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Rubber and rubber products — Confidence intervals for repeatability and reproducibility values determined by inter-laboratory tests

Caoutchouc et produits en caoutchouc — Intervalles de confiance de répétabilité et de reproductibilité déterminées par essais interlaboratoires

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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 main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 11753, which is a Technical Report of type 3, was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

Introduction

The subject of confidence intervals of repeatability and reproducibility determined in inter-laboratory tests was first discussed in Working Group 15 "Application of statistical methods" of ISO Technical Committee 45 "Rubber and rubber products" at its 1988 meeting in Paris. At the 1989 meeting of WG15 in Kuala Lumpur a first draft was considered, following which consultation took place with the chairman and members of ISO Technical Committee 69 "Application of statistical methods".

A revised draft was considered by WG15 at its 1992 meeting in Stockholm, where it was agreed that the text should be issued as a Type 3 Technical Report. This proposal, in Resolution 2207 of ISO/TC45 (document 45 N 5928) was approved unanimously by all ISO/TC45 members present at the 1992 meeting.

Rubber and rubber products — Confidence intervals for repeatability and reproducibility values determined by inter-laboratory tests

1 Scope

The precision values estimated from inter-laboratory tests vary on repetition of these tests. This document describes a method for obtaining confidence intervals for the unknown precision values.

2 Field of application

ISO 5725 describes the performance of inter-laboratory tests and states how data for the repeatability and reproducibility of a standardized test method can be obtained from the results of inter-laboratory tests.

The repeatability standard deviation s_r or repeatability limit r is used as a measure for repeatability, and the reproducibility standard deviation s_R or the reproducibility limit R as a measure for reproducibility.

Only estimates of the precision values are obtainable from inter-laboratory tests. On repetition of an inter-laboratory test slightly different estimates will be obtained owing to the random influences that are also present in inter-laboratory tests. ISO 5725 contains no data on the possible errors that may occur when the values of r and R are estimated.

However, knowledge of the deviation of determined estimates of repeatability and reproducibility from the true values is very important, since it enables the following two groups of questions to be answered:

a) Questions relating to the planning of inter-laboratory tests:

How extensive should the inter-laboratory test be, i.e. how many laboratories, materials and individual values are needed to achieve a given degree of accuracy in estimating the precision values?

b) Questions relating to the application of results from inter-laboratory tests:

How accurate are the repeatability and reproducibility estimates determined in an inter-laboratory test? How long are the confidence intervals of the precision values sought?

The precision values determined in an inter-laboratory test are used to characterize the test method in the corresponding standard. It is important to know to how many decimal places they should be reported.

The confidence intervals of the precision values to be estimated, and hence the accuracy of the estimates, depend on the scope of testing: in general the precision of precision values is the greater and their confidence interval the narrower, the larger the number of laboratories p and number of individual values n determined for each test property level in each laboratory.

In this document it is assumed that values measured under repeatability conditions and in different laboratories are distributed normally; see Annex A1. Deviations from the normal distribution generally make the variability of the precision values somewhat greater and the confidence interval of these values correspondingly longer.

Note: The validity of the normal distributions of the deviations under repeatability and reproducibility conditions is a prerequisite for the relations stated in this standard; it is possible that this prerequisite is not fulfilled. In Section 7.3.1 of DIS 5725 Part 1 other unimodal distributions of the deviations under repeatability conditions are allowed also.

In addition it is necessary to comply with the condition, laid down in Section 10.1 of DIS 5725 Part 1 that the laboratories participating in the inter-laboratory test should be chosen at random.

On the assumption of normal distribution the confidence intervals are stated with a preselected error probability α. In this standard $\alpha = 0.10$. This means that there is a 10 % probability that the true value is outside the given confidence interval, with $\alpha/2 = 0.05$ or 5 % below the lower limit and likewise $\alpha/2 = 0.05$ or 5 % above the upper limit.

3 Definitions

The definitions used in this document are given in

ISO 3534-3:1985, Statistics - Vocabulary and symbols - Part 3: Design of experiments.

ISO 5725:1986, Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.

