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An American National Standard
Technical Association of the Pulp
and Paper Industry
Standard Method T 1200 os

Standard Practice for Interlaboratory Evaluation of Test Methods Used with Paper and Paper Products[1]

This standard is issued under the fixed designation D 1749; 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 The purpose of an interlaboratory study is to determine the variability in results caused by differences among laboratories following a prescribed test method, the consistency from material to material of this variability, and the type of additional standardization needed, if any. The study may be made to obtain information for improving the test method or to arrive at an estimate of the precision of an existing method for publication. It may also include a comparison of alternative test methods.

1.2 To achieve the objectives in 1.1 satisfactorily, it is essential that a sound statistical design be employed in the planning of an interlaboratory study. This practice gives the basic principles involved in the planning in order to make the data amenable to statistical analysis and interpretation.

1.3 This practice has been written for the task group chairman responsible for the preparation or the revision of a standard test method. It tells him what information he needs in order to properly plan an interlaboratory study (Sections 1-10), it outlines the procedure for conducting the study (Section 11), and it gives him background information for understanding the analysis (Section 12) and interpretation (Sections 13-15) of the results.

1.4 While the services of a statistician are not absolutely necessary for the design, analysis, and interpretation of interlaboratory studies, questions often arise that could be readily answered by a statistician familiar with the analysis used herein. Hence, the task group chairman should arrange whenever possible to consult with a statistician both during the planning and during the analysis and interpretation.

1.5 This practice is similar to TAPPI T1200, which details the analysis using a set of typical data.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 685 Practice for Conditioning Paper and Paper Products for Testing[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 178 Practice for Dealing With Outlying Observations[3]

E 456 Terminology Relating to Quality and Statistics[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

2.2 *TAPPI Standards:*

T 402 Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products[4]

T 1200 Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility[4]

T 1205 Dealing with outlying test results[4]

OUTLINE OF RECOMMENDED PROCEDURE

3. Formulating the Problem

3.1 If the objective of the task group was not clearly spelled out when the task group was established, determine this by task group discussion, and obtain approval from the chairman of the parent committee (Annex A1.1).

3.2 Refer to Terminology E 456 for terminology.

4. Preliminary Study Within One Laboratory

4.1 Investigate the several variables of the test method, both experimentally and theoretically, including the following (see also A1.2.1-A1.2.3).

4.1.1 Survey the literature and other sources of information for possible sources of variability in the application of the test method.

4.1.2 Determine how the result of measurement is affected by variations in the critical dimensions of instruments or critical steps in the procedure.

4.1.3 Determine how the result is affected by known variations in atmospheric conditions, unknown differences between operators, and by other variables.

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[1] This practice is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 15.09.

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

[4] Available from the Technical Association of the Pulp and Paper Industry, P.O. Box 105113, Atlanta, GA 30348.

4.2 Select what appears to be the optimum procedure. A choice between two procedures may be made using the "sensitivity criterion" (Annex A2).

4.3 Using papers with a wide range of values of the property under test (and possibly also with wide ranges in other properties), make a comparative study with other methods for measuring the property (see also A1.2.4 and A2.1). Evaluate the results using the sensitivity criterion, when applicable, or an absolute standard.

4.4 Draft instructions for the test method. Then observe, without comment, a laboratory technician performing a test according to this draft. Where he has difficulty, revise the draft. *It is extremely important that the directions for performing the test be clear, unambiguous, and sufficiently comprehensive.*

5. Study by Task Group

5.1 Prepare a definitive statement of the type of information the task group expects to obtain from the interlaboratory study (see 1.1 and Section 14).

5.2 Based on the study within one laboratory, prepare a master plan for the interlaboratory study (see 11.2). Circulate the proposed plan to all members of the task group and other competent authorities (including the manufacturers of the instruments involved) for comment and criticism. Discuss the plan in an open committee meeting.

5.3 Select the materials to be used in the interlaboratory studies that cover the range of the property to be measured and that represent all classes of material to which the method will be applied (see 10.2).

