INTERNATIONAL STANDARD

ISO 3086

Fourth edition
2006-04-15

Iron ores — Experimental methods for checking the bias of sampling

Minerais de fer — Méthodes expérimentales de contrôle de l'erreur systématique d'échantillonnage

Reference number
ISO 3086:2006(E)

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Published in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 3086 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 1, *Sampling*.

This fourth edition cancels and replaces the third edition (ISO 3086:1998), which has been technically revised.

Iron ores — Experimental methods for checking the bias of sampling

1 Scope

This International Standard specifies experimental methods for checking the bias of sampling of iron ores, when sampling is carried out in accordance with the methods specified in ISO 3082, having as reference a stopped-belt sampling method.

It is recommended that an inspection of the mechanical sampling system be carried out before conducting bias testing.

Sampling systems not completely in accordance with ISO 3082 are not always expected to be biased. Therefore, bias checking may be done when there is some disagreement about the importance of some departure from the conditions of ISO 3082. If one party argues that the bias is likely to be substantial under some particular set of conditions then bias testing should mostly be done when those conditions apply.

NOTE The method for analysis of experimental data described here may also be applied:

a) for checking the bias of sample preparation of iron ores, having as reference the methods for sampling preparation according to ISO 3082;

b) for checking the bias of size distribution of iron ores by sieving, having as reference the hand sieving methods according to ISO 4701;

c) for checking a possibly significant difference in the results obtained from the samples of one lot collected at different places, for example, a loading point and unloading point.

2 Normative references

The following referenced documents are indispensable for the application 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 3082:2000, *Iron ores — Sampling and sample preparation procedures*

ISO 3085:2002, *Iron ores — Experimental methods for checking the precision of sampling, sample preparation and measurement*

ISO 11323:2002, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

4 Principle

The results obtained from the method to be checked (referred to as method B) are compared with the results of a reference method (referred to as method A) which is considered to produce practically unbiased results, from technical and empirical viewpoints.

In the event of there being no significant difference, in a statistical sense, between the results obtained by method B and method A, method B may be adopted as a routine method. This difference is assessed by comparing a 90 % confidence interval for the true average bias with the relevant bias, δ (see 5.2).

5 General conditions

5.1 The number of paired sets of measurement shall not be less than ten. The number of further tests required depends on the results of the outlier test and of the statistical analysis of the confidence interval for the true average bias, based on at least ten paired sets.

NOTE A paired set of measurement is a paired measurement data of samples, which are sampled by methods A and B, and prepared and measured in the same way, for identical material.

5.2 The relevant bias, δ, which is considered large enough to justify the likely expense of reducing the average bias, shall be decided beforehand. As a guide, δ is likely to be less than σ_{SPM}, the standard deviation for sampling, sample preparation and measurement, determined according to ISO 3085.

NOTE If the experiment is aimed at checking sample preparation only, the value of δ is likely to be less than σ_{PM}, determined according to ISO 3085.

5.3 Quality characteristics, such as total iron content, moisture content, size distribution and physical properties, may be used.

6 Sampling and sample preparation methods

6.1 Sampling

The reference method, method A, for checking the bias of sampling is a stopped-belt sampling method in accordance with ISO 3082.

> **Method A:** take each increment from the full width and thickness of the ore stream on the stopped conveyor at a specified place, for a length of belt more than three times the nominal top size or 30 mm, whichever is the greater.

The method to be checked, method B, carried out according to ISO 3082 as far as possible, shall be compared with method A for the same material.

> **Method B:** sampling methods, such as sampling from moving conveyors with a mechanical sampler and sampling during the transfer to or from ships and wagons, are examples of method B.

Samples from Methods A and B shall be taken as close together as possible. This is particularly important for ore streams which are known to be variable.

6.2 Sample preparation

6.2.1 Increments obtained from one lot, in accordance with methods A and B, are made up into two gross samples, A and B.

6.2.2 The gross samples, A and B, are subjected, in the same manner, to sample preparation as specified in ISO 3082, and tested as specified in the relevant International Standards separately, and a pair of measurements obtained.

6.2.3 The above procedure is performed on ten or more lots (see 5.1).

When increments for methods A and B can be taken from closely adjacent portions of the ore, it is recommended that sample preparation and testing be carried out on individual increments or on combinations of a small number of adjacent increments. This allows comparisons of ten or more pairs of measurements to be made more quickly than if measurements were only made on entire lots. The above comparison of measurements should be made on pairs of increments taken from several lots, preferably of the same type of ore. However, it is not permitted to combine a number of paired results, originating from both increments and gross samples. It should be either a number of pairs from increments or from gross samples.

NOTE Given the cost and inconvenience of stopped-belt sampling, it is generally economic to conduct sample preparation and measurement in duplicate and with great care so that the number of stopped-belt samples might be reduced.

7 Analysis of experimental data

NOTE The procedures described in 7.1 to 7.5 are also shown in the form of a flowsheet in Annex A (normative).

7.1 Computation of the differences

7.1.1 Denote measurements obtained in accordance with methods A and B, by $x_{\mathrm{A}i}$ and $x_{\mathrm{B}i}$, respectively. When sampling preparation and measurement have been conducted in duplicate, these measurements will be averaged.

7.1.2 Calculate the difference, d_i, between x_{Ai} and x_{Bi} using the equation:

$$d_i = x_{\mathrm{B}i} - x_{\mathrm{A}i} \qquad i = 1, 2, \ldots k \tag{1}$$

where k is the number of paired sets of measurements.

7.2 Determination of the mean and the standard deviation of the differences

7.2.1 Calculate the mean of the differences, $\bar{d}$, with one decimal place more than that used in the measurements themselves:

$$\bar{d} = \frac{1}{k} \sum d_i \tag{2}$$

7.2.2 Calculate the sum of squares, SS_d, and the standard deviation of the differences, S_d, with one decimal place more than that used in the measurements themselves:

$$\mathrm{SS}_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 \tag{3}$$

$$S_d = \sqrt{\frac{\mathrm{SS}d}{(k-1)}} \tag{4}$$

7.3 Test for outliers – Grubbs' test

7.3.1 Sort d_i into ascending order.

7.3.2 Calculate the Grubbs' test statistics G_k and G_1, using the following equations:

$$G_k = \frac{d_k - \overline{d}}{S_d} \tag{5}$$

$$G_1 = \frac{\overline{d} - d_1}{S_d} \tag{6}$$

where

d_k is the largest value of d_i;

d_1 is the smallest value of d_i;

7.3.3 Choose the larger of G_k and G_1.

7.3.4 Compare the larger of G_k and G_1 with the critical value for Grubbs' test at the 5 % significance level according to Table 1.

