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Application of ISO 5725 for the determination of repeatability and reproducibility of precision tests performed in standardization work for chemical analysis of steel

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National foreword

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Application of ISO 5725 for the determination of repeatability and reproducibility of precision tests performed in standardization work for chemical analysis of steel

Application de la norme ISO 5725 pour la détermination de la répétabilité et la reproductibilité des essais de précision réalisés en travaux de normalisation pour l'analyse chimique de l'acier

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Contents

Page

Foreword

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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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The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

Application of ISO 5725 for the determination of repeatability and reproducibility of precision tests performed in standardization work for chemical analysis of steel

1 Scope

This document describes how to determine the repeatability and reproducibility of precision tests performed within standardization work using the chemical analysis method. Specifically, this document explains the procedure for calculating precision, using precision test data of ISO 5725-3:1994, Table D.2 for the precision test in ISO 9647:1989 as an example.

The procedure of the international test for determining precision is described in ISO 5725-2 and ISO 5725-3.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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

4 Precision test

4.1 Structure of the precision test

The structure of the precision test normally used within standardization work using chemical analysis is a 3-factor, staggered-nested structure, as shown in Figure 1.

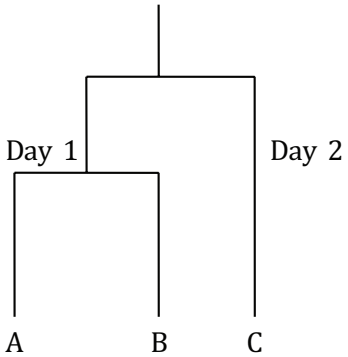


Figure 1 — Structure of the precision test

a) Both A and B (Day 1) are obtained under repeatability where independent test results are obtained with the same method on identical items in the same laboratory by the same operator using the same equipment within short intervals of time.

b) C (Day 2) is obtained under time-different intermediate precision conditions, except for the time factor. The measurement is performed by the same operator and, in addition, the measurements at a given level are performed using the same sample and equipment throughout.

4.2 Homogeneity of samples

For a precision test it is important to use homogeneous samples. Therefore, it is necessary to control the homogeneity of the samples selected for each precision test, if the samples are not certified reference materials, before starting a test in order to be sure that the heterogeneity level of the sample can be included in the expected precision values.

4.3 Number of laboratories and number of levels

In principle, the number of laboratories that participate in an international cooperative test is decided on the basis of the required precision. As this approach is often difficult to implement, the practical rule typically followed is the selection of 8 to 15 (or more) laboratories see ISO 5725-1:1994, 6.3.4, preferably in 5 countries. The number of levels depends on the range and scope of the method to be tested. A minimum of two levels by decade with the scheme (for example, 1–10) is required, and in the case of limited application ranges, three or more levels by decade (for example, 1–2–5–10) can be selected.

NOTE Fully-nested experiments offer higher reliability of repeatability than staggered-nested experiments. However, it will not improve the reliability of reproducibility significantly. From the standpoint of improving reliability, it is effective to increase the number of participating laboratories.

5 Representation of the experimental results

5.1 General

First, on the basis of precision test results, prepare the following tables and graphs.

5.2 Table of results and number of decimal places

Prepare a list of precision data.

In the list of data, the number of decimal places is the number required in the experiment plus one according to the convenor's requirement.

Table 1 shows an example of a list of data.

The data obtained by laboratory i are indicated by y_{ij} (j = 1, 2, 3).

The symbol p represents the number of laboratories participating in the experiment. (It should be noted that the number changes if outliers are deleted.)

Table 1 — Original results

Lab. no.	Day 1		Day 2
	A	B	C
1	y11	y12	y13
—	—	—	—
i	yi1	yi2	yi3
—	—	—	—
p	yp1	yp2	yp3

5.3 Graphical representation of the data

5.3.1 General

All data can be evaluated by graphical representation just to get an overview of the data population distribution. If there are laboratories which have obviously erroneous values for several levels, eliminating those laboratories as an outlier may be considered if deemed necessary.

