



## Rubber and rubber products — Determination of precision for test method standards

*Caoutchouc et produits en caoutchouc — Détermination de la fidélité de méthodes d'essai normalisées*

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The reasons which led to the decision to publish this document in the form of a technical Report type 3 are explained in the Introduction.

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## 0. INTRODUCTION

This document presents required guidelines for (1) conducting TC45 interlaboratory test programs, (2) gives a special nomenclature system needed for the application of statistical methods to rubber technology with its strong emphasis on multi-step physical measurement procedures and (3) gives the format for expressing the results of precision testing as precision clauses in test method standards. A completely worked out and calculated example is given for the measurement of rubber viscosity via the Mooney Test.

This document is published as an ISO Technical Report to make it more readily available to all who require it for background information in the use of precision results in TC45 test method standards.

The use of test method standards in science and technology requires careful consideration in assessing their general precision and, where pertinent, their accuracy. Clearly outlining the objectives and the uses of test method standards prior to the determination of test precision is essential. A critical requirement for this is the development of a standardized nomenclature system. This document addresses these, and other issues important in evaluating precision for ISO TC-45 test method standards.

## 1. SCOPE

This practice presents guidelines for preparing clear and meaningful precision statements for ISO/TC-45 test method standards. These guidelines expand upon the content of ISO 5725-81, they give needed definitions particular to rubber technology testing, explain the potential uses for standard test methods, and give the requirements for interlaboratory programs needed in precision formulation. They also give the format for expressing precision.

## 2. FIELD OF APPLICATION

This standard practice is devoted to test method precision assessment and is limited to test method standards that:

Have test results expressed in terms of a quantitative continuous variable.

Have been fully developed and are in routine use in a number of laboratories.

## 3. REFERENCES

ISO 5725-81 - Precision of Test Methods - Determination of Repeatability and Reproducibility by Interlaboratory Tests.

ISO Standards Handbook #3; Statistical Methods (1981). (This handbook contains the following ISO Standards: 2602, 2854, 2859, 3207, 3301, 3494, 3534, 3951 and 5725.)

## 4. GENERAL PRINCIPLES

### 4.1 Preliminary Discussion.

4.1.1 This document is prepared to accommodate a broad range of test method standards and uses of test method standards. As such, it may seem overly complex to a particular technologist that uses test method standards in a rather narrow part of this broad spectrum of uses. The narrow-range user should make use of those portions of this document that are applicable and ignore those parts that do not directly apply.

4.1.2 This standard practice is not a substitute for ISO 5725 but is a supplement to it in the field of rubber technology to give guidance in addressing rubber testing problems. Since ISO 5725 does not address the issue of accuracy or bias neither does this TC-45 Standard. However, for purposes of clarification a paragraph on the meaning of accuracy or bias as compared to

precision is given in Clause 4.4. Some discussion on the organization of an interlaboratory precision program is given in this standard, it being mainly a summarized version of Section Two of ISO 5725.

- 4.1.3 Although definitions for repeatability and reproducibility are given in ISO 5725 and in Clause 4.4 of this document, a few words of general discussion are merited at this point.

Repeatability refers to the ability of the same laboratory to obtain similar (test) results (values) under certain specified conditions. Reproducibility refers to the ability of different laboratories to obtain similar test results (values) under certain specified conditions. If test results closely agree, then good repeatability exists or good reproducibility exists.

- 4.1.4 The precision of a test method does not of necessity characterize a test with regard to how sensitive it is in measuring the basic property it is intended to measure. Precision may be good simply because the test method is insensitive to the basic property. A concept called "test sensitivity" has been defined in the statistical literature as the ratio of (1) the responsiveness of the test measurement to finite variations in the basic property in question to (2) the precision of the measurement. This standard does not address this issue.

- 4.1.5 Both repeatability and reproducibility should be determined under realistic or typical laboratory conditions. If extra-ordinary care is exercised or if extremely homogeneous materials are used or both, the resulting precision is overly-optimistic. Secondly as ordinarily determined repeatability has both an inherent test apparatus variability as well as a material variability. The sum of these two components suitable defined by full and clear explanations is the repeatability as normally quoted.

- 4.1.6 The statistical model for calculating repeatability and reproducibility is set forth in Clause 5 of Section One and in Section Three respectively of ISO 5725.

- 4.1.7 Annex C gives in very brief form the computational formulas for calculating repeatability and reproducibility.

- 4.1.8 Although not absolutely necessary for the use of this document, sampling terms tentatively adopted by WG15 are presented in Annex D for assistance to those engaged in inter and intra-laboratory testing.

## 4.2 Interlaboratory Distribution Scheme. (Test Pieces, Materials)

- 4.2.1 One of the key concepts that must be clearly understood by rubber technologists when contemplating interlaboratory precision testing is the matter of "what is distributed" to the participating laboratories. The "what" may be:

- I) Fully prepared test pieces (or test portions) requiring no further processing or action (preparation, adjustments, measurements) prior to inserting the pieces into (onto) a test machine or apparatus. (Example: Died out, gaged, dumbbells or rings for stress-strain testing.)
- II) Intermediate prepared materials, test pieces or items that require some minimal processing prior to insertion into or action by the test machine. (Example: Cured rubber sheets that must have dumbbells or

rings cut from them with subsequent gaging, prior to final stress-strain testing.)

- III) Specified (quantities of) raw materials that must be processed into final test pieces by a standardized procedure. (Example: Rubber, curatives, carbon black, oils and antioxidants that must be mixed, processing steps taken, cured sheets prepared, dumbbell, ring or other test pieces cut and gaged prior to stress-strain testing.)

- 4.2.2 The primary purpose of an interlaboratory program dictates whether scheme I, II or III is selected. If the attention is on the apparatus or test machine(s) in the various laboratories--how well these agree when testing the supplied test pieces--then I or perhaps II would be selected.

If, however, it is the total operational sequence of a test; such as mixing, processing, curing, die-cutting and gaging that is of interest, then III would be selected. Material distribution according to III would be called for in interlaboratory precision programs where producer-user acceptance testing of raw materials is of direct importance. An example would be carbon black or synthetic rubber producer-user testing.

In each case i.e. Class I, II, or III, it is necessary that the distribution of items or materials is made from a uniform source or lot; with a nominally good uniformity or homogeneity.

- 4.2.3 The amount of "within-laboratory" preparation or processing, after arrival of the circulated items or material, increases in the order I, II, III. Analytical chemistry and other simple physical tests often require no or very little "within-laboratory" preparation upon arrival of test portions and therefore make use of a Class I distribution scheme. Conversely, what may be called actual or quasi-performance tests require more complex "within-laboratory" preparation or processing and thus require a type III distribution. Performance implies the attainment of a certain minimal level of some specified or critical property--tensile strength or attained modulus in a standard compound for a raw material like carbon black or a synthetic rubber.
- 4.2.4 When considering a raw material of commerce, the type of test method will often indicate the scheme of interlaboratory distribution; SBR is a typical example. The quality of SBR may be ascertained by (a) certain "analytical" tests such as fatty acid content; (b) certain simple physical tests as such as Mooney viscosity; or (c) by certain performance tests, (minimum) tensile strength. Here categories (a), (b) and (c) correspond respectively to Class I, II, or III distribution schemes.

#### 4.3 Discussion of Repeatability (very short, short, long term).

- 4.3.1 In Clause 4.2 attention was mainly focussed on interlaboratory precision. In this clause within-laboratory precision (repeatability) is discussed. There are at least three different viewpoints that may be and have been expressed with regard to repeatability.

View 1: The smallest possible or "very short" time period is used to estimate the variation. The same material, apparatus and operator is used and repeat determinations are made within a period measured in minutes or at most within a period measured in hours.

View 2: A "short" time period is used for the repeated operations that produce test results. The same material and same operator (or set of operators) is employed but the time period for the repeat operations is most frequently measured in days.

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 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.

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

#### 4.4 Definitions.

4.4.1 accuracy, bias, precision. To set the stage for the more specific definitions to follow three general definitions are given. Although this standard does not address the issue of accuracy or bias its closely allied concept, definitions are presented to clearly show the difference between these two and precision.

4.4.1.1 accuracy: A concept that describes the degree of correspondence between an average measured value and an accepted reference or standard value for the material or phenomenon under test.

NOTE 1: The reference or standard value may be established by theory, by reference to an accepted standard, to another test method, or in some cases the average that could be obtained by applying the test method to all of the sampling units comprising a lot or batch of the material.

NOTE 2: The greater the accuracy the greater the degree of correspondence.

4.4.1.2 bias: The difference between the average measured test result and the accepted reference value.

NOTE: High accuracy implies a small or negligible bias and when bias exists increased testing does not increase accuracy but merely enhances the knowledge of the degree of bias.

4.4.1.3 precision: A testing or measurement concept that expresses the ability to generate (data) test results that agree with each other in absolute magnitude.

NOTE 1: The degree of agreement is normally measured inversely by the standard deviation, high precision corresponds to a low (small) standard deviation.

NOTE 2: High precision may exist simultaneously with a large bias or poor accuracy.

4.4.2 The following specific definitions are given for terms that will be required to accommodate test method standards in TC-45.

