Designation: D 6600 – 00

Standard Practice for Evaluating Test Sensitivity for Rubber Test Methods[1]

This standard is issued under the fixed designation D 6600; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 Testing to evaluate chemical constituents, chemical and physical properties of compounding materials, and compounded and cured rubbers may frequently be conducted by one or more test methods. When more than one test method is available, two questions arise: Which test method has the better (or best) response to or discrimination for the underlying fundamental property being evaluated? and Which test method has the least error? These two characteristics collectively determine one type of technical merit of test methods that may be designated as test sensitivity.

1.2 Although a comprehensive and detailed treatment, as given by this practice, is required for a full appreciation of test sensitivity, a simplified conceptual definition may be given here. Test sensitivity is the ratio of discrimination power for the fundamental property evaluated to the measurement error or uncertainty, expressed as a standard deviation. The greater the discriminating power and the lower the test error, the better is the test sensitivity. Borrowing from the terminology in electronics, this ratio has frequently been called the signal-to-noise ratio; the signal corresponding to the discrimination power and the noise corresponding to the test measurement error. Therefore, this practice describes how test sensitivity, generically defined as the signal-to-noise ratio, may be evaluated for test methods used in the rubber manufacturing industry, which measure typical physical and chemical properties, with exceptions as noted in 1.3.

1.3 This practice does not address the topic of sensitivity for threshold limits or minimum detection limits (MDL) in such applications as (*1*) the effect of intentional variations of compounding materials on measured compound properties or (*2*) the evaluation of low or trace constituent levels. Minimum detection limits are the subject of separate standards.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 The content of this practice is as follows:

2. Referenced Documents

2.1 *ASTM Standards:*

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

3. Terminology

3.1 A number of specialized terms or definitions are required for this practice. They are defined in a systematic or sequential order from simple terms to complex terms; the simple terms may be used in the definition of the more complex terms. This approach generates the most succinct and unambiguous definitions. Therefore, the definitions do not appear in the usual alphabetical sequence.

3.2 *Definitions:*

3.2.1 *fundamental property,* FP, *n*—the inherent or basic property (or constituent) that a test method is intended to evaluate.

3.2.2 *measured property,* MP, *n*—the property that the measuring instrument responds to; it is related to the FP by a functional relationship, MP = f (FP), that is known or that may be readily evaluated by experiment.

3.2.3 *reference material,* RM, *n*—a material (or other object) selected to serve as a common standard or benchmark for MP measurements for two or more test methods; the expected measurement value for each of the test methods, designated as

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

Current edition approved Nov. 10, 2000. Published January 2001.

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

the reference value, may be known (from other sources) or it may be unknown.

3.2.4 *calibration material*, CM, *n*—a material (or other object) selected to serve as a standard or benchmark reference material, with a fully documented FP reference value for a test method; the calibration material, along with several other similar materials with documented or FP reference values, may be used to calibrate a particular test method or may be used to evaluate test sensitivity.

3.2.4.1 *Discussion*—A fully documented FP or FP reference value implies that an equally documented measured property value may be obtained from a MP = f (FP) relationship. However, unless f = 1, the numerical values for the MP and the FP are not equal for any CM.

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

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

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

3.2.6 Although a simplified conceptual definition of test sensitivity was given in the Scope, a more detailed but still general definition using quantitative terms is helpful for preliminary discussion.

3.2.6.1 *test sensitivity* (generic), *n*—a derived quantity that indicates the level of technical merit of a test method; it is the ratio of the test discrimination power or signal, that is the magnitude of the change in the MP for some unit change in the related FP of interest, to the noise or standard deviation of the MP.

3.2.6.2 *Discussion*—This definition strictly applies to an absolute sensitivity, see 7.2. The change in the FP may be an actual measurement unit or a selected FP difference. The relation between the MP and the FP is of the form MP = f (FP).

4. Summary of Practice

4.1 This practice develops the necessary terminology and the required concepts for defining and evaluating test sensitivity for test methods. Sufficient background information is presented to place the standard on a firm conceptual and mathematical foundation. This allows for its broad application across both chemical and physical testing domains. The development of this practice draws heavily on the approach and techniques as given in the referenced literature.[3,4]

4.2 After the introduction of some general definitions, a brief review of the measurement process is presented succeeded by a development of the basic test sensitivity concepts. This is followed by defining two test sensitivity classifications, absolute and relative test sensitivity and two categories, (*1*) for a limited measured property range and (*2*) for an extended property range evaluation. For an extended property range for either classification, two types of test sensitivity may exist, (*1*) uniform or equal sensitivity across a range of properties or (*2*) nonuniform sensitivity which depends on the value of the measured properties across the selected range.

4.3 Annex A1 is an important part of this practice. It presents recommendations for using linear regression analysis for test sensitivity evaluation and recommendations for evaluating the precision of test sensitivity.

4.4 Appendix X1 is also an important adjunct to this practice. It gives two examples of relative test sensitivity calculations: (*1*) for a limited range or spot check program and (*2*) for an extended range test sensitivity program with a dependent (nonuniform) test sensitivity. Appendix X2 gives background on transformation of scale often needed for extended range sensitivity and for improved understanding, it also gives the derivation of the absolute test sensitivity for a simple analytical chemical test.

5. Significance and Use

5.1 Testing is conducted to make technical decisions on materials, processes, and products. With the continued growth in the available test methods for evaluating scientific and technical properties, a quantitative approach is needed to select test methods that have high (or highest) quality or technical merit. The procedures as defined in this practice may be used for this purpose to make testing as cost effective as possible.

5.2 One index of test method technical merit and implied sensitivity frequently used in the past has been test method precision. The precision is usually expressed as some multiple of the test measurement standard deviation for a defined testing domain. Although precision is a required quantity for test sensitivity, it is an incomplete characteristic (only one half of the necessary information) since it does not consider the discrimination power for the FP (or constituent) being evaluated.

5.3 Any attempt to evaluate relative test sensitivity for two different test methods on the basis of test measurement standard deviation ratios or variance ratios, which lack any discrimination power information content, constitutes an invalid quantitative basis for sensitivity, or technical merit evaluation. Coefficient of variation ratios (which are normalized to the mean) may constitute a valid test sensitivity evaluation only under the special condition where the two test methods under comparison are directly proportional or reciprocally related to each other. If the relationship between two test methods is nonlinear or linear with a nonzero intercept, the coefficient of variation ratios are not equivalent to the true test sensitivity as defined in this practice. See discussion of example in X1.1.4. The figure of merit defined by test sensitivity and its various classifications, categories, and types as introduced by this practice permits an authentic quantitative test sensitivity evaluation.

[3] Mandel, J., and Stiehler, R.D., *Journal of Research of National Bureau of Standards*, Vol 53, No. 3, September 1954. see also "Precision Measurement and Calibration—Statistical Concepts and Procedures," *Special Publication 300*, Vol 1, National Bureau of Standards, 1969, pp. 179–155). (The National Bureau of Standards is now the National Institute for Standards and Technology.)

[4] "The Statistical Analysis of Experimental Data", Chapters 13 and 14, J. Mandel, Interscience Publishers (John Wiley & Sons), 1964.

6. Measurement Process

6.1 *Brief Outline of the Measurement Process*—A measurement process involves three components: (*1*) a (chemical or physical) measurement system, (*2*) a chemical or physical property to be evaluated, and (*3*) a procedure or technique for producing the measured value. The FP to be determined or evaluated has two associated adjuncts: a measured quantity or parameter, MP, that can take on a range of numerical values and a relationship between FP and MP of the general functional form MP = f (FP). An implicit assumption is that the procedure or technique must be applicable across a range of material or system property values.

6.2 The fundamental property may be a defined characteristic, such as the percentage of some constituent in a material or it may be defined solely by the measuring process itself. For this latter situation the measurement and the property are identical, and MP = FP or f = 1. This is the usual case for many strictly technological measurement operations or tests, for example, the modulus of a rubber. The MP = f (FP) relationship must be monotonic; for every value of MP there must be a unique single value for FP. The relationship must be specific for any particular measuring process or test, and, if there are two different processes or tests for evaluating the FP, the relationship is generally different for each test.

7. Development of Test Sensitivity Concepts

7.1 *Test Domain*—The scope of any potential test sensitivity evaluation program should be established. Is the evaluation for a limited local testing situation, that is, one laboratory or test location? Or are the results to be applied on a global basis across numerous domestic or worldwide laboratories or locations? If local testing is the issue, the test measurements are conducted in one laboratory or location. For global testing, an interlaboratory test program (ITP) must be conducted. Two or more replicate test sensitivity evaluations are conducted in each participating laboratory and an overall or average test sensitivity is obtained across all laboratories. In the context of an ITP for global evaluation, each replicate sensitivity evaluation is defined as the entire set of operations that is required to calculate one estimated value for the test sensitivity. For additional background on the assessment of precision for the test sensitivity values attained, see A1.2 and also Practice D 4483.

7.2 *Test Sensitivity Classification*—There are two classifications for test sensitivity.

7.2.1 Class 1 is absolute test sensitivity, or ATS, where the word absolute is used in the sense that the measured property can be related to the FP by a relationship that gives absolute or direct values for FP from a knowledge of the MP. In evaluating test sensitivity for this class, two or more CMs are used each having documented values for the FP.

7.2.2 Class 2 is relative test sensitivity, or RTS, where the test sensitivity of Test Method 1 is compared to Test Method 2, on the basis of a ratio, using two or more RMs with different MP values. This class is used for physical test methods where no FPs can be evaluated.

7.3 *Absolute Test Sensitivity*—In this section absolute or direct test sensitivity is defined in a simplified manner by the use of Fig. 1.