Table 1 — Critical values for Grubbs' outlier test

k	**Critical value (5 %)**	k	**Critical value (5 %)**	k	**Critical value (5 %)**
6	1,887	12	2,412	18	2,651
7	2,020	13	2,462	19	2,681
8	2,126	14	2,507	20	2,709
9	2,215	15	2,549	21	2,733
10	2,290	16	2,585	22	2,758
11	2,355	17	2,620	23	2,781

NOTE Critical values for Grubbs' test for a wider range of numbers of observations, and for additional significance levels, are given in Grubbs, F. E. and Beck, G. (1972) Extension of sample sizes and percentage points for significance tests of outlying observations, *Technometrics* **14**, pp. 847-854.

7.3.4.1 If the larger of G_k and G_1 is less than or equal to the critical value, conclude that there is no outlier. Proceed with 7.5.

7.3.4.2 If the larger of G_k and G_1 is larger than the critical value:

7.3.4.2.1 If the larger is G_k, conclude that the largest value of the difference, d_k, is an outlier.

7.3.4.2.2 If the larger is G_1, conclude that the smallest value of the difference, d_1, is an outlier.

7.3.5 Exclude the outlier d_i, repeat the procedure described in 7.2 to 7.3.3.

7.3.6 Compare the larger of G_k and G_1 with the critical value for Grubbs' test at 5 % significance level according to Table 1.

7.3.6.1 If the larger of G_k and G_1 is less than or equal to the critical value, conclude that there is no outlier and proceed with 7.4.

7.3.6.2 If the larger of G_k and G_1 is larger than the critical value:

7.3.6.2.1 If the larger is G_k, conclude that the largest value of the difference, d_k, is an outlier.

7.3.6.2.2 If the larger is G_1, conclude that the smallest value of the difference, d_1, is an outlier.

7.3.7 If at least 60 % of the initial set of data remain, proceed with 7.3.5.

7.3.8 If not, stop the outlier test, reinstate all outliers and proceed with 7.5.

7.4 Selection of data for use in statistical test for bias

7.4.1 Consideration of outliers whose causes are assignable

Once outliers have been detected by Grubbs' test, consideration should be given to assignable causes for those outliers, such as change in the level of moisture, partial blockage of a cutter opening, or changes in characteristics of the material being sampled.

For each outlier whose cause can be determined with reasonable confidence: If the cause is likely to occur in the future then reinstate the outlier, but if the cause is not likely to occur in the future then exclude the outlier.

7.4.2 Consideration of outliers whose causes are not assignable

If the cause of an outlier could not be determined with reasonable confidence then the outlier should be excluded.

7.4.3 Consideration of amount of data remaining

If at least 10 paired sets of measurements remain, proceed with 7.5. If not, carry out more sampling and testing to complete at least 10 paired sets of measurements, reinstate the outliers excluded, except those which have an assignable cause and are not likely to occur in the future, and repeat 7.1 to 7.4 since differences previously classified as outliers may or may not be found to be outliers when Grubbs' test is applied to the larger set of data.

7.5 Statistical test for bias

7.5.1 Determination of the confidence interval for $\bar{d}$

7.5.1.1 Calculate the mean and standard deviation of the differences which have not been rejected as outliers.

7.5.1.2 Calculate the lower limit of the confidence interval LL and the upper limit of the confidence interval UL with the same number of decimal places of that used in the measurements themselves, using the equations:

$$\mathrm{LL} = \bar{d} - t\frac{s_d}{\sqrt{k}} \tag{7}$$

$$\mathrm{UL} = \bar{d} + t\frac{s_d}{\sqrt{k}} \tag{8}$$

where

t is the value of Student's t distribution for $(k - 1)$ degrees of freedom and is given in Table 2;

k is the number of paired sets of measurements which have not been rejected as outliers.

Table 2 is prepared in such a way that when entering with a number of paired sets of measurement, k, the corresponding t value has already (k – 1) degrees of freedom.

7.5.2 Interpretation of confidence interval

Plot on a horizontal scale, with 0 (zero) in the centre, the values of LL, UL, $-\delta$ and $+\delta$.

Check if the interval between LL and UL is entirely contained in the interval between $-\delta$ and $+\delta$.

If this happens, any bias is not large enough to justify the likely expense of reducing it. Stop the test and conclude that method B may be adopted as a routine method.

If this does not happen, check if the interval between LL and UL includes 0.

If 0 is not included in this interval, then conclude that method B cannot be adopted as a routine method and the sampling system shall be adjusted.

If 0 is included in this interval, then more sampling and testing are necessary. After each new pair of results, or, if desired, several new pairs of results to reduce mobilization costs, repeat the procedure from 7.1 to 7.5 until the test conclusion on acceptance or rejection of the routine method is definite. (See Annex A.)

Table 2 — Value of *t* at 10 % level of significance (two-sided test)

Number of paired sets of measurements k	t	Number of paired sets of measurements k	t
10	1,833	26	1,708
11	1,812	27	1,706
12	1,796	28	1,703
13	1,782	29	1,701
14	1,771	30	1,699
15	1,761	31	1,697
16	1,753	32	1,696
17	1,746	33	1,694
18	1,740	34	1,692
19	1,734	35	1,691
20	1,729	40	1,685
21	1,725	50	1,677
22	1,721	81	1,664
23	1,717	121	1,658
24	1,714	241	1,651
25	1,711	∞	1,645

NOTE 1 Table 2 was based on Table 1 of ISO 2602:1980, *Statistical interpretation of test results — Estimation of the mean — Confidence interval*.

NOTE 2 Tables of t are available in a large number of statistical textbooks.

8 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) names of supervisor and personnel who performed the experiment;

c) site of experiment;

d) date of issue of test report;

e) period of experiment;

f) measured characteristic and reference to the International Standard(s) used;

g) details of the lots investigated;

h) details of sampling and sample preparation;

i) results of outlier test and conclusions;

j) t value and conclusion;

k) comments and remarks by the supervisor;

l) action taken based on the results.

