INTERNATIONAL STANDARD

ISO 3085

Fourth edition
2002-03-01

Iron ores — Experimental methods for checking the precision of sampling, sample preparation and measurement

Minerais de fer — Méthodes expérimentales de contrôle de la fidélité de l'échantillonnage, de préparation des échantillons et de mesurage

Reference number
ISO 3085:2002(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3085 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 which has been technically revised.

Annexes A and B of this International Standard are for information only.

Iron ores — Experimental methods for checking the precision of sampling, sample preparation and measurement

1 Scope

This International Standard specifies experimental methods for checking the precision of sampling, sample preparation and measurement of iron ores being carried out in accordance with the methods specified in ISO 3082 and the relevant ISO standards for measurement.

NOTE This International Standard may also be applied for the purpose of checking the precision of sampling, sample preparation and measurement separately.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3082:2000, *Iron ores — Sampling and sample preparation procedures*

ISO 3084:1998, *Iron ores — Experimental methods for evaluation of quality variation*

ISO 11323:—[1], *Iron ore and direct reduced iron — Vocabulary*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 11323 apply.

NOTE The precision of sampling is defined mathematically in annex A of ISO 3082:2000.

4 Principle

Sampling from twenty lots or more, preferably taking twice as many increments as specified in ISO 3082 and placing the increments alternately into two gross samples. If this is impracticable or the precision testing is carried out in conjunction with routine sampling, the normal number of increments specified in ISO 3082 may be used.

Preparation of separate test samples from each gross sample and determination of relevant quality characteristics.

Analysis of the experimental data obtained and calculation of the estimated value of the precision of sampling, sample preparation and measurement for each selected quality characteristic.

1) To be published. (Revision of ISO 11323:1996)

Comparison of the estimated precision with that specified in Table 1 of ISO 3082:2000 and necessary action if the estimated precision does not attain these specified values.

5 General conditions

5.1 Sampling

5.1.1 General

The sampling procedure to be followed shall be selected from the two methods of sampling, viz. periodic systematic sampling or stratified sampling, depending on the method of taking increments from the lot in accordance with ISO 3082.

5.1.2 Number of lots

To reach a reliable conclusion, it is recommended that the experiment be carried out on more than 20 lots of the same type of iron ore. However, if this is impracticable, at least 10 lots should be covered. If the number of lots for the experiment is not sufficient, each lot may be divided into several parts to produce more than 20 parts in total for the experiment, and the experiment should be carried out on each part, considering each part as a separate lot in accordance with ISO 3082.

5.1.3 Number of increments and number of gross samples

The number of increments required for the experiment shall preferably be twice the number specified in ISO 3082. Hence, if the number of increments required for routine sampling is n_1 and one gross sample is made up from these increments, the number of increments required for the experiment shall be $2n_1$ and two gross samples shall be constituted.

Alternatively, if the experiment is carried out as part of routine sampling, n_1 increments may be taken and two gross samples constituted, each comprising $n_1/2$ increments. In this case the sampling precision obtained will be for $n_1/2$ increments. The precision thus obtained must be divided by $\sqrt{2}$ to obtain the precision for gross samples comprising n_1 increments (see clause 7).

When the experiment is carried out with n_1 increments and n_1 is an odd number, an additional increment shall be taken in order to make the number of increments even.

5.2 Sample preparation and measurement

Sample preparation shall be carried out in accordance with ISO 3082. The measurement shall be carried out in accordance with the relevant ISO standards for chemical analysis, moisture content and size analysis of iron ores.

NOTE 1 For chemical analysis it is preferable to carry out a series of determinations on test samples for a lot over a period of several days, in order to maintain the independence of test results.

NOTE 2 The method of determination of any quality characteristic should remain the same throughout the experiment.

5.3 Replication of experiment

Even when a series of experiments has been conducted prior to regular sampling operations, the experiments should be carried out periodically to check for possible changes in quality variation and, at the same time, to control the precision of sampling, sample preparation and measurement. Because of the amount of work involved, it should be carried out as part of routine sampling, sample preparation and measurement.

5.4 Record of the experiment

For future reference and to avoid errors and omissions, it is recommended that detailed records of experiments be kept in a standardized format (see clause 9 and annex A).

6 Method of experiment

6.1 Sampling

6.1.1 Periodic systematic sampling

6.1.1.1 The number of increments, n_1, shall be determined in accordance with ISO 3082.

6.1.1.2 When $2n_1$ increments are taken, the sampling intervals, Δm, in tonnes, shall be calculated by dividing the mass, m_L, of the lot by $2n_1$, i.e. giving intervals equal to one-half of the sampling interval for routine sampling.

$$\Delta m = \frac{m_L}{2n_1}$$

Alternatively, when the experiment is carried out as part of routine sampling and n_1 increments are taken, the sampling interval, Δm, shall be calculated by dividing the mass, m_L, of the lot by n_1.

$$\Delta m = \frac{m_L}{n_1}$$

The sampling intervals thus calculated may be rounded down to the nearest 10 t.

6.1.1.3 The increments shall be taken at the sampling interval determined in 6.1.1.2, with a random start.

6.1.1.4 The increments shall be placed alternately in two containers. Thus, two gross samples, A and B, will be constituted.

EXAMPLE 1 See Figure 1.

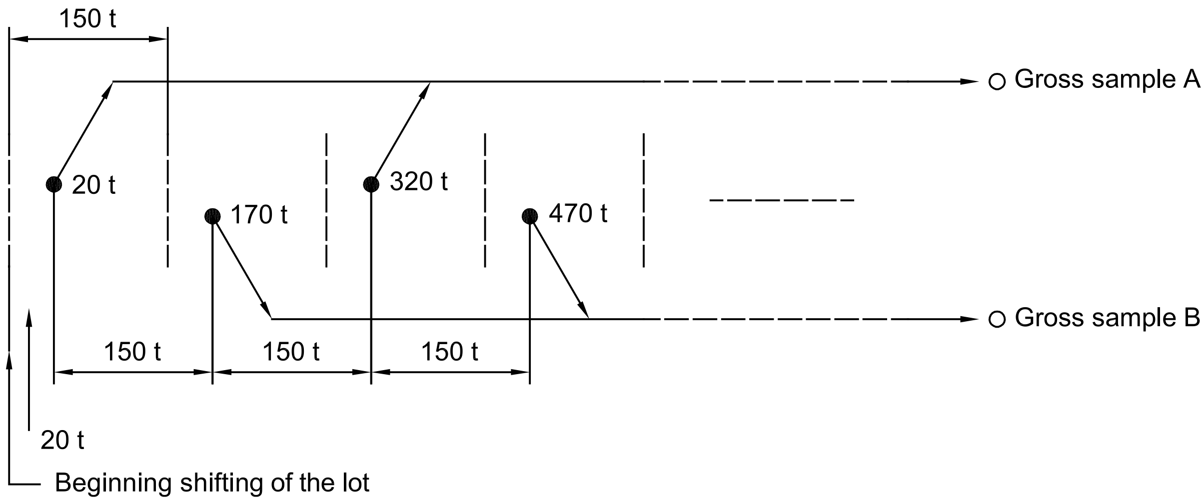
Suppose that a lot of 19 000 t is transferred by belt conveyors and the number of increments determined in accordance with ISO 3082 for routine sampling, n_1, is 60.