7.3.1 *Development of Absolute Test Sensitivity*—Fig. 1 is concerned with two types of properties: (*1*) a FP (or single criterion or constituent), the value of which is established by the use of a CM and (*2*) a MP obtained by applying the test method to the CM. A relationship or functionality exists between the MP and FP that may be nonlinear. In the application of a particular test, FP_1 corresponds to MP_1 and FP_2 corresponds to MP_2. Over a selected region of the relationship, designed by points *a* and *b*, the slope, K, of the illustrated curve is approximated by the relationship $K = \Delta(MP)/\Delta(FP)$. If the test measurement standard deviation for MP denoted as S_{MP}, is constant over this *a* to *b* range, the absolute test sensitivity designated as ψ_A is defined by Eq 1.

$$\psi_A = | K | / S_{MP} \quad (1)$$

The equation indicates that for the selected region of interest, test sensitivity will increase with the increase of the numerical (absolute) value of the slope, | K | and sensitivity will increase the more precise the MP measurement. Thus, ψ_A may be used as a criterion of technical merit to select one of a number of test methods to measure the FP provided that a functional relationship, MP = f (FP), can be established for each test method.

7.3.2 Absolute test sensitivity may not be uniform or constant across a broad range of MP or FP values. It is constant across a specified range, only if the direct (not transformed) MP versus FP relationship is linear and the test error S_{MP} is constant. With an assumed monotonic relationship between FP and MP, absolute test sensitivity, ψ_A, may be evaluated on the basis of (*1*) two or more CMs, (or objects) with different known FP values or (*2*) a theoretical relationship between MP and FP.

7.3.3 *Formal Development for* ψ_A—For the completely general case, a more formal mathematical development for absolute test sensitivity that does not involve the approximation of the slope using the deltas, Δ(MP) and (ΔFP), can be given in terms of differentials. When differentials are used, K = | *d*[MP]/d[FP] | and K is the tangent to the curve at some particular point. Appendix X2 outlines the derivation of the absolute test sensitivity for a simple analytical test on this more theoretical and formal basis.

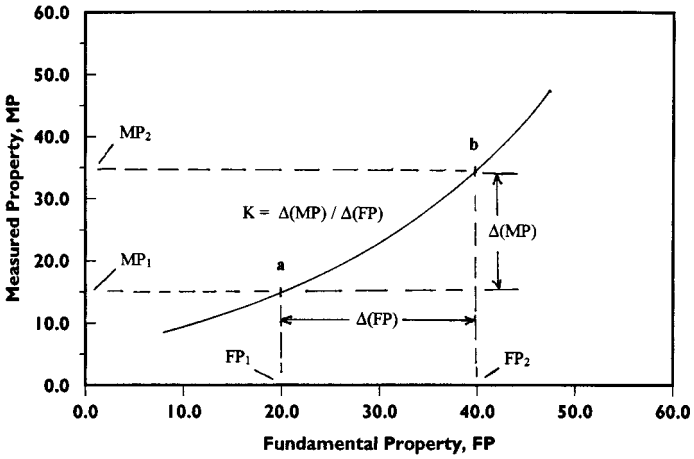

FIG. 1 Measured Versus Fundamental Property Relationship

7.4 *Absolute Test Sensitivity: Empirical Versus Theoretical*—Evaluating absolute test sensitivity requires that a well-established relationship exist between MP and FP. This can be obtained in one of two ways.

7.4.1 The empirical evaluation makes use of CMs, each with a different value for the FP designated as a FP calibration value; these values being certified by some recognized independent procedure or authority. The relationship is experimentally or empirically determined.

7.4.2 The theoretical evaluation is conducted by using the relationship between the MP and the FP, based on scientific or theoretical principles, for some measurement system that permits the calculation of FP calibration values for certain specified conditions. This will not be addressed by this practice since this practice is limited to experimental or empirical techniques as defined in 7.4.1.

7.5 *Relative Test Sensitivity*—When typical physical test methods are employed a relationship between MP and FP using CMs usually is not feasible or possible. The primary purpose of most if not all physical test methods is to make simple relative comparisons on the basis of MP values. Under these circumstances, it is not possible to evaluate absolute test sensitivity.

7.5.1 *Development of Relative Test Sensitivity*—If an absolute test sensitivity cannot be obtained, it is possible to evaluate the relative sensitivities of two or more test methods. This can be accomplished without knowledge of the MP = f (FP) relationship for each test method. The most simple and direct way to demonstrate how this is possible to assume that we have two test methods for which absolute test sensitivities are known. Fig. 2 illustrates the general relationship between Test Methods 1 and 2, with properties designated as MP1 and MP2 and the actual measured values of these two properties designated as MP_1 and MP_2. Since we know the two absolute test sensitivities, ψ_A 1 and ψ_A 2, we know the values for K_1, S_{MP1}, K_2, and S_{MP2} as given in Eq 2.

$$\psi_A\ 1 \mid K_1 \mid / S_{MP1} \text{ and } \psi_A\ 2 = \mid K_2 \mid / S_{MP2} \tag{2}$$

For test method comparison purposes, we form ratios of ψ_A 1 to ψ_A 2, and using the two relationships of Eq 2 we obtain

$$\psi_A\ 1 / \psi_A\ 2 = \mid K_1 / K_2 \mid [S_{mP1} / S_{MP2}] = \mid Ko \mid / [S_{MP1} / S_{MP2}] \tag{3}$$

The ratio $| K_1 / K_2 |$ which is defined as Ko, is obtained using numerical (absolute) values for K_1 and K_2 since positive values for the ratio are desired.

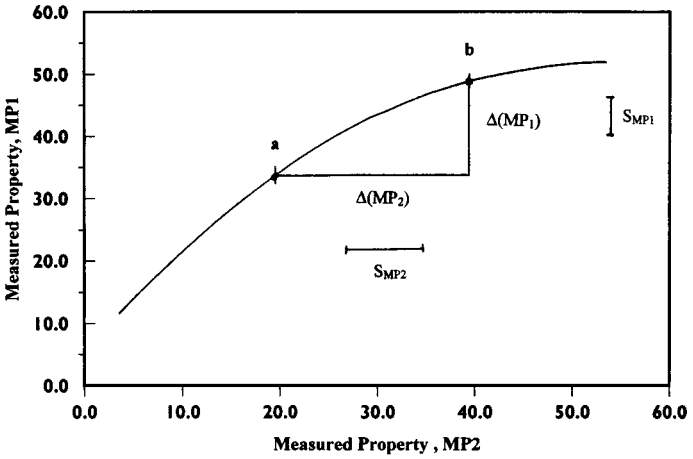

FIG. 2 Measured Property 1 Versus 2 Relationship

7.5.2 Fig. 2 illustrates the curvilinear relationship between MP1 and MP2 with the approximate slope given by $\Delta(MP_1)/\Delta(MP_2)$ and the magnitudes of S_{MP1} and S_{MP2} indicated by vertical and horizontal bars. The Ko may be evaluated as follows:

$$Ko = \mid K_1 / K_2 \mid = [\Delta(MP_1)/\Delta(FP)] / [\Delta(MP_2 / \Delta(FP)] = \mid \Delta(MP_1)/\Delta(MP_2) \mid \tag{4}$$

since the FP values, although unknown, are common to both MP_1 and MP_2 and the absolute value of $(\Delta MP_1)/\Delta(MP_2)$ is used. Thus Ko may be evaluated without any knowledge of the FPs; the requirements are (1) the relationship between MP1 and MP2 must be empirically known and (*2*) the measurements MP_1 and MP_2 must be made on the same set of RMs, each of which has a different fundamental property or FP value that may or may not be known. On this basis, the relative test sensitivity, for Test Method 1, or T1, compared to Test Method 2, or T2, designated as, ψ_R (T1/T2), is defined by Eq 5 as the ratio of T1 test sensitivity to T2 test sensitivity

$$\psi_R\ (T1/T2) = [\Delta(MP_1)/\Delta(MP_2)] / [S_{MP1} / S_{MP2}] = \mid Ko \mid / [S_{MP1} / S_{MP2}] \tag{5}$$

Unless otherwise needed, the excessive notation burden of the parenthetical term (T1/T2) is dropped to avoid confusion, and it is understood that the symbol ψ_R indicates a comparison of (numerator) Test Method 1 to (denominator) Test Method 2.

7.5.3 If ψ_R is above unity, Test Method 1 is more sensitive then Test Method 2. If ψ_R is below unity, Test Method 2 is more sensitive than Test Method 1. Again, the relative test sensitivity is applicable to a particular intermediate range of MP_1 and MP_2 values unless the plot of MP_1 versus MP_2 is linear and the ratio (S_{MP1}/S_{MP2}) is constant across the experimental range under study. The relative test sensitivity can be expressed in more formal mathematical terms by the use of differentials rather than deltas (ΔMP_1) and $\Delta(MP_2)$; (see Appendix X2).

7.6 *Test Sensitivity Categories and Types*—For each of the two classes of test sensitivity, there are two sensitivity categories and for Category 2 there are two types of test sensitivity.

7.6.1 *Category 1* is designated as a limited range or spot check test sensitivity. This is an assessment of absolute test sensitivity by a procedure that uses two (or perhaps three) different CMs for the FP values or for an assessment of relative test sensitivity by the use of two (or three) different RMs. This is in essence a spot check in a selected MP range.

7.6.2 *Category 2* is an extended range test sensitivity, a more comprehensive evaluation assesment over a substantial part or all of the entire working range of MP vs FP values or MP1 versus MP2 values, as customarily used in routine testing. Evaluating a Category 2 absolute test sensitivity requires several CMs; the recommended number is 4 to 6, with several measurements of MP for each established CM value for the FP. Evaluating a Category 2 relative test sensitivity also requires several RMs, the recommended number is 4 to 6, with several measurements of MP for each RM.

7.6.3 Category 2 test sensitivity may or may not be uniform or constant across a broad range of the MP. Thus there are two types of sensitivity.

