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

This standard is issued under the fixed designation D6600; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers testing to evaluate chemical constituents, chemical and physical properties of compounding materials, and compounded and cured rubbers, which 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:

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

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 *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

[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.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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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.

3.2.1.1 *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.

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

3.2.3 *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.

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

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

[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.

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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.

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 D4483.

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*) an FP (or single criterion or constituent), the value of which is established by the use of a CM and (*2*) an 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 = Δ(MP)/Δ(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

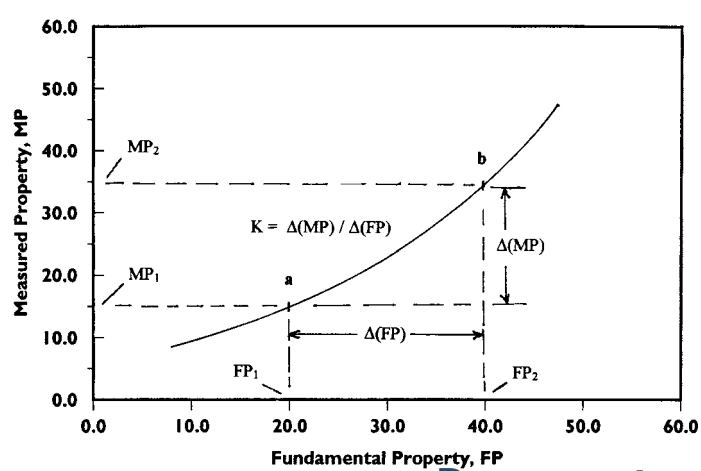

FIG. 1 Measured Versus Fundamental Property Relationship

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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.

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 an 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 is 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.

$$\psi_A\,1 = |\,K_1\,|/S_{MP1} \text{ and } \psi_A\,2 = |\,K_2\,|/S_{MP2} \qquad (2)$$

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

$$\psi_A\,1/\psi_A\,2 = |\,K_1/K_2\,|\,[S_{MP1}/S_{MP2}] = |\,Ko\,|/[S_{MP1}/S_{MP2}] \qquad (3)$$

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.

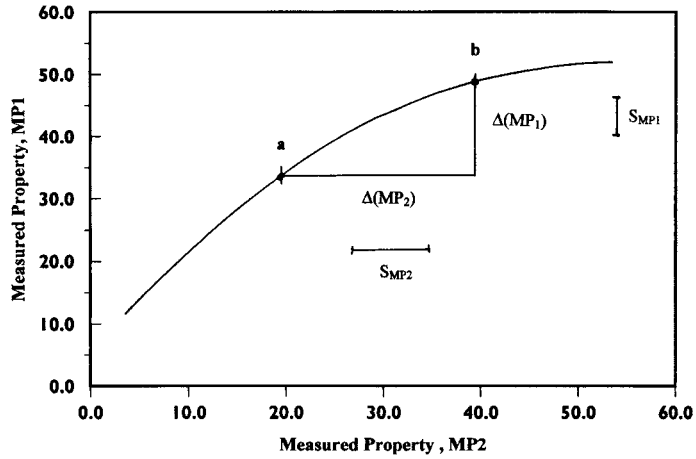

FIG. 2 Measured Property 1 Versus 2 Relationship

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:

$$Ko = |\,K_1/K_2\,| = [\Delta(MP_1)/\Delta(FP)]/[\Delta(MP_2/\Delta(FP)] = |\Delta(MP_1)/\Delta(MP_2)| \qquad (4)$$

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

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

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 the relative test

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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 assessment over a substantial part or all of the entire working range of MP versus 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

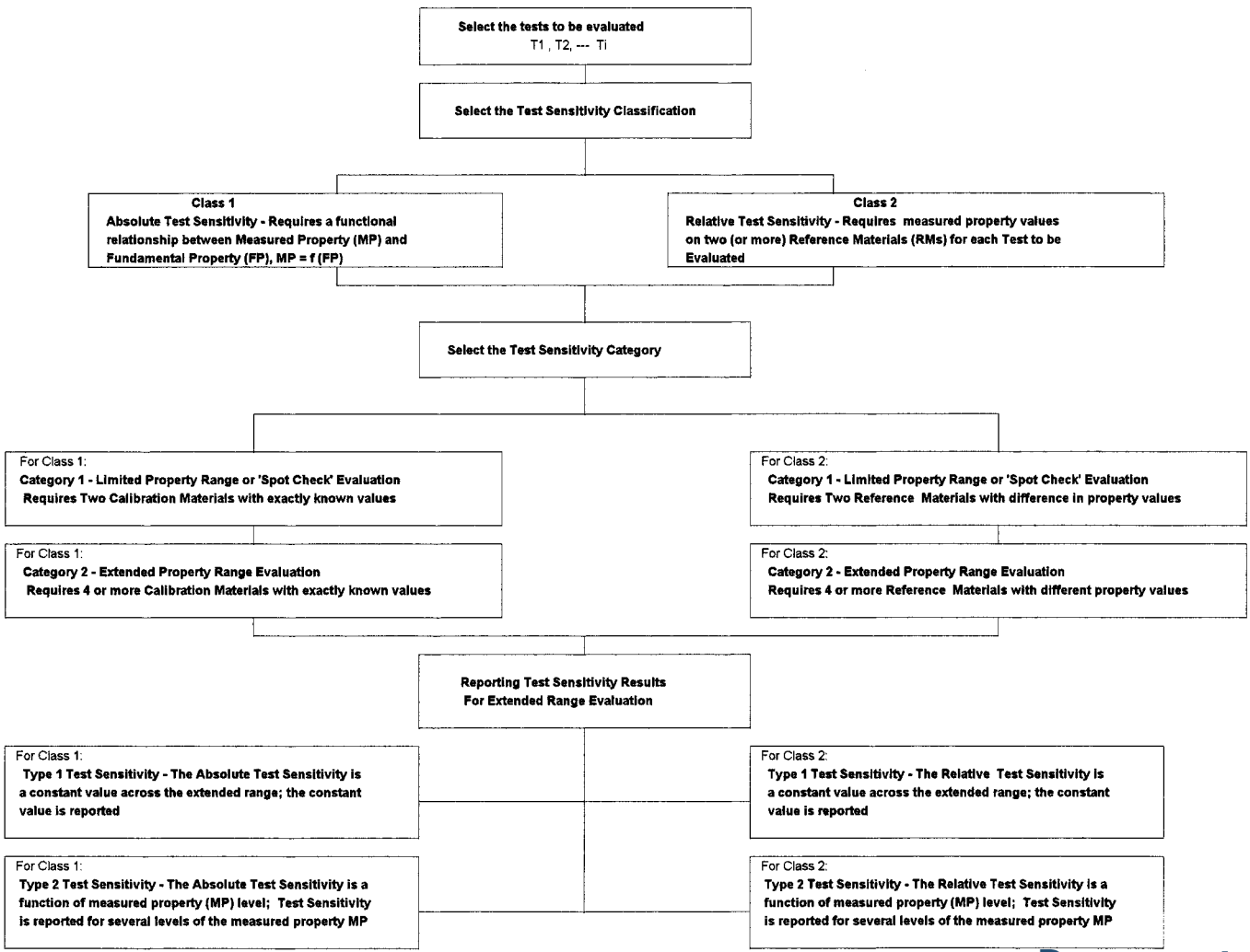

FIG. 3 Outline of the Steps in a Test Sensitivity Evaluation Program for a Local Test Domain

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