When $2n_1$ increments are taken, the sampling interval for the experiment, Δm, is given by the equation

$$\Delta m = \frac{m_L}{2n_1} = \frac{19\,000}{60 \times 2} = 158 \rightarrow 150$$

Thus, increments are taken at 150 t intervals. The point for taking the first increment from the first sampling interval of 150 t is determined by a random selection method. If the point for taking the first increments is determined as 20 t from the beginning of handling the lot, subsequent increments should be taken at the point $20 + i\Delta m$, where $i = 1, 2, ..., 2n_1$ (170 t, 320 t and so on). Since the whole lot size is 19 000 t, 126 increments shall be taken.

The increments are placed alternately in two containers, and two gross samples, A and B, are constituted, each composed of 63 increments.



Key

Solid circles indicate increments taken from stratum.

Open circles indicate gross samples.

Figure 1 — Schematic diagram for example 1

6.1.2 Stratified sampling

6.1.2.1 The number of increments, n_3, to be taken from each stratum shall be calculated from the number of strata, n_4, forming one lot and the number of increments determined in accordance with ISO 3082, n_1, using the equation

$$n_3 = \frac{n_1}{n_4}$$

NOTE Examples of strata, based on time, mass or space, include production periods, production masses, holds in vessels, wagons in a train or containers.

The number of increments thus calculated shall be rounded up to the next higher whole number if $2n_1$ increments are taken, or to the next higher whole even number if n_1 increments are taken.

6.1.2.2 When $2n_1$ increments are taken, $2n_3$ increments shall be taken from each stratum and shall be separated at random into two partial samples, each of n_3 increments.

Alternatively, when the experiment is carried out as part of routine sampling and n_1 increments are taken, n_3 increments shall be taken from each stratum and be separated at random into two partial samples, each of $n_3/2$ increments.

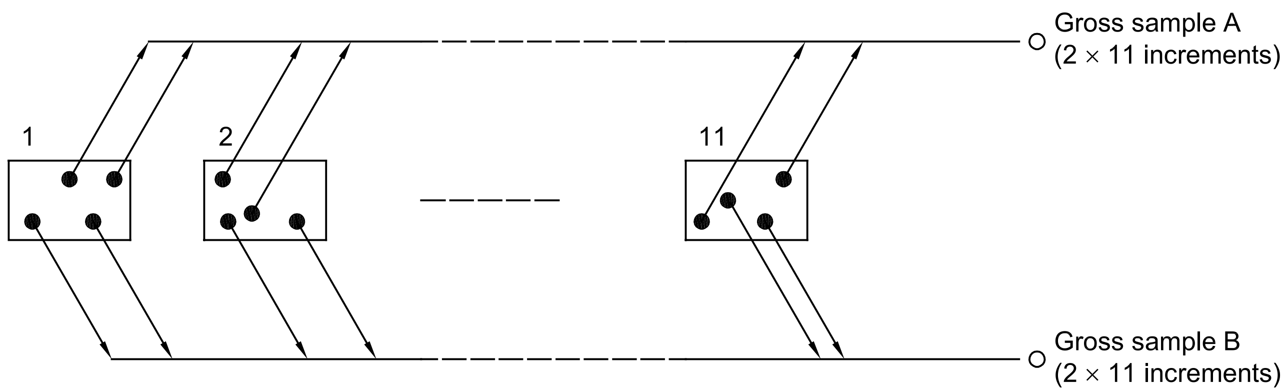
6.1.2.3 The two partial samples from each stratum shall be combined into two gross samples, A and B, respectively.

NOTE If the mass varies from stratum to stratum, the number of increments to be taken from each stratum shall be varied in proportion to the mass of ore in each stratum. This method is called “proportional stratified sampling”.

EXAMPLE 1 See Figure 2.

Suppose that a lot is divided in 11 strata each of 60 t and the number of increments, n_1, determined for the entire lot ($60 \times 11 = 660$ t) in accordance with ISO 3082 is 20. Thus, the number of increments to be taken from each stratum is

$$n_3 = \frac{n_1}{n_4} = \frac{20}{11} = 1{,}8 \rightarrow 2$$



Key

Boxes indicate strata.

Solid circles indicate increments taken from stratum.

Open circles indicate gross samples.

Figure 2 — Schematic diagram for example 2

When $2n_1$ increments are taken, four ($2n_3 = 2 \times 2$) increments are taken from each stratum and separated at random into two partial samples, each consisting of two increments.

The two partial samples from each of the 11 strata are combined into two gross samples, A and B respectively, each comprising 22 ($2n_4 = 2 \times 11$) increments.

6.2 Sample preparation and measurement

6.2.1 General

The two gross samples A and B taken in accordance with 6.1 shall be prepared separately and subjected to testing by either method 1, method 2 or method 3 described below.

6.2.2 Method 1

The two gross samples A and B shall be divided separately. The resulting four test samples, A_1, A_2, B_1 and B_2, shall be tested in duplicate. The eight tests shall be run in random order. See Figure 3.

NOTE Method 1 allows the precision of sampling, sample preparation and measurement to be separately estimated.

6.2.3 Method 2

Gross sample A shall be divided to prepare two test samples, A_1 and A_2 and one test sample shall be prepared from gross sample B. See Figure 4.

Test sample A_1 shall be tested in duplicate and single tests shall be conducted on test samples A_2 and B.

NOTE Method 2 also allows the precision of sampling, sample preparation and measurement to be separately estimated. However, the estimates are less precise than those obtained by method 1.

6.2.4 Method 3

One test sample shall be prepared from each of the two gross samples A and B, and single tests shall be conducted on each sample. See Figure 5.

NOTE Using method 3, only the overall precision of sampling, sample preparation and measurement is obtained.