6. Pilot Study

6.1 If the method to be evaluated is new or represents an extensive modification of an existing method, a pilot study involving two or three materials and four or five laboratories should expose any ambiguities or misleading directions in the procedure.

7. First Interlaboratory Study

7.1 For the first interlaboratory study use a minimum number of materials (six) to cover the range. Include all of the laboratories that will participate in the main interlaboratory study (see 10.1). This study is to train the participants in the test method, to clarify the procedure, to eliminate laboratories that cannot comply with the procedure because of nonstandard conditions or equipment (as determined by means of a questionnaire), and, together with the main study, to give an indication of any change in laboratory performance with time.

8. Main Interlaboratory Study

8.1 For the main interlaboratory study follow Section 7, only use a maximum number of materials. Include only those laboratories that participated in the first interlaboratory study and that can meet the standard requirements. Base any statements of the precision of the test method on this main study, as analyzed and interpreted in accordance with the "linear model" (see 12.3 and Section 14).

8.2 Sometimes, because of factors beyond the control of the task group, it will not be possible to run the full study described in Section 10. Useful information may still be obtained from the data if the interlaboratory study is designed in accordance with Section 11 and complies with Section 10 to the greatest extent practicable.

9. Decision on Standardization

9.1 This will be based on the results of the main interlaboratory study (as discussed in Section 14). The decision could be (*1*) abandon the test method, (*2*) use the test method as it is, (*3*) rewrite the procedure to eliminate some of the variability, or (*4*) provide one or two standards for "calibration" of the test method.

9.2 It should be realized that if, as a result of the study, any step in the procedure is changed significantly, the degree of concordance of previously obtained data becomes questionable, possibly to a degree that a further study will be required.

INTERLABORATORY STUDIES (1,2)[5]

10. Independent Variables

10.1 The selection of the various levels at which each independent variable is taken shall, whenever possible, be based on preliminary work or previous experience. The main independent variables to be considered in an interlaboratory study include the following:

10.1.1 *Laboratories*—The participating laboratories shall have skilled personnel and adequate equipment for carrying out the tests. If necessary, skill in the particular methods involved in the interlaboratory study shall be acquired by preliminary experimentation (Section 7). Obtain assurance from each participating laboratory that it is properly equipped to follow all the details of the procedure and is willing to assign the work to skilled personnel.

10.1.2 *Number of Participants*—Use as many laboratories as practicable, preferably 20 to 30, subject to the amount of work involved in preparing samples for distribution to the participating laboratories, and the increase in sampling variability due to the larger amount of material required. If fewer than ten laboratories are prepared to use the test method, include all the laboratories that will do so. In the latter case, in order to increase the number of participants, it may be desirable to have two operators in each laboratory. It should be noted, however, that the "operator effect" (that is, difference between operators in the same laboratory) can vary sharply from laboratory to laboratory, depending on the degree of supervision and control maintained within a laboratory, and is therefore usually not a suitable variable for investigation in an interlaboratory study to evaluate a test method.

NOTE 1—The data required from each laboratory are held to a minimum in this practice. This should stimulate the participation of an adequate number of laboratories to provide valid measures of precision. In view of the referee nature of TAPPI and ASTM methods, the importance of assessing their reproducibility between laboratories cannot be overemphasized. This assessment requires the participation of as many laboratories as possible.

NOTE 2—If it is necessary to have two operators in each of one or more laboratories, the two operators must evaluate the method independently in

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

the fullest sense of the word, interpreted as using different samples, different reagents, different apparatus where possible, and performing the work on different calendar days.

10.2 *Materials*—The evaluation of a test method shall be made over the entire range of values of the measured property and for a reasonably representative group of the materials of the type(s) to which the test applies. If possible, select the materials to give results that fall at approximately equal intervals in the applicable range of the test method.