The definitions for repeatability and reproducibility are basically the same as appearing in ISO 5725. However, the wording has been modified to improve the understanding. The definitions proposed are briefer and one or more notes have been added to clarify the meaning and differentiate among the various types of repeatability and reproducibility. The three time scales of repeatability and reproducibility discussed in 4.3.1 are reduced to two for the sake of simplification.

Two preliminary definitions, that define the "numbers" produced by testing methods, are required. These are given first.

4.4.2.1 determination: The application of the complete test procedure to one test piece or test portion to produce one numerical (test) measured value to be used to form an average or median.

4.4.2.2 test result: The average or median of a specified number of determinations; it is the reported value for a test.

4.4.2.3 repeatability,  $r$ : an established value, below which the absolute difference between two "within-laboratory" test results may be expected to lie, with a specified probability.

NOTE 1: The two test results are obtained with the same method on nominally identical test materials under the same conditions (same operator, apparatus, laboratory and specified time period) and in the absence of other indications the probability is 95%.

NOTE 2: The "established value" may be also called a "critical difference."

4.4.2.4 reproducibility,  $R$ : an established value, below which the absolute difference between two "between-laboratory" test results may be expected to lie, with a specified probability.

NOTE 1: The two test results are obtained with the same method on nominally identical test materials under different conditions (different laboratories, operators, apparatus and in a specified time period) and in the absence of other indications the probability is 95%.

NOTE 2: The essential characteristic of reproducibility is the different laboratories in which the testing is conducted.

4.4.2.5 (short-term) repeatability,  $r_{ST}$ : a repeatability estimate obtained under a short or brief time period.

NOTE: The time period may be minutes, hours or days and needs to be specified for each test method standard.

4.4.2.6 (long-term) repeatability,  $r_{LT}$ : a repeatability estimate obtained over a long time period.

NOTE 1: The time period may be days, weeks, months and needs to be specified for each test method standard.

NOTE 2: Events that influence long-term repeatability are the use of different operators, environmental factors (i.e., seasonal variations in temperature, humidity, etc.), and the recalibration and/or adjustment of equipment.

4.4.2.7 (short-term) reproducibility,  $R_{ST}$ : a reproducibility estimate obtained over a short time period.

NOTE: The time period may be minutes, hours, days and needs to be specified for each test method standard.

4.4.2.8 (long-term) reproducibility,  $R_{LT}$ : a reproducibility estimate obtained over a long period of time.

NOTE 1: The time period may be weeks, months, and needs to be specified for each test method standard.

NOTE 2: Events that influence long-term reproducibility are different operators, environmental factors (i.e., seasonal variations in temperature, humidity, etc.), and the recalibration and/or adjustment of equipment.

If additional determinations are made the original (invalid) data are to be discarded and only the new determinations used for a decision.

4.4.2.9 (type 1) repeatability: Type 1,  $r$ , a repeatability estimate obtained in an interlaboratory program where the material(s) distributed to all laboratories is (are) in a prepared state ready for testing (with perhaps some minimal preparation steps required), i.e. Class I or II.

4.4.2.10 (type 1) reproducibility: Type 1  $R$ , a reproducibility estimate obtained in an interlaboratory program where the material(s) distributed to all laboratories is (are) in a prepared state ready for testing (with perhaps some minimal preparation steps required), i.e. Class I or II.

4.4.2.11 (type 2) repeatability: Type 2  $r$ , a repeatability estimate obtained in an interlaboratory program where some or all of the material(s) distributed to all laboratories require a specified operation or series of operations, to produce the final test samples, portions, or test pieces prior to applying the test method to the material(s) or item(s) under test, to produce one test result (value), i.e. Class III.

4.4.2.12 (type 2) reproducibility: Type 2  $R$ , a reproducibility estimate obtained in an interlaboratory program where some or all of the material(s) distributed to all laboratories require a specified operation or series of operations, to produce the final test samples, portions, or test pieces prior to applying the test method to the material(s) or item(s) under test, to produce one test result (value), i.e. Class III.

4.4.2.13 relative repeatability and reproducibility: It is often appropriate to express repeatability and reproducibility on a relative basis--as a percent of a certain mean value. This is equivalent to a coefficient of variation. Such expression is important when  $r$  and  $R$  vary with the mean level of the property being measured. Relative values for  $r$  and  $R$  might be unambiguously expressed as percentages (%) alongside the actual measured values in usual test result units, thus

$$r = 0.60 \text{ mPa} \quad r = 1.8 \%$$

However, there is one important exception to this that can cause confusion--many technical properties are expressed as a percentage; i.e., % Cu, % tensile strength retained, % elongation. To avoid this ambiguity the following symbols are defined by the use of the parenthesis.

4.4.2.14  $(r_{ST})$  or  $(r_{LT})$ : repeatability estimate expressed as percentage of the mean of the property for which the estimate was obtained.

4.4.2.15  $(R_{ST})$  or  $(R_{LT})$ : reproducibility estimate expressed as percentage of the mean of the property for which the estimate was obtained.

4.4.2.16 acceptance difference, (duplicate determinations),  $AD_2$ : An established value, below which the difference between two "within laboratory" determinations may be expected to lie, with a specified probability.

NOTE 1: The two test determinations are obtained at the "same" time (side-by-side) with identical test material, operators and apparatus and in the absence of other indications the probability is 95%.

NOTE 2: If the calculated difference lies (below) the acceptance difference, the two values are accepted for averaging and the average is reported as the test result; if the calculated difference exceeds the acceptance difference, additional determinations are made to produce acceptable data.

If additional determinations are made the original (invalid) data are to be discarded and only the new determinations used for a decision.

4.4.2.17 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) may be expected to lie, with a specified probability.

NOTE 1: The specified number of determinations are obtained at the "same" time (side-by-side) with identical test material, operators and apparatus and in the absence of other indications the probability is 95%.

NOTE 2: If the calculated maximum range lies within the critical range or below the acceptance difference, all of the determinations are accepted for averaging or selection of a median value and the average or median is reported as the test result; if the maximum range exceeds the acceptance interval, additional determinations are made to produce acceptable data.

If additional determinations are made the original (invalid) data are to be discarded and only the new determinations used for a decision.

#### 4.5 Discussion of Annex A.

Annex A is presented as an attempt to clarify the concepts discussed in Clauses 4.2, 4.3, and the definitions given in Clause 4.4. It puts into simple flowchart form the various ways repeatability and reproducibility may be estimated. The way chosen is dictated by the objectives or use of test method standard in question, or in some circumstances the choice may be made by the Task Group, Panel, Working Group or Subcommittee. The flowcharts are intended to present the concepts in a way that can be used by nonstatistician technologists; to assist in the organization of interlaboratory programs.

An important added feature not found in ISO 5725 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 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.

The "acceptable difference" repeatability will be calculated for determinations in the same way that ordinary repeatability is calculated for test results. Thus, an extra set of calculations can be performed for individual determinations to permit estimates of  $AD_2$  or  $AD_x$  to be obtained. See Annex A.

For any given test method standard a given Working Group or Subcommittee will normally choose one type of repeatability and reproducibility whether short term or long term. Annex A is structured to accomodate a wide range of possible standards and uses of standards but only a small subpart of it applies to any specific standard.

## 5. ORGANIZING AN INTERLABORATORY PRECISION PROGRAM

5.1 Task Group or Panel. A task group or panel of experts should be organized to conduct the program. This should consist of a chairman, a statistical expert and members well-experienced with the standard in question. The panel chairman should insure that all instructions of the program are clearly communicated to all laboratories in the program. A supervisor in each laboratory should be chosen.

5.2 Type of Precision. The panel should make the following initial decisions.

- a) The type of precision to be obtained; Type 1 or Type 2.
- b) The time period of the repeatability and reproducibility estimate; short (minutes, hours, days) or long (weeks, months). Define the time period.
- c) Whether acceptance intervals are desired or needed.

These decisions set the stage for important but secondary decisions that naturally evolve from the structure of the program.

5.3 Laboratories and Materials. The number of laboratories should be determined. The number of materials, each comprising a different level of the measured property, should be selected.

The number of laboratories available is seldom large, and if the test method is complex, or expensive to run, the problem is complicated further. Thus, the problem is finding and obtaining the cooperation of enough qualified laboratories to produce meaningful estimates of precision, rather than selection from a group of available laboratories.

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 of the reproducibility of the test method.

The number and type of materials to be included will depend on (1) the range of the property and how precision varies over that range; (2) the different types of materials to which the test method is applied; (3) the difficulty (expense) in performing the tests; (4) the commercial or legal need for obtaining a reliable estimate of precision.

An interlaboratory study should include at least three materials, and for development of broadly applicable precision statements, five or more materials should be included. The term "materials" is used in a broad generic sense. Materials may be raw or natural substances, manufactured products,

etc. For each level of material, an adequate quantity (sample) of homogeneous material should be available for subdivision and distribution by random allocation to the participating laboratories. This supply of sample material should include a reserve of 50% beyond the requirements for possible later use in retesting in one or more laboratories. When the material(s) to be tested is (are) not homogeneous, it is important to prepare the samples in the manner prescribed by the method, preferably starting with one batch of commercial material for each level. Some modifications may be necessary to ensure that the amount of material available is sufficient to cover the experiment and keep a stock in reserve.