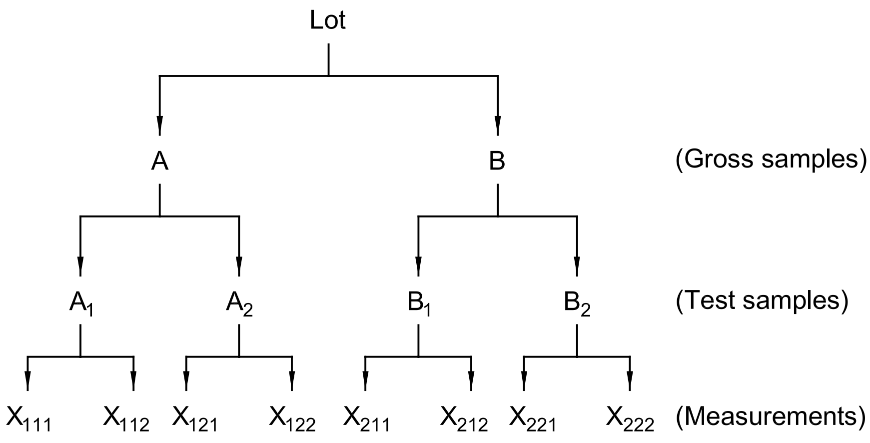


Figure 3 — Flowsheet for method 1

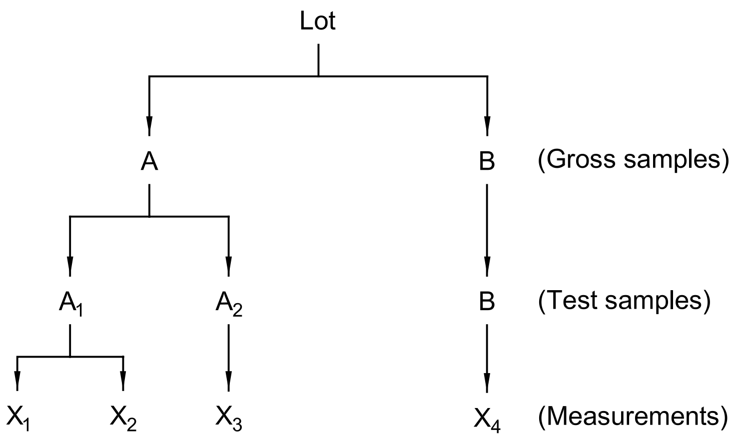


Figure 4 — Flowsheet for method 2

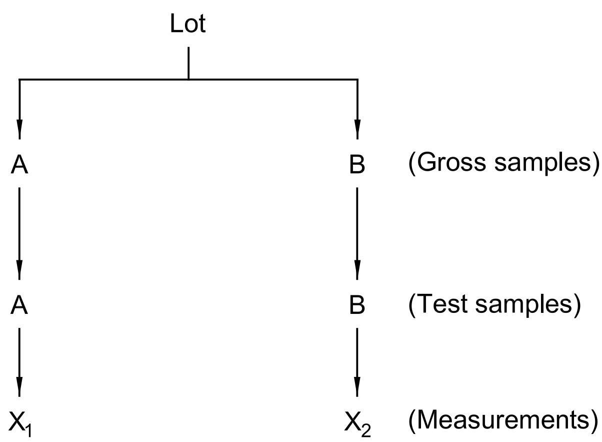


Figure 5 — Flowsheet for method 3

7 Analysis of experimental data

7.1 General

The method for analysis of experimental data shall be as specified in 7.2 to 7.4 depending on the method of sample preparation and measurement, regardless of whether the method of sampling is periodic, systematic or stratified.

7.2 Method 1

7.2.1 The estimated values of precision at the 95 % probability level (hereinafter referred to simply as precision) of sampling, sample preparation and measurement shall be calculated according to 7.2.2 to 7.2.10.

Annex A shows an example of application of method 1.

7.2.2 Denote the four measurements (such as % Fe), for the two gross samples A and B, as x_{111}, x_{112}, x_{121}, x_{122}, and x_{211}, x_{212}, x_{221}, x_{222}.

7.2.3 Calculate the mean, $\bar{x}_{ij.}$, and the range, R_1, for each pair of duplicate measurements using equations (1) and (2) respectively.

$$\bar{x}_{ij.} = \frac{1}{2}\left(x_{ij1} + x_{ij2}\right) \tag{1}$$

$$R_1 = \left|x_{ij1} - x_{ij2}\right| \tag{2}$$

where

$i = 1$ and 2 and stands for A and B gross samples;

$j = 1$ and 2 and stands for test samples.

7.2.4 Calculate the mean, $\bar{\bar{x}}_{i..}$, and the range, R_2, for each pair of duplicate samples, using equations (3) and (4) respectively.

$$\bar{\bar{x}}_{i..} = \frac{1}{2}\left(\bar{x}_{i1.} + \bar{x}_{i2.}\right) \tag{3}$$

$$R_2 = \left|\bar{x}_{i1.} - \bar{x}_{i2.}\right| \tag{4}$$

7.2.5 Calculate the mean, $\bar{\bar{\bar{x}}}$, and the range, R_3, for each pair of gross samples, A and B, using equations (5) and (6) respectively.

$$\bar{\bar{\bar{x}}} = \frac{1}{2}\left(\bar{\bar{x}}_{1..} + \bar{\bar{x}}_{2..}\right) \tag{5}$$

$$R_3 = \left|\bar{\bar{x}}_{1..} - \bar{\bar{x}}_{2..}\right| \tag{6}$$

7.2.6 Calculate the overall mean, $\bar{\bar{\bar{x}}}$, and the means of ranges, $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$, using equations (7) to (10).

$$\bar{\bar{\bar{x}}} = \frac{1}{n}\sum \bar{\bar{\bar{x}}} \tag{7}$$

$$\bar{R}_1 = \frac{1}{4n}\sum R_1 \qquad (8)$$

$$\bar{R}_2 = \frac{1}{2n}\sum R_2 \qquad (9)$$

$$\bar{R}_3 = \frac{1}{n}\sum R_3 \qquad (10)$$

where n is the number of lots.

Calculate the control limits for ranges as follows and construct range control charts.

Upper control limits for R-charts

$D_4\bar{R}_1$ (for R_1), $D_4\bar{R}_2$ (for R_2), $D_4\bar{R}_3$ (for R_3)

where $D_4 = 3{,}267$ (for a pair of measurements).

7.2.7 When all of the values of R_3, R_2 and R_1 are within the upper control limits of the R-charts, it is an indication that the processes of sampling, sample preparation and measurement of samples are in a state of statistical control.

On the other hand, when several values of R_3, R_2 or R_1 fall outside the respective upper control limits, the process (such as sampling, sample preparation or measurement) under investigation is not in a state of statistical control and should be checked in order to detect assignable causes. Such values should be excluded and the means of ranges recalculated.

7.2.8 When $2n_1$ increments are taken, calculate the estimated values of the standard deviations of measurement, $\hat{\sigma}_M$, sample preparation, $\hat{\sigma}_P$, and sampling, $\hat{\sigma}_S$, using equations (11) to (13) respectively:

$$\hat{\sigma}_M{}^2 = \left(\bar{R}_1/d_2\right)^2 \qquad (11)$$

$$\hat{\sigma}_P{}^2 = \left(\bar{R}_2/d_2\right)^2 - \frac{1}{2}\hat{\sigma}_M{}^2 \qquad (12)$$

$$\hat{\sigma}_S{}^2 = \left(\bar{R}_3/d_2\right)^2 - \frac{1}{2}\hat{\sigma}_P{}^2 - \frac{1}{4}\hat{\sigma}_M{}^2 \qquad (13)$$

where $1/d_2 = 0{,}886\,2$(for a pair of measurements).