7.6.3.1 *Uniform Test Sensitivity (Type 1)* is a test sensitivity

that is uniform or constant across the entire experimental range as investigated. This requires a constant value for the (S_{MP1}/S_{MP2}) ratio across this range.

7.6.3.2 *Nonuniform or dependent Test Sensitivity (Type 2)* is a test sensitivity that depends on, or is correlated with, the value of either MP across the experimental range. The ratio, S_{MP1}/S_{MP2}, can usually be expressed as a linear function of either MP (used as the *x* variable) in the MP1 versus MP2 relationship.

8. Steps in Conducting a Test Sensitivity Evaluation Program

8.1 *Initial Decisions*—A test sensitivity program requires decisions on a number of preliminary issues Decisions as indicated by 8.1.1-8.1.4 are required prior to any actual testing. The subsequent required steps are dependent on what decisions were made for 8.1.1-8.1.4, and these steps are given on the basis of a local evaluation program in four sections of this practice. Fig. 3 is an outline diagram that may help in the decision process. For absolute test sensitivity, 8.2.1 lists the steps for a spot check and 8.2.2 gives the steps for an extended range program. For relative test sensitivity, 8.3.1 is for a spot check and 8.3.2 for an extended range program. Although there is some repetition of the instructions for the execution of the program in these four sections, this arrangement allows the user of this practice to go directly to the section pertinent to the requirements for the test sensitivity to be evaluated. Recommendations for a global evaluation program are found in A1.2.

8.1.1 *Tests to Be Evaluated*—Select the test method(s) to be evaluated. For most programs, this would be two or more test methods since even for absolute test sensitivity there is an implication that a comparison of two or more tests is the goal of the program. Ensure that the procedure for each test method is well-established and documented.

8.1.2 *Test Domain*—The scope of the test sensitivity program should be established; local for testing in one laboratory or test location or global for numerous domestic or worldwide laboratories or locations.

8.1.3 *Class of Test Sensitivity*—The test sensitivity classification must be selected; Class 1 is an absolute test sensitivity and Class 2 is a relative test sensitivity.

8.1.4 *Category of Test Sensitivity*—Select the evaluation procedure, Category 1 for a limited range or spot check program or Category 2 for an extended range program. For Category 2 extended range evaluations for either absolute or

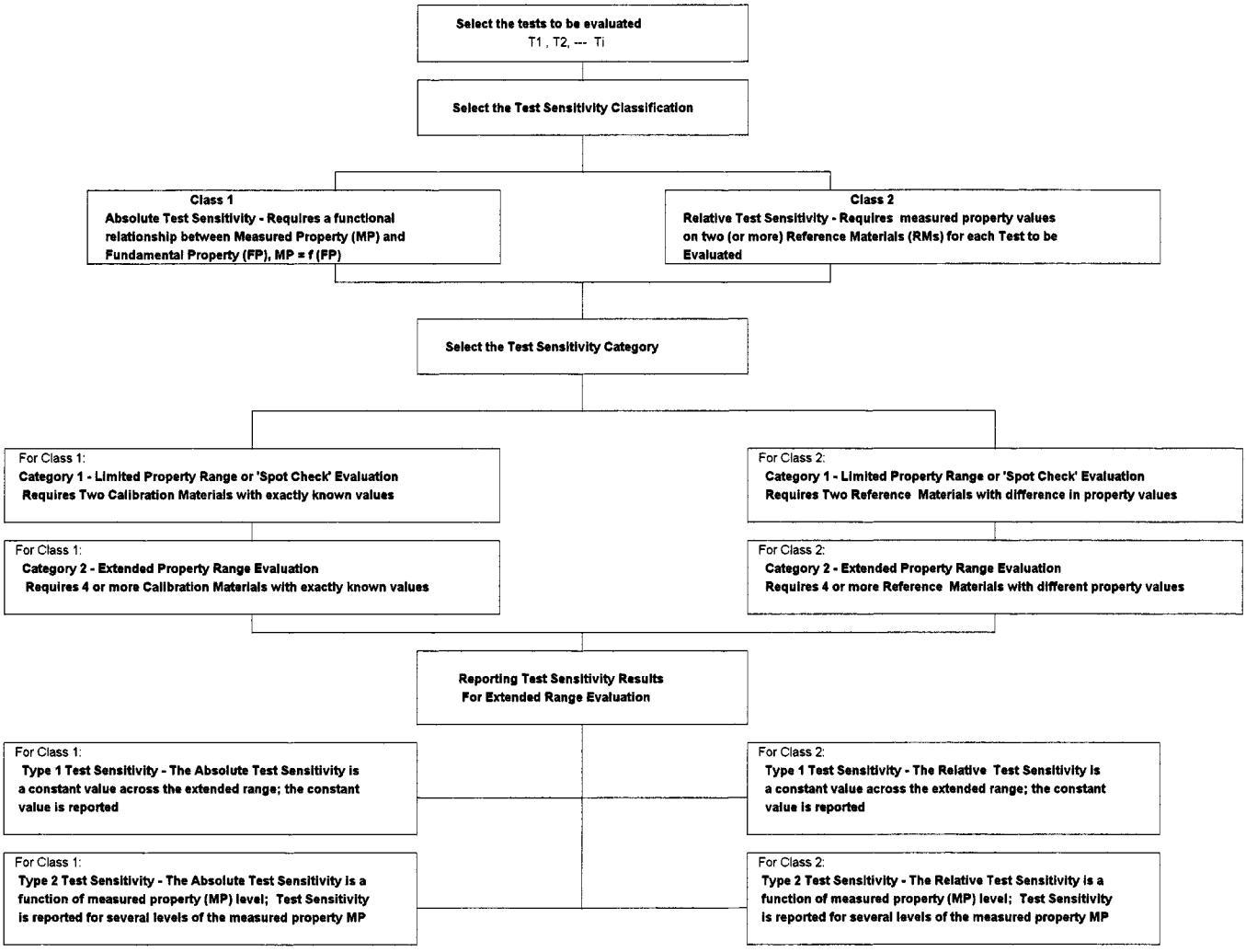

FIG. 3 Outline of the Steps in a Test Sensitivity Evaluation Program for a Local Test Domain

relative test sensitivity, the final evaluated test sensitivity may not be uniform across the range under study. For reporting test sensitivity, this requires a tabulation of values for ψ_A at selected values of the FP or ψ_R at selected values for the MP.

8.2 *Absolute Test Sensitivity*—Follow the steps in accordance with 8.2.1 or 8.2.2 for this classification.

8.2.1 *Limited Range or Spot Check*—Select the two (or three) CMs to be used. The difference between the MP values for the two (or three) CM should be large enough to permit a good evaluation of the slope K. The FP calibration values for each CM must be known to an accuracy sufficient for the purposes of the sensitivity evaluation program. This implies that the uncertainty region for the (certified) FP calibration values be one fourth or less of the uncertainty for the MP values.

8.2.1.1 *Replication for MP Measurements*—For each calibration material conduct sufficient replicate MP measurements to establish a good estimate of the MP average and standard deviation of the measurement process. The absolute minimum number of replicates is four; five or six replicates is much better. For each CM calculate the standard deviation for the replicate MP measurements. Calculate the average or pooled variance across all the CMs used. The square root of this calculation is defined as S_{MP}.

8.2.1.2 *Establish the MP Versus FP Relationship*—Determine the slope or K for each test method under review. Refer to 7.3.1.

8.2.1.3 *Calculate the Absolute Test Sensitivity*—For each test method under consideration, the absolute test sensitivity, ψ_A, is obtained from the values for | K | and S_{MP} by the use of Eq 1. If several test methods are being evaluated, prepare a table of ψ_A values for each test method for a review of results.

8.2.2 *Extended Range*—Select the number of CMs to be used. For a good extended range evaluation, four or more CMs are required as a minimum; five or six are better. The selected CMs should span the range with approximately equal differences between each successive CM in an ascending value order. The FP calibration values for each CM must be known to an accuracy sufficient for the purposes of the sensitivity evaluation program. This implies that the uncertainty region for the (certified) FP calibration values be one fourth or less of the uncertainty for the MP values.

8.2.2.1 *Replication for MP Measurements*—For each CM, conduct sufficient replicate MP measurements to establish a good estimate of the MP average and the standard deviation of the measurement process. The absolute minimum number of replicates is four; five or six replicates is much better. Calculate the variance and standard deviation for each set of replicate MP measurements on each calibration material.

8.2.2.2 *MP Measurement Standard Deviations*—Determine if there is a relationship (linear or otherwise) between the MP standard deviation for each CM and the MP average, for each CM. If a statistically significant relationship exists, then ψ_A is nonuniform and varies as the level of the MP or FP varies across the range examined. This variation must be taken into account in calculating ψ_A by establishing a regression equation that relates S_{MP} to MP across the range of values used in the program. This is of the form $S_{MP} = ao + a_1(MP)$, where intercept ao and slope a_1 are evaluated from a regression analysis assuming linearity as an approximation of the relationship.

8.2.2.3 If there is no significant relationship between the MP individual standard deviations (for each CM) and the MP, calculate the average or pooled variance for the MP across all the CMs used. The square root of this calculation is defined as S_{MP}.

8.2.2.4 *Establish the MP vs FP Relationship*—Generate the plot of the MP versus the FP and examine its nature, linear or curvilinear. The ideal outcome is a linear relationship. For curvilinear relationships, perform transformations (on one or both variables) to obtain a linear functionality. See Appendix X2 for recommendations on applicable transformations. Once a satisfactory linear relationship is found based on visual examination, conduct a linear regression analysis. For the MP vs FP relationship, the calculation results are expressed in terms of the constant or intercept, bo and the slope or linear regression coefficient, b_1, where the subscript 1 may be replaced by one or more symbols that refer directly to the two measured properties, MP and FP. Also calculate the correlation coefficient, R (or R^2) and the standard deviation (or standard error of estimate), Syx, about the fitted line. Follow the procedure in accordance with A1.1.1 and A1.1.2. For each test method under review, the MP vs FP slope or regression b coefficient is equal to K.

