Designation: D 4678 – 04

Standard Practice for Rubber—Preparation, Testing, Acceptance, Documentation, and Use of Reference Materials[1]

This standard is issued under the fixed designation D 4678; 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 materials used on an industry-wide basis as reference materials, which are vitally important to conduct product, specification, and development testing in the rubber and carbon black industry. This practice describes the steps necessary to ensure that any candidate material, that has a perceived need, can become a Reference Material. The practice sets forth the recommendations on the preparation steps for these materials, on the testing that shall be conducted to permit acceptance of any candidate material, and on how the documentation needed for the acceptance shall be recorded for future use and review.

1.2 This practice shall be administered by ASTM Committee D11 in consultation on all matters with Committee D24.

1.2.1 Important sections of this practice are as follows:

	Section
Significance and Use	3
Preparation of Industry Reference Materials	4
Overview of Industry Reference Material Testing	5
Chemical and Physical Specifications for IRM	6
Reference Material Documentation	7
Assignment and Tabulation of Reference Material Numbers	8
Typical Reference Material Use	9
Recommended Package Size for IRM	Annex A1
Recommended Sampling Plans for Homogeneity Testing of an IRM	Annex A2
Test Plan and Analysis for Homogeneity of an IRM	Annex A3
Test Plan and Analysis to Evaluate an Accepted Reference Value	Annex A4
Statistical Model(s) for IRM Testing	Annex A5
Example of Annex Calculations for a Typical IRM	Appendix X1
Two-Way Analysis of Variance for Calculating *Sr*	Appendix X2
Inventory of NIST Rubber Compounding SRMs	Appendix X3

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

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process

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

E 826 Practice for Testing Homogeneity of Materials for Development of Reference Materials

3. Significance and Use

3.1 Reference materials are vitally important in product and specification testing, in research and development work, in technical service work, and in quality control operations in the rubber and carbon black industries. They are especially valuable for referee purposes.

3.2 *Categories, Classes, and Types of Reference Materials (RM)*:

3.2.1 Reference materials are divided into two categories:

3.2.1.1 *Industry Reference Materials (IRM)*—Materials that have been prepared according to a specified production process to generate a uniform lot; the parameters that define the quality of the lot are evaluated by a specified measurement program.

3.2.1.2 *Common-Source Reference Materials (CRM)*—Materials that have been prepared to be as uniform as possible but do not have established property (parameter) values; the knowledge of a common or single source is sufficient for certain less critical applications.

3.2.2 Industry reference materials (IRMs) are divided into additional classes and types according to the method of evaluating the lot parameters and according to the production process for generating the lot material. These are explained

[1] This practice is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

Current edition approved Dec. 1, 2004. Published January 2005. Originally approved in 1987. Last previous edition approved in 2001 as D 4678 – 94 (2001).

[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.

more fully (refer to Annex A3 and Annex A4 for more details on the discussion in Section 3).

3.2.3 The following lot parameters are important for reference material use:

3.2.3.1 *Accepted Reference Value (AR Value)*—An average IRM property or parameter value established by way of a specified test program.

3.2.3.2 *Test Lot Limits (TL Limits)*—These are limits defined as ±3 times the standard deviation of individual IRM test results across the entire lot for the property or parameter(s) that defines lot quality; the measurements are conducted in the laboratory of the organization producing the IRM.

3.2.3.3 Although the limits as defined in 3.2.3.2 are given in terms of ±3 times the standard deviation, the rejection of individual portions of the lot as being outlier or non-typical portions in assessing the homogeneity of the lot is done on the basis of ±2 times the appropriate standard deviation, that is, on the basis of a 95 % confidence interval. See Annex A3 and Annex A4 for more information and the evaluation procedures.

3.2.4 All IRMs have an AR value and TL limits; however the AR value may be obtained in one of two ways to produce one of two classes of AR values:

3.2.4.1 *Global AR Value*—This AR value is obtained from an interlaboratory test program where the word "global" indicates an average value across many laboratories.

3.2.4.2 *Local AR Value*—This is an AR value obtained in one laboratory or at one location, usually the laboratory responsible for preparation of the homogeneous lot.

3.2.5 An additional parameter is of importance for IRMs that have a global AR value:

3.2.5.1 *Between-Laboratory Limits (BL)*—The group of laboratories that conduct interlaboratory testing to establish an AR-value are not equivalent to a system or population typical of industrial production operations that use the usual ±3 standard deviation limits. Such production operations are systems that have been purged of all assignable causes of variation and are in a state of 'statistical control' with only random variations that cannot be removed. Thus, the recommended limits on all IRMs are the ±2 standard deviation limits that pertain to a 95 % confidence level. If for serious reasons that can be totally justified, ±3 standard deviation limits are required, these may be used provided that full and complete documentation is supplied to justify the limits.