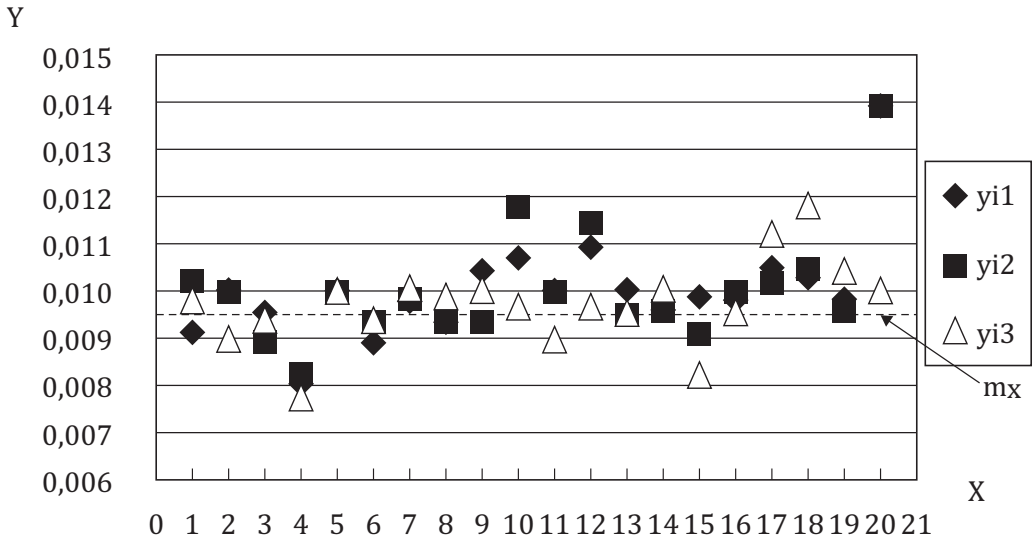
5.3.2 Data plot

Draw a graph for each of the levels by plotting the data as follows.

a) For each of the laboratories in Table 1, plot Day 1 to 1 (= yi1), Day 1 to 2 (= yi2) and Day 2 (= yi3) using different symbols.

b) Indicate the average m_x for each level.

An example of the graph for the original results is shown in Figure 2.



Key

X laboratory no.

Y contents % (mass fraction)

Figure 2 — Original results

6 Statistical evaluation

The general flow chart of the statistical evaluation is shown in Figure 3.

Perform Cochran's test and Grubbs' test following the procedure shown below to detected the outliers and delete them. The flow chart diagram of the tests is shown in Figure 4.