If $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ as calculated from equations (12) and (13) is found to be negative, $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ must be replaced by zero. When n_1 increments are taken in accordance with 5.1.2, the estimated value of the standard deviation of sampling, $\hat{\sigma}_S$ from equation (13) shall be divided by $\sqrt{2}$ to obtain the standard deviation of sampling for gross samples comprising n_1 increments. The estimated values of the standard deviations of measurement and sample preparation may be calculated using equations (11) and (12).

NOTE As an alternative to using ISO 3084, the quality variation, σ_W, can be determined from the standard deviation of sampling, σ_S, as follows:

$$\sigma_W = \sqrt{n_1}\sigma_S$$

7.2.9 Calculate the estimated values of the precision of sampling $(\beta_S = 2\hat{\sigma}_S)$, sample preparation $\left(\beta_P = 2\hat{\sigma}_P\right)$ and measurement $\left(\beta_M = 2\hat{\sigma}_M\right)$.

7.2.10 Calculate the estimated value of the overall precision of sampling, sample preparation and measurement $(\beta_{SPM} = 2\hat{\sigma}_{SPM})$, using equation (14):

$$\hat{\sigma}_{SPM} = \sqrt{\hat{\sigma}_S{}^2 + \hat{\sigma}_P{}^2 + \hat{\sigma}_M{}^2} \quad (14)$$

7.3 Method 2

7.3.1 The estimated values of precision of sampling, sample preparation and measurement shall be calculated in accordance with 7.3.2 to 7.3.10.

7.3.2 Denote the four measurements as follows:

x_1, x_2 are the duplicate measurements of test sample A_1 prepared from gross sample A;

x_3 is the single measurement of test sample A_2 prepared from gross sample A;

x_4 is the single measurement of test sample B prepared from gross sample B.

7.3.3 Calculate the mean, $\bar{x}$, and the range, R_1, for each pair of duplicate measurements using equations (15) and (16).

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \quad (15)$$

$$R_1 = |x_1 - x_2| \quad (16)$$

7.3.4 Calculate the mean, $\bar{\bar{x}}$, and the range, R_2, using equations (17) and (18).

$$\bar{\bar{x}} = \frac{1}{2}(\bar{x} + x_3) \quad (17)$$

$$R_2 = |\bar{x} - x_3| \quad (18)$$

7.3.5 Calculate the mean, $\bar{\bar{\bar{x}}}$, and the range, R_3, for each pair of gross samples, A and B, using equations (19) and (20).

$$\bar{\bar{\bar{x}}} = \frac{1}{2}\left(\bar{\bar{x}} + x_4\right) \quad (19)$$

$$R_3 = \left|\bar{\bar{x}} - x_4\right| \quad (20)$$

7.3.6 Calculate the overall mean, $\bar{\bar{\bar{\bar{x}}}}$, and the means of ranges, $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$, using equations (7), (21), (22) and (10) respectively.

$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{n}\sum \bar{\bar{\bar{x}}} \quad (7)$$

$$\bar{R}_1 = \frac{1}{n}\sum R_1 \quad (21)$$

$$\bar{R}_2 = \frac{1}{n}\sum R_2 \quad (22)$$

$$\bar{R}_3 = \frac{1}{n}\sum R_3 \qquad (10)$$

where n is the number of lots.

Calculate the control limits for ranges as in 7.2.6.

7.3.7 When all the values of R_3, R_2 and R_1 are within the upper control limits of the R-charts, it is an indication that the processes of sampling, sample preparation and measurement of samples are in a state of statistical control.

On the other hand, when several values of R_3, R_2 or R_1 fall outside the respective upper control limits, the process (such as sampling, sample preparation, or measurement) under investigation is not in a state of statistical control and should be checked in order to detect assignable causes. Such values should be excluded and the means of ranges recalculated.

7.3.8 When $2n_1$ increments are taken, calculate the estimated values of the standard deviations of measurement, $\hat{\sigma}_M$, sample preparation, $\hat{\sigma}_P$, and sampling, $\hat{\sigma}_S$, using equations (11), (23) and (24) respectively.

$$\hat{\sigma}_M{}^2 = \left(\bar{R}_1/d_2\right)^2 \qquad (11)$$

$$\hat{\sigma}_P{}^2 = \left(\bar{R}_2/d_2\right)^2 - \frac{3}{4}\hat{\sigma}_M{}^2 \qquad (23)$$

$$\hat{\sigma}_S{}^2 = \left(\bar{R}_3/d_2\right)^2 - \frac{3}{4}\hat{\sigma}_P{}^2 - \frac{11}{16}\hat{\sigma}_M{}^2 \qquad (24)$$

where $1/d_2 = 0{,}886\,2$ (for a pair of measurements).

If $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ as calculated from equations (23) and (24) is found to be negative, $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ must be replaced by zero.

When n_1 increments are taken in accordance with 5.1.2, the estimated value of the standard deviation of sampling, $\hat{\sigma}_S$ from equation (24) shall be divided by $\sqrt{2}$ to obtain the standard deviation of sampling for gross samples comprising n_1 increments. The estimated values of the standard deviations of measurement and sample preparation may be calculated using equations (11) and (23).

NOTE As an alternative to using ISO 3084, the quality variation, σ_W, can be determined from the standard deviation of sampling, σ_S, as follows:

$$\sigma_W = \sqrt{n_1}\sigma_S$$

7.3.9 Calculate the estimated values of the precision of sampling $(\beta_S = 2\hat{\sigma}_S)$, sample preparation $(\beta_P = 2\hat{\sigma}_P)$ and measurement $(\beta_M = 2\hat{\sigma}_M)$.

7.3.10 Calculate the estimated value of the overall precision of sampling, sample preparation and measurement $(\beta_{SPM} = 2\hat{\sigma}_{SPM})$, using equation (14):

$$\hat{\sigma}_{SPM} = \sqrt{\hat{\sigma}_S{}^2 + \hat{\sigma}_P{}^2 + \hat{\sigma}_M{}^2} \qquad (14)$$

7.4 Method 3

7.4.1 When method 3 is applied, the estimated values of precision of sampling, sample preparation and measurement cannot be separated and only the overall precision, $2\hat{\sigma}_{SPM}$, of sampling, sample preparation and measurement is obtained.

The relationship between these precision values is

$$\hat{\sigma}_{SPM}^2 = \hat{\sigma}_S^2 + \hat{\sigma}_P^2 + \hat{\sigma}_M^2 \quad (25)$$

The estimated value of precision shall be calculated in accordance with 7.4.2 to 7.4.6.