3.2.6 The homogeneity or uniformity of the lot, which determines the magnitude of the TL limits, may be designated as one of two different levels of uniformity. The key factor that determines the level of uniformity is the capability of blending the IRM portions or parts that constitute the lot, to ensure a high degree of uniformity from the blending process. IRMs that cannot be blended will have an extra residual amount of variation (portion to portion) that lowers the level of uniformity.

3.2.6.1 *Uniformity Level 1 (UL-1)*—This is the most uniform or highest level of homogeneity that can be attained by the use of *a specified test* for measuring the parameter that defines lot quality; it is obtained by the use of a blended material and is referred to as a Type *B* (*B* = blended) IRM.

3.2.6.2 *Uniformity Level 2 (UL-2)*—This is the lesser degree of uniformity that is attained by the use of*a specified test* for measuring the parameter that defines lot quality; it is normally obtained for non-blended materials and is referred to as a Type *NB* (not blended) IRM.

3.3 IRMs have a number of use applications in the technical areas, as cited in 3.1.

3.3.1 *Single Laboratory Self Evaluation*—The IRM may be used in a given laboratory (or with a given test system) to compare the test results within the laboratory to the accepted reference value for the IRM. An IRM can also be used for internal statistical quality control (SQC) operations.

3.3.2 *Multi-Laboratory Evaluation*—The IRM may be used between two or more laboratories to determine if the test systems in the laboratories are operating within selected control limits.

3.3.3 One or more IRMs may be used in the preparation of compounds to be used for evaluating non-reference materials in compound testing and performance.

3.3.4 Reference liquid IRMs may be used for immersion testing of various candidate or other reference compounds. Such immersion testing is important due to the deleterious influences of immersion liquids on rubber compounds.

3.3.5 IRMs may also be used to eliminate interlaboratory testing variation known as "test bias:" a difference between two (or more) laboratories that is essentially constant between the laboratories for a given test property level, irrespective of the time of the test comparisons. In such applications a differential test measurement value, (IRM – experimental material), becomes a corrected test result; this corrected value is used as the measure of performance rather than the "as-measured" test value on the experimental material of interest.

3.4 Average values play an important role in various operations and decisions in this practice. For this practice, "average" is defined as the arithmetic mean.

3.5 The various characteristics of IRMs and CRMs (categories, classes, types) are listed in summary form in Table 1.

3.6 This practice and the IRM program it describes is being developed to replace a standardization program conducted by the National Institute of Standards and Technology (NIST) that was begun in 1948 and is being phased out. The standard materials developed by the NIST program are referred to as Standard Reference Materials or SRM.

3.7 To provide for some continuity in the "phase-out—phase-in" operation, Appendix X3 lists the rubber and compounding materials still carried by NIST on an interim basis.

TABLE 1 Categories of Reference Materials[A]

	IRM				CRM
AR Value	Global		Local		None
Homogeneity (TL Limits)	Type *B* (UL-1)	Type *NB* (UL-2) or (UL-1)	Type *B* (UL-1)	Type *NB* (UL-2) or (UL-1)	Single Source Material

[A] AR value = accepted reference value.
TL limits = test lot limits.
Global = AR value obtained from an interlaboratory test program.
Local = AR value obtained from one laboratory.
Type-B = IRM that has been blended to ensure high uniformity.
Type-NB = IRM that cannot be blended.
UL-1 and UL-2 = levels of uniformity in the IRM lot; UL-1 is higher uniformity than UL-2.
See Annex A3 and Annex A4 for more information.

Appendix X3 will be retained in this practice until such time as NIST materials are no longer used.

3.8 It is not feasible to write into this practice all the necessary specifications, modes of preparation, sampling, and testing protocols, for the wide variety of materials that will eventually become IRM. Therefore this practice is published to give general guidelines for IRMs.

3.9 A permanent IRM Steering Committee within Subcommittee D11.20 shall be constituted by Subcommittee D11.90 in consultation with Committee D24 to assist in the utilization of this practice and to make technical and, where required, policy decisions regarding the preparation and administration of IRM. The IRM Steering Committee shall have members of both Committees D11 and D24.

