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Rubber — Determination of precision of test methods

Caoutchouc — Détermination de la fidélité des méthodes d'essai

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Introduction

The procedures used for several years by ISO/TC 45/SC 2 for estimating precision of test methods by means of interlaboratory tests (ISO/TR 9272) were closely related to ASTM D4483. ISO/TR 9272 was found to have serious flaws which users were using work-arounds to counteract. It became clear that ISO/TR 9272 needed to be replaced and it was concluded that the best option was to base a new standard on ISO 5725 (all parts) with specific choices and variations of procedures to suit the particular requirements of rubbers.

Rubber — Determination of precision of test methods

1 Scope

This document provides guidelines and specifies requirements for estimating the precision of rubber test methods by means of interlaboratory test programmes based on the procedures given in ISO 5725 (all parts).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

ISO 3534-2, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 5725-4, *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method*

ISO 5725-5, *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*

ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534-1, ISO 3534-2, ISO 5725 (all parts), and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
day-to-day repeatability
precision under the conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment

Note 1 to entry: The time interval between repeated tests is normally between one and seven days.

3.2
type 1 precision
precision determined directly on a target material

Note 1 to entry: Prepared test pieces or test portions of the target material (class of elements) drawn from a homogeneous source are tested, with no processing or other operations required prior to testing.

3.3
type 2 precision
precision determined indirectly for a target material

Note 1 to entry: The target material is usually combined with a number of homogeneous ancillary materials to form a composite material and testing is conducted on samples of this and the property response of the target material is determined.

4 Symbols and abbreviated terms

D_{ij}	day-to-day effect, the day-to-day variance component of which is σ_D^2
h values	Mandel's between-laboratory consistency test statistic
k values	Mandel's within-laboratory consistency test statistic
L_i	between-laboratory effect, the between-laboratory variance component of which is σ_L^2
M_{ijk}	repeatability effect, the repeatability variance component of which is σ_M^2
n	number of measurements
p	number of laboratories
q	number of days
r	repeatability
r_D	day-to-day repeatability
R	reproducibility
(r)	relative repeatability
(r_D)	relative day-to-day repeatability
(R)	relative reproducibility
s_M^2	repeatability variance
s_{rD}^2	day-to-day repeatability variance
s_R^2	reproducibility variance
s_D^2	day-to-day variance
s_L^2	between-laboratory variance
s	standard deviation of data
s_r	repeatability standard deviation
s_{rD}	day-to-day repeatability standard deviation
s_R	reproducibility standard deviation
S_T	total sum of squares
S_L	between-laboratory sum of squares
S_D	day-to-day sum of squares
S_M	repeatability sum of squares
T	total sum of data
V_L	between-laboratory mean square
V_D	day-to-day mean square
V_M	repeatability mean square

y_{ijk}	data i, j, k: each data of laboratory, day, repeat
$\bar{y}$	mean value of data
$\bar{\bar{y}}$	mean value of $\bar{y}$
ϕ_T	total degree of freedom
ϕ_L	between-laboratory degree of freedom
ϕ_D	day-to-day degree of freedom
ϕ_M	repeatability degree of freedom
μ	population mean
σ_M^2	repeatability variance component
σ_D^2	day-to-day variance component
σ_L^2	between-laboratory variance component

5 Interlaboratory test programme

To evaluate precision for test method standards by means of interlaboratory test programmes (ITPs), use either one of the two methods:

a) Method A, where three precisions, namely the repeatability, the day-to-day repeatability and the reproducibility, are calculated in accordance with ISO 5725-3;

b) Method B, where two precisions, namely the day-to-day repeatability and the reproducibility, are calculated in accordance with ISO 5725-2.

NOTE If two or more results are available from within-a-day repeated tests, method A is applicable to evaluate the variance of measurement errors.

6 Procedures

6.1 Application

A standard measurement method is taken to mean an established international test method for rubber.

A determination of the precision of a test method is normally conducted with a selected group of materials typical of those used with that method, and by a group of volunteer laboratories that have experience of the method.

Caution is necessary in applying precision results for a particular test method to product testing for commercial product accepted procedures. For this purpose, the precision estimates should be obtained from special programmes that are specific to the product in question and carried out by the interested laboratories.

6.2 Repeatability conditions

Repeatability conditions are where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

NOTE "Short interval of time" indicates that tests are repeated within a day.

Identical test items is interpreted as nominally identical, i.e. no intentional differences.

For rubbers, repeatability can be dependent on the magnitude or level of the measured property and is usually reported for each of a number of materials having particular property levels.