10.2.1 *Number of Materials*—Use as many materials as practicable, consistent with economic considerations of time of preparation and testing, if possible 20 or more materials, but a minimum of six materials for a single-scale instrument, and a minimum of five materials per scale for a multiple-scale instrument, the five or six materials covering the useful range of the scale at approximately equal intervals. The more widely different the types of materials to be included in the study, the more materials per scale will be required.

10.2.2 *Sampling of Materials*—Sample each material so that the variability among the specimens of that material will be minimized. To do this, take all of the specimens from a small area of a single roll, avoiding the edges of the roll. Usually specimens taken adjacent to each other in the machine direction are more nearly alike than those adjacent in the cross direction. As a further refinement for physical tests, each sheet or specimen may be weighed individually to check that the weight is within tolerable limits and to exclude any that exceed these limits. The study of sampling variability, such as machine and cross-direction variabilities within a sheet, is not a proper part of an interlaboratory study for the evaluation of a test method, but should be done in a single laboratory preliminary to the selection of materials for inclusion in the study.

10.2.3 *Aging of Samples*—If the samples are of such a nature that their properties may change noticeably in the course of days or a few weeks, coordinate the timing of the tests among the participating laboratories, so that the effect of aging is not confounded with the differences among laboratories.

10.2.4 *Conditioning of Samples*—Especially for physical tests, preconditioning (see TAPPI Standard T 402/Practice D 685) of the samples at low relative humidity prior to conditioning and testing at 50 % relative humidity will avoid confounding hysteresis effects with the differences among laboratories. Whether preconditioning and conditioning should be left to each participating laboratory or done at the laboratory where the samples are selected, depends on whether the objective of the interlaboratory study is to obtain information for improving the test method or to arrive at an estimate of the precision of the test method as applied.

10.3 *Order of Testing*—The order and timing of replicate determinations shall be designed to simulate the anticipated testing procedure. Thus, while in many situations variability among replicate determinations is greater when measurements are made at different times than when they are made as part of a group, nevertheless, if the normal testing procedure is to group replicate measurements, they should likewise be grouped in the interlaboratory study. Between group variability, as for example, day-to-day variability, may depend markedly on the degree of control over the testing environment and the amount of supervision received. On the other hand, within-group replications error has been found to be consistent among laboratories using the same type of test equipment and degree of skill in operating it.

10.3.1 *Number of Replicate Tests*—The number of specimens that shall be tested by each laboratory for each material will normally be two or three for a chemical test and three or four for a physical test. The number may be as small as two when there is little danger that specimens or results will be lost or questionable test values will be obtained, or as many as ten when test values are apt to vary among replicates or the number of laboratories or materials is insufficient (see 10.3.2).

10.3.2 *Total Extent of An Interlaboratory Study*—For the optimum yield of information comparable with the amount of work involved, the following conditions should hold (subject to the considerations of the above sections):

$$npq = 720 \text{ approximately}$$

$$np = 30 \text{ or more for each material}$$

$$n = 2 \text{ or more}$$

where:

n = number of replicate measurements per material per laboratory,
p = number of laboratories, and
q = number of materials.

Thus if only 10 laboratories participate, it is suggested that each be asked to make at least 4 replicate determinations on each of 18 materials. If 30 laboratories participate only 2 replicates per material would be required by the second condition above, and then by the first condition only 12 materials would be needed. Where a choice exists between the number of replicates and the number of materials, it is best to minimize the number of replicates.

11. Design

11.1 *Basic Design*—It is advisable to keep the design as simple as possible in order to obtain estimates of within- and between-laboratory variability that are unconfounded by secondary effects. The basic design is represented by a two-way classification table in which the rows represent the laboratories, the columns materials, and each cell (that is, the intersection of a row with a column) contains the replicate determinations made by a particular laboratory (the row) on a particular material (the column) (see Practice E 691).

11.2 *Master Plan*—Use the plan agreed upon between the task group after careful discussion (see 5.2). This plan should include detailed instructions for:

11.2.1 The care of test specimens, including prominent instructions for preconditioning and conditioning when required (see 10.2.4).