4 Calculation of confidence intervals

4.1 Confidence interval of the repeatability

The results of an inter-laboratory test for a test property level with p laboratories and n_i measurements in i. laboratory can be evaluated by a variance analysis with simple classification and random effects. Assuming that the deviations of the results obtained in the laboratories have a normal distribution, the confidence interval of the true repeatability value r' can be calculated from the χ^2 distribution (see Annex A2):

(1) $$r\,A_{r,1} < r' < r\,A_{r,2}$$

The following is true of the confidence interval of the quotient r'/r:

(1a) $$A_{r,1} < r'/r < A_{r,2}$$

where (1b) $$A_{r,1} = \sqrt{\nu_2/\chi^2(\nu_2,Q)}$$

and (1c) $$A_{r,2} = \sqrt{\nu_2/\chi^2(\nu_2,P)}$$

Estimated repeatability standard deviation	s_r
True repeatability standard deviation	σ_r
Estimated repeatability limit	$r = 2{,}8\ s_r$
True repeatability limit	$r' = 2{,}8\ \sigma_r$
Number of laboratories	p
Number of measurements in i.laboratory (for i = 1, 2 ... p)	n_i
total number measurements per material level	$N = \Sigma\, n_i$
Degree of freedom of the repeat measurements	$\nu_2 = \Sigma\, n_i - p$
Probability of the χ^2 fractiles for the upper limit	$P = 0{,}05$
Probability of the χ^2 fractiles for the lower limit	$Q = 0{,}95$
Fractiles of the χ^2 distribution of ν and P	$\chi^2(\nu,P)$

The χ^2 fractiles $\chi^2(\nu_2,P)$ and $\chi^2(\nu_2,Q)$ can be taken from tables in standard literature. However, the approximation formula in Annex B can be used also.

The χ^2 fractiles are defined by

(2) $$w\ (\ \chi^2 \leq \chi^2(\nu,P)\ ) = P$$

i.e. there is a probability P that the random quantity χ^2 of a χ^2 distribution with degree of freedom ν is smaller than or equal to the fractiles $\chi^2(\nu,P)$, tabulated for ν and P.

If $Q = 1 - (\alpha/2) = 0.95$ and $P = \alpha/2 = 0.05$ are chosen for the probabilities, a probability of error of 10 % is obtained for the confidence interval, i.e. there is a probability of 10 % that the quotient r'/r sought is not covered by the confidence interval.

In the orthogonal case, i.e. $n_i = n$ for all $i = 1, 2 \ldots p$, the following is true

(3a) $N = p\ n$ and (3b) $\nu_2 = p\ (n-1)$.

The approximation formula given in Annex B was used to calculate the confidence limits $A_{r,1}$ and $A_{r,2}$ for n = 2, 3, 5 and 9 test results per laboratory and p = 8 to 60 laboratories, which are given in Table 1 (see next page) and in Figure 1 (see page 14).

Table 1: Confidence intervals $A_{r,1}$ and $A_{r,2}$ for r'/r

n	p	ν_2	$\chi^2(\nu_2,5\%)$	$\chi^2(\nu_2,95\%)$	$A_{r,1}$	$A_{r,2}$
2	8	8	2.73	15.51	0.72	1.71
2	10	10	3.94	18.31	0.74	1.59
2	12	12	5.23	21.03	0.76	1.52
2	14	14	6.57	23.68	0.77	1.46
2	16	16	7.96	26.30	0.78	1.42
2	18	18	9.39	28.87	0.79	1.38
2	20	20	10.85	31.41	0.80	1.36
2	25	25	14.61	37.65	0.81	1.31
2	30	30	18.49	43.77	0.83	1.27
2	35	35	22.47	49.80	0.84	1.25
2	40	40	26.51	55.76	0.85	1.23
2	50	50	34.76	67.50	0.86	1.20
2	60	60	43.19	79.08	0.87	1.18
3	8	16	7.96	26.30	0.78	1.42
3	10	20	10.85	31.41	0.80	1.36
3	12	24	13.85	36.42	0.81	1.32
3	14	28	16.93	41.34	0.82	1.29
3	16	32	20.07	46.19	0.83	1.26
3	18	36	23.27	51.00	0.84	1.24
3	20	40	26.51	55.76	0.85	1.23
3	25	50	34.76	67.50	0.86	1.20
3	30	60	43.19	79.08	0.87	1.18
3	35	70	51.74	90.53	0.88	1.16
3	40	80	60.39	101.88	0.89	1.15
3	50	100	77.93	124.34	0.90	1.13
3	60	120	95.70	146.57	0.90	1.12
5	8	32	20.07	46.19	0.83	1.26
5	10	40	26.51	55.76	0.85	1.23
5	12	48	33.10	65.17	0.86	1.20
5	14	56	39.80	74.47	0.87	1.19
5	16	64	46.59	83.68	0.87	1.17
5	18	72	53.46	92.81	0.88	1.16
5	20	80	60.39	101.88	0.89	1.15
5	25	100	77.93	124.34	0.90	1.13
5	30	120	95.70	146.57	0.90	1.12
5	35	140	113.66	168.61	0.91	1.11
5	40	160	131.76	190.52	0.92	1.10
5	50	200	168.28	233.99	0.92	1.09
5	60	240	205.14	277.14	0.93	1.08
9	8	64	46.59	83.68	0.87	1.17
9	10	80	60.39	101.88	0.89	1.15
9	12	96	74.40	119.87	0.89	1.14
9	14	112	88.57	137.70	0.90	1.12
9	16	128	102.87	155.40	0.91	1.12
9	18	144	117.27	173.00	0.91	1.11
9	20	160	131.76	190.52	0.92	1.10
9	25	200	168.28	233.99	0.92	1.09
9	30	240	205.14	277.14	0.93	1.08
9	35	280	242.25	320.03	0.94	1.08
9	40	320	279.56	362.72	0.94	1.07
9	50	400	354.64	447.63	0.95	1.06
9	60	480	430.20	532.08	0.95	1.06