8.2.2.5 *Calculate the Uniform Absolute Test Sensitivity*—If S_{MP} is invariant or equal across the range of MP values, use the standard deviation obtained from the pooled variance for the MP measurements, as the value for S_{MP}. Refer to Eq 1. This calculation gives the uniform test sensitivity.

8.2.2.6 *Calculate the NonUniform or Dependent Absolute Test Sensitivity*—If the individual MP standard deviations (across the CMs used) is a function of either property, the denominator of Eq 1 varies with the value of MP. This requires an expression of the form $S_{MP} = [ao + a_1(MP)]$ as developed in 8.2.2.2 where the numerical values as obtained from analysis are substituted for ao and a_1. On this basis ψ_A is reported as a dependent absolute test sensitivity and a table of values should be prepared giving ψ_A for each of several selected MP values across the experimental range. If several test methods are being evaluated, prepare a table of ψ_A values at some reference value for each method, such as the middle of the MP range. This permits a common basis comparative review of the test sensitivities for all the methods under consideration.

8.3 *Relative Test Sensitivity*—To evaluate relative test sensitivity, follow the steps in accordance with 8.3.1 and 8.3.2.

8.3.1 *Limited Range or Spot Check*—Select the two (or three) RMs to be used. Although it is not required that a certified FP value be known for each it is appropriate to know an approximate value for the MP or FP for each RM. The difference between the MP values for the two (or three) RMs should be large enough to permit a reliable evaluation of the slope or Ko as given in Eq 4.

8.3.1.1 *Replication for MP Measurements*—For each RM, conduct sufficient replicate MP measurements, for each of the test methods under review, to establish a good estimate of the standard deviation of the measurement process. The absolute

minimum number of replicates is four; five or six replicates is much better especially for a limited range evaluation. For each test method, calculate the standard deviation for each set of replicate MP measurements and calculate the average or pooled variance across all the RMs used. The square root of the pooled variance for Test Method 1, or T1, is defined as S_{MP1} and the square root of the pooled variance for Test Method 2, or T2, is defined as S_{MP2}.

8.3.1.2 *Establish the MP1 Versus MP2 Relationship*—Determine the slope or Ko for each test method under review using Δ(MP) or delta values as given by Eq 4. This assumes that only two RMs are being used. If three RMs are used, a slope may be determined by linear regression analysis assuming that linearity is a good approximation of the MP1 versus MP2 functionality.

8.3.1.3 *Calculate the Relative Test Sensitivity*—For each test method under consideration, ψ_R is obtained from the values for | Ko | and the ratio (S_{MP1}/S_{MP2}) by the use of Eq 5. If several test methods are being evaluated, select one of the test methods as a reference or standard method and use this as T2 (the denominator in the T1/T2 ratio) for all relative test sensitivity calculations. Thus, for the three test methods, that are three ψ_R values; ψ_R(T1/T2), Test Method 1 compared to Test Method 2; and ψ_R(T3/T2), Test Method 3 compared to Test Method 2; and ψ_R(T2/T2) which by definition is 1.00. The numerical values for ψ_R (T1/T2) and ψ_R (T3/T2) may be compared to 1.00 to determine which of the three test methods has the highest test sensitivity.

8.3.2 *Extended Range*—Select the number of RMs to be used. For a good extended range evaluation, four or more RMs are required as a minimum; five or six are better. The selected RMs should span the range with approximately equal differences between each successive RM in an ascending value order. Thus approximate values for the MP or FP should be known for each RM.

8.3.2.1 *Replication for MP Measurements*—For each RM, conduct sufficient replicate (MP1, MP2, and so forth) measurements to establish a good estimate of the standard deviation of the measurement process for all MPs. The absolute minimum number of replicates is four; five or six replicates is much better. If more than two test methods are being evaluated, one test method should be selected as the reference or standard method and used as a reference for a comparative review of ψ_R for all test methods. For each test method, calculate the variance and standard deviation for each set of replicate MP measurements on each RM.

8.3.2.2 *MP Measurement Standard Deviations*—For each test method, determine if there is a relationship (linear or otherwise) between the standard deviation (for each RM) and either or both MPs. This is for general background information. Next calculate the ratio (S_{MP1}/S_{MP2}) for each RM and determine if this ratio is a function of either MP. If a statistically significant relationship exists, then ψ_R is nonuniform and varies with the level of the MP across the range examined. Establish a regression equation of the form (S_{MP1}/S_{MP2}) = ao + a_1(MP), where ao and a_1 are evaluated from a regression analysis assuming linearity as an approximation of the relationship.

8.3.2.3 *Establish the MP1 Versus MP2 Relationship*—The next operation is to establish a relationship between the two MPs. For this relationship or plot, the *x* variable should be the MP with the smaller pooled variance for the MP measurements across all RM. Select this MP and construct a plot of the other MP (as *y*) and the MP with smaller pooled variance (as *x*) and examine its nature, linear or curvilinear. The ideal outcome is a linear relationship. For curvilinear relationships, transformations, on one or both variables, may be made to obtain a linear functionality. See X2.1.3 for recommendations on applicable transformations.

8.3.2.4 *Evaluating Ko*—Once a satisfactory linear relationship is found based on visual examination, conduct a linear regression analysis. For the MP1 versus MP2 relationship, the calculation results are expressed in terms of the constant or intercept, bo and the slope or linear regression coefficient, b_1, where the subscript 1 may be replaced by one or more symbols that refer directly to the two MPs, MP1 and MP2. Also calculate the correlation coefficient, R (or R^2) and the standard deviation (or standard error of estimate), Sy.x, about the fitted line. Follow the procedure in accordance with A1.1.1 and A1.1.2. The slope or Ko of Eq 4 is equal to the regression coefficient, b_1, for each test method under review. Refer to 7.5.1 and 7.5.2.

8.3.2.5 *Calculate the Uniform Relative Test Sensitivity*—If (S_{MP1}/S_{MP2}) is invariant (equal) across the range of values, use the overall average (S_{MP1}/S_{MP2}) obtained from the pooled variances, to calculate the uniform relative test sensitivity ψ_R.

8.3.2.6 *Calculate the NonUniform or Dependent Relative Test Sensitivity*—If the ratio (S_{MP1}/S_{MP2}) varies with the value of either MP, an expression of the form (S_{MPl}/S_{MP2}) = [ao + a_1(MP)], as developed in 8.3.2.2, is required and the term [ao + a_1 (MP)] must be substituted for the denominator of Eq 5. This assumes that a linear expression is a good approximation. Numerical values are used for ao and a_1 as obtained from analysis and the term MP may represent transformed values. On this basis, ψ_R is reported as a nonuniform or dependent test sensitivity and a table of values should be prepared giving ψ_R for a number of selected MP values across the experimental range. If several test methods are being evaluated, prepare a table of ψ_R values at some reference value for each test method, such as the middle of the MP range. This permits a common basis comparative review of the test sensitivities for all the methods under consideration.

9. Report for Test Sensitivity Evaluation

9.1 A report of the results for a test sensitivity evaluation should be prepared. This is required due to the various classes, categories, and types of test sensitivity that may be under investigation. Report the following information:

9.1.1 Test method(s) under investigation,

9.1.2 The CMs or RMs used for the program, certified FP values, and approximate RM values,

9.1.3 The classification, absolute or relative test sensitivity,

9.1.4 The category, limited range (spot check) or extended range (list the range),

9.1.5 The type of test sensitivity obtained, uniform or nonuniform (dependent),

9.1.6 Any transformations made,

9.1.7 A tabulation of the one or more uniform or nonuniform (dependent) test sensitivities, and

9.1.8 Standard deviation of the test sensitivities, if evaluated.

10. Keywords

10.1 absolute test sensitivity; calibration material; reference material; relative test sensitivity; signal-to-noise ratio; test sensitivity

ANNEX

(Mandatory Information)

A1. BACKGROUND ON: USE OF LINEAR REGRESSION ANALYSIS AND PRECISION OF TEST SENSITIVITY EVALUATION

A1.1 Linear Regression Analysis: MP Versus FP and MP1 Versus MP2

A1.1.1 This annex applies to an extended range or Category 2 test sensitivity. Once an apparent linear functionality between MP and FP or MP1 and MP2 has been found (if necessary by applying transformations), a decision on the goodness of fit for the particular selected functionality can be made. The recommended procedure is not exact but a first order approximation that should be suitable for most circumstances.

A1.1.2 For a absolute sensitivity, plot the individual values of MP used as the *y* variable versus the FP value for each CM, and, for a relativity sensitivity, plot the individual values of MP1 (for each RM) versus the individual values of MP2 (for each RM), that is, do not use the average values for each RM as the *x* or *y* variable values. Conduct a linear regression analysis on this individual value *x* and *y* database. Evaluate the slope b_1, the intercept *b*o, the standard error of the estimate, *S*yx, and the correlation coefficient, R. Form a ratio of the variance of the regression estimate (standard error of estimate squared) to the pooled variance (across all calibration materials or reference materials) for the MP used as the *y* variable. For good fit, these two variances should be approximately equal. However, if the ratio of these two variances is of the order of 4 or less, the goodness of fit can be considered as acceptable and the particular functionality adopted as a reasonable approximation for evaluating the slope K or Ko. If the ratio is above 4, another better fitting functionality should be found.

