INTERNATIONAL STANDARD

ISO 3085

Fifth edition
2019-08

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 3085:2019(E)

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

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

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This fifth edition cancels and replaces the fourth edition (ISO 3085:2002), which has been technically revised. The main change compared to the previous edition is the use of the mean square difference between assay pairs, as described in the Introduction.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The key change between this document and the previous edition is the use of the mean square difference between assay pairs to estimate the numerical value of the precision instead of the mean difference between assay pairs, noting that the use of mean square differences was included in ISO 3085:1996, Annex B, as an alternative method only. The use of mean square differences avoids overestimating the sampling system's capability, thereby limiting the opportunity for improvement. In addition, when possible measurement outliers are identified, the process (such as sampling, sample preparation or measurement) under investigation may not be in a state of statistical control and should be checked in order to detect assignable causes. If these assignable causes can be identified, then the set of measurements should be repeated after the assignable causes have been corrected. Otherwise, data assessment should proceed without eliminating the outliers.

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

1 Scope

This document 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.

This document can also be applied for the purpose of checking the precision of sampling, sample preparation and measurement separately.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3082:2017, *Iron ores — Sampling and sample preparation procedures*

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

ISO 11323, *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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Sampling from 20 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.

Comparison of the estimated precision with that specified in ISO 3082:2017, Table 1, and necessary action taken 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, namely 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 shall 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.

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.

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 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 using mass basis sampling, 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, see Formula (1):

$$\Delta m = \frac{m_L}{2n_1} \tag{1}$$

Alternatively, when the experiment is carried out as part of routine mass basis 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, see Formula (2):

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

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

6.1.1.3 When $2n_1$ increments are taken using time basis sampling, the sampling intervals, Δt, in minutes, shall be calculated using Formula (3), i.e. giving intervals equal to one-half of the sampling interval for routine sampling:

$$\Delta t = \frac{60 m_L}{2 q_{max} n_1} \tag{3}$$

where q_{max} is the maximum flow rate, expressed in tonnes per hour, of ore on the conveyor belt.

Alternatively, when the experiment is carried out as part of routine time basis sampling and n_1 increments are taken, the sampling interval, Δt, shall be calculated using Formula (4):

$$\Delta t = \frac{60 m_L}{q_{max} n_1} \tag{4}$$

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

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

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

EXAMPLE 1 See Figure 1.

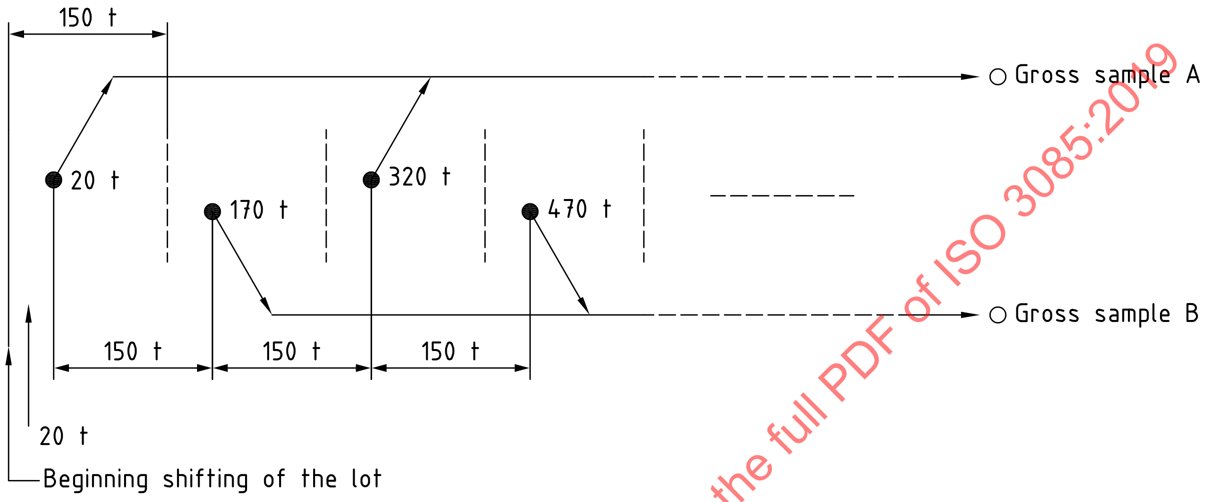
A lot of 19 000 t is transferred by belt conveyors and the number of increments determined in accordance with ISO 3082 for routine mass basis sampling, n_1, is 60.

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

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

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 are 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 are taken.

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



NOTE 1 Solid circles indicate increments taken from strata.

NOTE 2 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 Formula (5):

$$n_3 = \frac{n_1}{n_4} \quad (5)$$

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.

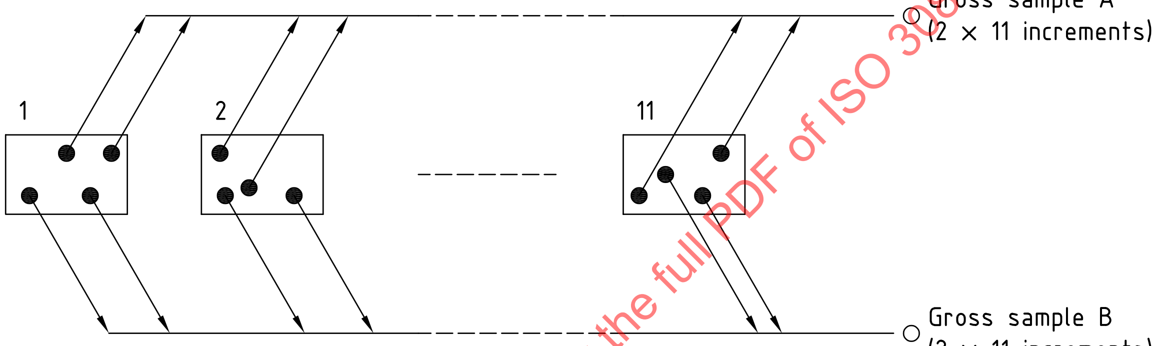
6.1.2.3 The two partial samples from each stratum shall be combined into two gross samples, A and B, respectively.