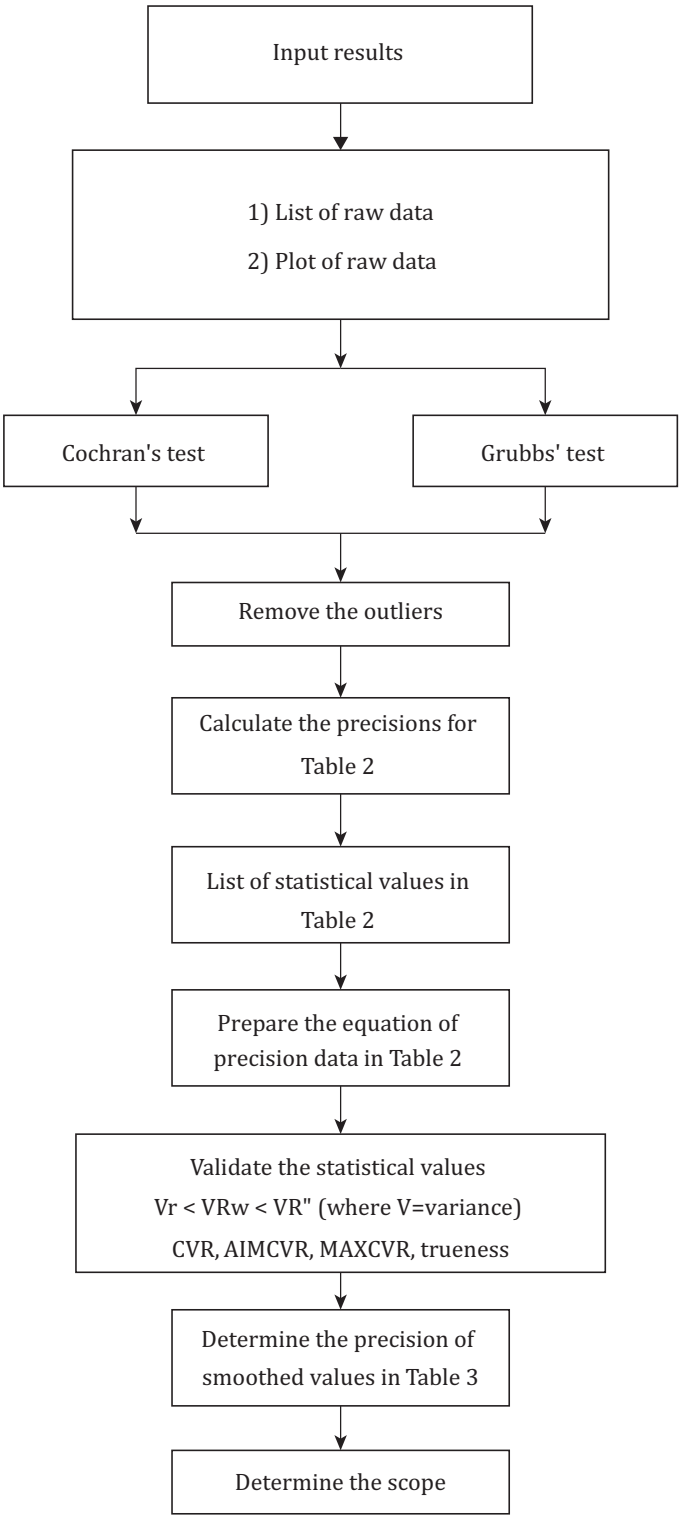


Figure 3 — Flow for determining the precision

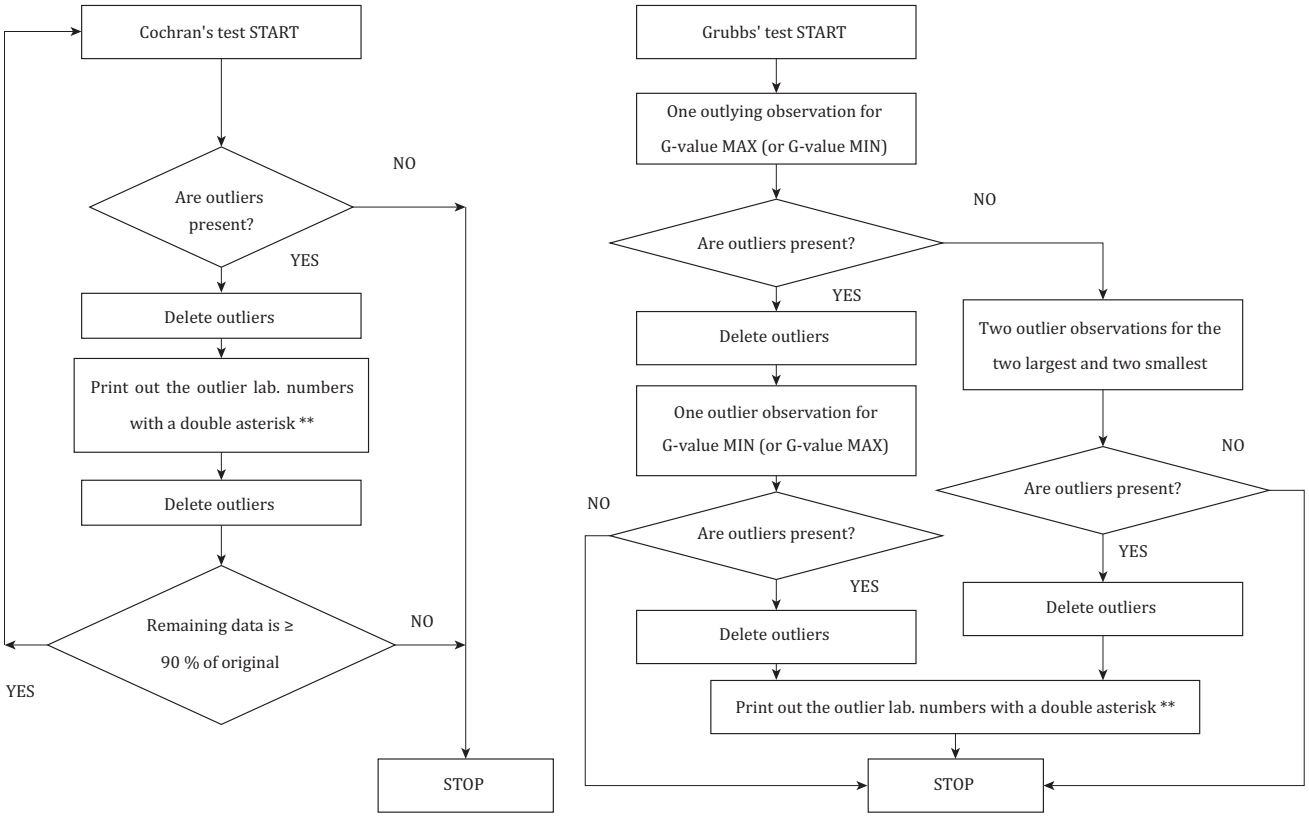


Figure 4 — Flow diagram of the Cochran's test and Grubbs' test

6.1 Cochran's test

See ISO 5725-2:1994, 7.3.3.