A1.1.3 One of the assumptions in classic linear regression analysis is that each *x* value is known with zero or very minimal error compared to the variation in the *y* variable. The typical relative test sensitivity evaluation does not conform to this requirement since both measured properties are subject to test error. The recommended procedure to address this is to select for the *x* variable, the measured property that has the lowest pooled variance across all the CMs or RMs used in the program. This produces a minimum error estimate for the linear regression *b* coefficient or slope of the MP versus FP or MP1 versus MP2 relationship.

A1.1.4 Most relative test sensitivity programs are conducted with one of the measured properties preselected as the basis or standard for comparison, that is, T2 in the parameter ψ_R(T1/T2), which is the MP that corresponds to the *x* value. If T2 has a higher pooled variance than T1, the procedure to follow is to conduct the regression analysis on the basis *x* = T1 = MP1 and *y* = T2 = MP2 and obtain the slope or regression coefficient designated as *b* (T2/T1). This coefficient is the inverse of the coefficient *b* (T1/T2), and after the analysis has been conducted, the reciprocal of *b* (T2/T1) is obtained and used as the best estimate of *b* (T1/T2). This operation reduces the perturbing influence of the *x* variation on the *b* coefficient estimate. If the variance ratio, (T with larger variance/ T with smaller variance) is not substantially different from unity (of the order of 4 or less), the difference in the *b* coefficient estimates is not great.

A1.2 Precision of Test Sensitivity Evaluation

A1.2.1 Test sensitivity is evaluated on the basis of experimentally measured parameters, and the precision of any test sensitivity estimate depends on the precision of these measurements. For ψ_A there are two parameters; the slope of MP versus FP relationship, K, and the standard deviation S_{MP}. For ψ_R there are three parameters; the slope of the MP1 versus MP2 relationship, Ko, and the standard deviations S_{MP1} and S_{MP2}. It is possible to relate the uncertainty in ψ_A and ψ_R to the uncertainties in the measured parameters by means of propagation of random error equations. However, this is not addressed in this practice for two reasons: (*1*) it adds a measure of complexity that is beyond the scope of this practice and (*2*) it may not yield a true estimate of the real uncertainty in evaluating ψ_A and ψ_R.

A1.2.2 The system-of-causes that generates uncertainty in either ψ_A or ψ_R includes variation in individual set up operations in addition to the actual test measurement variation as such. The actual CMs or RMs used, the condition of these materials, the operators used for the testing, and the ambient laboratory operating conditions (accuracy of instrument calibrations) all contribute to total uncertainty in any ψ_A and ψ_R value. For a reliable estimate of the uncertainty for ψ_A and ψ_R, such components of variation must be included for a realistic precision program.

A1.2.3 The recommended procedure for evaluating test sensitivity uncertainty or confidence levels is as follows:

A1.2.3.1 *Local Evaluation of Test Sensitivity*—For local absolute or relative test sensitivity, repeat the total evaluation of either sensitivity a sufficient number of times to obtain a good estimate of the standard deviation for either test sensitivity. If ψ_A or ψ_R can be fully evaluated in one day, conduct at least four separate complete evaluations (of the total operation)

over a several-day period. This gives a bare minimum degree of freedom estimate of test sensitivity standard deviation. It is much preferable to obtain six or more estimates for either sensitivity.

A1.2.3.2 Use these standard deviation estimates to calculate confidence intervals or to conduct statistical significance tests (*t*-Tests) for the difference ($\psi_A - 1.00$) or ($\psi_R - 1.00$), since unity for either ψ implies that there is no difference in the test sensitivity T1 versus T2. The use of more sophisticated multiple comparisons, such as the Duncan Range test, can be employed for comparing several (more than two) ψ_A or ψ_R estimates.

A1.2.3.3 *Global Evaluation of Test Sensitivity*—For a global evaluation of test sensitivity, the procedures and protocols as developed for interlaboratory precision should be followed. Refer to Practice D 4483. One or more experienced staff members in one laboratory should be selected to organize the global program. Select a number of laboratories that have good experience with the test methods. Sufficient quantity of a homogeneous lot of each RM should be set aside and samples sent to each participating laboratory. Two individual or separate test sensitivity evaluations (all required steps completed) should be conducted in each laboratory on the basis of this practice using the supplied RMs. The separate evaluations should be one week apart. The resulting database can be analyzed in accordance with the procedures in Practice D 4483. The outcome from such testing will give a global average for ψ_A or ψ_R and a between laboratory uncertainty or confidence limit on the average values.

APPENDIXES

(Nonmandatory Information)

X1. TWO EXAMPLES OF RELATIVE TEST SENSITIVITY EVALUATIONS

X1.1 Example 1: Relative Test Sensitivity: Limited—Range Processability Tests

X1.1.1 In this example, a limited range or spot check relative test sensitivity is evaluated for three different processability tests. The use of more than two tests illustrates the general procedure for multiple comparisons for relative sensitivity. The data and calculations are given in Table X1.1. The three processability tests that generate processability numbers are designated as P1, P2, and P3 and two reference materials (RM1 and RM2) are used with four replications of the processability number, (Proc number), R1 to R4 for each RM and each test. In Part 1 of the table, the average, the variance, and the standard deviation for the processability numbers are listed for each reference material and each test. For each test, the pooled (average) variance and the standard deviation obtained from the pooled variance as well as the coefficient of variation in percent are also listed.

X1.1.2 Part 2 of the table lists the calculated parameters and the relative test sensitivity or ψ_R values. For each test, the value of Δ is given, where Δ = (Average Proc Number RM2 − Average Proc number RM1), this corresponds to the Δ MP values as discussed in this practice. Also listed are the pooled standard deviation, the ratio of (Δ 1/Δ 2) which is equal to Ko (1,2), where 1 and 2 refer to P1 and P2 and the ratio (Δ 3/Δ 2) which is equal to Ko (3,2), where 3 and 2 refer to P3 and P2, the ratios S1/ S2 and S2/S3, where S1, S2, and S3 refer to the (pooled) standard deviations for P1, P2, and P3.

X1.1.3 Using these values, calculations for relative test sensitivity, give ψ_R(P1/P2), for P1 relative to P2 and ψ_R(P3/P2), for P3 relative to P2. We find that ψ_R(P1/P2) = 0.96 and ψ_R(P3/P2) = 1.26. To review all three processability tests on the same comparative scale, we assign unity to the test sensitivity value of the reference (or denominator) test, P2, since ψ_R(P2/P2) = 1.00. The last column of the Part 2 section of the table gives the comparative value, Compψ_R of P1 and P3 versus P2. Processability Tests P1 and P2 are very similar at 0.96 and 1.00; only a 4 % difference between them while P3 is the most sensitive test; 26 % more sensitive than P2.

X1.1.4 The coefficient of variation values for P1, P2, and P3 (that is 2.06, 1.37, and 1.30) can be used to determine an index of technical merit for the three processability tests based on precision alone. For this, technical merit is assumed to be proportional to the reciprocal of the coefficient of variation, that is, the higher the coefficient the less the technical merit. The reciprocal values are 0.49, 0.73, and 0.77 for P1, P2, and P3, respectively. When these three reciprocal coefficient of variation values are put on the same comparison basis as the Compψ_R values, this gives 0.67, 1.00, and 1.05 for P1/P2, P2/P2, and P3/P2. Although these precision technical merit indexes indicate P3 to be the best and P1 to be the worst processability test (assuming numerical differences to be statistically significant) which is the same qualitative ranking as the Compψ_R sensitivity values of 0.96, 1.00, and 1.26, the magnitude of the differences compared to the reference P2 test are substantially different for P1 and P3. This demonstrates the inability of the coefficient of variation (and standard deviation and variance as well) to give a useful quantitative indication of test sensitivity for this particular test. Refer to 4.3.

X1.1.5 As discussed in this practice, these ψ_R values are estimated values that are subject to sampling and inherent test error variation. The difference in ψ_R between 0.96 and 1.00 for P1 and P2 is probably within expected test error and these two processability tests are probably not statistically significant in regard to test sensitivity. The difference of 0.26 between P3 and P2 is probably statistically significant. For definitive statements on statistical significance for ψ_R values, sufficient repeat evaluations of the entire ψ_R evaluation process on a several day basis are required to generate a number of estimates (four or more) to be able to make decisions based on the usual statistical procedures for differences in mean values.

TABLE X1.1 Evaluation of Relative Test Sensitivity (RTS)—Limited Range or Spot Check—3 Processability Tests

Part 1 Baisc Processability Number Data for RM1 and RM2

	Processability Test P1							Processability Test P2						
RM or Polymer	R1	R2	R3	R4	Average	Variance	Standard Deviation	R1	R2	R3	R4	Average	Variance	Standard Deviation
RM1	4.50	4.65	4.60	4.70	4.61	0.00547	0.074	9.10	8.90	9.30	9.00	9.08	0.02188	0.148
RM2	3.10	3.00	2.90	3.10	3.03	0.00687	0.083	12.10	12.30	11.90	12.10	12.10	0.02000	0.141
				Δ	−1.59						Δ	3.03		
				Avg	3.82						Ave	10.59		
			Pooled Value=			0.00617	0.079			Pooled Value =			0.02094	0.145
			Coefficient of Variation, % =		2.06					Coefficient Variance % =		1.37		
			S1 = 0.079							S2 = 0.145				

	Processability Test P3						
RM or Polymer	R1	R2	R3	R4	Average	Variance	Standard Deviation
RM1	10.10	10.30	9.90	9.80	10.03	0.03688	0.192
RM2	14.20	14.10	14.30	14.00	14.15	0.01250	0.112
				Δ	4.13		
			Avg.		12.09		
			Pooled Value =			0.02469	0.157
			Coefficient of Variation, % =		1.30		
			S3 = 0.157				

S1 = Standard Deviation for P1
S2 = Standard Deviation for P2
S3 = Standard Deviation for P3

Ko (1,2) = (Δ1 / Δ2)
Ko (3,2) = (Δ3 / Δ2)