At each level,  $p$  separate containers (the number of laboratories) should be used where there is any danger of the material deteriorating when the container has once been opened. In the case of unstable materials, special instructions on storage and treatment should be prescribed.

5.4 Actual Organization of the Tests. The interlaboratory test plan is as shown in Figure 1, a table that indicates the laboratories, materials and replicates. With  $q$  levels and  $n$  replicates, each participating laboratory among the  $p$  total laboratories, has to carry out  $qn$  tests. A decision is necessary (for each test standard) as to whether a "replicate" as used in ISO 5725, is to be a "determination" or a "test result" as defined in this document. The performance of these tests should be organized and the operators instructed as follows:

- a) All  $qn$  tests should be performed by one and the same operator or operator set, using the same equipment throughout.
- b) Each group of  $n$  tests belonging to one level must be carried out under repeatability conditions, in a specified interval of time.
- c) It is not necessary that all  $qn$  tests be performed strictly within a short interval, the  $q$  groups of  $n$  tests may be carried out on different days.
- d) It is essential that a group of  $n$  tests under repeatability conditions be performed independently as if they were  $n$  tests on different materials.
- e) The number of replicates  $n$ , must be specified. Each replicate may be one test result or one determination according to the requirements of the test method standard. Normally,  $n$  is two. A larger number may be specified if necessary.

5.5 Instructions to Operators. The operators should receive no instructions other than those contained in the standard test method; these should suffice.

Prior to testing, the operators should be asked to comment on the standard and state whether the instructions contained in it are sufficiently clear.

All participating laboratories should report their test results to one more significant figure than is customary or prescribed in the Standard.

5.6 Reporting the Test Results. Each laboratory supervisor should write a full report on the tests containing the following particulars:

- a) The final test results, (avoid transcription and typing errors).
- b) The original individual observations or determination values from which the final results were derived. This is required if "acceptable difference" parameters ( $AD_2$  or  $AD_x$ ) are to be calculated.
- c) Comments and information about irregularities or disturbances that may have occurred during the test.

- d) The date(s) on which the samples were received and the date(s) and time(s) on which they were tested.
- e) Information about the equipment used, and other relevant information.

Additional information on this topic may be found in Clause 6 and Section Two of ISO 5725.

| Laboratory | Material |   |                 |   |
|------------|----------|---|-----------------|---|
|            | 1        | 2 | . . .           | q |
| 1          |          |   |                 |   |
| 2          |          |   |                 |   |
| 3          |          |   |                 |   |
| ⋮          |          |   | $Y_1 \dots Y_n$ |   |
| p          |          |   |                 |   |

FIGURE 1  
Layout of Uniform Level Precision Program

with: p laboratories (i→p)  
 q levels (j→q)  
 n replicates per cell (k→n)  
 and Y = test result

Cell (ij) contains  $n_{ij}$  results  $Y_{ijk}$  ( $k = 1, 2 \dots n_{ij}$ )

## 6. ANALYSIS OF INTERLABORATORY PROGRAM TEST DATA

General Comments. Detailed instructions for interlaboratory precision data analysis are given in Section Three of ISO 5725. Two tasks are recognized:

- a) Collecting all the data produced in the program and preparing a report for use by the statistician. The data should be put into table form as shown in Figure 1 in the report.
- b) The second task is the formal analysis following Section Three of ISO 5725. The flowchart diagrams of Section Three will assist in the analysis.

## 7. FORMAT FOR PRECISION SECTION (CLAUSE) OF STANDARDS

7.1 General. The results of the formal analysis according to Section Three of ISO 5725 should be contained in a specific section or clause of the test method standard with the heading -- Precision.

7.2 Introductory Subclause. This shall consist of one or more paragraphs that give the pertinent details of the interlaboratory program. Following this one or more tables of results that give the actual precision parameters are presented.

These introductory paragraphs should answer the following questions.

- a) What type precision was estimated, Type 1 or Type 2?
- b) What is the time period for repeatability, reproducibility--short term (define), long term (define)?
- c) What is a test result--how many determinations--average or median?
- d) How many laboratories participated? (p)
- e) How many materials? (q)
- f) How many replicates? (n) What is a replicate?
- g) At what time was the interlaboratory program conducted (month, year)?
- h) Are there any unusual results that the reader should be aware of.
- i) How do  $r$  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.) (This information may be given in graphs.)

7.3 Table of Precision Parameters. A table with the general format as for Table I should be prepared. This includes the following information:

- a) ISO designation.
- b) Type precision, time period used for  $r$  and  $R$ .
- c) Measured property.
- d) Materials with mean level and units of measurement.
- e)  $r$ , ( $r$ ),  $R$ , ( $R$ ) and for completeness of record the within and between laboratory standard deviation  $s_r$  and  $S_R$ . An example follows.

TABLE I

ISO XXXX - Type 1 - Precision\*  
(Measured Property = XXXX)

| Material                       | Mean Level  | Within Labs |     |         | Between Labs |     |         |
|--------------------------------|-------------|-------------|-----|---------|--------------|-----|---------|
|                                |             | $s_r$       | $r$ | ( $r$ ) | $S_R$        | $R$ | ( $R$ ) |
| A                              | XX<br>(mPa) | X           | X   | X       | X            | X   | X       |
| B                              | XX          | X           | X   | X       | X            | X   | X       |
| C                              | XX          | X           | X   | X       | X            | X   | X       |
| D                              | XX          | X           | X   | X       | X            | X   | X       |
| Pooled or<br>Average<br>Values | XX          | X           | X   | X       | X            | X   | X       |

$s_r$  = within lab standard deviation

$S_R$  = standard deviation for total between lab variation

$r$  = repeatability (in measurement units)

( $r$ ) = repeatability (in percent)\*\*

$R$  = reproducibility (in measurement units)

( $R$ ) = reproducibility (in percent)\*\*

$p = XX$ ,  $q = 4$ ,  $n = 2$

\* The time period for precision is days.

\*\* If actual measurement units are (%), these values represent percent relative, i.e. percent of a percent.

Pooled or average values for all tabulated parameters may be given if appropriate.

NOTE: If  $AD_2$  or  $AD_x$  is determined, the results may be given in a table similar to Table I above.

7.4 Statements or recommendations for use and interpretation of statistical parameters  $r$  and  $R$ .

7.4.1 Clause 18.3 of ISO 5725 modified slightly to fit TC-45 requirements, gives the following:

- (i) "The difference between two single test results or determinations found on identical test material under the repeatability conditions prescribed for a particular test, will exceed the repeatability on average not more than once in 20 cases in the normal and correct operation of method.
- (ii) The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on average not more than once in 20 cases in the normal and correct operation of method."

These two statements may be added to the Precision Section.

7.4.2 Alternatively statements of the following form may be prepared for use in the Precision clause of any test method standard.

7.4.2.1 Repeatability. The repeatability of test xxxx has been established as xxxx. Two single test results (or determinations) that differ by more than xxxx (expressed in appropriate terms), must be considered suspect, i.e. to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

NOTE: Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

7.4.2.2 Reproducibility. The reproducibility of test xxxx has been established as xxxx. Two single test results (or determinations) produced in separate laboratories, that differ by more than xxxx, (expressed in appropriate terms) must be considered as suspect, i.e. that they represent different sample populations. Such a decision dictates that appropriate investigative or technical/commercial actions be taken.

ANNEX AFlowchart Explanation of  
Repeatability/Reproducibility

A.1 This annex illustrates, in flowchart form, the concepts and definitions discussed in Clauses 4.4 and 4.5.

A.2 The flowchart example in this annex is given for typical stress-strain testing.

A.3 Symbols Used.

$s_r$  = standard deviation within laboratories.

$S_R$  = total standard deviation (among) between laboratories.

$W$  = range (maximum-minimum value).

$\bar{x}_{rep_k}$  = average or median test result value, for the  $k^{th}$  replication of a test in a particular laboratory.

$\bar{x}_{rep_i}$  = average or median test result, for the  $i^{th}$  replication of a test among several laboratories.

$\bar{\bar{x}}$  = grand mean of several ( $\bar{x}$ ).

$TS_m$  =  $m^{th}$  (single) test piece or portion, the measurement of which produces a determination.

$r$  = repeatability (in test measurement units).

$R$  = reproducibility (in test measurement units).

$(r)$  = repeatability (as a percentage of the mean test result value).

$(R)$  = reproducibility (as a percentage of the mean test result value).

$f$  = factor for multiplication of range ( $w$ ) to obtain  $r_{AD}$ .

Subscript Notation.

ST = short term (time span must be defined for particular standard).

LT = long term (time span must be defined for particular standard).

SV = single value(s) (produced by one test piece).