7.4.2 Calculate the mean, $\bar{x}$ and the range, R_1, for each pair of measurements using equations (15) and (16).

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \quad (15)$$

$$R_1 = |x_1 - x_2| \quad (16)$$

Where x_1, x_2 are the measurements of test samples A and B respectively.

Calculate the overall mean, $\bar{\bar{x}}$, and the mean range, $\bar{R}$, using equations (26) and (27).

$$\bar{\bar{x}} = \frac{1}{n}\sum \bar{x} \quad (26)$$

$$\bar{R} = \frac{1}{n}\sum R \quad (27)$$

where n is the number of lots.

7.4.3 Calculate the control limits for range as follows and construct the control chart.

Upper control limit for R-chart

$$D_4\bar{R}$$

where $D_4 = 3{,}267$ (for a pair of measurements).

7.4.4 When all of the values of R are within the upper control limit of the R-chart, it is an indication that the overall process of sampling, sample preparation and measurement is in a state of statistical control.

On the other hand, when several values of R fall outside the respective upper control limits, the overall process under investigation is not in a state of statistical control and should be checked in order to detect assignable causes. Such values should be excluded and the mean of ranges recalculated.

7.4.5 When $2n_1$ increments are taken, calculate the estimated value of the overall standard deviation, $\hat{\sigma}_{SPM}$, using equation (28).

$$\hat{\sigma}_{SPM}{}^2 = (\bar{R}/d_2)^2 \quad (28)$$

where $1/d_2 = 0{,}886\,2$ (for a pair of measurements).

7.4.6 Calculate the estimated value of the overall precision, $2\hat{\sigma}_{SPM}$.

When n_1 increments are taken in accordance with 5.1.2, is not possible to convert the estimated value of the overall standard deviation, $\hat{\sigma}_{SPM}$, to the corresponding value for gross samples comprising n_1 increments, because the standard deviation of sampling cannot be separately estimated.

8 Interpretation of results and action

8.1 Interpretation of results

Compare the estimated value of the overall precision of sampling, sample preparation and measurement, $2\hat{\sigma}_{SPM}$, obtained by 7.2 (method 1), 7.3 (method 2) or 7.4 (method 3) with the overall precision, β_{SPM} specified in Table 1 of ISO 3082:2000. When the estimated value of the precision does not attain the value specified in ISO 3082, one or more of the following actions shall be taken.

8.2 Actions

8.2.1 Checking for changes in quality variation

Check for changes in quality variation of the iron ore in accordance with the method given in ISO 3084. When it is confirmed that there is a significant change in quality variation of the iron ore in question, the number of increments, n_1, to be taken from the lot must be changed in accordance with the revised quality variation using Table 2 of ISO 3082:2000.

8.2.2 Increasing number of increments

In the case of periodic systematic or stratified sampling, a greater number, n'_1, of increments may be collected from the lot. This will improve the precision of sampling in proportion to

$$\sqrt{n_1/n'_1}$$

8.2.3 Increasing mass of increments

Increasing the mass of increments generally improves precision. However, an increase in increment mass above a certain value will not significantly improve the precision of sampling.

8.2.4 Checking the sample preparation and measurement procedures

When methods 1 and 2 are applied, and the individual precision of sampling, sample preparation and measurement are estimated, it is possible to check whether one of these stages shows poor precision. Sample preparation and measurement procedures need to be checked carefully, because improvement of sample preparation operations and repeatability of the measurement method helps in obtaining better overall precision.

9 Test report

The test report shall include the following information:

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

b) site of experiment;

c) date of issue of the test report;

d) period of experiment;

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

f) details of the lots investigated;

g) details of sampling and sample preparation;

h) estimated values of the precision of sampling, sample preparation and measurement obtained by this experiment;

i) comments and remarks of the supervisor;

j) action taken based on the results.

Annex A
(informative)

Example of experiment on periodic systematic sampling by method 1

This example is based on an experiment conducted by a consumer of iron ores.

Sampling	periodic systematic sampling
Sample preparation	method 1
Quality characteristic	total iron (% Fe)

Table A.1 shows particulars of the experiment and analysis results of iron determinations. Table A.2 shows the records of % Fe and the process of calculation of $\hat{\sigma}_M$, $\hat{\sigma}_P$ and $\hat{\sigma}_S$.

Figure A.1 shows the control charts for mean and range for $\bar{x}$, $\bar{\bar{x}}$, $\bar{\bar{\bar{x}}}$ and R_1, R_2, R_3. $\bar{x}$ charts are shown only for information to indicate the fluctuation of the mean values on the chart. The control limits for the mean have been calculated using the following formulae.

Control limits for $\bar{x}$ chart

$$\bar{\bar{\bar{x}}} \pm A_2 \bar{R}_1 \quad \bar{\bar{\bar{x}}} \pm A_2 \bar{R}_2 \quad \bar{\bar{\bar{x}}} \pm A_2 \bar{R}_3$$

Where $A_2 = 1{,}88$.

The numbers of cases where points of data are situated outside the three sigma control limits are recorded in the bottom space of Table A.2, and the corresponding data are identified by asterisks.

The values of estimated standard deviations and precision of sampling, sample preparation and measurement for this example are the following.

Standard deviation and precision of sampling:

$$\hat{\sigma}_S = 0{,}23 \text{ (\% Fe)} \qquad \beta_S = 2\hat{\sigma}_S = 0{,}46 \text{ (\% Fe)}$$

Standard deviation and precision of sample preparation:

$$\hat{\sigma}_P = 0{,}11 \text{ (\% Fe)} \qquad \beta_P = 2\hat{\sigma}_P = 0{,}22 \text{ (\% Fe)}$$

Standard deviation and precision of measurement:

$$\hat{\sigma}_M = 0{,}077 \text{ (\% Fe)} \qquad \beta_M = 2\hat{\sigma}_M = 0{,}154 \text{ (\% Fe)}$$

The overall standard deviation of sampling, sample preparation and measurement, calculated by equation (14), is:

$$\hat{\sigma}_{SPM} = 0{,}27 \text{ (\% Fe) and } \beta_{SPM} = 0{,}54 \text{ (\% Fe)}$$

This value of β_{SPM} satisfies the overall precision shown in Table 1 of ISO 3082:2000 and therefore no action needs to be taken on the sampling, sample preparation and measurement procedures.

Table A.1 — Example of recording particulars of experiment

(Name of the company and works)

Report on checking the precision of sampling, sample preparation and measurement

Period of experiment: ....

Site of experiment: (location identification) ....

Characteristic measured and iron content (% Fe), ISO 2597-1:1994

International Standard used

Lots investigated

Source and type of ore: ....

Loading point: ....

