Pooled SDev		0.328	0.270	0.878	0.366	T4 =	0.75500	0.58500	5.39500	0.80500
				Pooled Variance			0.1079	0.0731	0.7707	0.1342

TABLE A6.34 (5-R2-OD): k-values: 2 % Sig Outliers Removed

Lab #	Matl 1	Matl 2	Matl 3	Matl 4
1	**2.37**		1.53	0.97
2	0.00	1.31	0.81	0.97
3	1.08	1.31	0.81	1.74
4		0.00		
5	0.43	0.00	0.89	0.39
6	0.22	1.31	1.53	0.19
7	0.00	0.26	0.48	0.97
8	0.00	1.31	0.00	
9		1.05		
Pooled SDev	0.328	0.270	0.878	0.366
k(crit) 2% Sig Level at n=2 , indicated p ;				
p =	7	8	7	6
k(crit) =	2.04	2.07	2.04	2.00
Lab#>k(crit)	NA	NA	NA	NA

TABLE A6.35 (6-R2-OD): Mooney Viscosity Calculations for Precision: Final Results

		n =	2	2	2	2
		p =	7	8	7	6
			Matl 1	Matl 2	Matl 3	Matl 4
		T1 =	354.850	549.350	521.850	595.150
		T2 =	17993.648	37724.903	38991.558	59037.563
		T4 =	0.75500	0.58500	5.39500	0.80500
Calc 1	(Sr)^2	= T4 / p =	0.1079	0.0731	0.7707	0.1342
	(SL)^2 = { [p T2 - (T1)^2] / p (p - 1) } - [(Sr)^2 / 2]					
Calc 2	(SL)^2 =		0.8273	0.2098	14.2213	0.6613
	(SR)^2 = (SL)^2 + (Sr)^2					
Calc 3	(SR)^2 =		0.9351	0.2829	14.9920	0.7955
	r = 2.8 [(Sr)^2]^0.5 = Repeatability					
Calc 4	r =		0.920	0.757	2.458	1.026
	R = 2.8 [(SR)^2]^0.5 = Reproducibility					
Calc 5	R =		2.71	1.49	10.84	2.50
Material Averages			50.69	68.67	74.55	99.19
Standard Deviation, Sr =			0.328	0.270	0.878	0.366
Standard Deviation, SR =			0.967	0.532	3.872	0.892
Relative Precision:			Matl 1	Matl 2	Matl 3	Matl 4
	(r) ==>		1.81	1.10	3.30	1.03
	(R) ==>		5.34	2.17	14.54	2.52
Step 1: Outliers at 5% Significance Level for Materials 1 to 4						
			Matl 1	Matl 2	Matl 3	Matl 4
For h :	Lab #		9	1	9	9
For k :	Lab #		4	none	4	4
Step 2 : Outliers at 2% Significance Level for Materials 1 to 4						
			Matl 1	Matl 2	Matl 3	Matl 4
For h:	Lab #		none	none	none	8
For k :	Lab #		1(a)	none	none	none

(a) Note: Cell values for Lab 1 Material 1 not deleted for 2 % sig k-value. See Annex A6 for discussion.

TABLE A6.36 Replacement Values for Outliers at Both 5 % and 2 % Significance level

Part A : AOT Cell - Parameter Replacement Values, PRV				
1. AOT : PRV for Cell Average Outliers				
1		69.7 (0.30)		
8				***101.2 (1.00)***
9	49.4 (0.20)		69.0 (2.00)	96.5 (1.80)
Note: Cell PRV Cell Averages listed with individual Cell Range in ( )				
2. AOT : PRV for Cell Range Outliers				
1	***0.80 (49.35)***			
4	0.85 (50.25)		2.20 (77.25)	1.20 (96.50)
Note: Cell PRV Cell Ranges listed with individual Cell Averages in ( )				

Part B : AOT Cell - Data Replacement Values, DRV				
3. AOT : DRV for Cell Average Outliers				
Lab Number	**Matl 1**	**Matl 2**	**Matl 3**	**Matl 4**
1		69.6 , 70.0		
8				***100.7, 101.7***
9	49.3, 49.5		68.0, 70.0	95.6, 97.4
4. AOT : DRV for Cell Range Outliers				
Lab Number	**Matl 1**	**Matl 2**	**Matl 3**	**Matl 4**
1	***49.8, 49.0***			
4	49.8, 50.7		76.2, 78.4	95.9, 97.1

Note: 2% sig level AOT replacements in bold and italic.

TABLE A6.37 Comparison of Outlier Handling Procedures

Part 1 Outlier Procedure	Repeatability , r				Pooled Precision
	Matl 1	Matl 2	Matl 3	Matl 4	
Original Database (No Outliers Deleted)	1.29	0.74	3.43	2.54	2.26
AOT Outlier Replacement, Option 2 (a)	0.88	0.76	2.92	1.55	1.75
Outliers Deleted, Option 1 (a)	0.92	0.76	2.46	1.03	1.46

Part 2 Outlier Procedure	Reproducibility , R				Pooled Precision
	Matl 1	Matl 2	Matl 3	Matl 4	
Original Database (No Outliers Deleted)	3.37	1.97	15.15	8.84	8.98
AOT Outlier Replacement, Option 2 (a)	2.64	1.76	11.27	4.66	6.30
Outliers Deleted, Option 1 (a)	2.71	1.49	10.84	2.50	5.77

(a) Final precision results.
Note: See Table A6.36 for Materials (and Labs) with Outliers.

TABLE A6.38 Relative Reduction Factors: Precision Parameters, r and R

Part 1 Outlier Procedure	Reduction Factor for Repeatability , r				Pooled Precision
	Matl 1	Matl 2	Matl 3	Matl 4	
Original Database (No Outliers Deleted)	1.0	1.0	1.0	1.0	1.0
AOT Outlier Replacement, Option 2 (a)	0.68	1.0	0.85	0.61	0.78
Outliers Deleted, Option 1 (a)	0.71	1.0	0.72	0.41	0.65

Part 2 Outlier Procedure	Reduction Factor for Reproducibility , R				Pooled Precision
	Matl 1	Matl 2	Matl 3	Matl 4	
Original Database (No Outliers Deleted)	1.0	1.0	1.0	1.0	1.0
AOT Outlier Replacement, Option 2 (a)	0.78	0.89	0.74	0.53	0.70
Outliers Deleted, Option 1 (a)	0.80	0.76	0.72	0.28	0.64

(a) Final precision results.
Reduction factor = (Prec Revised Database / Prec Original Database)
Note: See Table A6.36 for Materials (and Labs) with Outliers.

TABLE A6.39 Precision for Mooney Viscosity

		Within Laboratories			Between Laboratories			
Material	Mean Level	Sr	r	(r)	SR	R	(R)	No. Labs (b)
1 - SBR 1712	50.7	0.328	0.92	1.81	0.967	2.71	5.35	7
2 - IIR (Butyl)	68.7	0.270	0.76	1.10	0.532	1.49	2.17	8
3- SBR-BMB	74.6	0.878	2.46	3.29	3.87	10.84	14.5	7
4- NR	99.2	0.366	1.03	1.03	0.892	2.50	2.52	6
Pooled or Avg Val (a)	72.9	0.328	0.918	1.26	0.819	2.29	3.14	

(a) Pooled values calculated for Materials 1, 2, and 4 only; SBR-BMB omitted. See A6.8.5 for details.
(b) Number of labs after outliers deleted, (Option 1); 3 step analysis.
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)