Δ = (RM2 Average Proc Number - RM1 Average Proc Number) as obtained for each processability test

Part 2 Calculated Parameters and ψ_R Values

Test	Δ	Pooled Standard Deviation	Ko (1,2)	Ko (3,2)	S1/S2	S2/S3	Comp ψ_R
P1	−1.59	0.079	−0.525		0.545		0.96
P2	3.03	0.145					1.00
P3	4.13	0.157		1.363		1.083	1.26

Comp ψR = Value for P1 and P3 on Basis of ψ_R value for P2 =1.00

Calculation of ψ_R Values:

ψ_R(P1/P2) = Ko (1,2) / (S1 / S2) = | (−1.59 / 3.03) | (0.079 / 0.145) = 0.96

ψ_R(P3/P2) = Ko (3,2) / (S3 /S2) = | (4.13 / 3.03) | / (0.157 /0.145) = 1.26

X1.2 Example 2: Relative Test Sensitivity: Extended Range—Compliance vs Modulus

X1.2.1 In this example, the relative test sensitivity is assessed for two different physical properties for evaluating characteristics such as the degree of cure for rubbers. Property or Test 1 is a compliance test , that is, the deformation of strain of a test specimen under a fixed or constant force and property or Test 2 is a modulus test, the stress under a fixed (constant) extension or strain. The magnitude of ψ_R is to be evaluated over a range of values for each test; high compliance corresponds to low modulus and vice versa. Table X1.2 contains the data as generated by the evaluation program. Six RMs or rubbers designated as A to F were used with a range of compliance and modulus values, in deformation and stress units characteristic of the two tests. For each test and each RM, four replicates were evaluated, R1 to R4. Part 1 of Table X1.2 gives values for these measurements, as well as the average and standard deviation for each RM for both properties.

X1.2.2 Plot (a) of Fig. X1.1 illustrates the curvilinear inverse relationship for compliance versus modulus. For each RM, all four replicates have been shown in the plot. Since a linear relationship is required to simplify the ψ_R evaluation, a transformation of the compliance and modulus is needed. Part 2 of the table shows the data obtained by a log transformation of both properties; the average, variance, standard deviation, and coefficient of variation of the log transformations are listed. Plot (*b*) of Fig. X1.1 shows that a reasonably good linear relationship is obtained for log compliance versus log modulus. A transformation of the original data values for compliance and modulus can be made without concern about the potential influence on the relative sensitivity since it is shown in Appendix X2 that such a transformation does not alter the relative sensitivity.

X1.2.3 Part 3 of Table X1.2 illustrates the results of a sorting operation on the transformed data of Part 2; a sorting low to high, on the value of RM average for log compliance and log modulus. This indicates that variance, standard deviation, and coefficient of variation (among the four replicates for each RM) increases as compliance increases and conversely the variation decreases as modulus increases. This dependence of variation on RM average value suggested the log transformation as discussed in X1.2.2. This behavior will be subsequently discussed concerning the nonconsistancy of ψ_R for this measurement system over the range of RM values.

X1.2.4 Table X1.3 lists the log data values for all four replicates for each of the six RMs in a tabular format that is convenient for linear regression analysis. This evaluation (as in most other such evaluations) does not obey the ′zero error in x’ regression assumption since both physical properties are subject to test error. The Appendix X1 recommended procedure to address this is to select for the *x* variable, the measured property that has the smaller pooled variance across all the RMs used in the program.

X1.2.5 A review of the pooled variance in Part 2 of Table X1.2 shows that the pooled variance for log compliance is 0.0000791 and for log modulus is 0.0000253 or a compliance to modulus variance ratio of 3,13. Thus modulus should be the *x* variable; it had been initially selected for the plots of Fig. X1.1. Although not strictly required, part 1 of Table X1.3 is presented to illustrate the dual regression calculation procedure as discussed in Annex A1. It gives the results of two sets of linear regression calculations; No. 1 for log compliance versus log modulus (as *y* and *x*) and No 2 for log modulus versus log compliance (as *y* and *x*). The regression *X* coefficients or slopes indicated by b (C/M) for calculation No 1 and b (M/C) for No 2 are given in the tabular output where C = compliance and M = modulus.

X1.2.6 The value for b (C/M) is −1.844, and for the inverse regression, b (M/C) is −0.540. Since the two b coefficients stand in an inverse relationship to each other, the reciprocal of b (M/C) gives a second estimate of b (C/M) and 1/−0.540 = −1.853. The difference between −1.844 and −1.853 represents the influence (on the slope estimate) of *x* variation being generated by modulus values versus *x* variation generated by compliance. Both estimates are reasonably close since the variance ratio is not very large compared to unity. The better b coefficient estimate is –1.844 and this is used. The slope or the b coefficient is equal to d[MP1]/d[MP2] (or Δ[MP1]/Δ[MP2]) as discussed in this practice and therefore is equal to Ko.

X1.2.7 Part 2 of Table X1.3 illustrates the evaluation of the (S_{MP1}/S_{MP2}) ratio as previously discussed in this practice; this ratio is indicated in Part 2 as (S1/S2), where S1 is for log compliance and S2 is for log modulus. The Part 2 tabulation also lists the averages and standard deviations for log compliance and log modulus and the S1 to S2 ratio for each RM. Fig. X1.2 plot (a) shows the standard deviation for log compliance versus log compliance and in plot (b) the standard deviation of log modulus vs log modulus. These of course show the opposite slopes as expected. Fig. X1.3 shows that the (S1/S2) ratio also is a linear function of either log compliance or log modulus, again with opposite slopes. To the right of the Table X1.3, Part 2 tabulation is the output of the linear regression analysis for (S1/S2) = ao + a_1(log modulus). The slope a_1, designated as “X coefficient” in the printout is −1.89 and ao, designated as the constant, is 2.76.

X1.2.8 We now turn to evaluating the relative test sensitivity, ψ_R(T1/T2), where T1 = compliance and T2 = modulus or ψ_R (C/M). This is equal to | Ko | /(S1/S2). We have seen that the ratio (S1/S2) changes with the level of either compliance or modulus, and therefore, ψ_R(C/M) does not have a fixed or constant value. As previously indicated, it is a function of log modulus and is given as follows:

$$\psi_R\,(\mathrm{C/M}) = |\,\mathrm{Ko}\,| / [2.76 - 1.89\,(\log \mathrm{modulus})] \qquad \text{(X1.1)}$$

The enclosed tabulation in Part 3 of Table X1.3 lists values for ψ_R(C/M) at selected log modulus values that span the experimental range for this program. At the lowest log modulus of 0.40 (high compliance), ψ_R(C/M) is 0.92 and the modulus test is slightly more sensitive than the compliance test. As modulus increases, ψ_R(C/M) increases above unity and the compliance test becomes more sensitive than the modulus test. At the highest log modulus level of 0.80 the compliance test has a 47 % margin in test sensitivity compared to the modulus test.

X1.2.9 Again, as in the case of Example 1, for the spot check relative test sensitivity, any extended range tabulated ψ_R(C/M) value in Part 3 is an estimate from one replication of

TABLE X1.2 Relative Test Sensitivity—Extended Range—Compliance Versus Modulus—Initial Data Review

Part 1 Data as Obtained from Evaluation Program

RM or rubber	Compliance				Compliance Average	Compliance Standard Deviation	Modulus				Mod Average	Mod Standard Deviation
	R1	R2	R3	R4			R1	R2	R3	R4		
A	8.30	8.25	8.15	8.30	8.25	0.0707	5.45	5.52	5.52	5.53	5.51	0.0370
B	28.55	27.55	29.50	28.15	28.44	0.8189	2.74	2.82	2.72	2.77	2.76	0.0435
C	11.25	11.02	11.35	11.25	11.22	0.1399	4.56	4.64	4.61	4.63	4.61	0.0356
D	14.15	13.95	13.71	13.95	13.94	0.1800	4.12	4.18	4.20	4.17	4.17	0.0340
E	24.65	23,60	24.83	25.10	24.55	0.6566	3.09	3.14	3.06	3.04	3.08	0.0435
F	20.25	19.40	19.27	20.00	19.73	0.4704	3.49	3.55	3.59	3.49	3.53	0.0490
Averaged or Pooled	17.86	17.30	17.80	17.79	17.69		3.91	3.98	3.95	3.94	3.94	

R1, R2, and so forth = Replicates 1, 2, and so forth.