The purpose of the Cochran's test is to evaluate the interlaboratory repeatability variance. For that purpose, the intralaboratory repeatability variance is calculated and compared with those of other laboratories.

For each level j , perform the following calculations on each test data set. The test data sets are (A, B) and [(A+B)/2, C].

a) Obtain the standard deviation:

$$S_{ij} = \left| y_{ij1} - y_{ij2} \right| \Big/ \sqrt{2}$$

b) Calculate the C-value:

$$C = \frac{S_{\max}^2}{\sum_{i=1}^{p} S_i^2}$$

where

i is the identifier for a certain lab;

j is the identifier for a certain level;

p is the number of laboratories of level j (note that the number, p, changes if outliers are deleted);

$S_{\max}$ is the highest standard deviation in the set of level j.

c) Compare the calculated value with the value for n = 2 in the critical values table (see ISO 5725-2:1994, Table 4).

d) If the calculated value is larger than 1 % critical value, assume it is an outlier and delete the corresponding data. Then, repeat steps a) to c) for the remaining data.

e) Finish the test either when no outliers are detected or the remaining data are equal to or not less than 90 % of the original data.

NOTE A piece of statistical data greater than 1 % or 5 % of the critical value is called an "outlier" and "straggler", respectively.

6.2 Grubbs' test

6.2.1 General

See ISO 5725-2:1994, 7.3.4.

The purpose of the Grubbs' test is to evaluate the between-laboratory consistency. For that purpose, the cell average for each laboratory is obtained and evaluated in terms of the deviation from the overall average.

Using (A+B+C)/3 as the test data, perform the following calculations for each level j.

NOTE The symbols used in this clause are the same as those in 6.1

a) Obtain the average value $x_i = \bar{y}_{ij}$ of A-B-C.

b) Arrange the average values $x_i = \bar{y}_{ij}$ in ascending order.

$x_i \left(i = 1, 2, \ldots p\right)$

NOTE The number, p, changes if outliers are removed.

d) Obtain the unbiased variance for $x_i = \bar{y}_{ij}$:

$$\bar{x} = \frac{1}{p}\sum_{i=1}^{p} x_i \qquad S = \sqrt{\frac{1}{p-1}\sum_{i=1}^{p}(x_i - \bar{x})^2}$$

6.2.2 Grubbs' test for one outlier observation

See ISO 5725-2:1994, 7.3.4.1.

a) Calculate the following G-values and compare them with the appropriate value in the table of critical values (see ISO 5725-2:1994, Table 5). If the calculated value is larger than 1 % critical value, assume it is an outlier.

   1) Test of the maximum value: $G_p = (x_p - \bar{x}) \Big/ S$

   2) Test of the minimum value: $G_1 = (\bar{x} - x_1) \Big/ S$

b) If in a) above the maximum value (minimum value) is an outlier, remove it and apply the Grubb's test to the minimum value (maximum value).

c) Finish the test when no outliers are detected in steps a) and/or b). When no outliers are detected, conduct a further test for two outlier observations (6.2.2).

6.2.3 Grubbs' test for two outlier observations

See ISO 5725-2:1994, 7.3.4.2.

If in the above Grubbs' test [a) to c)] neither the maximum value nor the minimum value is an outlier, calculate the following G-values and compare them with the appropriate value in the table of critical values (see ISO 5725-2:1994, Table 5). If the calculated value is *smaller*, assume it is an outlier.

a) Test of the two largest observations: $G = S^2_{p-1,p} \Big/ S^2_0$

b) Test of the two smallest observations: $G = S^2_{1,2} \Big/ S^2_0$

where

$$S_0^2 = \sum_{i=1}^{p} (x_i - \bar{x})^2$$

$$S^2_{p-1,p} = \sum_{i=1}^{p-2} (x_i - \bar{x}_{p-1,p})^2$$

$$\bar{x}_{p-1,p} = \frac{1}{p-2} \sum_{i=1}^{p-2} x_i$$

$$S^2_{1,2} = \sum_{i=3}^{p} (x_i - \bar{x}_{1,2})^2$$

$$\bar{x}_{1,2} = \frac{1}{p-2} \sum_{i=3}^{p} x_i$$

6.3 Treatment of outlier observations

a) If a result is found to be an outlier in one of the tests, the entire laboratory data set of the appropriate level containing this result is discarded before starting the precision calculation described in 6.4.

b) If results from a laboratory are found to be outliers at several levels, consider removing the whole results from this laboratory.

c) Eliminating only a single data (A or B or C as labelled in 6.1) for a specific level of a specific laboratory is not done, since it influences the statistical calculations.