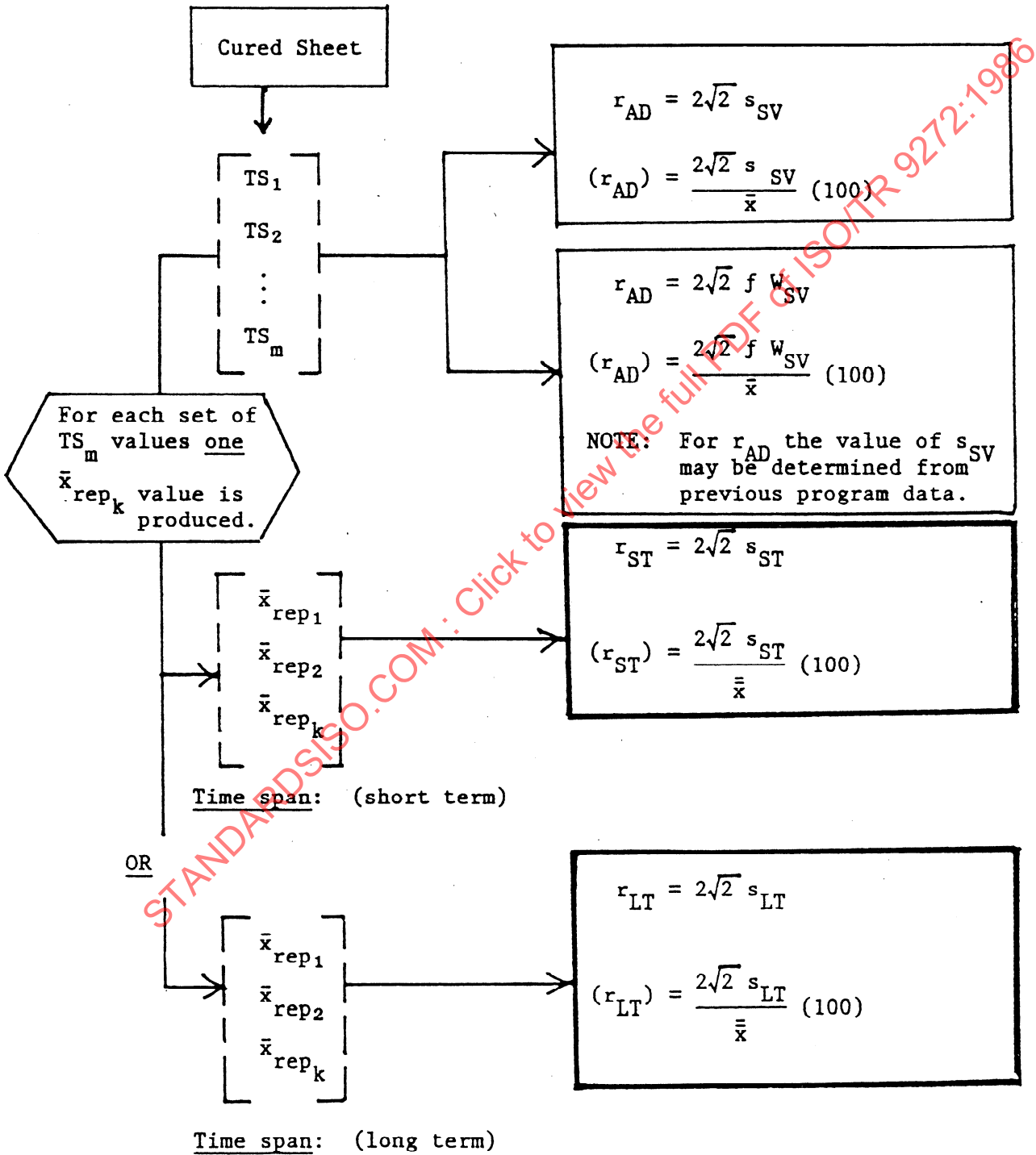
AD = acceptable difference, (acceptable for averaging).

The subscripts  $r$  and  $R$  are dropped from  $s_r$  and  $S_R$  in this Annex where the subscripts ST, LT, SV and AD are used to avoid the confusion of excessive notations. A lower case  $s$  is used for repeatability, an upper case  $S$  for reproducibility standard deviations.

A.4 Repeatability.

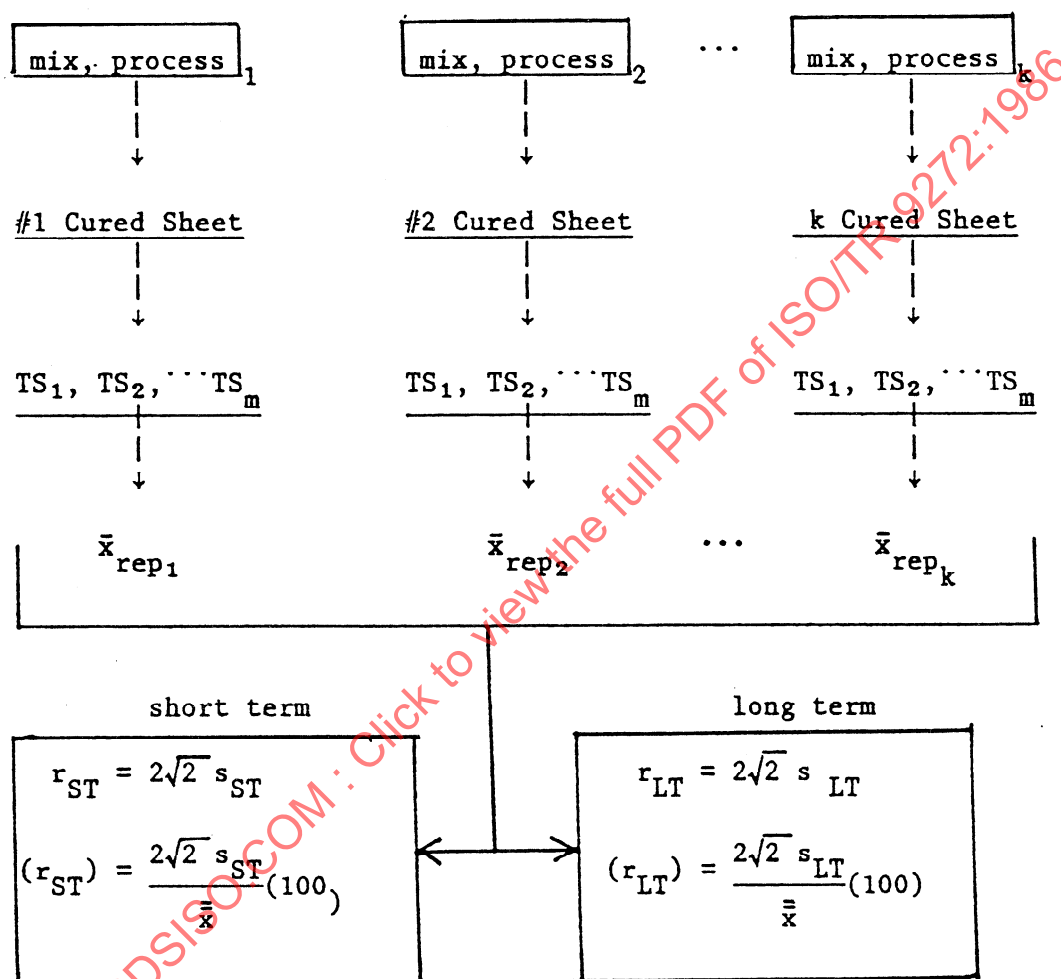
Type 1 Repeatability: This is applicable to any material and any laboratory where the focus of attention is on the variability associated with the operation of a particular test apparatus. A Class I or Class II distribution scheme is used.

Flowchart A



Type 2 Repeatability: This is applicable to any material and to any laboratory where the focus of attention is on the entire sequence of steps or total operation of the test, i.e. to mix, process, cure and prepare test pieces. A Class III distribution scheme is used.