Part 2 Log Data Value Transformation

RM or Rubber	Log Compliance				Average	Variance	Standard Deviation	Coefficient of Variation, %
	R1	R2	R3	R4				
A	0.919	0.916	0.911	0.919	0.916	0.0000139	0.00373	0.41
B	1.456	1.440	1.470	1.449	1.454	0.0001552	0.01246	0.86
C	1.051	1.042	1.055	1.051	1.050	0.0000294	0.00543	0.52
D	1.151	1.145	1.137	1.145	1.144	0.0000315	0.00561	0.49
E	1.392	1.373	1.395	1.400	1.390	0.0001379	0.01174	0.85
F	1.306	1.288	1.285	1.301	1.295	0.0001062	0.01031	0.80
Averaged or Pooled	1.212	1.201	1.209	1.211	1.208	0.0000791	0.00821	0.65

RM or rubber	Log Modulus				Average	Variance	Standard Deviation	Coefficent of Variation, %
	R1	R2	R3	R4				
A	0.736	0.742	0.742	0.743	0.741	0.0000086	0.00293	0.39
B	0.438	0.450	0.435	0.442	0.441	0.0000465	0.00682	1.54
C	0.659	0.667	0.664	0.666	0.664	0.0000113	0.00336	0.51
D	0.615	0.621	0.623	0.620	0.620	0.0000126	0.00356	0.57
E	0.490	0.497	0.486	0.483	0.489	0.0000368	0.00607	1.24
F	0.543	0.550	0.555	0.543	0.548	0.0000362	0.00602	1.10
Average or Pooled	0.580	0.588	0.584	0.583	0.584	0.0000253	0.00479	0.89

Part 3 Log Data Value Transformation—Sorted Values

Log Compliance - Sorted on Average Log Compliance

RM or Rubber	R1	R2	R3	R4	Average	Variance	Standard Variance	Coeficient of Variation, %
A	0.919	0.916	0.911	0.919	0.916	0.0000139	0.00373	0.41
C	1.051	1.042	1.055	1.051	1.050	0.0000294	0.00543	0.52
D	1.151	1.145	1.137	1.145	1.144	0.0000315	0.00561	0.49
F	1.306	1.288	1.285	1.301	1.295	0.0001062	0.01031	0.80
E	1.392	1.373	1.395	1.400	1.390	0.0001379	0.01174	0.85
B	1.456	1.440	1.470	1.449	1.454	0.0001552	0.01246	0.86
Average or Pooled	1.212	1.201	1.209	1.211	1.208	0.0000791	0.00821	0.65

Log Modulus - Sorted on Average Log Modulus

RM or Rubber	R!	R2	R3	R4	Average	Variance	Standard Deviation	Coefficient of Variation, %
B	0.438	0.450	0.435	0.442	0.441	0.0000465	0.00682	1.54
E	0.490	0.497	0.486	0.483	0.489	0.0000368	0.00607	1.24
F	0.543	0.550	0.555	0.543	0.548	0.0000362	0.00602	1.10
D	0.615	0.621	0.623	0.620	0.620	0.0000126	0.00356	0.57
C	0.659	0.667	0.664	0.666	0.664	0.0000113	0.00336	0.51
A	0.736	0.742	0.742	0.743	0.741	0.0000086	0.00293	0.39
Average or Pooled	0.580	0.588	0.584	0.583	0.584	0.0000253	0.00479	0.89

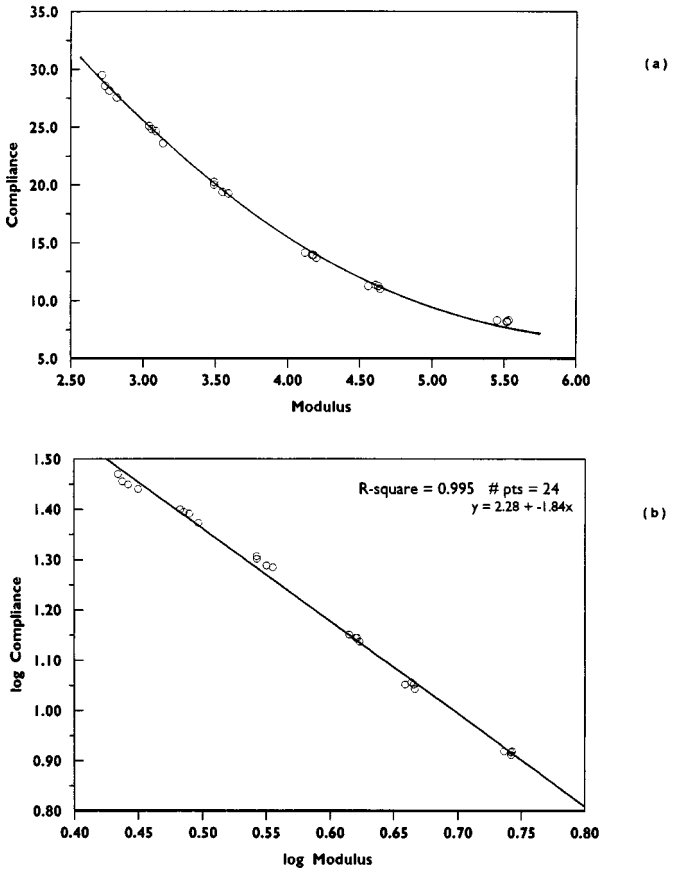

FIG. X1.1 (a) Compliance Versus Modulus, (B) Log Compliance Versus Modulus

the entire test sensitivity evaluation, that is, the use of six RMs and four test measurement replicates for each reference material. To be able to assign uncertainty or confidence limits on ψ_R(C/M), it would be necessary to repeat the entire relative test sensitivity program a sufficient number of times to be able to calculate reliable standard deviations for ψ_R(C/M) values at selected modulus or compliance levels.

TABLE X1.3 Relative Test Sensitivity (RTS)—Extended Range—Compliance Versus Modulus

Part 1 - Evaluating Ko

RM or Rubber	Log Modulus	Log Compliance
A	0.736	0.919
	0.742	0.916
	0.742	0.911
	0.743	0.919
B	0.438	1.456
	0.450	1.440
	0.435	1.470
	0.442	1.449
C	0.659	1.051
	0.667	1.042
	0.664	1.055
	0.666	1.051
D	0.615	1.151
	0.621	1.145
	0.623	1.137
	0.620	1.145
E	0.490	1.392
	0.497	1.373
	0.486	1.395
	0.483	1.400
F	0.543	1.306
	0.550	1.288
	0.555	1.285
	0.543	1.301
Pooled 'Within RM' Variance	0.0000253	0.0000791
Standard Deviation	0.00503	0.00889

Variance Ratio, max/min = 3.13

Linear Regression Calculations

Calculation No. 1 Y = log Compliance; X = log Modulus

Regression Output:

Constant	2.28	
Standard Error of Y, Estimate	0.0133	
R Squared	0.995	
Number of Observations	24	
Degress of Freedom	22	
X Coefficient = b(C/M) =		−1.844
Standard Error of Coefficient	0.026	

Calculation Number 2 Y = log Modulus; X = log Compliance

Regression Output:

Constant	1.236	
Standard Error of Y, Estimate	0.0072	
R Squared	0.995	
Number of Observations	24	
Degrees of Freedom	22	
X Coefficient = b(M/C) =		−0.540
Standard Error of Coefficient	0.0077	
Reciprocal b(M/C) = 1 / −0.540 =		−1.853

Therefore Ko = −1.844 = 1.84 (absolute)

Part 2 —Evaluating Functionality of Standard Deviation Ratio (S1/S2)

RM or Rubber	log Compliance Average	Log Compliance Standard Deviation	log Modulus Average	log Modulus Standard Deviation	Ratio: S1/S2
A	0.916	0.00373	0.741	0.00293	1.28
B	1.454	0.01246	0.441	0.00682	1.83
C	1.050	0.00543	0.664	0.00336	1.61
D	1.144	0.00561	0.620	0.00356	1.58
E	1.390	0.01174	0.489	0.00607	1.94
F	1.295	0.01031	0.548	0.00602	1.71

S1 = Standard Deviation (log Compliance)
S2 = Standard Deviation (log Modulus)

Defining Equation:
Ratio (S1/S2) = 2.76 −1.89 (log Mod)

Regression Output:
Y = Ratio S1/S2; X = log Mod

Constant	2.76	
Standard Error of Y, Estimate	0.0980	
R Squared	0.854	
Number of Observance	6	
Degrees of Freedom	4	
X Coefficient		−1.89
Standard Error of Coefficient		0.391

Part 3 - Evaluating Relative Test Sensitivity, Compliance / Modulus

ψ (C/M) = | Ko | / (S1/S2) = 1.84 / [2.76 − 1.89 (log Modulus)]

Tabulated Values

Log Mululus	ψ (C/M)
0.40	0.92
0.50	1.01
0.69	1.13
0.70	1.28
0.80	1.47

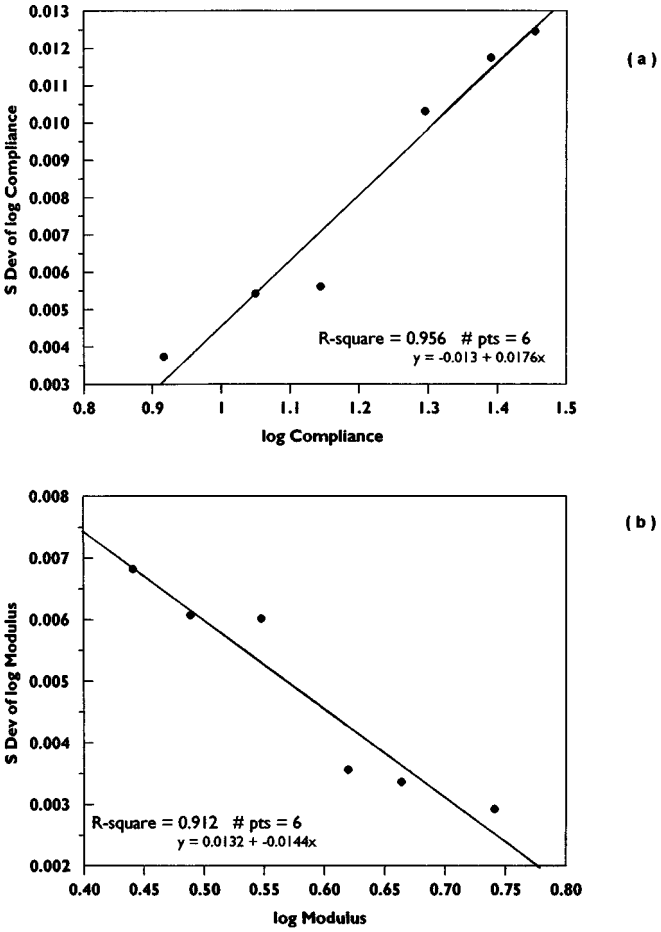

FIG. X1.2 (a) Standard Deviation Log Compliance Versus Log Compliance, (b) Standard Deviation Log Modulus Versus Log Modulus