Annex A
(normative)

Flowsheets of the statistical analysis

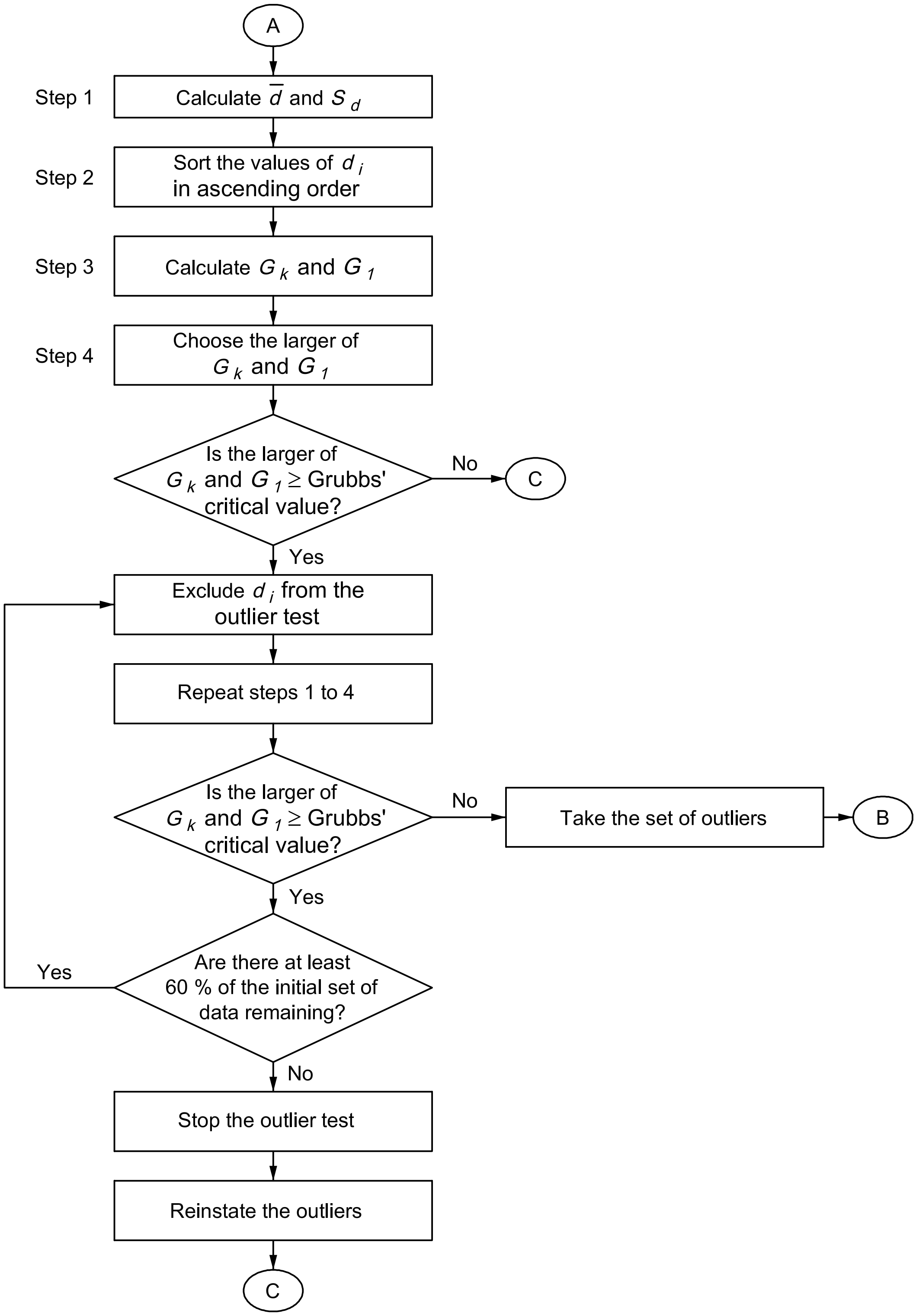


Figure A.1 — Flowsheet for Grubbs' outlier test

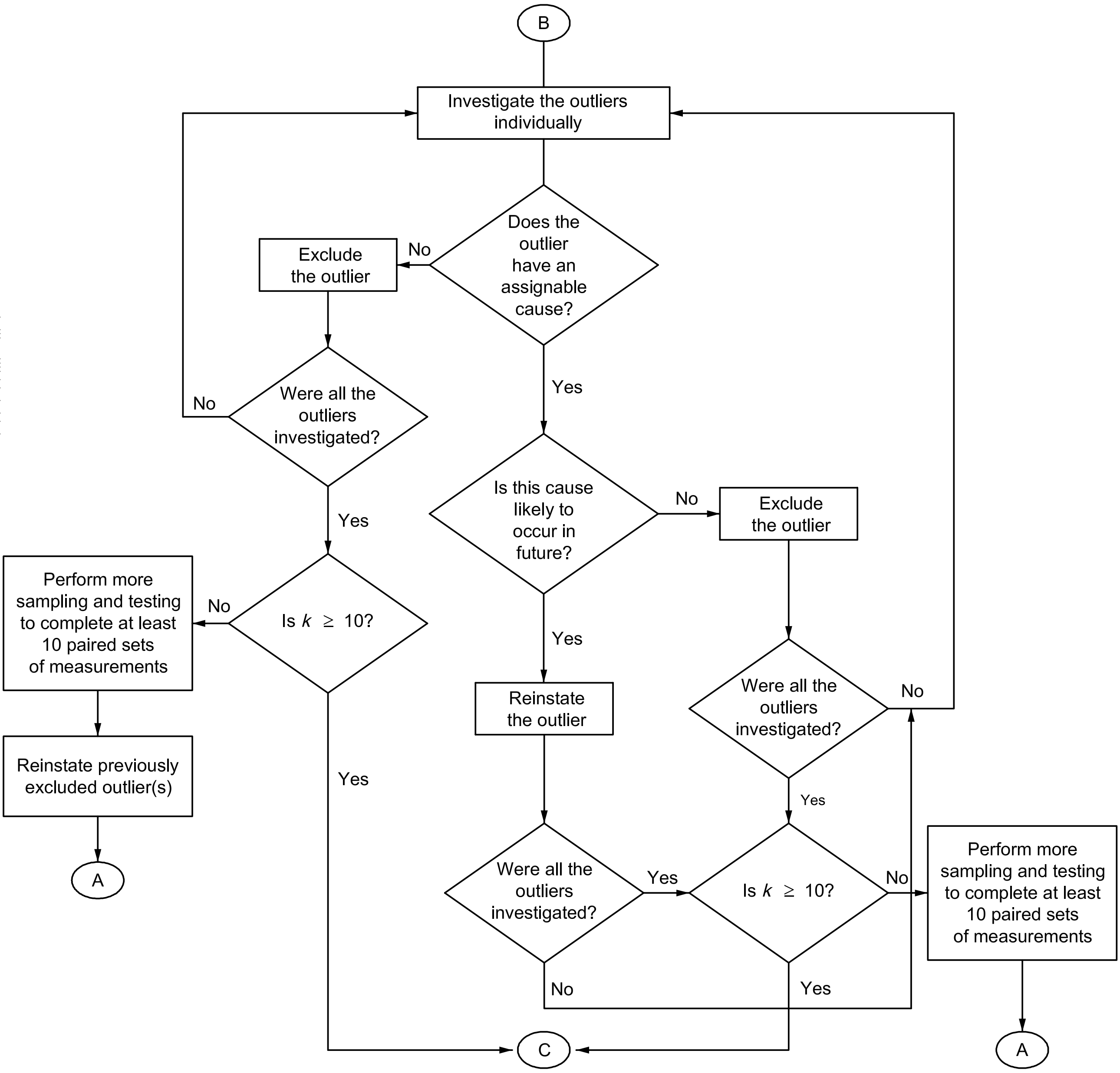


Figure A.2 — Flowsheet for consideration of outliers

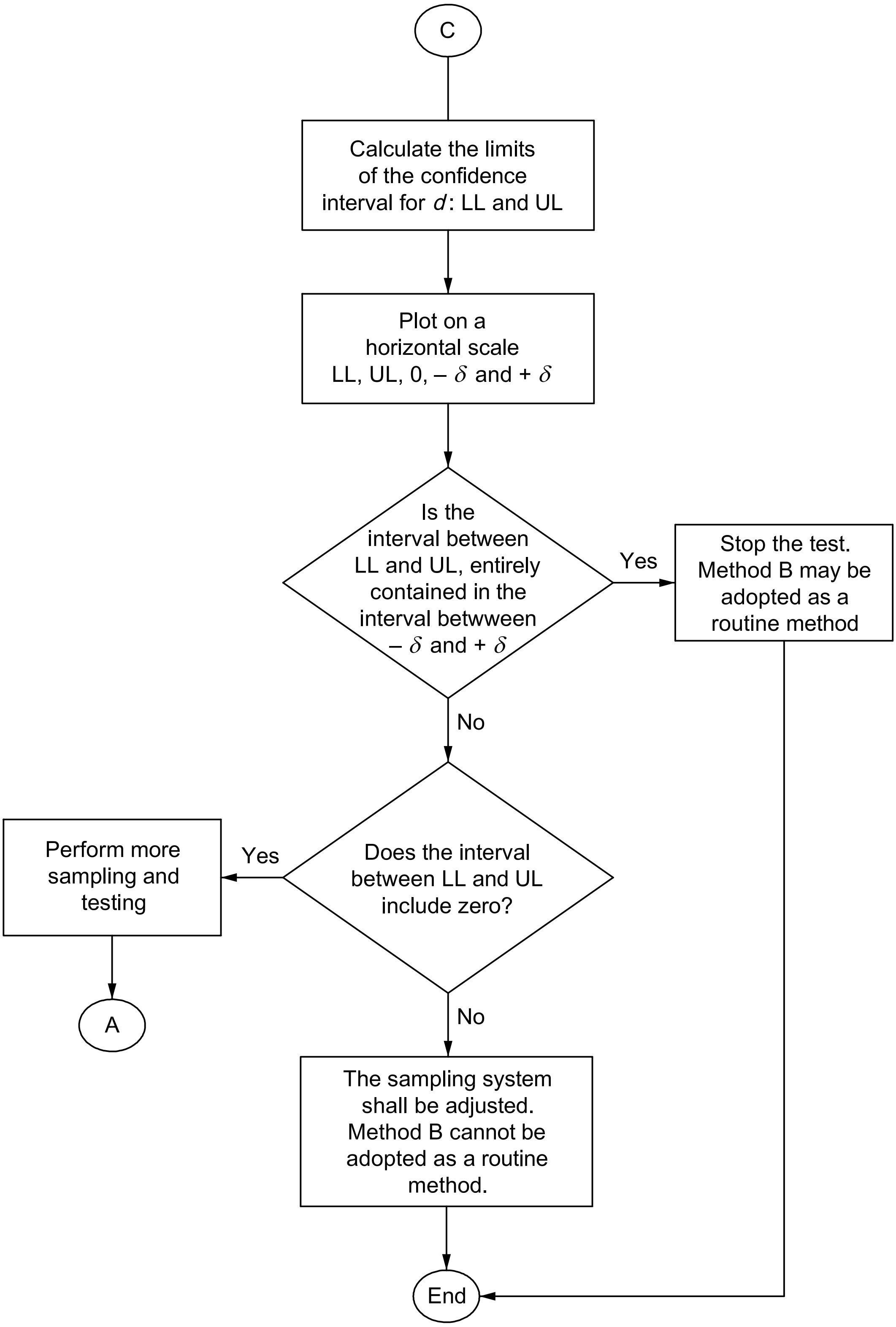


Figure A.3 — Flowsheet of the bias test

Annex B
(informative)

Numerical examples of experiments

The data shown in examples B.1 to B.5 was obtained from real experiments which have been altered by adding a constant to the results obtained by both methods (A and B) to protect the source of the data. However, the conditions of the experiments and the values of the relevant bias are examples only.

B.1 Numerical example 1

(δ: 0,10 % total iron content)

The numerical example shown in Table B.1 is the result of an experiment comparing mechanical sampling (method B) with the reference method A.

The magnitude of bias to be detected in the experiment is $\delta = 0{,}10$ % in total iron content.