6.3 Day-to-day repeatability conditions

Day-to-day repeatability conditions are where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment.

The "intervals of time" between repeated measurements of test results may be selected by the consensus of a particular testing community. For ISO/TC 45 and the international rubber manufacturing industry, the time interval between repeat tests is of the order of one to seven days, but most commonly seven days. For special tests (long ageing periods), however, replicate tests can require a longer time span.

NOTE The "repeatability" traditionally used by ISO/TC 45/SC 2 is equivalent to the day-to-day repeatability defined in this document.

6.4 Reproducibility conditions

Reproducibility conditions are where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment.

Identical test items is interpreted as nominally identical, i.e. no intentional differences.

For ISO/TC 45, different equipment means apparatus that might have different manufacturers but complies with the requirements of the test standard in question, including calibration.

For rubbers, reproducibility might be dependent on the magnitude or level of the measured property and is usually reported for each of a number of materials having particular property levels.

6.5 Testing elements

The element that is tested is either a test piece or a test sample as defined in the test method standard. The test method standard will also define the number of test elements to be tested to obtain a result for the property.

6.6 Planning

Select either type 1 precision or type 2 precision as defined in 3.2 and 3.3.

It is possible that a type 1 precision programme might be conducted on test pieces or portions that require some minimum processing or other simple operations prior to actual testing.

Unless circumstances dictate otherwise, using Type 1 precision is preferred.

For type 1 precision, the test pieces or test samples need to be produced from the same lot of material by the same procedures and then stored and conditioned in the same manner, in order to be nominally identical. This is best achieved by test pieces being prepared in one laboratory and distributed to the others with instructions for conditioning.

For type 2 precision, the properties of the composite material are directly related to the quality of properties of the target material. As an example, to determine the quality of a grade of SBR, a sample of the rubber plus curatives, fillers, antioxidants, etc. are mixed and cured. The precision of the resulting test pieces is determined and reflects sample preparation and the properties response of the target SBR.

The estimation of precision for rubber test methods is normally conducted using a balanced uniform level design with three or more materials sent to each participating laboratory with tests conducted to yield an independent test result by the same technician on each of two test days.

NOTE A balanced uniform level design is a plan for an interlaboratory test programme for precision, where all laboratories test all the materials selected for the programme and each laboratory conducts the same number of repeated tests, *n*, on each material.

The test method, materials, participating laboratories, test equipment and time interval for test in a laboratory are addressed in 6.1 to 6.6. Other aspects of planning are addressed in ISO 5725-1:1994, Clause 6.

6.7 Methodology

6.7.1 Method A

Method A determines the repeatability variance component (measurement error component) σ_M^2, the day-to-day variance component σ_D^2 and the between-laboratory variance component σ_L^2, by calculating the expected mean square in accordance with a suitable ANOVA table in ISO 5725-3, fully-nested experiments.

Then, the day-to-day repeatability variance s_{rD}^2 and the reproducibility variance s_R^2 are given by the following formulae:

$$s_{rD}^2 = \sigma_M^2 + \sigma_D^2$$

$$s_R^2 = \sigma_M^2 + \sigma_D^2 + \sigma_L^2$$

The repeatability, r, the day-to-day repeatability, r_D, and the reproducibility, R, are given by the following formulae:

$$r = 2{,}83\left(s_M^2\right)^{\frac{1}{2}} = 2{,}83\left(\sigma_M^2\right)^{\frac{1}{2}} = 2{,}83 s_M = 2{,}83\sigma_M$$

$$r_D = 2{,}83\left(s_{rD}^2\right)^{\frac{1}{2}} = 2{,}83 s_{rD}$$

$$R = 2{,}83\left(s_R^2\right)^{\frac{1}{2}} = 2{,}83 s_R$$

Calculations for method A shall be in accordance with Annex A. An example is given in D.3.

For rubber tests, it is usually possible to have two or more repeated tests within one day. In such cases, method A is preferred.

6.7.2 Method B

Method B determines the day-to-day variance (between-day variance), s_D^2, the between-laboratory variance s_L^2 and the reproducibility variance s_R^2 (which is equal to $s_L^2 + s_D^2$), according to the calculation procedures in ISO 5725-2.

The day-to-day repeatability, r_D, and the reproducibility, R, are given by the following formulae:

$$r_D = 2{,}83\left(s_D^2\right)^{\frac{1}{2}} = 2{,}83 s_D$$

$$R = 2{,}83\left(s_R^2\right)^{\frac{1}{2}} = 2{,}83 s_R$$

Calculations for method B shall be in accordance with Annex B. An example is given in D.4.