X2. BACKGROUND ON: TRANSFORMATION OF SCALE AND DERIVATION OF ABSOLUTE TEST SENSITIVITY FOR A SIMPLE ANALYTICAL TEST

X2.1 Transformation of Scale

X2.1.1 The operation of transforming the scale for MP values is important since it may be required to produce a linear relationship for transformed MP1 versus MP2 or transformed MP2. A linear relationship simplifies the evaluation of test sensitivity. This appendix demonstrates that relative test sensitivity, ψ_R, is not changed by a transformation of scale. If two test methods have different relationships given by Eq X2.1:

$$MP1 = f_1(FP);\ MP2 = f_2(FP) \tag{X2.1}$$

then ψ_R is given by Eq X2.2 or (Eq 5 in 7.5.2).

$$\psi_R = [\Delta(MP_1)/\Delta(MP_2)]/[S_{MP1}/S_{MP2}] = |\,Ko\,|\,/[S_{MP1}/S_{MP2}] \tag{X2.2}$$

For the next step, this can be expressed in more formal terms by using differentials rather than the deltas as follows:

$$\psi_R\,(T1/T2) = [d\,(MP_1)/d\,(MP_2)]\,/\,[S_{MP1}\,/\,S_{MP2}] = |Ko|\,/\,[S_{MP1}\,/\,S_{MP2}] \tag{X2.3}$$

The (T1/T2) notation is now included to avoid confusion in contrasting MP2 versus MP3 behavior as described as follows:

X2.1.2 If MP1 needs to be transformed, indicate this functionality as given by Eq X2.4:

$$MP3 = f_3(MP1) \tag{X2.4}$$

Then by definition, the relative test sensitivity of MP3 to MP2, is given as follows:

$$\psi_R\,(T3/T2) = [d\,(MP3)/d\,(MP2)]\,/\,[S_{MP3}\,/\,S_{MP2}] = |Ko|\,/\,[S_{MP3}\,/\,S_{MP2}] \tag{X2.5}$$

Since MP1 is functionality related to MP2, then MP3 is also related to MP2 and by differentiating Eq X2.4 with respect to MP2 the following equation is obtained:

$$d[MP3]/d[MP2] = d[f_3(MP1)]\,/\,d[MP2] \tag{X2.6}$$

Continuing the development, the right-hand side of X2.6 can be expressed as follows:

$$d[\,f_3(MP1)]\,/\,d[MP2] = d[f_3(MP1)]/d[MP1]\,[d(MP1)]\,/\,d[MP2] \tag{X2.7}$$

From the law of propagation of errors, the relationship between the test error of MP3 and MP1 is given as

$$S_{MP3} = |d[\,f_3(MP1)]\,/\,d[MP1]\,|S_{MP1} \tag{X2.8}$$

Introducing Eq X2.7 and Eq X2.8 into Eq X2.5 and using

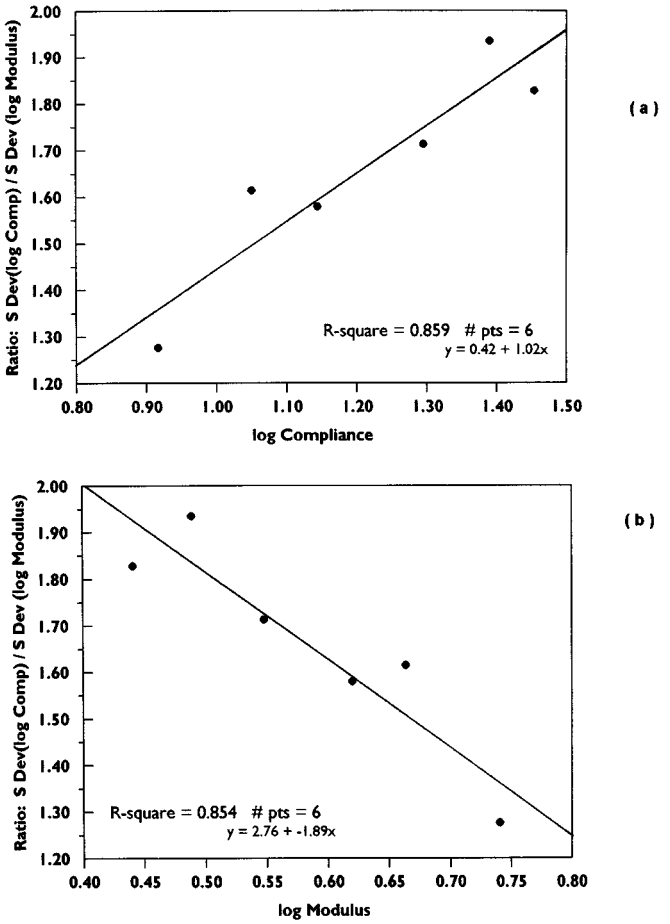

FIG. X1.3 (a) Ratio: Standard Deviation (Log Comp) / Standard Deviation (Log Modulus) Versus Log Compliance (b) Ratio: Standard Deviation (Log Comp) / Standard Deviation (Log Modulus) Versus Log Modulus

absolute values as indicated we obtain ψ_R (T3/T2) as defined by the ratio shown in Eq X2.9:

$$\psi_R\,(T3/T2) = \frac{|\, d[f_3(MP1)] / d[MP1]\,[d[MP1)] / d[MP2]\,|}{|\, d f_3(MP1)] / d[MP1]\,(S_{MP1} / S_{MP2})|} \quad (X2.9)$$

which simplifies to

$$\psi_R\,(T3/T2) = |[d[MP1)] / d[MP2\,]\,|/(\,S_{MP1} / S_{MP2}\,) \quad (X2.10)$$

The expression (ratio) as given in Eq X2.10 is the same as the ratio for the initial expression for the (T1/T2) comparison as given in Eq X2.3. The transformation of scale for MP1 (into MP3) has not changed the value of its relative sensitivity with respect to MP2. By the same reasoning, any transformation of MP2 will yield behavior equivalent to that found for MP1. Therefore, relative test sensitivity, ψ_R, is invariant with respect to scale transformation of either test method MP in the comparison.

X2.1.3 Transforming the MPs is usually a trial-and-error process. Typical transformations to produce linearity for the MP1 versus MP2 relationship are the use of the logarithm of either MP1 or MP2, or both, as well as the square root of either or both. Transformations also tend to reduce the perturbing influence of any non-normality in the underlying distributions for the MPs as well as frequently stabilizing or equalizing the variance across the range of values for the RMs.

X2.2 Deriving Absolute Sensitivity for a Simple Analytical Test

X2.2.1 In measurement techniques such as those employed in analytical chemistry, a material or constituent is determined by evaluating some quantity that hears a direct proportionality to the constituent. As an example in the classic analysis for total sulfur in rubber, the sulfur after appropriate chemical reactions is determined by the amount of precipitated barium sulfate. The development as given in X2.2 is devoted to a simple chemical test for the analysis or determination of one constituent. Under some circumstances, the procedure may also be applicable to simply physical tests.

X2.2.2 Constituent A, a chemical element in some matrix of materials, is to be determined by reacting A (in a solution of the matrix material) with a reagent to generate a carrier compound, C, where the proportion of A in C is fixed by the stoichiometry of the chemical reaction. This carrier compound is determined by weighing after separating it from the solution. The percentage of A in the matrix of materials (sample) is designated as A, %, and given as follows:

$$A, \% = [100\, W_C/W_S]\,[\{A\}/\{C\}\,] \quad (X2.11)$$

where:

W_C = mass of C as measured by the test,
W_S = mass of (matrix) sample used in the test,
{A} = equivalent mass of A, in chemical reaction to produce C, and
{C} = equivalent mass of C.

Eq X2.11 can be rewritten as follows:

$$A, \% = 100\, R_M\, R_{EM} \quad (X2.12)$$

where:

R_M = W_C/ W_S, ratio of the measured mass of C to the mass of the sample, and
R_{EM} = [{A}/{C}], ratio of the equivalent mass of A to the equivalent mass of C.

Based on Eq X2.12, the standard deviation for A, %, can be expressed as follows:

$$S(A, \%) = 100\, R_{EM}\, S\,(R_M) \quad (X2.13)$$

where:

S (A, %) = standard deviation in determining A, %, and
S (R_M) = standard deviation for measurement of R_M or (W_C/W_S).

X2.2.3 Eq X2.13 indicates that the precision for measuring A, %, is improved when R_{EM} is small and the standard deviation for the measurement of R_M (mass ratio carrier compound C to sample) is small. If Eq X2.12 is rearranged to give R_M in terms of A, %, the following is obtained:

$$R_M\,[1/100\, R_{EM}]\, A, \% \quad (X2.14)$$

Eq X2.14 shows that a plot of R_M (*y* axis) versus A(%) (*x* axis) yields a straight line with zero intercept and a slope of $[1/100\, R_{EM}]$. If the slope is designated as K, the $1 / K = 100\, R_{EM}$ and Eq X2.13 can be written as

$$S\,(A(\%)) = [1/K\,]\,S\,(R_M\,) = S\,(R_M\,) / K \quad (X2.15)$$

Thus high precision of A(%) is obtained when the ratio S

(R_M) / K is small or conversely when K / S (R_M) is large. Since the sensitivity to the constituent being analyzed is greater the higher the value for K / S (R_M), this ratio is defined as the test sensitivity and is given by Eq X2.16, using absolute or numerical (sign ignored) values for K

$$\textit{Absolute Test Sensitivity} = \psi_A = |K| / S(R_M) \quad \text{(X2.16)}$$

Therefore, although the technical merit of a test method requires that the MP, in this case R_M, has a small standard deviation, it also requires a high rate of change for R_M with rate or extent of change in A or A(%), that is, it requires the ability to discriminate or readily detect small changes in A. The greater this detection capability or discrimination power, the greater is K. In this appendix, R_M is equal to MP and A is equal to FP, in relation to the terminology used for the main text of this practice.