Transportation medium: ship

Number of lots: 20

Mass of lots: mean 9 920 t; minimum 7 000 t; maximum 13 000 t

Particulars of sampling

Nominal top size of lots: 110 mm

Type of increment: unit mass of ore on belt conveyor; for its full cross-section over a certain length of the flow

Nominal mass of increments; 25 kg

Number of increments from a lot: $2 \times 50 = 100$

Method of taking increment: stop belt conveyor at specified tonnage interval of ore discharge and collect all ore on the belt with a shovel at specified locations to obtain a 25 kg increment.

Sample preparation

Method of making up gross samples: place alternately individual increments taken successively in two containers, and make up gross samples A and B, each comprising 50 increments.

Mass of gross samples: mean 1 250 kg; minimum 1 220 kg; maximum 1 285 kg

Type of sample preparation: method 1 (duplicate samples)

Measurement of % Fe

Statistic	Experimental results	Commercial determination	Manifested at loading point
Mean	61,10	—	—
Minimum	59,90	—	—
Maximum	63,02	—	—

Estimated precision (% Fe)

$\hat{\sigma}_M = 0{,}077$ $\beta_M = 2\hat{\sigma}_M = 0{,}154$ $\hat{\sigma}_{SPM} = 0{,}27$

$\hat{\sigma}_P = 0{,}11$ $\beta_P = 2\hat{\sigma}_P = 0{,}22$ $\beta_{SPM} = 2\hat{\sigma}_{SPM} = 0{,}54$

$\hat{\sigma}_S = 0{,}23$ $\beta_S = 2\hat{\sigma}_S = 0{,}46$

Comments and remarks:..

....

Date: .... Reported by: ....

(Name of supervisor of experiment)

Table A.2 — Example of data sheet for checking precision

Source and type of ore ....

Characteristic measured: iron content .... Period of experiment: ....

Lot No.	Date of sampling	Size of lot	Number of increments		A_1 % Fe				A_2 % Fe			
		t	A	B	x_{111}	x_{112}	$\bar{x}_{11.}$	R_1	x_{121}	x_{122}	$\bar{x}_{12.}$	R_1
1		12 100	50	50	60,92	60,99	60,96	0,07	60,98	61,01	61	0,03
2		7 300	50	50	60,88	60,87	60,88*	0,01	61,02	61,02	61,02	—
3		10 700	50	50	60,82	60,76	60,79*	0,06	60,96	60,88	60,92*	0,08
4		13 000	50	50	61,4	61,3	61,35*	0,1	61,4	61,25	61,32*	0,15
5		11 500	50	50	62,04	62	62,02*	0,04	62,27	62,44	62,36*	0,17
6		10 000	50	50	62,7	62,92	62,81*	0,22	62,9	62,72	62,81*	0,18
7		11 200	50	50	60,94	60,98	60,96	0,04	60,8	60,85	60,82*	0,05
8		9 700	50	50	60,9	60,87	60,88*	0,03	61,02	61	61,01	0,02
9		8 600	50	50	61,2	61	61,1	0,2	61,08	61,08	61,08	—
10		9 300	50	50	60,94	61,07	61	0,13	61	61	61	—
11		8 300	50	50	59,94	59,9	59,92*	0,04	60,02	60,09	60,06*	0,07
12		10 500	50	50	60,08	60,04	60,06*	0,04	60,14	60,26	60,2*	0,12
13		8 200	50	50	60,38	60,23	60,3*	0,15	60,3	60,3	60,3*	—
14		10 600	50	50	61,1	61	61,05	0,1	61	61,02	61,01	0,02
15		9 100	50	50	62	61,93	61,96*	0,07	62,32	62,27	62,3*	0,05
16		10 400	50	50	60,72	60,78	60,75*	0,06	61,14	61,14	61,14	—
17		7 900	50	50	61,5	61,42	61,46*	0,08	62,02	62,07	62,04*	0,05
18		11 200	50	50	61,08	60,94	61,01	0,14	61,04	60,96	61	0,08
19		11 800	50	50	61,15	61,3	61,22	0,15	61,1	61,08	61,09	0,02
20		7 000	50	50	61,54	61,32	61,43*	0,22	61,5	61,26	61,38*	0,24
Sum		198 400	1 000	1 000	1 222,23	1 221,62	1 221,91	1,95	1 224,01	1 223,7	1 223,86	1,33
Mean		9 920	50	50	61,11	61,08	61,10	0,10	61,20	61,18	61,19	0,07

Calculation

$\hat{\sigma}^2{}_M = \left(0,886\,2\bar{R}_1\right)^2 = 0,005\,9$ $\left(0,886\,2\bar{R}_2\right)^2 = 0,032\,3$ $\left(0,886\,2\bar{R}_3\right)^2 = 0,072\,1$

$\hat{\sigma}_M = 0,077$ $\hat{\sigma}^2_P = 0,032\,3 - \dfrac{0,005\,9}{2} = 0,029\,4$ $\hat{\sigma}^2_S = 0,072\,1 - \dfrac{0,029\,4}{2} - \dfrac{0,005\,9}{4} = 0,056$

$\hat{\sigma}_P = 0,171$ $\hat{\sigma}_S = 0,237$

$\bar{\bar{\bar{x}}} \pm 1,88\,0\bar{R}_1 = 61,10 \pm 0,164$ (61,26 and 60,94) $\bar{\bar{\bar{x}}} \pm 1,880\bar{R}_2 = 61,10 \pm 0,382$ (61,48 and 60,72)

Adjustment for calculated values

Individual % Fe identified by asterisk (*) are outside the 3 sigma control limits.

Number of cases where % Fe fell outside the limits are

R_1: 0 out of 80 data (simplify as 0/80), R_2: 3/40, R_3: 0/20, $\bar{x}$: 57/80, $\bar{\bar{x}}$: 21/40, $\bar{\bar{\bar{x}}}$: 7/20

There are three outliers on the R_2 chart. Recalculate R_2 until no more outliers exist.

$\hat{\sigma}^2_M = 0,0059$ First adjustment for R_2: Second adjustment for R_2:

$\hat{\sigma}_M = 0,077$ $\bar{R}'_2 = 0,148$ $\bar{R}''_2 = 0,136$

$3,267\bar{R}'_2 = 0,484$ (one point outside the UCL) $3,267\bar{R}''_2 = 0,444$ (No point outside the UCL)

(see 7.2 and Figure 3)

Date: ....

Number of lots: 20 .... No. ....