In addition to the method stipulated in the guidelines, there is a method in which the Grubbs' test is carried out on the data after the elimination of outliers by the Cochran's test. It is desirable that the statistician or convener makes the final judgement after conducting both tests, if necessary, to identify outliers.

6.4 Calculation of precision

6.4.1 Carry out the calculation of precision data by following the steps described in 6.4.2 to 6.4.8, which are based on ISO 5725-3.

In this procedure, pay attention to the following points.

a) If the estimated value of variance becomes negative during the calculation, it is assumed to be zero. A negative estimated variance is due to a small degree of freedom. Therefore, it is desirable that the number of participating laboratories should be as many as possible. It is desirable that the factors causing the negative variance be analysed first in order to ascertain that nothing unusual exists.

b) The number of decimal places of the calculated precision data are the number required for the results of the related precision test plus one. Figures are not rounded during the calculations.

6.4.2 Mean value:

$$\bar{y}_{i(1)} = \frac{1}{2}(y_{i1} + y_{i2})$$

$$\bar{y}_{i(2)} = \frac{1}{3}(y_{i1} + y_{i2} + y_{i3})$$

$$\bar{\bar{y}} = \frac{1}{p}\sum_{i} \bar{y}_{i(2)}$$

6.4.3 Range:

$$w_{i(1)} = \left|y_{i1} - y_{i2}\right| \quad w_{i(2)} = \left|\bar{y}_{i(1)} - y_{i3}\right|$$

6.4.4 Total sum of squares:

$$SST = \sum_{i}\sum_{j}(y_{ij} - \bar{\bar{y}})^2 = SS0 + SS1 + SSe$$

where

$$SS0 = 3\sum_{i}(\bar{y}_{i(2)} - \bar{\bar{y}})^2$$

$$SS1 = \frac{2}{3}\sum_{i} w_{i(2)}{}^2$$

$$SSe = \frac{1}{2}\sum_{i} w_{i(1)}{}^2$$

Divide *SS*0, *SS*1 and *SSe*, respectively, by the appropriate degree of freedom (*SS*0 = p-1, *SS*1 = p, *SSe* = p) to obtain mean squares *MS*0, *MS*1 and *MSe*.

NOTE With the *SS*0 formula given in ISO 5725-3:1994, C.1, the value of *MS*0 can become negative as a result of improper rounding when computer processing is performed incorrectly. Therefore, it is not used in these guidelines.

6.4.5 Unbiased estimated values of $\sigma_{(0)}^2$, $\sigma_{(1)}^2$ and σ_r^2:

$$s_{(0)}^2 = \frac{1}{3}MS0 - \frac{5}{12}MS1 + \frac{1}{12}MSe$$

$$s_{(1)}^2 = \frac{3}{4}MS1 - \frac{3}{4}MSe$$

$$s_r^2 = MSe$$

NOTE Any estimated values that become negative during the calculation are assumed to be zero.

6.4.6 Repeatability variance:

$$s_r^2 = MSe$$

6.4.7 Intermediate variance:

$$s_{I(1)}^2 = s_r^2 + s_{(1)}^2$$

6.4.8 Reproducibility variance:

$$s_R^2 = s_r^2 + s_{(1)}^2 + s_{(0)}^2$$

6.5 Representation of the results of the statistical evaluations

6.5.1 The results of statistical calculations are shown in Table 2, which contains the eleven items described in 6.5.2 to 6.5.12.

6.5.2 General mean.

Designation: m

Definition: $\bar{\bar{y}}$

6.5.3 Repeatability.

Designation: $\sigma(r)$

Definition: s_r

6.5.4 Within-laboratory reproducibility.

Designation: $\sigma(R_w)$

Definition: $s_{I(1)}$

6.5.5 Reproducibility.

Designation: $\sigma(R)$

Definition: s_R

6.5.6 Repeatability limit.

Designation: r

Definition: $r = 2,8 \times \sigma(r)$

6.5.7 Within-laboratory reproducibility limit

Designation: R_w

Definition: $R_w = 2,8 \times \sigma(R_w)$

6.5.8 Reproducibility limit.

Designation: R

Definition: $R = 2,8 \times \sigma(R)$

6.5.9 Coefficient of variation.

Designation: $CV(R)$

Definition: $CV(R) = \dfrac{\sigma(R)}{m} \times 100$

6.5.10 Aimed coefficient of variation.

Designation: $AIMCV(R)$

This is obtained from regression Formula (1), which is derived from the mean line shown in Figure 5:

$$\log CVR = -0,346\,6 \log m + \log 1,477\,21 \qquad (1)$$

6.5.11 Maximum coefficient of variation.

Designation: $MAXCV(R)$

This is obtained from regression Formula (2), which is derived from the mean+1s.d. line shown in Figure 5:

$$m > 0,001\,\% \quad \log CVR = -0,346\,6 \log m + \log 3,246\,70 \tag{2}$$

$$m \leq 0,001\,\% \quad CVR = 35,71\left(\text{constant}\right)$$

NOTE 1 Regression Formulae (1) and (2) are based on the experimental data. Note that the reproducibility limit (R) and unit (µg/g) in Figure 5 are expressed as reproducibility and %, respectively, in this document.

NOTE 2 Regression Formula (1), which is the regression formula for the value of R, is represented by "mean" in Figure 5.

NOTE 3 Regression Formula (2), which is the regression formula for the value of R plus the value of the standard deviation σ, is represented by "mean +1σ" in Figure 5.

6.5.12 Trueness.

Concerning trueness, the following method is used.

If many levels are judged to have a significant bias in trueness, it is advisable to check if there are any problems with test method and procedure (or values of the reference materials).

(a) For each level, calculate the difference between the reference material value, μ, and the measured mean value:

$$\hat{\delta} = \bar{y} - \mu$$

(b) Calculate the values of $\hat{\delta} - A\sigma_R$ and $\hat{\delta} + A\sigma_R$ (n = 3, number of measurements, p = number of laboratories):

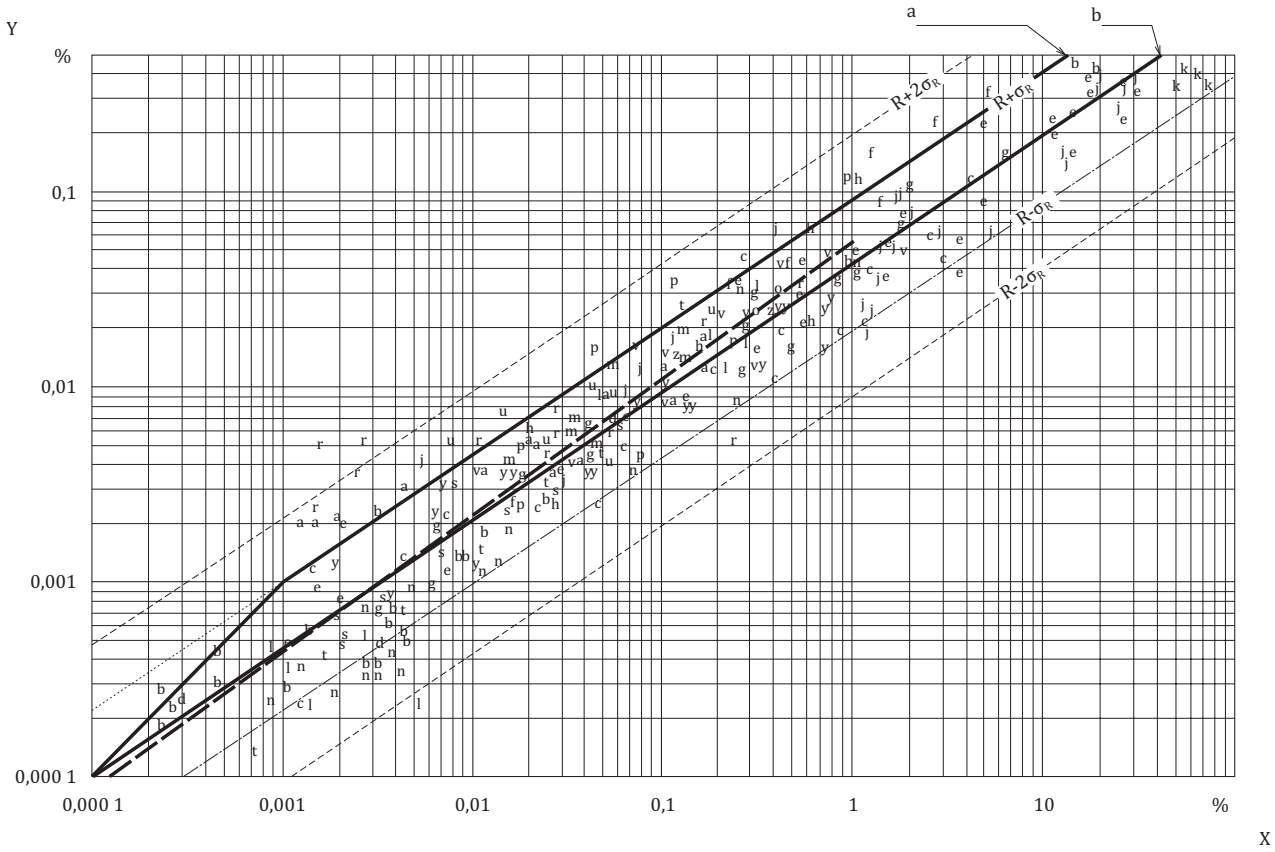
$$A = 1,96\sqrt{\frac{n(r^2 - 1) + 1}{r^2 pn}} \qquad r = \sigma_R / \sigma_r$$