Table B.1 — Experimental data

Lot	**Total iron content** (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	63,71	63,75	− 0,04	0,001 6
2	62,98	62,95	0,03	0,000 9
3	63,24	63,70	− 0,46	0,211 6
4	63,77	63,93	− 0,16	0,025 6
5	60,01	60,82	− 0,81	0,656 1
6	63,82	63,99	− 0,17	0,028 9
7	63,85	64,09	− 0,24	0,057 6
8	64,20	64,21	− 0,01	0,000 1
9	64,08	64,12	− 0,04	0,001 6
10	64,07	64,27	− 0,20	0,040 0
		Sum	− 2,10	1,024 0

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-2{,}10}{10} = -0{,}210$$

$$\mathrm{SS}_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 1{,}024 - \frac{(-2{,}10)^2}{10} = 0{,}583$$

$$s_d = \sqrt{\frac{\mathrm{SS}_d}{(k-1)}} = \sqrt{0{,}583/9} = 0{,}255$$

Test of outlier:

Sorted values of d_i: – 0,81; – 0,46; – 0,24; – 0,20; – 0,17; – 0,16; – 0,04; – 0,04; – 0,01; 0,03

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}03 - (-0{,}210)}{0{,}255} = 0{,}941$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0{,}210 - (-0{,}81)}{0{,}255} = 2{,}353$$

The larger of G_k or G_1 = 2,353

From Table 1, for 10 paired sets of measurements, the Grubbs' critical value is 2,290.

As $G_1 > 2{,}290$, it is concluded that $d_i = -0{,}81$ is an outlier.

The outlier test is then applied to the remaining 9 pairs of data.

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-1{,}29}{9} = -0{,}143$$

$$\mathrm{SS}_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 0{,}368 - \frac{(-1{,}29)^2}{9} = 0{,}183$$

$$S_d = \sqrt{\frac{\mathrm{SS}_d}{(k-1)}} = \sqrt{0{,}183/8} = 0{,}151$$

Test of outlier:

Sorted values of d_i: – 0,46; – 0,24; – 0,20; – 0,17; – 0,16; – 0,04; – 0,04; – 0,01; 0,03

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}03 - (-0{,}143)}{0{,}151} = 1{,}146$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0{,}143 - (-0{,}46)}{0{,}151} = 2{,}099$$

The larger of G_k or $G_1 = 2{,}099$

From Table 1, for 9 paired sets of measurements, the Grubbs' critical value is 2,215.

As $G_1 < 2{,}215$, it is concluded that there is no additional outlier.

Consideration of outlier

Consideration showed that the outlier ($d_i = -0{,}81$) had an assignable cause, i.e. there was a change in the characteristics of the material being sampled. Since this cause is likely to occur in the future, the pair of data should be retained. The original data (Table B.1) will therefore be submitted to the bias test.

Bias test:

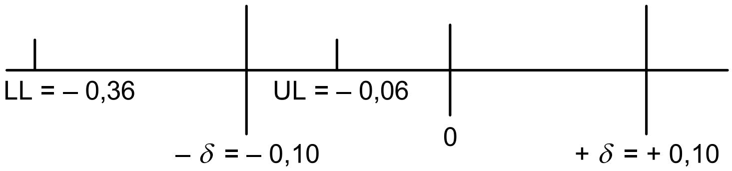
The original values of $\overline{d}$; SS_d and S_d are retained.

$$\mathrm{LL} = \overline{d} - t\frac{S_d}{\sqrt{k}} = -0{,}210 - 1{,}833\,\frac{0{,}255}{\sqrt{10}} = -0{,}36$$

$$\mathrm{UL} = \overline{d} + t\frac{S_d}{\sqrt{k}} = -0{,}210 + 1{,}833\,\frac{0{,}255}{\sqrt{10}} = -0{,}06$$

The value of $t = 1{,}833$ is taken from Table 2.

Plotting on a horizontal scale:



The interval between LL and UL is not entirely contained in the interval between $-\delta$ and $+\delta$ and does not include 0 (zero).

Therefore, there is a significant bias in method B and it cannot be adopted as a routine method. The sampling system shall be adjusted.

B.2 Numerical example 2

(δ: 0,20 % total iron content)

The numerical example shown in Tables B.2, B.3 and B.4 is the result of an experiment comparing mechanical sampling (method B), carried out in accordance with ISO 3082, with the reference method A.

The magnitude of bias to be detected in the experiment is $\delta = 0{,}20$ % in total iron content.

Table B.2 — Experimental data (10 lots)

Lot	**Total iron content** (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	62,36	62,36	0	0
2	62,18	62,21	− 0,03	0,000 9
3	62,22	62,44	− 0,22	0,048 4
4	62,32	62,27	0,05	0,002 5
5	62,43	62,51	− 0,08	0,006 4
6	62,72	62,74	− 0,02	0,000 4
7	63,58	63,79	− 0,21	0,044 1
8	63,64	63,77	− 0,13	0,016 9
9	63,85	64,15	− 0,30	0,090 0
10	63,21	63,93	− 0,72	0,518 4
		Sum	− 1,66	0,728 0

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-1{,}66}{10} = -0{,}166$$

$$SS_d = \sum d_i^{\ 2} - \frac{1}{k}(\sum d_i)^2 = 0{,}728\ 0 - \frac{(-1{,}66)^2}{10} = 0{,}452$$

$$S_d = \sqrt{\frac{SS_d}{(k-1)}} = \sqrt{\frac{0{,}452}{9}} = 0{,}224$$

Test of outlier

Sorted values of d_i: – 0,72; – 0,30; – 0,22; – 0,21; – 0,13; – 0,08; – 0,03; – 0,02; 0,00; 0,05

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}05 - (-0{,}166)}{0{,}224} = 0{,}964$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0{,}166 - (-0{,}72)}{0{,}224} = 2{,}473$$

The larger of G_k or $G_1 = 2{,}473$

From Table 1, for 10 paired sets of measurements, the Grubbs' critical value is 2,290.

As $G_1 > 2{,}290$, it is concluded that $d_i = -0{,}72$ is an outlier.

The outlier test is then applied to the remaining 9 pairs of data.

