INTERNATIONAL STANDARD

ISO 19983

First edition
2017-07

Rubber — Determination of precision of test methods

Caoutchouc — Détermination de la fidélité des méthodes d'essai

Reference number
ISO 19983:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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
$\sigma_M{}^2$	repeatability variance component
$\sigma_D{}^2$	day-to-day variance component
$\sigma_L{}^2$	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.

When there are two or more data from repeated tests within the same day, estimate the median values or the mean values, as appropriate, and apply them for the method B procedures.

6.8 Treatment of outliers

For treating outliers, this document adopts two measures called Mandel's h and k statistics. The h statistic is a parameter used to review the difference between averages, while the k statistics is a parameter used to review the difference between variances. It may be noted that, as well as describing the variability of the measurement method, these help in laboratory evaluation. The calculation of h and k statistic values and the table of their critical values at 5 % significance level shall be in accordance with Annex C.

If outliers are found at the 5 % significance level, there are two options:

a) discard all of the data from suspected laboratories, or

b) replace the data from suspected laboratories by repeating a single-day test at the same sites.

An example is given in D.2.

For method A, Mandel's h and k statistics are calculated by average values of repeated test data within the same day. For method B, the data are calculated without averaging or other treatment.

7 Report

Each summary precision-data table should have a heading to indicate:

— the type of precision, type 1 or type 2, used;

— the property measured and its measurement units.

For each material tested, the following shall be recorded:

a) the material identification;

b) the mean level of measured property;

c) the repeatability standard deviation, s_r;

d) the repeatability, r, in measurement units;

e) the relative repeatability, (r), in per cent of the mean level;

f) the day-to-day repeatability standard deviation, $s_{r\mathrm{D}}$;

g) the day-to-day repeatability, r_D, in measurement units;

h) the relative day-to-day repeatability, (r_D), in per cent of the mean level;

i) the reproducibility standard deviation, s_R;

j) the reproducibility, R, in measurement units;

k) the relative reproducibility, (R), in per cent of the mean level;

l) the number of laboratories in the final database used to determine the precision.

NOTE Guidance on how to use precision results is provided in Annex E.

Annex A
(normative)

Calculations for method A

A.1 General

When ITP data contain the results from repeated tests within a day, and especially when the dispersion of single-day repeated test results is in question, adopt method A for evaluation without using the mean values or the median values.

To evaluate ITP results with method A, a set of data of r-time results [indicated by k (k = 1, 2, ..., n)] from two-day tests [indicated by j (j = 1, 2, ..., q); in this document q = 2, so (j = 1, 2)] in p laboratories [indicated by i (i = 1, 2, ..., p)] is required to generate a precision table (see Table A.1).

Table A.1 — Basic data for method A

		Measurement		
Laboratory	**Day**	1	...	n
1	Day1			
	Day2			
2	Day1			
	Day2			
...	Day1			
	Day2			
i	Day1		y_{i1k}	
	Day2		y_{i2k}	
...	Day1			
	Day2			
p	Day1			
	Day2			
There is a total of p laboratories: i = 1, 2, ..., p. There is a total of two-day results for each laboratory: j = 1, 2. There is a total of r measurements for each day: k = 1, 2, ..., n.				

A.2 Generation of an ANOVA table

The following is the data structure for method A:

$$y_{ijk} = \mu + L_i + D_{ij} + M_{ijk}$$

where

μ is the population mean;

L_i is the between-laboratory variance, whose day-to-day variance component is $\sigma_L{}^2$, i.e. $V(L_i) = \sigma_L{}^2$;

D_{ij} is the day-to-day variance, whose day-to-day variance component is $\sigma_D{}^2$, i.e. $V(D_{ij}) = \sigma_D{}^2$;

M_{ijk} is the repeatability variance (measurement errors), whose repeatability variance component is $\sigma_M{}^2$, i.e. $V(M_{ijk}) = \sigma_M{}^2$;

where T_{ij}, T_i and T are defined as

$$T_{ij} = \sum_k y_{ijk} \text{ ,}$$

$$T_i = \sum_j \sum_k y_{ijk} \text{ , and}$$

$$T = \sum_i \sum_j \sum_k y_{ijk} \text{ ,}$$

the total sum of squares, S_T, the total between-laboratory sum of squares, S_L, the total day-to-day (between days) sum of squares, S_D, and the total repeatability (measurement errors) sum of squares, S_M, are determined by the following formulae:

$$S_T = \sum_i \sum_j \sum_k y_{ijk}{}^2 - \frac{T^2}{pqn}$$

$$S_L = \sum_i \frac{T_i{}^2}{qn} - \frac{T^2}{pqn}$$

$$S_D = \sum_i \sum_j \frac{T_{ij}{}^2}{n} - \sum_i \frac{T_i{}^2}{qn}$$

$$S_M = \sum_i \sum_j \sum_k y_{ijk}{}^2 - \sum_i \sum_j \frac{T_{ij}{}^2}{n}$$

while each degree of freedom ϕ_T, ϕ_L, ϕ_D and ϕ_M is

$$\phi_T = pqn - 1 = 2pn - 1 \text{ ,}$$

$$\phi_L = p - 1 \text{ ,}$$

$$\phi_D = p(q-1) = p \text{ and}$$

$$\phi_M = pq(n-1) = 2p(n-1)$$

Using these values, generate an ANOVA table as Table A.2.

Table A.2 — ANOVA table for a three-factor fully-nested experiment

Source	Sum of squares	Degree of freedom	Mean square	Expected mean square
Laboratory	S_L	$p - 1$	$V_L = S_L/(p - 1)$	$\sigma_M^2 + n\,\sigma_D^2 + 2n\sigma_L^2$
Day	S_D	p	$V_D = S_D/p$	$\sigma_M^2 + n\,\sigma_D^2$
Measurement	S_M	$2p(n - 1)$	$V_M = S_M/2p(n - 1)$	σ_M^2
Total	S_T	$2pn - 1$		
NOTE The number of days q is fixed as 2.				

A.3 Estimation of variance components

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, from the mean squares V_L, V_D, and V_M, respectively, as

$$\sigma_L^2 = \frac{1}{2n}(V_L - V_D),$$

$$\sigma_D^2 = \frac{1}{n}(V_D - V_M), \text{ and}$$

$$\sigma_M^2 = V_M$$

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 then given by the following formulae:

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

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

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

Annex B
(normative)

Calculations for method B

When evaluating ITP results with method B, a set of data from two-day tests ($j = 1, 2$) in p laboratories [indicated by i ($i = 1, 2, ..., p$)] is required to generate a precision table (see Table B.1). If there are two or more data from repeated tests within a day, estimate the median values or the mean values and apply them for the procedure.

Table B.1 — Basic data for method B

Laboratory	Day 1	Day 2	Means of day
1	y_{11}	y_{12}	
2	y_{21}	y_{22}	
…			
i	y_{i1}	y_{i2}	$\overline{y_{i\bullet}}$
…			
p	y_{p1}	y_{p2}	
There is a total of p laboratories: $i = 1, 2, ..., p$			
There is a total of two-day results for each laboratory: $j = 1, 2$			

The following is the data structure for method B:

$$y_{ij} = \mu + L_i + D_{ij}$$

where

μ is the mean population;

L_i is the between-laboratory variance, whose between-laboratory variance component is σ_L^2, i.e. $V(L_i) = \sigma_L^2$;

D_{ij} is the day-to-day variance, whose day-to-day variance component is σ_D^2, i.e. $V(D_{ij}) = \sigma_D^2$.

Calculate the day-to-day variance (between-day variance), s_D^2, and the between-laboratory variance, s_L^2, by applying the values in the obtained table to the following formulae:

$$s_D{}^2 = \frac{1}{2p}\sum_{i=1}^{p}\left(y_{i1} - y_{i2}\right)^2$$

$$s_L{}^2 = \frac{1}{p-1}\sum_{i=1}^{p}\left(\bar{y}_{ij} - \bar{\bar{y}}_j\right)^2 - \frac{s_D{}^2}{2}$$

$$= \left[\frac{p\sum\left(\bar{y}_i\right)^2 - \left(\sum\bar{y}_i\right)^2}{p\left(p-1\right)}\right] - \frac{s_D{}^2}{2}$$

NOTE This procedure assumes that the number of days, q, is fixed as 2.

From these values, the reproducibility variance, s_R^2, is obtained by the following formula:

$$s_R^2 = s_L^2 + s_D^2$$

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

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

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

Annex C
(normative)

Calculating the *h* and *k* values (Mandel's statistics)

C.1 General

When evaluating outliers, two measures called Mandel's *h* and *k* statistics are used. It may be noted that, as well as describing the variability of the measurement method, these help in laboratory evaluation.