11.2.2 The adjustment and calibration of the test apparatus.

11.2.3 The order of testing the specimens.

11.2.4 The performance of the test.

11.2.5 The recording of results on the report form (see 11.4), including number of decimal places to be used which, when possible, should be one more than is required to be reported by the test method under study.

11.2.6 The completion of a check list covering the requirements for each critical part of the apparatus or step in the test method (see 11.4).

11.3 *Allocation of Specimens*—Allocate and distribute the specimens from a single place. Prepare from each sample of material enough specimens to provide the required test material for all participating laboratories and a sufficient number of additional specimens for replacement of any lost or spoiled specimens. Label each specimen by means of a code symbol and record the identification of the specimens for future reference. Completely randomize the specimens of a particular test material before dividing them into the required number of groups for assignment to the laboratories.

11.3.1 The complete randomization of specimens, as specified above, ensures that between-specimen variability is the same within laboratories as between laboratories, and greatly simplifies the statistical analysis and its interpretation. When each laboratory is given a single sheet for each material from which it is expected to obtain all specimens, between-laboratory variability is confounded with between-sheet variability. On the other hand, when blocks of adjacent specimens (which presumably are more alike) are assigned one from each block to each laboratory, the within-laboratory replication error is confounded with block-to-block variability and cannot be compared with the between-laboratory variability, which does not include the block-to-block variability.

11.4 *Report Form and Questionnaire*—Supply each participating laboratory with a form for reporting the data (Annex A3) to ensure that all pertinent data and information are reported in a uniform manner by all participants. In addition to providing space for reporting the measurements and the usual information, such as relative humidity, temperature, and type of instrument, include with the report a check list and questionnaire, the answers to which will tell whether the general instructions and the several critical steps in the procedure have been followed correctly.

12. Analysis of Data

12.1 *Responsibility for Analysis*—The person or persons who provide the detailed design for the interlaboratory test shall also assume responsibility for the analysis of the data, either by carrying out the analysis, or by preparing written instructions for guidance in the statistical treatment of the data.

12.2 *Method of Analysis*—If the study covers several physical or chemical tests, or both, first analyze the data for each test separately. Carry out the analysis for each test in two steps: (*1*) analysis of within-cell replications, and (*2*) analysis of cell averages. Use the procedure for these analyses explained in detail in Practice E 691.

12.3 *Model for the Analysis*:

12.3.1 The analysis of the data is made in accordance with the“ linear model,” which assumes that systematic differences may exist between sets of measurements made by different laboratories, and that these systematic differences are linear functions of the magnitude of the measurements.

12.3.2 Consider a particular laboratory and suppose that for each material the average value obtained by this laboratory is plotted against the average obtained for this material by all the laboratories combined. Then, according to the linear model, the points corresponding to the various materials will lie on a straight line except for random errors. The same applies for each of the laboratories. Thus the results of the interlaboratory study may be represented by a set of straight lines with common abscissas. Each laboratory is characterized by the slope, *b*, and the location parameter, *u*, of its straight line, and the departure, *d*, of each experimental point from the line (Fig. 1). The last may be expressed by the standard deviation, *s* (*d*), or variance *V* (*d*).

12.3.3 The departure of an experimental point from its line (within-laboratory variability), *d*, may be divided into two parts: that explainable by replication error, *e*, and that which is not, λ. The latter may be thought of as the differential response of that laboratory due either to some interfering property of that material (such as permeability, or response to relative humidity) or as a result of instrument scale error. Then:

$$V(d) = V(\lambda) + V(e)/n$$

13. Cleaning Up the Data

13.1 *Wild Results*—An individual wild result is apparent from an abnormally high standard deviation in the corresponding cell. If it appears from an examination of the standard deviations in all cells that wild results are not characteristic of the method, then omit the occasional wild result from the analysis. (See TAPPI Standard T 1205, or Practice E 178.