A % Fe		B_1 % Fe				B_2 % Fe				B % Fe			
$\bar{\bar{x}}_{1..}$	R_2	x_{211}	x_{212}	$\bar{x}_{21.}$	R_1	x_{221}	x_{222}	$\bar{x}_{22.}$	R_1	$\bar{\bar{x}}_{2..}$	R_2	$\bar{\bar{\bar{x}}}$	R_3
60,98	0,04	61,4	61,34	61,37*	0,06	61,28	61,35	61,32*	0,07	61,34	0,05	61,16	0,36
60,95	0,14	60,27	60,1	60,18*	0,17	60,04	59,93	59,98*	0,11	60,08*	0,2	60,52*	0,87
60,86	0,13	60,7	60,67	60,68*	0,03	60,82	60,6	60,71*	0,22	60,7*	0,03	60,78	0,16
61,34	0,03	61,94	61,97	61,96*	0,03	61,6	61,43	61,52*	0,17	61,74*	0,44	61,54	0,4
62,19*	0,34	61,92	61,77	61,84*	0,15	62,51	62,52	62,52*	0,01	62,18*	0,68[a]	62,18*	0,01
62,81*	—	63,02	62,94	62,98*	0,08	62,98	62,92	62,95*	0,06	62,96*	0,03	62,88*	0,15
60,89	0,14	61,14	61,2	61,17	0,06	60,94	61,03	60,98	0,09	61,08	0,19	60,98	0,19
60,94	0,13	60,9	60,88	60,89*	0,02	60,7	60,5	60,6*	0,2	60,74	0,29	60,84	0,2
61,09	0,02	60,88	60,64	60,76*	0,24	60,6	60,55	60,58*	0,05	60,67*	0,18	60,88	0,42
61	—	61	61	61	—	59,95	59,87	59,91*	0,08	60,46*	1,09[a]	60,73	0,54
59,99*	0,14	59,96	60,02	59,99*	0,06	59,98	59,9	59,94*	0,08	59,96*	0,05	59,98*	0,03
60,13*	0,14	60,52	60,6	60,56*	0,08	60,46	60,35	60,4*	0,11	60,48*	0,16	60,3*	0,35
60,3*	—	60,28	60,18	60,23*	0,1	60,29	60,32	60,3*	0,03	60,26*	0,07	60,28*	0,04
61,03	0,04	60,84	60,66	60,75*	0,18	61,12	60,96	61,04	0,16	60,9	0,29	60,96	0,13
62,13*	0,34	61,8	61,74	61,77*	0,06	61,74	61,71	61,72*	0,03	61,74*	0,05	61,94*	0,39
60,94	0,39	60,82	60,74	60,78*	0,06	60,56	60,38	60,47*	0,18	60,62*	0,31	60,78	0,32
61,75*	0,58	61,06	61,04	61,05	0,02	61,16	61,25	61,2	0,09	61,12	0,15	61,44	0,63
61	0,01	60,78	60,8	60,79*	0,02	60,88	60,89	60,88*	0,01	60,84	0,09	60,92	0,16
61,16	0,13	62	62,05	62,02*	0,05	61,21	61,12	61,16	0,09	61,59*	0,86*	61,38	0,43
61,4	0,05	61,86	61,6	61,73*	0,26	61,66	61,58	61,62*	0,08	61,68*	0,11	61,54	0,28
1 222,88	2,79	1 223,09	1 221,94	1 222,5	1,75	1 220,48	1 219,16	1 219,8	1,92	1 221,14	5,32	1 222,01	6,06
61,14	0,14	61,15	61,10	61,12	0,08	61,02	60,96	60,99	0,10	61,06	0,26	61,10	0,30

$\bar{\bar{\bar{x}}} = 61{,}10$ $\bar{R}_1 = 0{,}087$ $3{,}267\bar{R}_1 = 0{,}284$

$\bar{R}_2 = 0{,}203$ $3{,}267\bar{R}_2 = 0{,}664$

$\bar{R}_3 = 0{,}303$ $3{,}267\bar{R}_3 = 0{,}991$

$\bar{\bar{\bar{x}}} \pm 1{,}880\bar{R}_3 = 61{,}10 \pm 0{,}570$ (61,67 and 60,53)

$\left(0{,}886\ 2\bar{R}''_2\right)^2 = 0{,}014\ 5$ $\left(0{,}886\ 2\bar{R}'_3\right)^2 = 0{,}060\ 7$

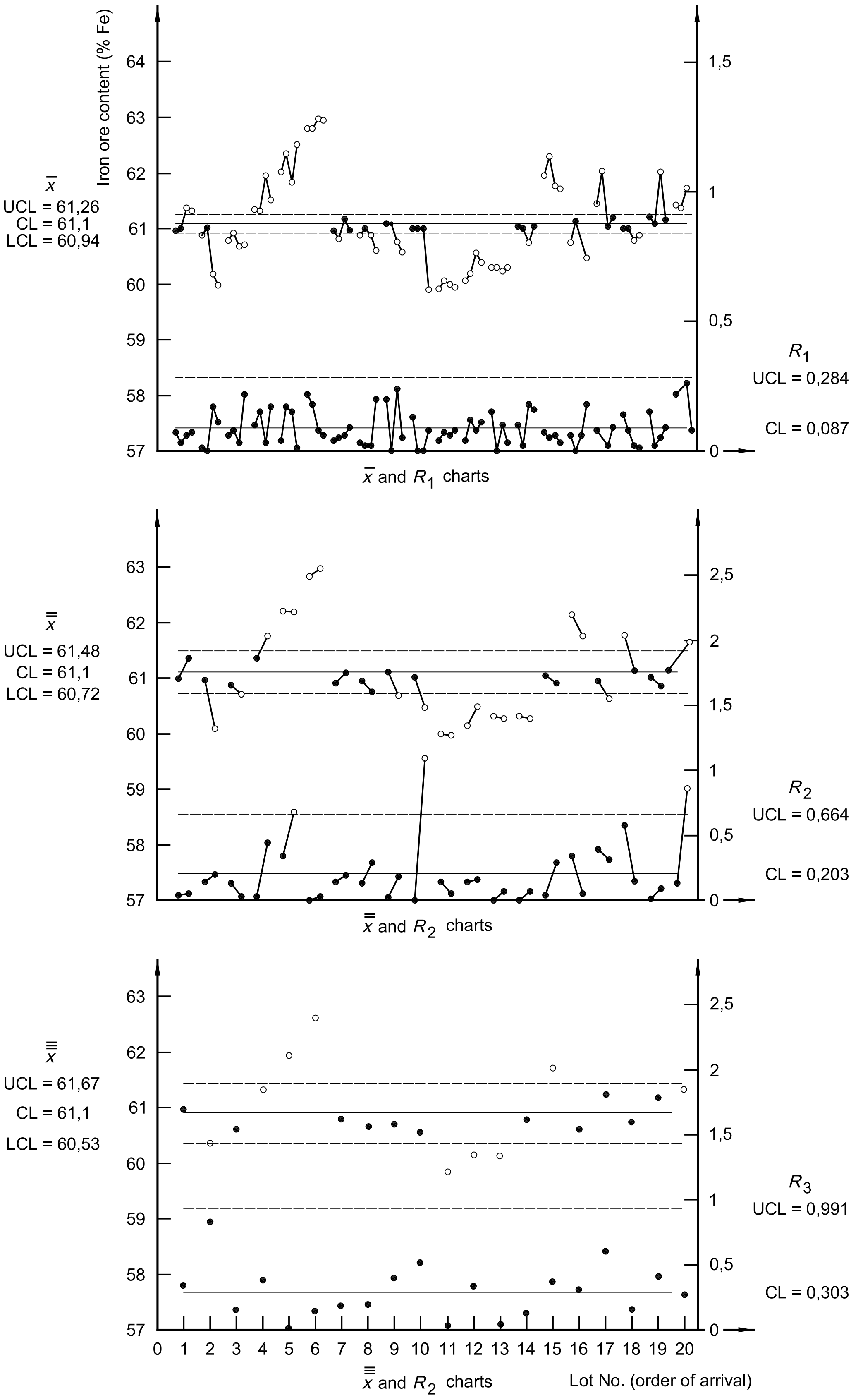
$\hat{\sigma}_P = 0{,}107\ 5$ $\hat{\sigma}_S = 0{,}231\ 2$

Comments and remarks:

Recorded by ....