C.2 *h*-value, for the between-laboratory dispersion

Calculate the between-laboratory consistency statistic, *h*, for each laboratory by dividing the cell deviation (cell mean minus the grand mean for that level) by the standard deviation among cell means:

$$h_i = \frac{\bar{y}_{i\bullet} - \bar{\bar{y}}}{\sqrt{\frac{1}{(p-1)} \sum_{i=1}^{p} \left(\bar{y}_{i\bullet} - \bar{\bar{y}} \right)^2}}$$

in which $\bar{y}_{i\bullet}$ is defined in Table C.1.

$$\bar{\bar{y}} = \frac{\sum_{i=1}^{p} \bar{y}_{i\bullet}}{p}$$

C.3 *k*-value, for the within-laboratory dispersion

Calculate the within-laboratory consistency statistic, *k*, by first calculating the pooled within-cell standard deviation

$$\sqrt{\frac{\sum_{i=1}^{p} s_i^2}{p}}$$

and then calculate

$$k_i = s_i \sqrt{\frac{p}{\sum_{i=1}^{p} s_i^2}}$$

for each laboratory.

Table C.1 — Format for statistics analyses

Laboratory	Day 1	Day 2	Means of day	Standard deviations of day
1				
2				
… i …	y_{i1}	y_{i2}	$\bar{y}_{i\bullet}$	s_i
p				

C.4 Statistical table

Indicators for Mandel's *h* and *k* statistic at the 5 % significance level are given in Table C.2.

Table C.2 — Critical *h*-values and *k*-values at 5 % significance level

p	h	k
3	1,15	1,65
4	1,42	1,76
5	1,57	1,81
6	1,66	1,85
7	1,71	1,87
8	1,75	1,88
9	1,78	1,90
10	1,80	1,90
11	1,82	1,91
12	1,83	1,92
13	1,84	1,92
14	1,85	1,92
15	1,86	1,93
16	1,86	1,93
17	1,87	1,93
18	1,88	1,93
19	1,88	1,93
20	1,89	1,94
NOTE The day number is fixed as 2.		

Annex D
(informative)

Example of general precision determination

D.1 General

This annex presents a detailed example of a precision determination with method A for the ITP of ISO 37, which was performed in 2002. The number of participating laboratory was eight, the number of the set of test was two days and the number of measurement was five. In addition, D.4 gives another example of how the same data as in Table D.1 can be treated with method B using averaged Day 1 and Day 2 results.

D.2 Identification of outliers

Table D.1 shows the results of the ITP (for Type 1A dumbbells), as well as supplementary statistics.

Table D.1 — Results of the ITP for ISO 37 and supplementary statistics

Laboratory	Day	Measurement					Mean of data	Mean of day	Standard deviation of day
							$\bar{y}_{ij\bullet}$	$\bar{y}_{i\bullet\bullet}$	s_i
1	Day 1	31,60	32,55	32,40	32,52	31,48	**32,110**	**32,295**	**0,262**
	Day 2	32,98	33,33	30,02	33,00	33,07	**32,480**		
2	Day 1	34,66	32,17	33,02	33,24	33,55	**33,328**	**32,839**	**0,692**
	Day 2	31,80	32,75	31,74	31,33	34,13	**32,350**		
3	Day 1	34,50	33,80	34,10	33,90	31,20	**33,500**	**34,090**	**0,834**
	Day 2	35,20	35,50	33,40	35,30	34,00	**34,680**		
4	Day 1	34,70	32,70	34,30	32,50	33,30	**33,500**	**33,870**	**0,523**
	Day 2	33,10	35,00	34,00	33,20	35,90	**34,240**		
5	Day 1	33,47	35,56	32,09	33,20	33,27	**33,518**	**33,256**	**0,371**
	Day 2	32,98	30,97	32,72	34,04	34,26	**32,994**		
6	Day 1	31,29	30,89	31,43	30,53	31,98	**31,224**	**31,385**	**0,228**
	Day 2	31,20	30,73	31,39	32,45	31,96	**31,546**		
7	Day 1	34,60	31,70	34,70	30,90	32,20	**32,820**	**32,550**	**0,382**
	Day 2	33,00	33,10	31,90	32,00	31,40	**32,280**		
8	Day 1	34,70	32,70	34,30	32,50	33,30	**33,500**	**33,870**	**0,523**
	Day 2	33,10	35,00	34,00	33,20	35,90	**34,240**		

Mandel's *h* and *k* statistics are calculated by average values of repeated test data within the same day.