Calculating $\bar{d}$ and S_d for the remaining 9 paired sets of measurements:

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-0{,}94}{9} = -0{,}104$$

$$SS_d = \sum d_i^{\ 2} - \frac{1}{k}(\sum d_i)^2 = 0{,}209\ 6 - \frac{(-0{,}94)^2}{9} = 0{,}111$$

$$S_d = \sqrt{\frac{SS_d}{(k-1)}} = \sqrt{\frac{0{,}111}{8}} = 0{,}118$$

Test of outlier

Sorted values of d_i: – 0,30; – 0,22; – 0,21; – 0,13; – 0,08; – 0,03; – 0,02; 0,00; 0,05

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}05 - (-0{,}104)}{0{,}118} = 1{,}305$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0{,}104 - (-0{,}30)}{0{,}118} = 1{,}661$$

The larger of G_k or $G_1 = 1{,}661$.

From Table 1, for 9 paired sets of measurements, the Grubbs' critical value is 2,215.

As $G_1 < 2{,}215$, it is concluded that there is no additional outlier.

Consideration of outlier

Consideration showed that the outlier ($d_i = -0{,}72$) does not have an assignable cause. More sampling and testing is required to complete at least 10 pairs of results. The pair of results for lot 10 is reinstated with the new data, because the result of the outlier test may be different now. See Table B.3.

Table B.3 — Experimental data 8 (11 lots)

Lot	**Total iron content** (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	62,36	62,36	0	0
2	62,18	62,21	− 0,03	0,000 9
3	62,22	62,44	− 0,22	0,048 4
4	62,32	62,27	0,05	0,002 5
5	62,43	62,51	− 0,08	0,006 4
6	62,72	62,74	− 0,02	0,000 4
7	63,58	63,79	− 0,21	0,044 1
8	63,64	63,77	− 0,13	0,016 9
9	63,85	64,15	− 0,30	0,090 0
10	63,21	63,93	− 0,72	0,518 4
11	63,53	63,50	0,03	0,000 9
		Sum	− 1,63	0,728 9

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-1{,}63}{11} = -0{,}148$$

$$\mathrm{SS}_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 0{,}728\,9 - \frac{(-1{,}63)^2}{11} = 0{,}487$$

$$S_d = \sqrt{\frac{\mathrm{SS}_d}{(k-1)}} = \sqrt{0{,}487/10} = 0{,}221$$

Test of outlier

Sorted values of d_i: − 0,72; − 0,30; − 0,22; − 0,21; − 0,13; − 0,08; − 0,03; − 0,02; 0,00; 0,03; 0,05

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}05 - (-0{,}148)}{0{,}221} = 0{,}896$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0{,}148 - (-0{,}72)}{0{,}221} = 2{,}588$$

The larger of G_k or $G_1 = 2{,}588$

From Table 1, for 11 paired sets of measurements, the Grubbs' critical value is 2,355.

As $G_1 > 2{,}355$, it is concluded that $d_i = -0{,}72$ is still an outlier and shall be excluded from the outlier test.

The outlier test is then applied to the remaining 10 pairs of data.

Table B.4 — Experimental data

Lot	Total iron content (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	62,36	62,36	0	0
2	62,18	62,21	− 0,03	0,000 9
3	62,22	62,44	− 0,22	0,048 4
4	62,32	62,27	0,05	0,002 5
5	62,43	62,51	− 0,08	0,006 4
6	62,72	62,74	− 0,02	0,000 4
7	63,58	63,79	− 0,21	0,044 1
8	63,64	63,77	− 0,13	0,016 9
9	63,85	64,15	− 0,30	0,090 0
10	excluded			
11	63,53	63,50	0,03	0,000 9
		Sum	− 0,91	0,210 5

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-0{,}91}{10} = -0{,}091$$

$$SS_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 0{,}210\,5 - \frac{(-0{,}91)^2}{10} = 0{,}128$$

$$S_d = \sqrt{\frac{SS_d}{(k-1)}} = \sqrt{0{,}128/9} = 0{,}119\,1 = 0{,}119$$

Test of outlier

Sorted values of d_i: − 0,30; − 0,22; − 0,21; − 0,13; − 0,08; − 0,03; − 0,03; − 0,02; 0,00; 0,05

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}05 - (-0{,}091)}{0{,}119} = 1{,}185$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0{,}091 - (-0{,}30)}{0{,}119} = 1{,}756$$

The larger of G_k or G_1 = 1,756.

From Table 1, for 10 paired sets of measurements, the Grubbs' critical value is 2,290.

As $G_1 < 2{,}290$, it is concluded that there is no additional outlier.

Bias test

As the set of data has not changed, the last values of $\bar{d}$; SS_d and S_d are retained.

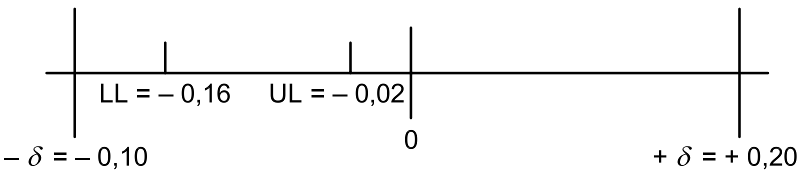
Determination of the confidence interval for $\bar{d}$:

$$\text{LL} = \bar{d} - t\frac{S_d}{\sqrt{k}} = -0{,}091 - 1{,}833\,\frac{0{,}119}{\sqrt{10}} = -0{,}16$$

$$\text{UL} = \bar{d} + t\frac{S_d}{\sqrt{k}} = -0{,}091 + 1{,}833\,\frac{0{,}119}{\sqrt{10}} = -0{,}02$$

The value of $t = 1{,}833$ is taken from Table 2.

Plotting on a horizontal scale:



The interval between LL and UL is entirely contained in the interval between $-\delta$ and $+\delta$.

Therefore, the bias is not large enough to justify the likely expense of reducing it.

Method B may be adopted as a routine method.

B.3 Numerical example 3

(δ: 0,30 % + 6,3 mm fraction)

The numerical example shown in Table B.5 is the result of an experiment comparing mechanical sampling (method B) with the reference method A.

The magnitude of bias to be detected in the experiment is $\delta = 0{,}30$ % in + 6,3 mm fraction.