4.2 Confidence interval of the reproducibility

A χ^2 distribution can also be assumed for estimation of the confidence interval of the reproducibility limit R, but in this case it is only approximately true (see Annex A.3). On the assumption of orthogonality the degree of freedom ν_3 for this distribution can be calculated from the following equation:

$$\nu_3 = \frac{n^2\ (1+\gamma^2)^2\ \nu_1\ \nu_2}{(n+\gamma^2)^2\ \nu_2 + (n-1)^2\ \gamma^4\ \nu_1} \tag{4}$$

If there is a marked deviation from orthogonality, i.e. if the number of measurements per laboratory varies greatly, the method given in Annex A.3.2 can be used. The degrees of freedom $\nu_1 = p - 1$ and $\nu_2 = p(\Sigma n_i - 1)$ are obtained from the number of laboratories p and number of individual measurements n_i per laboratory.

The degree of freedom ν_3 depends on a further parameter γ' or g':

(5a) $\gamma' = \sigma_r / \sigma_L$ or $g' = \sigma_r / \sigma_R$

As the true values σ_r, σ_R and σ_L are not known, the estimated values s_r, s_R and s_L are used as approximations, $\gamma' = \sigma_r/\sigma_L$ being estimated by $\gamma = s_r/s_L$.

From the definition of σ_R^2 or s_R^2 (see ISO 5725) it follows that

(6a) $\sigma_R^2 = \sigma_L^2 + \sigma_r^2$ or (6b) $s_R^2 = s_L^2 + s_r^2$

Therefore

(7a) $\gamma = \dfrac{s_r}{s_L} = \dfrac{s_r}{\sqrt{s_R^2 - s_r^2}} = \dfrac{g}{\sqrt{1-g^2}}$ and (7b) $g = \dfrac{s_r}{s_R}$

After an inter-laboratory test has been evaluated the estimated values s_r^2 and s_R^2 from which γ can be calculated according to (7a) are known. In the planning of an inter-laboratory test assumptions for γ must be made in order to fix the number of laboratories (see Section 5.1).

After the degree of freedom ν_3 has been calculated, the confidence interval for the reproducibility limit R is obtained analogously to that of the repeatability limit r, as described in Section 4.1. The following is valid for the confidence interval of the quotient R'/R:

(8) $$A_{R,1} < R'/R < A_{R,2}$$

where (8a) $$A_{R,1} = \sqrt{\nu_3/\chi^2(\nu_3,Q)}$$

and (8b) $$A_{R,2} = \sqrt{\nu_3/\chi^2(\nu_3,P)}$$

Note: The degree of freedom ν_3 in Equation 4 is generally not an integer number. In reading χ^2 fractiles $\chi^2(\nu_3,P)$ from a χ^2 table it is necessary whether to interpolate or to take the nearest smaller number. However, it is also possible to enter the non-integral value ν_3 in the approximation formula given in Annex B for the χ^2 fractiles.

If $Q = 1 - (\alpha/2) = 0.95$ and $P = \alpha/2 = 0.05$ are chosen for the probabilities, a probability of error of 10 % is obtained for the confidence interval, i.e. there is a probability of 10 % that the quotient R'/R sought is not covered by the confidence interval.

The approximation formula Equation 8 was used with the aid of the approximation formula given in Annex B for the χ^2 distribution, to calculate the confidence limits $A_{R,1}$ and $A_{R,2}$ given in Table 2 for n = 2, 5 and 15 measurement results per laboratory, p = 8 to 60 laboratories and γ = 0.05, 0.33, 0.67 and 1.00 and in Figure 2 for n=2 (see page 15).