13.2 Study the values of *b* and *u* for the various laboratories. It is helpful to plot these values using a control-chart type plot (see Practice E 691). Occasionally, a single laboratory (or a small group of laboratories) is discrepant in one or both of these parameters, while all others are in much closer agreement. An investigation of the causes of such discrepancies is then indicated, and the analysis of variance carried out by omitting the discrepant laboratory (or laboratories) may be more meaningful than that based on its inclusion.

13.3 Study the values of *V* (*d*) or *s* (*d*) for the various laboratories. A laboratory with an obviously high value not approached by any other laboratory should be omitted from the analysis.

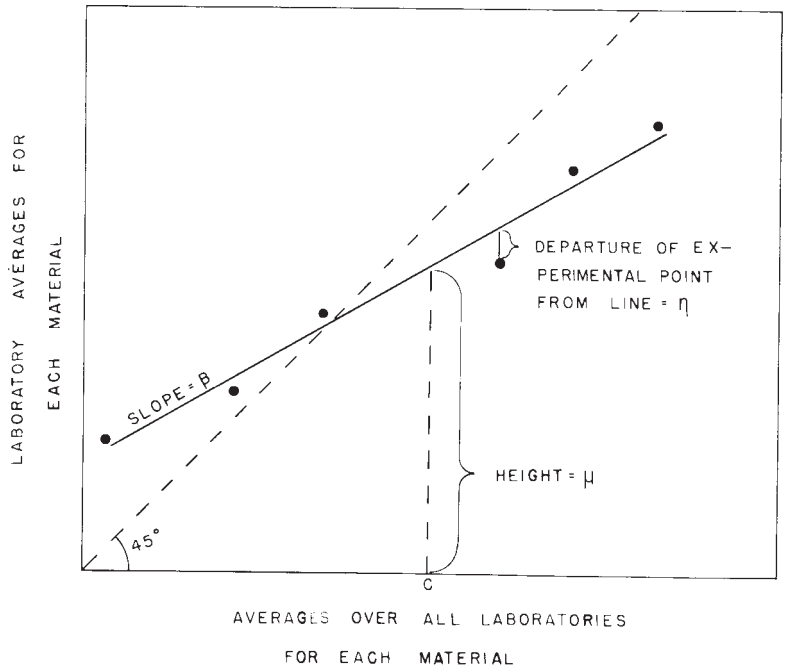
14. Interpretation of the Analysis

14.1 In interpreting the results of the analysis, the points of major interest are the relative importance of the various sources of error, the steps required to improve precision, if necessary, and the need for standard samples. For these purposes, the following procedure is recommended:

14.2 Compare *V* (λ) and *V* (*e*): the relation between these two quantities will reveal how much can be expected from mere replication of measurements. Replication is useful only to the point of making *V* (*e*)/*n* small with respect to *V* (λ).

14.3 Compare the total between-laboratory variability with the within-laboratory variability for various values of the measured property, keeping in mind that the effect of *V* (*e*) will depend on the number of replications that is called for by the test method.

14.3.1 If the total between-laboratory variability is small compared with the within-laboratory variability throughout the table, all of the laboratories are essentially in agreement, and



c— average of all materials and all laboratories [mdit]d[med] may be divided into two parts: that which can be accounted for by replication error (e) and that due to differential response of that laboratory to that material (λ).

FIG. 1 Linear Model

only refinement of the procedure to reduce $V(\lambda)$ can improve the precision of the test method.

14.3.2 If $V(\lambda)$ is so large that the test method is not sufficiently precise to be useful, the possible causes of a large $V(\lambda)$ should be investigated. Perhaps types of materials were included in the round robin for which the test method was not designed. Perhaps the test method as written fails to call for the control of important interfering conditions or fails to correct for significant interfering properties.