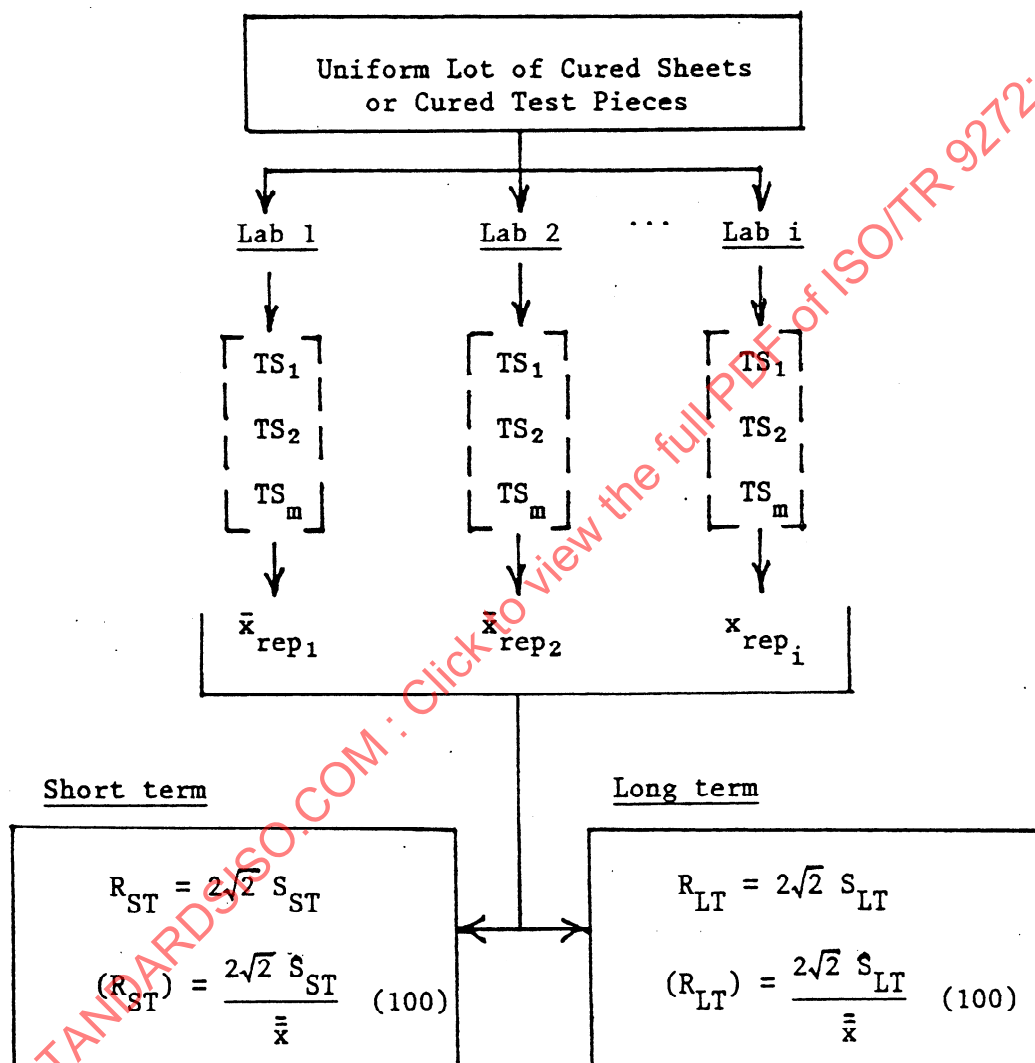
Flowchart B



A.5 Reproducibility.

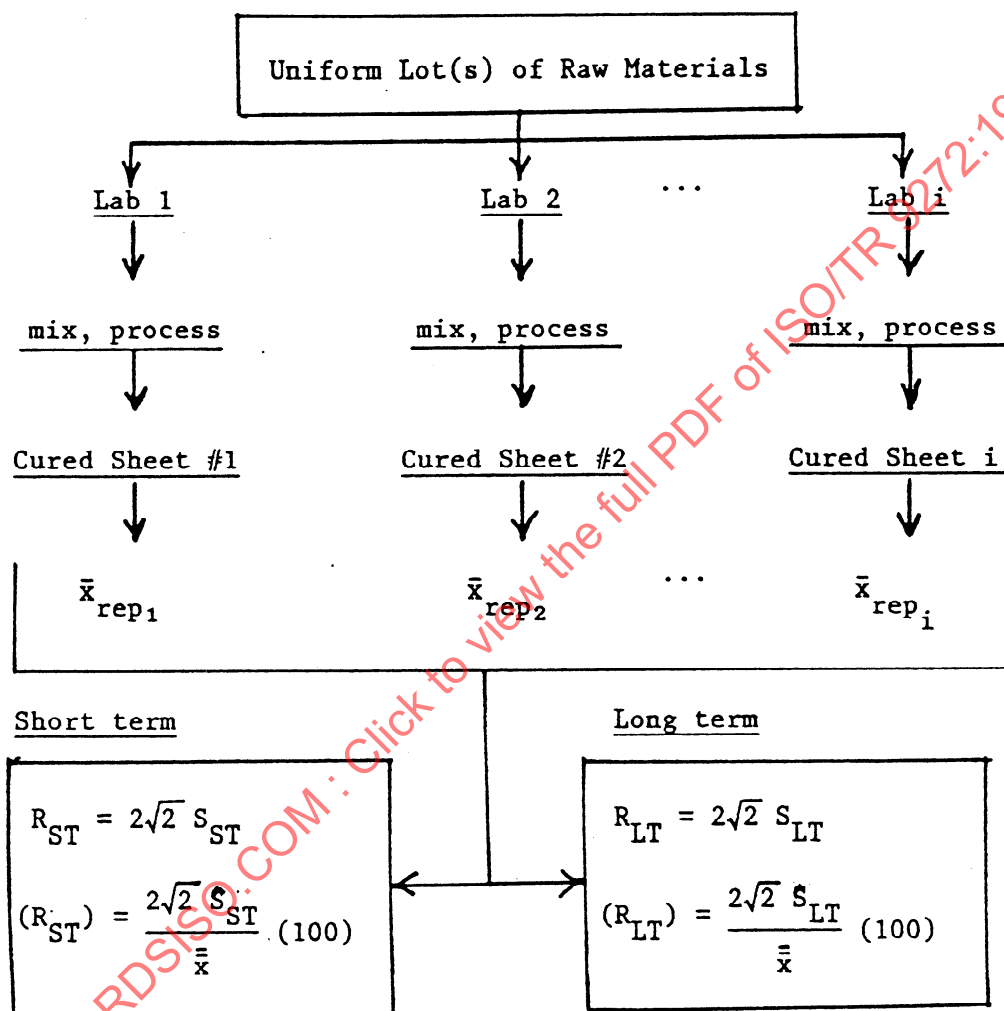
Type 1 Reproducibility: This is applicable to any material where the focus of attention is on the variability associated with the operation of a particular apparatus or test in a number of laboratories. A Class I or Class II distribution is used.

Flowchart C



Type 2 Reproducibility: This is applicable to any material where the focus of attention is on the variability, among laboratories, associated with the entire sequence of steps or total operation of the test, i.e. to mix, process, cure and prepare test pieces. A Class III distribution is used.

Flowchart D



ANNEX BAn Example of Precision Calculation  
Mooney Viscosity Test

B.1 Introduction. The measurement of raw or compounded rubber viscosity is a commonly used test method in the rubber industry. ISO 289, "Determination of Viscosity of Natural and Synthetic Rubbers by the Shearing Disk Viscometer" describes the procedure for this test method. Although the testing program described here was conducted in North America alone, it illustrates testing typical of the world-wide rubber industry and typical of ISO 289 or of ASTM D1646.

The interlaboratory effort consisted of a uniform level program with seven materials tested by way of two replicate (single) test measurements i.e. determinations, each conducted on a separate day. A test result is a single determination. A total of 11 laboratories participated. Thus,  $p = 11$ ,  $q = 7$ ,  $n = 2$ . The materials are described in some detail in Table B1. A Type 1 precision was measured on a short-term basis, i.e. a period of a few days separated replicate test measurements.

TABLE B1

Materials and Test Conditions Used

| <u>Material Number</u> | <u>Material Description</u>                          | <u>Test Temp (°C)</u> | <u>Other Details</u> |
|------------------------|------------------------------------------------------|-----------------------|----------------------|
| 1                      | SBR 1500                                             | 100                   | ML (1+4)             |
| 2                      | SBR 1712                                             | 100                   | ML (1+4)             |
| 3                      | EPDM                                                 | 125                   | ML (1+4)             |
| 4*                     | IIR (NBS-SRM 388j)*                                  | 100                   | ML (1+8)             |
| 5                      | Compounded Blend 1500/1505                           | 100                   | ML (1+4)             |
| 6                      | SBR Black Master Batch<br>(1712, 65 N339, 50 HA Oil) | 100                   | ML (1+4)             |
| 7                      | NR                                                   | 100                   | ML (1+4)             |

\* This IIR (butyl rubber) is a Standard Reference Material No. 388 Lot j, as furnished by the National Bureau of Standards (USA). Measurements are made on unmassed samples.

B.2 Data and Preliminary Calculations. Following the format of Section Three of ISO 5725, the raw data and various calculated values therefrom are displayed in Tables A, B, C (as called for in ISO 5725) which are respectively, Original Test Results, Measures of Cell (Spread) Variation, ( $s_{ij}$ ) and Cell Averages.

Table A: This lists, in Mooney torque units, the duplicate test results in each cell. The material averages are given at the bottom of each material or level column.

Table B: This lists the standard deviations of each cell ( $s_{ij}$ ). At the bottom of this table the calculations and analysis are outlined for Cochran's Maximum Variance Test as described in Clause 12 of ISO 5725. As a result of this analysis, two "cell standard deviations" are declared as outliers. These are for Lab 2 - Material 1, and Lab 11 - Material 3. Although these are officially designated as "Stragglers" in ISO 5725 (they are significant at the 95% Confidence Level but not at the 99%) they are rejected here and were not included in the "within laboratory" or repeatability calculations.

NOTE: Decisions on whether to use the 95% or 99% Confidence Level for rejection of potential outliers must be made by the panel of experts conducting the analysis for any test method standard.

Table C: This lists the cell averages. There are some suspected values that disagree rather sharply with the material average; Lab 10 - Material 1 and Lab 11 - Material 7. Material 6 shows some fairly sharp deviations from the material average; Lab 11 on the low end, Lab 8 on the high end are especially noted.

A Dixons Outlier Test is performed as described in Clause 13 of ISO 5725 and this is presented in Table C2. In this table, the cell averages are placed in ascending numerical order for each material. The calculated and critical (95%) Q values ( $Q_{11}$ ) are shown at the bottom of the table.