Checked by..

(Name of supervisor of experiment)



Iron ore content (% Fe)
x̄
UCL = 61,26
CL = 61,1
LCL = 60,94
64
63
62
61
60
59
58
57
1,5
1
0,5
0
R1
UCL = 0,284
CL = 0,087
x̄ and R1 charts
x̿
UCL = 61,48
CL = 61,1
LCL = 60,72
63
62
61
60
59
58
57
2,5
2
1,5
1
0,5
0
R2
UCL = 0,664
CL = 0,203
x̿ and R2 charts
UCL = 61,67
CL = 61,1
LCL = 60,53
63
62
61
60
59
58
57
2,5
2
1,5
1
0,5
0
R3
UCL = 0,991
CL = 0,303
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
x and R2 charts
Lot No. (order of arrival)

Key

° Data outside the control limit

UCL Upper control limit

CL Central line

LCL Lower control limit

Figure A.1 — Example of control charts for mean and range (graphical presentation of data in Table A.2)

Annex B
(informative)

Alternative method for analysis of experimental data

B.1 General

When the experimental data do not contain any rogue values, this alternative method may be used in place of the method described in clause 7.

B.2 Method 1

See Figure 3.

The following procedure may be used in place of 7.2.6 to 7.2.8.

B.2.1 Calculate the overall mean, $\overline{\overline{\overline{x}}}$, and the variances, ${\hat{\sigma}_1}^2$, ${\hat{\sigma}_2}^2$ and ${\hat{\sigma}_3}^2$ in accordance with the sum of squares of the ranges using equations (7) and (B.1) to (B.6).

$$\overline{\overline{\overline{x}}} = \frac{1}{n}\sum \overline{\overline{x}} \quad (7)$$

$$\sum {R_1}^2 = \sum \left| \overline{x}_{ij1} - \overline{x}_{ij2} \right|^2 \quad (\text{B.1})$$

$$\sum {R_2}^2 = \sum \left| \overline{x}_{i1} - \overline{x}_{i2} \right|^2 \quad (\text{B.2})$$

$$\sum {R_3}^2 = \sum \left| \overline{\overline{x}}_{1} - \overline{\overline{x}}_{2} \right|^2 \quad (\text{B.3})$$

$${\hat{\sigma}_1}^2 = \frac{1}{8n}\sum {R_1}^2 \quad (\text{B.4})$$

$${\hat{\sigma}_2}^2 = \frac{1}{4n}\sum {R_2}^2 \quad (\text{B.5})$$

$${\hat{\sigma}_3}^2 = \frac{1}{2n}\sum {R_3}^2 \quad (\text{B.6})$$

B.2.2 Calculate the estimated values of the variances of measurement, ${\hat{\sigma}_\text{M}}^2$, sample preparation, $\hat{\sigma}_\text{P}$ and sampling, ${\hat{\sigma}_\text{S}}^2$, using equations (B.7) to (B.9).

$${\hat{\sigma}_\text{M}}^2 = {\hat{\sigma}_1}^2 \quad (\text{B.7})$$

$${\hat{\sigma}_\text{P}}^2 = {\hat{\sigma}_2}^2 - \frac{1}{2}{\hat{\sigma}_\text{M}}^2 \quad (\text{B.8})$$

$${\hat{\sigma}_\text{S}}^2 = {\hat{\sigma}_3}^2 - \frac{1}{2}{\hat{\sigma}_2}^2 \quad (\text{B.9})$$

B.3 Method 2

See Figure 4.

The following procedure may be used in place of 7.3.6 to 7.3.8.

B.3.1 Calculate the overall mean, $\overline{\overline{\overline{\overline{x}}}}$, and the variances, $\hat{\sigma}_1{}^2$, $\hat{\sigma}_2{}^2$ and $\hat{\sigma}_3{}^2$ in accordance with the sum of squares of the ranges using equations (7) and (B.10) to (B.12).

$$\overline{\overline{\overline{\overline{x}}}} = \frac{1}{n}\sum \overline{\overline{\overline{x}}} \tag{7}$$

$$\hat{\sigma}_1{}^2 = \frac{1}{2n}\sum R_1{}^2 \tag{B.10}$$

$$\hat{\sigma}_2{}^2 = \frac{1}{2n}\sum R_2{}^2 \tag{B.11}$$

$$\hat{\sigma}_3{}^2 = \frac{1}{2n}\sum R_3{}^2 \tag{B.12}$$

where n is the number of lots.

B.3.2 Calculate the estimated values of the variances of measurement, $\hat{\sigma}_M{}^2$, sample preparation, $\hat{\sigma}_P{}^2$ and sampling $\hat{\sigma}_S{}^2$ using equations (B.7), (B.13) and (B.14).

$$\hat{\sigma}_M^2 = \hat{\sigma}_1{}^2 \tag{B.7}$$

$$\hat{\sigma}_P^2 = \hat{\sigma}_2{}^2 - \frac{3}{4}\hat{\sigma}_M^2 \tag{B.13}$$

$$\hat{\sigma}_S^2 = \hat{\sigma}_3{}^2 - \frac{3}{4}\hat{\sigma}_P^2 - \frac{11}{16}\hat{\sigma}_M^2 \tag{B.14}$$

B.4 Method 3

The following procedure may be used in place of 7.4.3 and the latter half of 7.4.2 and 7.4.4.

Calculate the overall mean, $\overline{\overline{x}}$, and the estimated value of the overall variance, $\hat{\sigma}_{SPM}^2$, in accordance with the sum of squares of the ranges using equations (26) and (B.15).

$$\overline{\overline{x}} = \frac{1}{n}\sum \overline{x} \tag{26}$$

$$\hat{\sigma}^2{}_{SPM} = \frac{1}{2n}\sum R^2 \tag{B.15}$$

where n is the number of lots.

ICS 73.060.10

Price based on 21 pages