(c) If 0 is included in the range $\hat{\delta} - A\sigma_R \leq \delta \leq \hat{\delta} + A\sigma_R$, judge that the measuring method has no bias. Otherwise, judge that the method has bias, and enter an asterisk (*) in the TRU line.

For sample 3 reported in Table 2, $\bar{y}$ = 0,105 9, μ = 0,10, $A\sigma_R$ = 0,000 98 and 0 is not included in the range $\hat{\delta} - A\sigma_R \leq \delta \leq \hat{\delta} + A\sigma_R$. Hence, it can be judged that there is a positive bias. For the reference material value, μ, however, it is advisable to consider the uncertainty given to it.

Table 2 — Results of the statistical analysis

Sample		**1**	**2**	**3**	**4**	**5**	**6**
Cochran's test	C1	Correct	20**	Correct	Correct	Correct	2**
	C2	20**	Correct	Correct	Correct	20**	20**
Grubbs's test	G	Correct	Correct	Correct	Correct	Correct	Correct
No. discarded		1	1	0	0	1	2
Mean	%	0,009 798	0,037 863	0,105 900	0,213 900	0,516 368	0,747 278
σ(r)	%	0,000 381	0,000 540	0,001 739	0,003 588	0,006 237	0,006 318
σ(Rw)	%	0,000 603	0,000 848	0,002 305	0,005 693	0,006 436	0,006 318
σ(R)	%	0,000 801	0,001 062	0,002 650	0,007 307	0,009 412	0,014 725
r	%	0,001 067	0,001 512	0,004 869	0,010 046	0,017 464	0,017 690
Rw	%	0,001 688	0,002 374	0,006 454	0,015 940	0,018 021	0,017 690
R	%	0,002 243	0,002 974	0,007 420	0,020 460	0,026 354	0,041 230
CV(R)	%	8,175 138	2,804 849	2,502 361	3,416 082	1,822 731	1,970 485
AIMCV(R)	%	7,340 303	4,594 443	3,216 720	2,521 106	1,857 507	1,634 155
MAXCV(R)	%	16,132 955	10,097 941	7,069 899	5,541 038	4,082 540	3,591 644
TRU				*			
NOTE C1 dataset : (A, B), C2 dataset : [(A+B)/2,C]							



Key

a	mean+1 s.d.	X	element content, C
b	mean	Y	reproducibility, R
---	"R" converted from CVR=21-log C(%)		

Figure 5 — Graphical representation of the statistical calculation results

6.6 Functions linking the level and the precision parameters

6.6.1 Use the method shown in ISO 5725-2:1994, 7.5 to obtain a regression formula. Since there are no definite rules for the use of a straight line or logarithm in the regression formula, the working group may make its own judgement. As long as there is some factor of change that can technically be explained, a jagged line (= a line with different slopes) may be used. However, to the extent possible, using a straight line is recommended. When assessing the functions linking the level and the precision parameters follow the steps described in 6.6.2 to 6.6.6.

6.6.2 Draw the values of r, R_w and R for each level.

6.6.3 Indicate the regression lines for r, R_w and R.

6.6.4 Edit the mathematical expression and correlation coefficient of the regression formula.

6.6.5 It is not appropriate to use a regression formula when the correlation coefficient is less than 0,65. In this case, use the permissible tolerance α:

$$\alpha = 2,8 \times \sqrt{\frac{1}{n}\sum_{i=1}^{n} \beta_i{}^2}$$

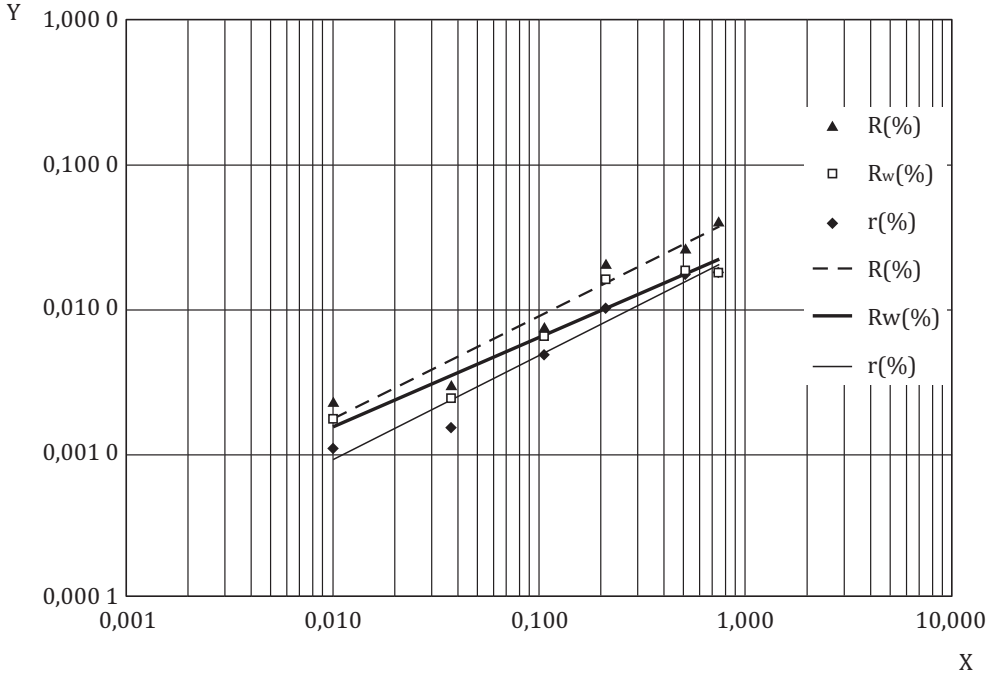
where

β is the statistical values of σ (r),σ (R_w) and σ (R) for each level.

The working group may decide that 2, 8 times the highest value of the statistical parameter under concern is a constant value, if necessary.

6.6.6 If the correlation lines of limits (r, R_w) or (R, R_w) intersect, an additional test is carried out, or if the results are adopted, the regression line of R_w alone is represented beyond the point of intersection, with the note that $R_w = r$ or $R_w = R$.

Figure 6 shows an example of the graphical representation of the precision data.



lgR = 0,7147lgx-1,339 1 R = 0,972 6

lgRw = 0,6232lgx-1,576 8 R = 0,962 8

lgr = 0,7287lgx-1,602 0 R = 0,979 5

Key

X precision % (mass fraction)

Y contents % (mass fraction)

Figure 6 — Graphical representation of the precision data

7 Determining smoothed precision and scope

On the basis of an overall judgement on the calculation results shown in Clause 6, determine the precision of the smoothed value and scope.

In this procedure, pay attention to the following points.

(a) The scope is within the range of the precision test.

(b) The scope is decided with consideration to *CV*(*R*). It is advisable to unconditionally adopt *CV*(*R*) when it is lower than *AIMCV*(*R*) and reject it when it is higher than *MAXCV*(*R*). If *CV*(*R*) is between *AIMCV*(*R*) and *MAXCV*(*R*). The working group may determine what to do, giving consideration to the economy of the experiment, although it depends more or less on the circumstances.

After determining the scope, obtain the smoothed values *r*, R_w, *R*, *CV*(*R*), *AIMCV*(*R*) and *MAXCV*(*R*) for the specified levels (for example, 5 including min. and max. content for scope) from the regression formulae and edit them in a table having a format similar to that shown in Table 3.

Table 3 — Results for the repeatability and reproducibility limits

Content mass fraction %	**Repeatability limit** *r* mass fraction %	**Reproducibility limits**		*CV(R)*	*AIMCV(R)*	*MAXCV(R)*
		R_w mass fraction %	*R* mass fraction %			
0,01	0,001	0,002	0,002	6,1	7,3	16,0
0,05	0,003	0,004	0,005	3,8	4,2	9,2
0,10	0,005	0,006	0,009	3,2	3,3	7,2
0,50	0,015	0,017	0,028	2,0	1,9	4,1
1,00	0,025	0,027	0,046	1,6	1,5	3,2

Bibliography

[1] ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*

[2] ISO 5725-2:1994, *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*

[3] ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

[4] ISO 9647:1989, *Steel and iron — Determination of vanadium content — Flame atomic absorption spectrometric method*

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