The cell average analysis indicates that Lab 2 - Material 1 (42.25) is significant as an outlier at the 95% Confidence Level, (excessive low value) and that Lab 11 - Material 7 is likewise too low at (92.1). However, no significant outlier exists for Material 6 due to the broad distribution of values, i.e. 62.6 to 81.35, a range of 18.75. There is excessive variation among the 11 laboratories for this material; the variation however is well distributed across all 11 laboratories and not manifested as one (or perhaps two) excessive values.

TABLE A  
Original Test Results  
(Level (Material))

| Laboratory | 1           | 2          | 3          | 4          | 5          | 6          | 7            |
|------------|-------------|------------|------------|------------|------------|------------|--------------|
| 1          | 46.0, 47.0* | 51.0, 51.0 | 68.0, 67.0 | 69.0, 69.0 | 68.0, 69.0 | 76.0, 76.0 | 99.0, 101.0  |
| 2          | 46.8, 50.4  | 49.2, 50.0 | 68.4, 69.6 | 68.3, 68.3 | 68.9, 69.6 | 75.8, 75.2 | 98.0, 100.0  |
| 3          | 46.9, 46.9  | 48.8, 49.9 | 68.1, 67.8 | 70.0, 70.3 | 69.0, 69.1 | 72.3, 74.2 | 100.0, 99.5  |
| 4          | 47.0, 46.0  | 51.0, 51.0 | 66.0, 66.0 | 68.0, 68.5 | 70.0, 70.0 | 69.0, 70.0 | 97.5, 98.0   |
| 5          | 45.6, 46.5  | 50.4, 49.9 | 65.1, 65.8 | 68.1, 68.6 | 68.3, 67.5 | 72.6, 73.6 | 98.7, 99.6   |
| 6          | 48.5, 47.0  | 51.0, 49.5 | 67.0, 66.0 | 68.0, 68.0 | 68.5, 67.0 | 79.0, 75.5 | 98.0, 95.0   |
| 7          | 46.2, 46.3  | 50.3, 50.1 | 68.0, 68.5 | 68.5, 68.5 | 68.7, 68.1 | 76.0, 77.1 | 100.2, 100.4 |
| 8          | 48.2, 48.9  | 52.4, 52.3 | 69.0, 70.0 | 69.5, 69.0 | 69.2, 70.2 | 80.4, 82.3 | 99.0, 99.1   |
| 9          | 46.0, 46.4  | 50.8, 50.8 | 69.0, 69.7 | 69.5, 69.4 | 68.9, 69.3 | 71.8, 72.4 | 98.9, 99.4   |
| 10         | 42.0, 42.5  | 51.0, 51.0 | 70.0, 71.0 | 69.0, 68.5 | 71.0, 70.5 | 76.0, 76.0 | 104.0, 103.0 |
| 11         | 46.0, 45.4  | 48.1, 48.3 | 70.0, 66.7 | 69.0, 68.6 | 68.3, 67.0 | 63.6, 61.6 | 93.0, 91.2   |
| Average    | 46.48       | 50.36      | 68.03      | 68.80      | 68.91      | 73.93      | 98.72        |

\* Tabulated values, (Mooney) torque units.

TABLE B  
Measures of Cell Variation

(Table entries are  $s_{ij}$ )

| Laboratory                | Level (Material) |        |        |        |        |        |        |
|---------------------------|------------------|--------|--------|--------|--------|--------|--------|
|                           | 1                | 2      | 3      | 4      | 5      | 6      | 7      |
| 1                         | 0.707            | 0.     | 0.707  | 0.     | 0.707  | 0.     | 1.414  |
| 2                         | 2.546*           | 0.566  | 0.849  | 0.     | 0.495  | 0.424  | 1.414  |
| 3                         | 0.               | 0.778  | 0.212  | 0.212  | 0.0707 | 1.344  | 0.354  |
| 4                         | 0.707            | 0.     | 0.     | 0.354  | 0.     | 0.707  | 0.707  |
| 5                         | 0.636            | 0.354  | 0.495  | 0.354  | 0.566  | 0.707  | 0.636  |
| 6                         | 1.061            | 1.061  | 0.707  | 0.     | 1.061  | 2.475  | 2.121  |
| 7                         | 0.071            | 0.141  | 0.354  | 0.     | 0.424  | 0.778  | 0.141  |
| 8                         | 0.495            | 0.0707 | 0.707  | 0.354  | 0.707  | 1.344  | 0.0707 |
| 9                         | 0.283            | 0.     | 0.495  | 0.0707 | 0.283  | 0.424  | 0.354  |
| 10                        | 0.354            | 0.     | 0.707  | 0.354  | 0.354  | 0.     | 0.707  |
| 11                        | 0.424            | 0.141  | 2.334* | 0.283  | 0.919  | 1.414  | 1.273  |
| $\Sigma s^2_{ij} =$       | 9.648            | 2.221  | 8.784  | 0.631  | 3.926  | 13.703 | 11.798 |
| $s^2_{\max} =$            | 6.482            | 1.126  | 5.448  | 0.125  | 1.126  | 6.126  | 4.499  |
| $C_{\text{calc}} =$       | 0.672            | 0.507  | 0.620  | 0.199  | 0.287  | 0.447  | 0.381  |
| (95%) $C_{\text{crit}} =$ | 0.570            | 0.570  | 0.570  | 0.570  | 0.570  | 0.570  | 0.570  |
| (99%) $C_{\text{crit}} =$ | 0.680            | 0.680  | 0.680  | 0.680  | 0.680  | 0.680  | 0.680  |

\* = Outlier at 95% Confidence Level.

TABLE C

Cell Averages

Level (Material)

| <u>Laboratory</u> | <u>1</u> | <u>2</u> | <u>3</u> | <u>4</u> | <u>5</u> | <u>6</u> | <u>7</u> | <u>Avg.</u> |
|-------------------|----------|----------|----------|----------|----------|----------|----------|-------------|
| 1                 | 46.5     | 51.0     | 67.5     | 69.0     | 68.5     | 76.0     | 100.0    | 68.36       |
| 2                 | 48.6     | 49.6     | 69.0     | 68.3     | 69.25    | 75.5     | 99.0     | 68.46       |
| 3                 | 46.9     | 49.35    | 67.95    | 70.15    | 69.05    | 73.25    | 99.75    | 68.06       |
| 4                 | 46.5     | 51.0     | 66.0     | 68.25    | 70.0     | 69.5     | 97.5     | 66.96       |
| 5                 | 46.05    | 50.15    | 65.45    | 68.35    | 67.9     | 73.1     | 99.15    | 67.16       |
| 6                 | 47.75    | 50.25    | 66.5     | 68.0     | 67.75    | 77.25    | 96.5     | 68.71       |
| 7                 | 46.25    | 50.2     | 68.25    | 68.5     | 68.4     | 76.55    | 100.3    | 68.35       |
| 8                 | 48.55    | 52.35    | 69.5     | 69.25    | 69.7     | 81.35    | 99.05    | 69.96       |
| 9                 | 46.2     | 50.8     | 69.35    | 69.45    | 69.1     | 72.1     | 99.15    | 68.02       |
| 10                | 42.25    | 51.0     | 70.5     | 68.75    | 70.75    | 76.0     | 103.5    | 68.96       |
| 11                | 45.7     | 48.2     | 68.35    | 68.8     | 67.65    | 62.6     | 92.1     | 64.77       |
| Average           | 46.48    | 50.36    | 68.03    | 68.8     | 68.91    | 73.93    | 98.73    | 67.88       |

TABLE C2

Dixon's Outlier Test ( $H = 11$ )  
Outliers for Cell Average

| Ascending<br>Order Value | 1      | 2     | 3     | 4     | 5     | 6     | 7     |
|--------------------------|--------|-------|-------|-------|-------|-------|-------|
| 1                        | 42.25* | 48.2  | 65.45 | 68.0  | 67.65 | 62.6  | 92.1* |
| 2                        | 45.7   | 49.35 | 66.0  | 68.25 | 67.75 | 69.5  | 96.5  |
| 3                        | 46.05  | 49.6  | 66.5  | 68.3  | 67.9  | 72.1  | 97.5  |
| 4                        | 46.2   | 50.15 | 67.5  | 68.35 | 68.4  | 73.1  | 99.0  |
| 5                        | 46.25  | 50.2  | 67.95 | 68.5  | 68.5  | 73.25 | 99.05 |
| 6                        | 46.5   | 50.25 | 68.25 | 68.75 | 69.05 | 75.5  | 99.15 |
| 7                        | 46.5   | 50.8  | 68.35 | 68.8  | 69.1  | 76.0  | 99.15 |
| 8                        | 46.9   | 51.0  | 69.0  | 69.0  | 69.25 | 76.0  | 99.75 |
| 9                        | 47.75  | 51.0  | 69.35 | 69.25 | 69.7  | 76.55 | 100.0 |
| 10                       | 48.55  | 51.0  | 69.5  | 69.45 | 70.0  | 77.25 | 100.3 |
| 11                       | 48.6   | 52.35 | 70.5  | 70.15 | 70.75 | 81.35 | 103.5 |

| End Examined                   | L                                    | U                                    | none | none | none | L                                  | L                                  |
|--------------------------------|--------------------------------------|--------------------------------------|------|------|------|------------------------------------|------------------------------------|
| $Q_{11} =$                     | $\frac{45.7 - 42.25}{48.55 - 42.25}$ | $\frac{52.35 - 51.0}{52.35 - 49.35}$ | (a)  | (a)  | (a)  | $\frac{69.5 - 62.6}{77.25 - 62.6}$ | $\frac{96.5 - 92.1}{100.3 - 92.1}$ |
| (Calc) $Q_{11} =$              | 0.548                                | 0.450                                |      |      |      | 0.471                              | 0.537                              |
| $Q_{11} \text{ (Crit 95\%)} =$ | 0.502                                | 0.502                                |      |      |      | 0.502                              | 0.502                              |

\* Outlier at 95% Confidence Level.