4. Preparation of Industry Reference Materials

4.1 *Basic Preparation Steps*:

4.1.1 An IRM should be prepared in a way that ensures that the entire quantity or lot of the material is as homogeneous, in composition and vital performance properties, as is possible.

4.1.2 For particulate and liquid materials this implies a thorough physical blending operation during or after the manufacturing steps, or both.

4.1.3 For materials not easily blended after manufacture, two options to ensure homogeneity are recommended:

4.1.3.1 Use highly homogeneous components or other materials that are required in the manufacturing steps or conduct certain blending operations at intermediate manufacturing steps to ensure maximum homogeneity.

4.1.3.2 Use intensive statistical quality control procedures to ensure a specified degree of homogeneity among the packets, bales, or other discrete units of the material.

4.1.4 Examples, as cited in 4.1.3.1, are such materials as accelerators, antioxidants, sulfur, and reference test (liquid) fuels.

4.1.5 Examples, as cited in 4.1.3.2, are various synthetic rubbers.

4.2 *Packaging of Industry Reference Materials*:

4.2.1 Industry reference materials should be packaged preferably in small quantities or packages. The packages shall be consecutively numbered as they are filled. Nominally the size should be the smallest amount that the average user of the material would require for normal volume testing. High volume users could therefore order multiple package lots. The use of such minimum volume (mass) packages will of course vary, but Annex A1 gives recommended masses or volumes.

4.2.2 Industry reference materials shall be suitably packaged to prevent or retard the change of IRM values with the passage of time or inadvertent exposure to heat, light, moisture, or combinations thereof, in normal storage. The stringency of this requirement varies with the type of IRM. All precautions shall be taken to make IRMs as stable as possible.

4.2.3 Packages shall be dispensed by the manufacturing or distribution organization with a document that shall furnish the following general information:

4.2.3.1 Name and number of the IRM,

4.2.3.2 Name of the manufacturer,

4.2.3.3 Date of manufacture or preparation,

4.2.3.4 Storage conditions, and

4.2.3.5 Reference to ASTM research report for documentation of testing.

4.2.4 For each test property measured to assess lot quality report the following:

4.2.4.1 Accepted reference value,

4.2.4.2 Test lot limits, and

4.2.4.3 Between-laboratory limits.

4.3 *Packaging of Common–Source Reference Materials*:

4.3.1 CRMs shall be packaged and dispensed in the same manner as for IRMs. Each CRM package shall be furnished with a documentation sheet with the following information:

4.3.1.1 Name and number of the CRM,

4.3.1.2 Name of manufacturer,

4.3.1.3 Date of manufacture or preparation,

4.3.1.4 Storage conditions, and

4.3.1.5 Reference to ASTM research report.

5. Overview of Industry Reference Material Testing

5.1 Testing is conducted to *(1)* demonstrate the uniformity of the IRM lot to some selected limits and evaluate the test lot limits, and *(2)* to establish an accepted reference value for the lot and as a secondary goal to evaluate the between-laboratory limits for interlaboratory testing of the IRM where this is applicable.

5.2 *Testing for Homogeneity*:

5.2.1 Homogeneity testing is ideally conducted in one highly qualified laboratory, which is usually the laboratory of the organization that produces the IRM. The lot size is determined and samples are drawn from the lot. Guidance for the size and number of samples is given in Annex A2. The samples taken from the lot are tested according to the instructions given in Annex A3. This latter annex also addresses the concept of different uniformity levels for an IRM and the importance of this in IRM development and use.

5.2.2 It is important that each sample represents a fraction or portion of the total lot that can be physically separated from the remainder of the lot, in the event that the portion represented by the sample is judged to be significantly different from the remainder of the lot and is therefore rejected.

5.2.3 Those portions of the lot that are shown to be significantly different from the remainder or bulk of the lot shall be rejected.

5.2.4 If, in the statistical analysis of Annex A3, a substantial fraction (25 to 30 %) of the lot is declared to be not acceptable for lack of homogeneity, retesting may be permitted. This retesting shall include all suspected portions and a number of accepted homogeneous portions or parts equal in number to the suspect portions. The retest shall be conducted according to Annex A3.

5.2.5 If on retesting and analysis of the newly generated data these same portions are again found to be unequal in property value to the accepted portions by standard statistical tests, they shall be rejected. If the suspected portions are found to be equal to the accepted portions in property values, they may be accepted as part of the lot.

5.3 *Testing for an Accepted Reference Value*—Testing for an accepted reference value may be undertaken once a homogeneous lot has been achieved. The detailed instructions for conducting the interlaboratory program and analyzing the data