14.3.3 If the between-laboratory variability is not negligible, examine its two components separately (see Practice E 691). If the term in $V(b)$ may be neglected, the lines will be, for all practical purposes, parallel. In that case, the calibration of the test method at a single point will suffice to obtain the maximum possible agreement among the laboratories. On the other hand, if the term in *V(b)* becomes appreciable anywhere, the lines for the different laboratories, will tend to criss-cross at random, and the test method will require calibration at two points. There is one exception: if the slopes are correlated with the averages, the lines just happen to converge, and calibration will be required at a single point as far away from the point of convergence as is practicable.

14.3.4 In general, the term in $V(u)$ will not be negligible. If the variation in this term is small, the place of the required calibration point or points in the range of the measured quantity is immaterial, except that when two points are required, they should be located as far apart as practicable. If the variation in the $V(u)$ term is appreciable, the lines will partially or completely converge, and the calibration point or points should be located to avoid the area of convergence.

14.3.5 In summary, if between-laboratory variability is greater than can be tolerated for practical application of the test method, the test method must have better standardization. This can be done by using one or two standard reference materials to calibrate the test method at appropriately chosen values of the measured quantity.

14.4 Conclusions reached in accordance with 14.2 and 14.3 about useful amount of replication and possible value of standard samples should be tempered by findings in Section 13 of wild results and discrepant laboratories, and by judgment as to how representative were the materials and laboratories involved in the interlaboratory study.

15. Statement of Precision of Test Method

15.1 Refer to Practice E 177 for establishing the precision and bias statement.

15.2 Use of the procedures given in Practice E 691 and the associated computer program is recommended.

16. Additional Information

16.1 The design recommended in this practice is for an interlaboratory study that will yield information about the overall laboratory-to-laboratory variability, which includes the variability caused by different personnel, equipment, and environment. The statement of precision which is given at the end of a standard test method should be written in terms of this overall variability. However, during the development of a test method, separate estimates for the various components of this variability will help indicate where improvement is desirable. Because of the flexibility of the design recommended here, it can just as easily be used to study the systematic causes of these components as the overall laboratory-to-laboratory variability. This is done by redefining the word “laboratory” so that each systematic cause (operator, instrument, day, etc.) becomes

a "laboratory." An analysis of the parameters, *u*, *b*, *V* (*d*), of these "laboratories" will generally yield more useful information than a conventional analysis of variance based on a necessarily complex hierarchical design.

ANNEXES

(Mandatory Information)

A1. TECHNIQUE OF EVALUATING AN INSTRUMENT OR A TEST METHOD WITHIN ONE LABORATORY (3)

A1.1 Possible Objectives of Evaluation of a Test Method

A1.1.1 To determine if the test method actually measures the property intended, and how accurately.

A1.1.2 To determine if test results correlate with results obtained by other commonly used test methods to measure that property.

A1.1.3 To determine the limits and fields within which the test method has value, and

A1.1.4 To determine if certain defined faults of the test method can and should be corrected.

A1.2 First Steps in Evaluation of Method

A1.2.1 Establish a clear understanding of the definition and significance of the measured property. Reconcile or recognize divergent interpretations of the property.

A1.2.2 Compare this with a known reliable test method or standards if possible.

A1.2.3 Examine individual principles and features of the test method for compatibility with intended use.

A1.2.4 If reliable methods or standards are available and if individual features cannot be usefully examined, compare with subjective test methods and with other available test methods, remembering that correlation is useful only if good for small differences in the property being measured as well as for large differences.

A2. THE SENSITIVITY CRITERION (4,5)

A2.1 Description of Term

A2.1.1 "*Sensitivity*" is a measure of performance which is useful in the evaluation of test methods. Its application can be pictured in terms of an analogy with a radio receiver. Thus, an instrument or test method receives a signal in the form of a sample, and noise in the form of experimental error. The test method detects and amplifies the desired signal (component to be determined) and yields a message in the form of a measured value. Sensitivity, in terms of this analogy, is a measure that increases directly with amplification and inversely with noise. **(6)**

A2.1.2 Consider two alternative methods, *A* and *B*, for measurement of a property, *Q*. For example, *Q* may be smoothness and *A* and *B* may be Bekk seconds and Sheffield number, respectively. Since both *A* and *B* are a measure of *Q*, they are functionally related to *Q* and therefore also functionally related to each other. The curve in Fig. A2.1 represents such a relationship. As can be seen from the figure, determination of the sensitivity ratio requires a knowledge of the slope $\Delta A/\Delta B$ and of the standard deviations, σ_A and σ_B.