(a) No calculations warranted due to lack of suspected outliers.

End Examined: L = Lower end of ordered values sequence; U = upper end of ordered values sequence.

- B.3 Calculation of r and R. We now proceed to calculate r and R. The calculations are illustrated for each material 1 to 7 in Tables D1 to D7. The calculations are performed according to Clause 14.8 of ISO 5725, with, however, the use of  $s^2_i$  rather than  $w^2_i$  ( $w$  = range or difference of two cell values). This is the uniform level,  $n = 2$ , calculation. Also to avoid confusion, the computational formula symbols  $S_1$ ,  $S_2$  and  $S_3$  of Clause 14.8 of ISO 5725 are replaced with  $(Sum_1)$ ,  $(Sum_2)$  and  $(Sum_3)$ . The use of Sum in place of S avoids confusing the computational terms with S, the standard deviation between laboratories.

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TABLE D1

Level 1

| <u>Laboratory</u>                          | <u>s<sub>i</sub></u> | <u><math>\bar{y}_i</math></u> |                           |
|--------------------------------------------|----------------------|-------------------------------|---------------------------|
| 1                                          | 0.707                | 46.5                          |                           |
| 2                                          | *                    | 48.6                          |                           |
| 3                                          | 0                    | 46.9                          | n = 2                     |
| 4                                          | 0.707                | 46.5                          | p = 10 (1 lab omitted)    |
| 5                                          | 0.636                | 46.05                         | f = 2                     |
| 6                                          | 1.061                | 47.75                         | * rejected values omitted |
| 7                                          | 0.071                | 46.25                         |                           |
| 8                                          | 0.495                | 48.55                         |                           |
| 9                                          | 0.283                | 46.2                          |                           |
| 10                                         | 0.354                | *                             |                           |
| 11                                         | <u>0.424</u>         | <u>45.7</u>                   |                           |
| (Sum <sub>1</sub> ) = $\Sigma \bar{y}_1$   |                      |                               | - 469.00                  |
| (Sum <sub>2</sub> ) = $\Sigma \bar{y}_1^2$ |                      |                               | 22,005.83                 |
| (Sum <sub>3</sub> ) = $\Sigma s_i^2$       |                      |                               | 3.1654                    |

---


$$s_r^2 = (\text{Sum}_3)/p = 3.1654/10 = 0.3165$$

$$s_L^2 = \left[ \frac{p(\text{Sum}_2) - (\text{Sum}_1)^2}{p(p-1)} \right] - \frac{s_r^2}{n}$$

$$s_L^2 = \left[ \frac{10 (22,005.83) - 219,961.}{10 (9)} \right] - \frac{0.3165}{2}$$

$$s_L^2 = 1.0811 - 0.1583 = 0.9228$$

$$s_r = 0.563$$

$$s_L = 0.961$$

$$r = f\sqrt{2} \sqrt{s_r^2} = 2.83 \sqrt{0.3165} = 1.592$$

$$R = f\sqrt{2} \sqrt{s_L^2 + s_r^2} = 2.83 S_R = 2.83 (1.113)$$

$$R = 2.83 \sqrt{0.9228 + 0.3165} = 3.151$$

TABLE D2

Level 2

| Laboratory                               | $s_i$  | $\bar{y}_i$ |
|------------------------------------------|--------|-------------|
| 1                                        | 0      | 51.0        |
| 2                                        | 0.566  | 49.6        |
| 3                                        | 0.778  | 49.35       |
| 4                                        | 0      | 51.0        |
| 5                                        | 0.354  | 50.15       |
| 6                                        | 1.061  | 50.25       |
| 7                                        | 0.141  | 50.2        |
| 8                                        | 0.0707 | 52.35       |
| 9                                        | 0.     | 50.8        |
| 10                                       | 0.     | 51.0        |
| 11                                       | 0.141  | 48.2        |
| (Sum <sub>1</sub> ) = $\sum \bar{y}_i$   | -      | 553.90      |
| (Sum <sub>2</sub> ) = $\sum \bar{y}_i^2$ | -      | 27,903.110  |
| (Sum <sub>3</sub> ) = $\sum s_i^2$       | 2.221  | -           |

$$s_r^2 = (\text{Sum}_3)/p = 2.221/11 = 0.202$$

$$s_L^2 = \left[ \frac{11 (27903.11) - 306805.21}{11 (10)} \right] - \frac{0.202}{2} = 1.1727 - 0.101 = 1.0717$$

$$s_r = 0.449$$

$$s_L = 1.035$$

$$r = 2.83 \sqrt{0.202} = 1.272$$

$$R = 2.83 \sqrt{1.0717 + 0.202} = 3.194$$

TABLE D3

Level 3

| <u>Laboratory</u>                          | <u>s<sub>i</sub></u> | <u><math>\bar{y}_i</math></u> |
|--------------------------------------------|----------------------|-------------------------------|
| 1                                          | 0.707                | 67.5                          |
| 2                                          | 0.849                | 69.                           |
| 3                                          | 0.212                | 67.95                         |
| 4                                          | 0.                   | 66.                           |
| 5                                          | 0.495                | 65.45                         |
| 6                                          | 0.707                | 66.5                          |
| 7                                          | 0.354                | 68.25                         |
| 8                                          | 0.707                | 69.5                          |
| 9                                          | 0.495                | 69.35                         |
| 10                                         | 0.707                | 70.5                          |
| 11                                         | <u>*</u>             | <u>68.35</u>                  |
| <hr/>                                      |                      |                               |
| (Sum <sub>1</sub> ) = $\Sigma \bar{y}_i$   | -                    | 748.35                        |
| (Sum <sub>2</sub> ) = $\Sigma \bar{y}_i^2$ | -                    | 50,936.113                    |
| (Sum <sub>3</sub> ) = $\Sigma s_i^2$       | 3.3364               | -                             |

$$s_r^2 = (\text{Sum}_3)/p = 3.3364/10 = 0.3364$$

$$s_L^2 = \left[ \frac{11 (50936.11) - 560027.72}{11 (10)} \right] - \frac{0.3364}{2} = 2.449 - 0.1682$$

$$= 2.282$$

$$s_r = 0.580$$

$$s_L = 1.511$$

$$r = 2.83 \sqrt{0.3364} = 1.641$$

$$R = 2.83 \sqrt{2.282 + 0.3364} = 4.579$$

\* rejected value omitted

TABLE D4

Level 4

| <u>Laboratory</u>                          | <u>s<sub>i</sub></u> | <u><math>\bar{y}_i</math></u> |
|--------------------------------------------|----------------------|-------------------------------|
| 1                                          | 0.                   | 69.                           |
| 2                                          | 0.                   | 68.3                          |
| 3                                          | 0.212                | 70.15                         |
| 4                                          | 0.354                | 68.25                         |
| 5                                          | 0.354                | 68.35                         |
| 6                                          | 0.                   | 68.                           |
| 7                                          | 0.                   | 68.5                          |
| 8                                          | 0.354                | 69.25                         |
| 9                                          | 0.0707               | 69.45                         |
| 10                                         | 0.354                | 68.75                         |
| 11                                         | <u>0.283</u>         | <u>68.8</u>                   |
| <hr/>                                      |                      |                               |
| (Sum <sub>1</sub> ) = $\Sigma \bar{y}_i$   | -                    | 756.80                        |
| (Sum <sub>2</sub> ) = $\Sigma \bar{y}_i^2$ | -                    | 52,071.815                    |
| (Sum <sub>3</sub> ) = $\Sigma s_i^2$       | 0.6313               | -                             |

$$s_r^2 = (\text{Sum}_3)/p = 0.6313/11 = 0.0574$$

$$s_L^2 = \left[ \frac{11 (52071.815) - 572746.24}{11 (10)} \right] - \frac{0.0574}{2} = 0.3688$$

$$s_r = 0.2396$$

$$s_L = 0.6073$$

$$r = 2.83 \sqrt{0.0574} = 0.673$$

$$R = 2.83 \sqrt{0.3688 + 0.0574} = 1.848$$

TABLE D5

Level 5

| <u>Laboratory</u>                          | <u>s<sub>i</sub></u> | <u><math>\bar{y}_i</math></u> |
|--------------------------------------------|----------------------|-------------------------------|
| 1                                          | 0.707                | 68.5                          |
| 2                                          | 0.495                | 69.25                         |
| 3                                          | 0.0707               | 69.05                         |
| 4                                          | 0.                   | 70.                           |
| 5                                          | 0.566                | 67.9                          |
| 6                                          | 1.061                | 67.75                         |
| 7                                          | 0.424                | 68.4                          |
| 8                                          | 0.707                | 69.7                          |
| 9                                          | 0.283                | 69.1                          |
| 10                                         | 0.354                | 70.75                         |
| 11                                         | <u>0.919</u>         | <u>67.65</u>                  |
| (Sum <sub>1</sub> ) = $\Sigma \bar{y}_i$   | -                    | 758.05                        |
| (Sum <sub>2</sub> ) = $\Sigma \bar{y}_i^2$ | -                    | 52,249.73                     |
| (Sum <sub>3</sub> ) = $\Sigma s_i^2$       | 3.9255               | -                             |