Using those data in Table D.1, the h values for between-laboratory dispersion and the k values for within-laboratory dispersion were determined by the formula below. For this programme, p_j is 8:

$$h_i = \frac{\bar{y}_{i\bullet} - \bar{\bar{y}}}{\sqrt{\frac{1}{(p-1)}\sum_{i=1}^{p}\left(\bar{y}_{i\bullet} - \bar{\bar{y}}\right)^2}}$$

$$k_i = s_i\sqrt{\frac{p}{\sum_{i=1}^{p} s_i^2}}$$

The obtained h-values and k-values are shown in Table D.2 and Table D.3.

Table D.2 — Obtained *h*-values

Laboratory	h_i
1	−0,78
2	−0,19
3	1,15
4	0,91
5	0,25
6	−1,75
7	−0,50
8	0,91

Table D.3 — Obtained *k*-values

Laboratory	k_i
1	0,51
2	1,34
3	1,62
4	1,02
5	0,72
6	0,44
7	0,74
8	1,02

Each h and k value was compared with the corresponding critical values. According to Table C.2, the critical h value is 1,75 and the critical k value is 1,88 when p is 8. Since none in Tables D.2 and D.3 exceeded those values, it was concluded that there was no outlier in the results and that all the data were valid.

D.3 Precision analyses with method A

Table D.4 shows the ITP results and the supplementary statistics that were required for precision evaluation with method A.

Table D.4 — The ITP results and the supplementary statistics (method A)

Laboratory	Day	Measurement						
		M1	M2	M3	M4	M5	$T_{ij.}$	$T_{i..}$
1	Day 1	31,60	32,55	32,40	32,52	31,48	160,55	322,95
	Day 2	32,98	33,33	30,02	33,00	33,07	162,40	
2	Day 1	34,66	32,17	33,02	33,24	33,55	166,64	328,39
	Day 2	31,80	32,75	31,74	31,33	34,13	161,75	
3	Day 1	34,50	33,80	34,10	33,90	31,20	167,50	340,90
	Day 2	35,20	35,50	33,40	35,30	34,00	173,40	
4	Day 1	34,70	32,70	34,30	32,50	33,30	167,50	338,70
	Day 2	33,10	35,00	34,00	33,20	35,90	171,20	
5	Day 1	33,47	35,56	32,09	33,20	33,27	167,59	332,56
	Day 2	32,98	30,97	32,72	34,04	34,26	164,97	
6	Day 1	31,29	30,89	31,43	30,53	31,98	156,12	313,85
	Day 2	31,20	30,73	31,39	32,45	31,96	157,73	
7	Day 1	34,60	31,70	34,70	30,90	32,20	164,10	325,50
	Day 2	33,00	33,10	31,90	32,00	31,40	161,40	
8	Day 1	34,70	32,70	34,30	32,50	33,30	167,50	338,70
	Day 2	33,10	35,00	34,00	33,20	35,90	171,20	

Those values were applied to the following formulae to generate an ANOVA table (Table D.5):

$$T=\sum_i\sum_j\sum_k y_{ijk}=2\ 641{,}55$$

$$S_\mathrm{T}=\sum_i\sum_j\sum_k y_{ijk}{}^2-\frac{T^2}{pqn}=87\ 370{,}854\ 7-\frac{2\ 641{,}55^2}{8\times2\times5}=148{,}525$$

$$S_\mathrm{L}=\sum_i\frac{T_i{}^2}{qn}-\frac{T^2}{pqn}=\frac{872\ 833{,}110\ 7}{2\times5}-\frac{2\ 641{,}55^2}{8\times2\times5}=60{,}981$$

$$S_\mathrm{D}=\sum_i\sum_j\frac{T_{ij}{}^2}{n}-\sum_i\frac{T_i{}^2}{qn}=\frac{436\ 469{,}690\ 9}{5}-\frac{872\ 833{,}110\ 7}{2\times5}=10{,}627$$

$$S_\mathrm{M}=\sum_i\sum_j\sum_k y_{ijk}{}^2-\sum_i\sum_i\frac{T_{ij}{}^2}{n}=87\ 370{,}854\ 7-\frac{436\ 469{,}690\ 9}{5}=76{,}917$$

Table D.5 — Obtained ANOVA table

Source	Sum of squares	Degree of freedom	Mean square	Expected mean square
Laboratory	60,981	7	8,712	$\sigma_\mathrm{M}{}^2+5\sigma_\mathrm{D}{}^2+10\sigma_\mathrm{L}{}^2$
Day	10,627	8	1,328	$\sigma_\mathrm{M}{}^2+5\sigma_\mathrm{D}{}^2$
Measurement	76,917	64	1,202	$\sigma_\mathrm{M}{}^2$
Total	148,525	79		