A2.2 Steps in the Comparison of Two Test Methods of Procedures by Means of the Sensitivity Criterion

A2.2.1 Determine the relationship between the results of the two test methods so that the slope of this relationship can be evaluated at each point (that is, at each level of the measured property). If no relationship can be found, the two test methods are not measuring the same property, and the sensitivity cannot be used for comparing them.

A2.2.2 For each test method, determine the standard deviation and how it varies with the magnitude of the measurement. Remember that in order to obtain a good estimate of standard deviation, considerable replication is required.

A2.2.2.1 The nature of the standard deviation occurring in the definition of sensitivity depends on the nature of the comparison between the two test methods. In a within-one-laboratory study, the interest is centered on the intrinsic merit of each instrument. Therefore, the standard deviation used in the evaluation of sensitivity must be a measure of instrumental fluctuations only. Consequently, all errors that are not related to the instrument, such as variability in machine and cross-direction, sheet-to-sheet variability, and systematic operator biases, are eliminated. The remaining error is used in the estimation of the standard deviation.

A2.2.2.2 If two test methods are compared on an interlaboratory basis, as when deciding which shall be chosen as the standard test method, the standard deviations to be used are the total standard deviations (or standard errors) obtained from the interlaboratory study.

A2.2.3 Compute the ratio of sensitivities in accordance with the equations given in Fig. A2.1. Method A is more sensitive wherever the absolute value of the ratio is appreciably greater than unity.

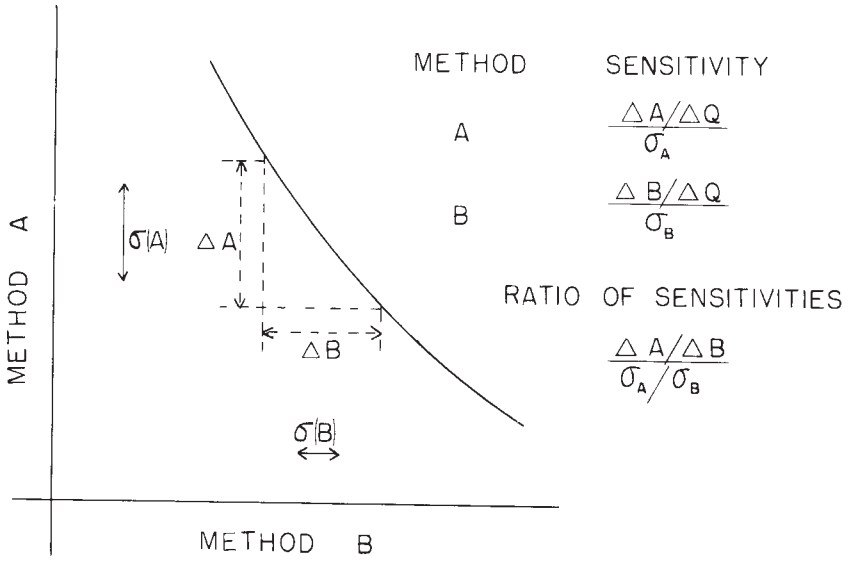


FIG. A2.1 Comparison of Two Test Methods for Measurement of Property Q

A3. SAMPLE DATA SHEET

Set No. _____ Observer _____ Date _____ Sheet No. _____

Time	Specimen Code No.	Scale Reading	Corrected Scale Reading	Time	Specimen Code No.	Scale Reading	Corrected Scale Reading
RH				RH			
				Temp			
Temp							

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