$$s_r^2 = (\text{Sum}_3)/p = 3.925/11 = 0.3569$$

$$s_L^2 = \left[ \frac{11 (52249.73) - 574639.80}{11 (10)} \right] = - \frac{0.3569}{2} = 0.7964$$

$$s_r = 0.5974$$

$$s_L = 0.8924$$

$$r = 2.83 \sqrt{0.3569} = 1.691$$

$$R = 2.83 \sqrt{0.7964 + 0.3569} = 3.039$$

TABLE D6

Level 6

| Laboratory                                 | $s_i$        | $\bar{y}_i$ |
|--------------------------------------------|--------------|-------------|
| 1                                          | 0.           | 76.         |
| 2                                          | 0.424        | 75.5        |
| 3                                          | 1.344        | 73.25       |
| 4                                          | 0.707        | 69.5        |
| 5                                          | 0.707        | 73.1        |
| 6                                          | 2.475        | 77.25       |
| 7                                          | 0.778        | 76.55       |
| 8                                          | 1.344        | 81.35       |
| 9                                          | 0.424        | 72.1        |
| 10                                         | 0.           | 76.         |
| 11                                         | <u>1.414</u> | <u>62.6</u> |
| (Sum <sub>1</sub> ) = $\Sigma \bar{y}_i$   | -            | 813.20      |
| (Sum <sub>2</sub> ) = $\Sigma \bar{y}_i^2$ | -            | 60,354.13   |
| (Sum <sub>3</sub> ) = $\Sigma s_i^2$       | 13.703       | -           |

$s_r^2 = (\text{Sum}_3)/p = 13.703/11 = 1.246$

$s_L^2 = \left[ \frac{11 (60354.13) - 661294.24}{11 (10)} \right] - \frac{1.246}{2} = 23.02$

$s_r = 1.115$

$s_L = 4.798$

$r = 2.83 \sqrt{1.246} = 3.155$

$R = 2.83 \sqrt{23.02 + 1.246} = 13.94$

TABLE D7

Level 7

| <u>Laboratory</u>                          | <u>s<sub>i</sub></u> | <u><math>\bar{y}_i</math></u> |
|--------------------------------------------|----------------------|-------------------------------|
| 1                                          | 1.414                | 100.                          |
| 2                                          | 1.414                | 99.                           |
| 3                                          | 0.354                | 99.75                         |
| 4                                          | 0.707                | 97.5                          |
| 5                                          | 0.636                | 99.15                         |
| 6                                          | 2.121                | 96.5                          |
| 7                                          | 0.141                | 100.3                         |
| 8                                          | 0.0707               | 99.05                         |
| 9                                          | 0.354                | 99.15                         |
| 10                                         | 0.707                | 103.5                         |
| 11                                         | <u>1.273</u>         | <u>*</u>                      |
| (Sum <sub>1</sub> ) = $\Sigma \bar{y}_i$   | -                    | 993.90                        |
| (Sum <sub>2</sub> ) = $\Sigma \bar{y}_i^2$ | -                    | 98,814.25                     |
| (Sum <sub>3</sub> ) = $\Sigma s_i^2$       | 11.798               |                               |

$$s_r^2 = (\text{Sum}_3)/p = 11.798/11 = 1.073$$

$$s_L^2 = \left[ \frac{10 (98814.25) - 987837.21}{10 (9)} \right] - \frac{1.073}{2} = 2.856$$

$$s_r = 1.036$$

$$s_L = 1.690$$

$$r = 2.83 \sqrt{1.073} = 2.932$$

$$R = 2.83 \sqrt{2.856 + 1.073} = 5.610$$

\* rejected value omitted

- B.4 Expressing the Precision Calculation. In this section of Annex B the results of the data tabulation, calculations and analysis as illustrated in Clause B1 to B3 are presented in the form of a typical Precision Section or Clause. Some of the information given in the Introduction to Annex B is repeated so as to present a complete "Precision Section" example.

The Supplementary Discussion, Clause 4.5, may not be required in a brief Precision Statement for a standard like (RS) 289. It is included here as an example of the types of information that may be gained from a more detailed analysis of precision testing.

XX.\* Precision.

- X.1 A Type 1 interlaboratory precision program was conducted. Both repeatability and reproducibility are short term, a period of a few days separate replicate test results. Eleven laboratories participated and seven materials or viscosity levels were used. Thus  $p = 11$ ,  $q = 7$ ,  $n = 2$ . A test result is the value obtained from a single determination, with the Mooney viscometer. Acceptable difference values were not measured.
- X.2 Details on the materials used and on test parameter variations among the seven materials are given in Table B1.

TABLE B1

Materials and Test Conditions Used

| <u>Material Number</u> | <u>Material Description</u>                          | <u>Test Temp (°C)</u> | <u>Other Details</u> |
|------------------------|------------------------------------------------------|-----------------------|----------------------|
| 1                      | SBR 1500                                             | 100                   | ML (1+4)             |
| 2                      | SBR 1712                                             | 100                   | ML (1+4)             |
| 3                      | EPDM                                                 | 125                   | ML (1+4)             |
| 4*                     | IIR (NBS-SRM 388j)**                                 | 100                   | ML (1+8)             |
| 5                      | Compounded Blend 1500/1505                           | 100                   | ML (1+4)             |
| 6                      | SBR Black Master Batch<br>(1712, 65 N339, 50 HA Oil) | 100                   | ML (1+4)             |
| 7                      | NR                                                   | 100                   | ML (1+4)             |

\*\* This IIR (butyl rubber) is a Standard Reference Material No. 388 Lot 3, as furnished by the National Bureau of Standards (USA). Viscosity is measured on unmassed specimens (samples). Measurements are made on unmassed samples.

\* X represents the actual clause number of any particular standard.

- X.3 The results of the precision calculations are presented in Table B2 for each of the seven materials with the materials arranged in ascending "mean level" order.

TABLE B2

ISO (RS) 289 Type 1-Precision\*

## Mooney Viscosity

|    | Material                 | Mean Level  | Within Labs  |              |             | Between Labs |              |             |
|----|--------------------------|-------------|--------------|--------------|-------------|--------------|--------------|-------------|
|    |                          |             | $s_r$        | r            | (r)         | $S_R$        | R            | (R)         |
| 1. | (SBR 1500)               | 46.5***     | 0.563        | 1.592        | 3.42        | 1.113        | 3.151        | 6.78        |
| 2. | (SBR 1712)               | 50.4        | 0.449        | 1.272        | 2.52        | 1.129        | 3.194        | 6.34        |
| 3. | EPDM                     | 68.0        | 0.580        | 1.641        | 2.41        | 1.618        | 4.579        | 6.73        |
| 4. | IIR (388j)               | 68.8        | 0.240        | 0.673        | 0.98        | 0.653        | 1.848        | 2.69        |
| 5. | Blend 1500/1505          | 68.9        | 0.597        | 1.691        | 2.45        | 1.074        | 3.040        | 4.41        |
| 6. | SBR-BMB                  | 73.9        | 1.115        | 3.155        | 4.27        | 4.930        | 13.94        | 18.86       |
| 7. | NR                       | <u>98.7</u> | <u>1.036</u> | <u>2.932</u> | <u>2.97</u> | <u>1.982</u> | <u>5.610</u> | <u>5.68</u> |
|    | Pooled or Average Values | 67.9        | 0.716        | 2.025        | 2.98        | 1.332**      | 3.77**       | 5.55**      |

\* This is short-term precision (days) with  $p = 11$ ,  $q = 7$ ,  $n = 2$ .

\*\* Values excluding Material #6 (SBR-BMB).

\*\*\* Mooney Torque units.

$s_r$  = within lab day-to-day standard deviation.

$S_R$  = total between lab standard deviation.

r = repeatability (in measurement units).

(r) = repeatability (in percent).

R = reproducibility (in measurement units).

(R) = reproducibility (in percent)

X.4 Figure B1 shows that there is reasonably good linear correlation between r and R versus the mean level of Mooney viscosity. Linear regression calculations yield the following results for a relation:  $r$  or  $R = a_0 + a_1 MV$ , where MV = Mooney viscosity.

| Type Regression | $a_0$ | $a_1$ | Correlation Coefficient |
|-----------------|-------|-------|-------------------------|
| r versus MV     | -0.16 | 0.033 | 0.77                    |
| R versus MV     | 0.32  | 0.052 | 0.87                    |

For these calculations IIR was omitted. The reason for this is discussed below. Such a linear dependence of r or R on MV with essentially zero intercepts indicates that r and R expressed on a relative or percent basis