Table B.5 — Experimental data

Lot	+ **6,3 mm fraction content** (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	2,62	2,63	− 0,01	0,000 1
2	3,76	2,79	0,97	0,940 9
3	1,75	1,83	− 0,08	0,006 4
4	3,08	4,04	− 0,96	0,921 6
5	1,80	2,33	− 0,53	0,280 9
6	2,38	2,82	− 0,44	0,193 6
7	2,64	3,21	− 0,57	0,324 9
8	2,25	2,08	0,17	0,028 9
9	2,35	2,48	− 0,13	0,016 9
10	2,31	2,34	− 0,03	0,000 9
		Sum	− 1,61	2,715 1

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-1{,}61}{10} = -0{,}161$$

$$\mathrm{SS}_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 2{,}715\,1 - \frac{(-1{,}61)^2}{10} = 2{,}456$$

$$S_d = \sqrt{\frac{\mathrm{SS}_d}{(k-1)}} = \sqrt{2{,}456/9} = 0{,}522$$

Test of outlier

Sorted values of d_i: – 0,96; – 0,57; – 0,53; – 0,44; – 0,13; – 0,08; – 0,03; – 0,01; 0,17; 0,97

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}97 - (-0{,}161)}{0{,}522} = 2{,}167$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0{,}161 - (-0{,}96)}{0{,}522} = 1{,}531$$

The larger of G_k or $G_1 = 2{,}167$.

From Table 1, for 10 paired sets of measurements, the Grubbs' critical value is 2,290.

As $G_k < 2{,}290$, there is no outlier.

Bias test

As there is no outlier, the values of $\bar{d}$; SS_d and S_d are retained.

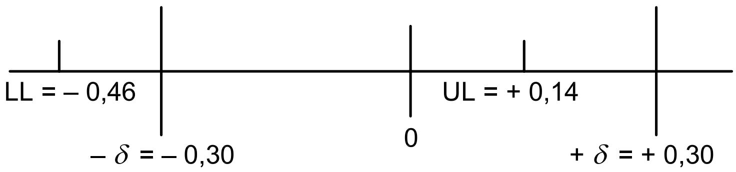
Determination of the confidence interval for $\bar{d}$:

$$\mathrm{LL} = \bar{d} - t\frac{S_d}{\sqrt{k}} = -0{,}161 - 1{,}833\,\frac{0{,}522}{\sqrt{10}} = -0{,}46$$

$$\mathrm{UL} = \bar{d} + t\frac{S_d}{\sqrt{k}} = -0{,}161 + 1{,}833\,\frac{0{,}522}{\sqrt{10}} = 0{,}14$$

The value of $t = 1{,}833$ is taken from Table 2.

Plotting on a horizontal scale:



The interval between LL and UL is not entirely contained in the interval between $-\delta$ and $+\delta$ but it includes 0 (zero).

Therefore, more sampling and testing are necessary.

B.4 Numerical example 4

(δ: 0,30 % moisture content)

The numerical example shown in Table B.6 is the result of an experiment for checking the effects of particle size and mass of test samples upon moisture content. In this experiment, mass samples of 1 kg and particle size – 10 mm (method B) were compared with mass samples of 5 kg and particle size – 22,4 mm (method A).

The magnitude of significant difference to be detected in the experiment is $\delta = 0,30$ % in moisture content.

Table B.6 — Experimental data

Lot	Name of iron ore	Moisture content (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
		x_{Bi}	x_{Ai}		
1	A	1,89	2,00	– 0,11	0,012 1
2	A	1,64	1,68	– 0,04	0,001 6
3	A	1,80	1,67	0,13	0,016 9
4	B	5,34	5,42	– 0,08	0,006 4
5	B	5,22	5,29	– 0,07	0,004 9
6	C	3,27	3,04	0,23	0,052 9
7	C	3,75	3,90	– 0,15	0,022 5
8	D	4,36	4,65	– 0,29	0,084 1
9	E	4,08	4,00	0,08	0,006 4
10	C	3,70	3,89	– 0,19	0,036 1
			Sum	– 0,49	0,243 9

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{-0,49}{10} = -0,049$$

$$\mathrm{SS}_d = \sum d_i^{\,2} - \frac{1}{k}\left(\sum d_i\right)^2 = 0,243\,9 - \frac{(-0,49)^2}{10} = 0,220$$

$$S_d = \sqrt{\frac{\mathrm{SS}_d}{(k-1)}} = \sqrt{\frac{0,220}{9}} = 0,156$$

Test of outlier

Sorted values of d_i: – 0,29; – 0,19; – 0,15; – 0,11; – 0,08; – 0,07; – 0,04; 0,08; 0,13; 0,23

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0,23 - (-0,049)}{0,156} = 1,788$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{-0,049 - (-0,29)}{0,156} = 1,545$$

The larger of G_k or $G_1 = 1,788$.

From Table 1, for 10 paired sets of measurements, the Grubbs' critical value is 2,290.

As $G_k < 2{,}290$, there is no outlier.

Bias test

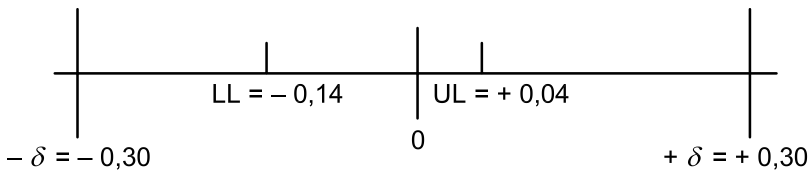
Determination of the confidence interval for $\bar{d}$:

$$\text{LL} = \bar{d} - t\frac{S_d}{\sqrt{k}} = -0{,}049 - 1{,}833\,\frac{0{,}156}{\sqrt{10}} = -0{,}14$$

$$\text{UL} = \bar{d} + t\frac{S_d}{\sqrt{k}} = -0{,}049 + 1{,}833\,\frac{0{,}156}{\sqrt{10}} = 0{,}04$$

The value of $t = 1{,}833$ is taken from Table 2.

Plotting on a horizontal scale:



The interval between LL and UL is entirely contained in the interval between $-\delta$ and $+\delta$.

Therefore, it is concluded that there is no significant difference between the two procedures.

B.5 Numerical example 5

(δ: 0,30 % total iron content)

The numerical example shown in Tables B.7 and B.8 is the result of an experiment comparing mechanical sampling (method B), carried out in accordance with ISO 3082, with the reference method A.

The magnitude of bias to be detected in the experiment is $\delta = 0{,}30$ % in total iron content.

Table B.7 — Experimental data (10 lots)

Lot	Total iron content (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	66,88	66,88	0,20	0,040 0
2	67,53	67,24	0,29	0,084 1
3	65,20	64,96	0,24	0,057 6
4	68,35	68,13	0,22	0,048 4
5	66,57	66,84	− 0,27	0,072 9
6	66,14	66,16	− 0,02	0,000 4
7	66,19	65,96	0,23	0,052 9
8	66,35	66,32	0,03	0,000 9
9	67,00	66,70	0,30	0,090 0
10	66,40	66,26	0,14	0,019 6
		Sum	1,36	0,466 8

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{1,36}{10} = 0,136$$

$$SS_d = \sum {d_i}^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 0,466\,8 - \frac{(1,36)^2}{10} = 0,282$$

$$s_d = \sqrt{\frac{SS_d}{(k-1)}} = \sqrt{\frac{0,282}{9}} = 0,177$$

Test of outlier

Sorted values of d_i: − 0,27; − 0,02; 0,03; 0,14; 0,20; 0,22; 0,23; 0,24; 0,29; 0,30.

$$G_k = \frac{d_k - \bar{d}}{s_d} = \frac{0,30 - 0,136}{0,177} = 0,927$$

$$G_1 = \frac{\bar{d} - d_1}{s_d} = \frac{0,136 - (-0,27)}{0,177} = 2,294$$

The larger of G_k or $G_1 = 2,294$

From Table 1, for 10 paired sets of measurements, the Grubbs' critical value is 2,290.

As $G_1 > 2,290$, it is concluded that $d_i = -0,27$ is an outlier.

The outlier test is then applied to the remaining 9 paired sets of results.

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{1,63}{9} = 0,181$$

$$SS_d = \sum d_i^{\ 2} - \frac{1}{k}(\sum d_i)^2 = 0{,}394 - \frac{(1{,}63)^2}{9} = 0{,}099$$

$$S_d = \sqrt{\frac{SS_d}{(k-1)}} = \sqrt{\frac{0{,}099}{8}} = 0{,}111$$

Test of outlier

Sorted values of d_i: – 0,02; 0,03; 0,14; 0,20; 0,22; 0,23; 0,24; 0,29; 0,30

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}30 - 0{,}181}{0{,}111} = 1{,}072$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{0{,}181 - (-0{,}02)}{0{,}111} = 1{,}811$$

The larger of G_k or $G_1 = 1{,}811$

From Table 1, for 9 paired sets of measurements, the Grubbs' critical value is 2,215.

As $G_1 < 2{,}215$, it is concluded that there is no additional outlier.

Consideration of outlier

Consideration showed that the outlier ($d_i = -0{,}27$) has an assignable cause, namely a sample contamination, which is not likely to occur in future and the pair of results for lot 5 shall be excluded. Therefore, more sampling and testing were performed and a new pair of results was obtained as shown in Table B.8.

Table B.8 — Experimental data (11 lots, excluding lot 5)

Lot	Total iron content (%)		$d_i = x_{Bi} - x_{Ai}$	d_i^2
	x_{Bi}	x_{Ai}		
1	66,88	66,68	0,20	0,040 0
2	67,53	67,24	0,29	0,084 1
3	65,20	64,96	0,24	0,057 6
4	68,35	68,13	0,22	0,048 4
5	excluded			
6	66,14	66,16	– 0,02	0,000 4
7	66,19	65,96	0,23	0,052 9
8	66,35	66,32	0,03	0,000 9
9	67,00	66,70	0,30	0,090 0
10	66,40	66,26	0,14	0,019 6
11	66,83	66,91	– 0,08	0,006 4
		Sum	1,55	0,400 3

Test of outlier

$$\bar{d} = \frac{1}{k}\sum d_i = \frac{1{,}55}{10} = 0{,}155$$

$$\mathrm{SS}_d = \sum d_i^2 - \frac{1}{k}\left(\sum d_i\right)^2 = 0{,}400\,3 - \frac{(1{,}55)^2}{10} = 0{,}160$$

$$S_d = \sqrt{\frac{\mathrm{SS}_d}{(k-1)}} = \sqrt{\frac{0{,}160}{9}} = 0{,}133$$

Sorted values of d_i: – 0,08; – 0,02; 0,03; 0,14; 0,20; 0,22; 0,23; 0,24; 0,29; 0,30.

$$G_k = \frac{d_k - \bar{d}}{S_d} = \frac{0{,}30 - 0{,}155}{0{,}133} = 1{,}090$$

$$G_1 = \frac{\bar{d} - d_1}{S_d} = \frac{0{,}155 - (-\,0{,}08)}{0{,}133} = 1{,}767$$

The larger of G_k or $G_1 = 1{,}767$.

From Table 1, for 10 paired sets of measurements, the Grubbs' critical value is 2,290.

As $G_1 < 2{,}290$, it is concluded that there is no outlier.

Bias test

As the set of data has not changed, the last values of $\bar{d}$; SS_d and S_d are retained.

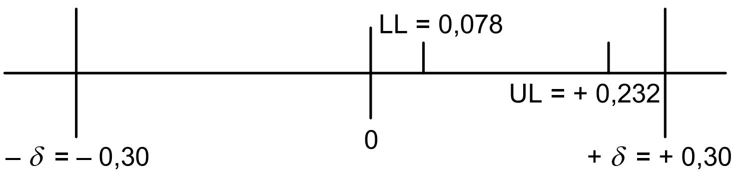
Determination of the confidence interval for $\bar{d}$:

$$\mathrm{LL} = \bar{d} - t\frac{S_d}{\sqrt{k}} = 0{,}155 - 1{,}833\,\frac{0{,}133}{\sqrt{10}} = 0{,}08$$

$$\mathrm{UL} = \bar{d} + t\frac{S_d}{\sqrt{k}} = 0{,}155 + 1{,}833\,\frac{0{,}133}{\sqrt{10}} = 0{,}23$$

The value of $t = 1{,}833$ is taken from Table 2.

Plotting on a horizontal scale:



The interval between LL and UL is entirely contained in the interval between $-\,\delta$ and $+\,\delta$.

Therefore, the bias is not large enough to justify the likely expense of reducing it.

Method B may be adopted as a routine method.

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