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 2 See Figure 2.

A lot is divided in 11 strata each of 60 t and the number of increments, n_1, determined for the entire lot (60 × 11 = 660 t) in accordance with ISO 3082 is 20. Thus, the number of increments to be taken from each stratum is shown by the formula:

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



NOTE 1 Boxes indicate strata.

NOTE 2 Solid circles indicate increments taken from strata.

NOTE 3 Open circles indicate gross samples.

Figure 2 — Schematic diagram for example 2

When $2n_1$ increments are taken, four ($2n_3$ = 2 × 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 × 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 in 6.2.2 to 6.2.4.

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.

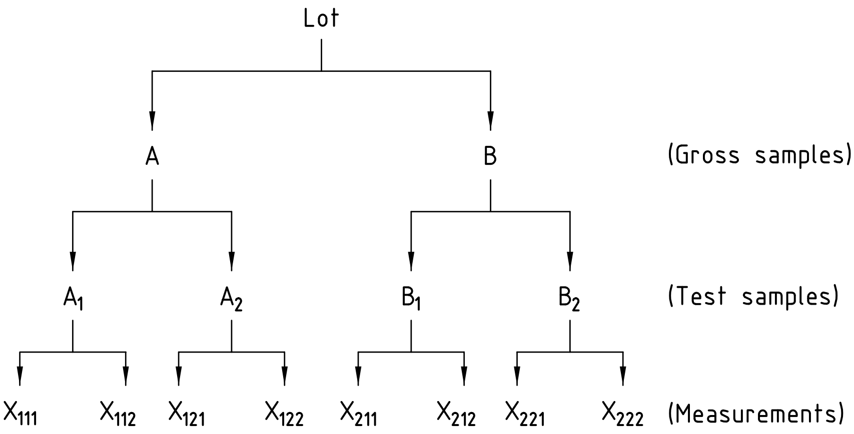


Figure 3 — Flowsheet for method 1

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.

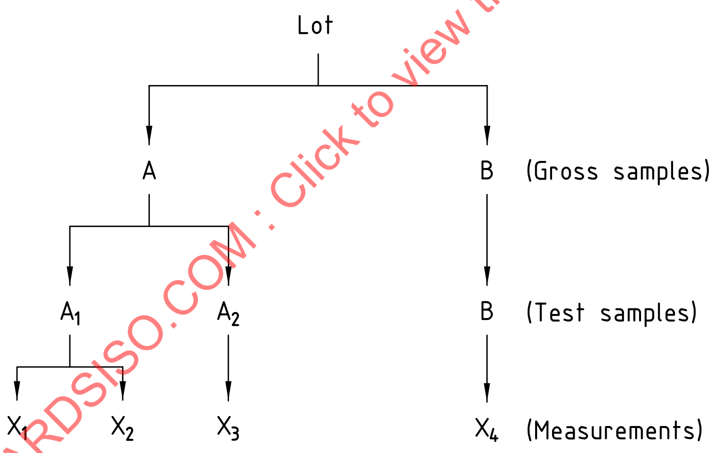


Figure 4 — Flowsheet for method 2

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.

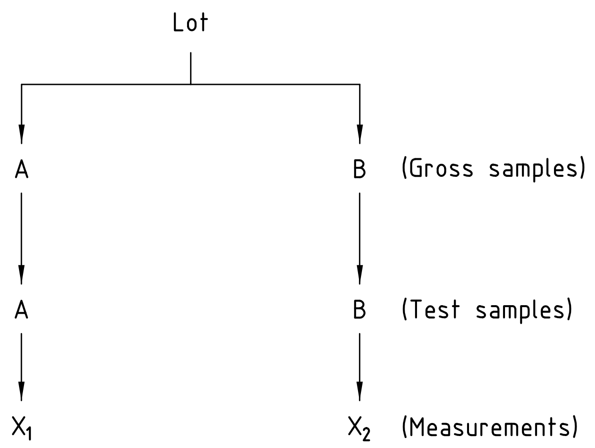


Figure 5 — Flowsheet for method 3

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 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 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 Formulae (6) and (7), respectively:

$$\bar{x}_{ij.} = \frac{1}{2}\left(x_{ij1} + x_{ij2}\right) \quad (6)$$

$$R_1 = \left|x_{ij1} - x_{ij2}\right| \quad (7)$$

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 test samples, using Formulae (8) and (9), respectively:

$$\bar{\bar{x}}_{i..} = \frac{1}{2}\left(\bar{x}_{i1.} + \bar{x}_{i2.}\right) \tag{8}$$

$$R_2 = \left|\bar{x}_{i1.} - \bar{x}_{i2.}\right| \tag{9}$$

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 Formulae (10) and (11), respectively:

$$\bar{\bar{\bar{x}}} = \frac{1}{2}\left(\bar{\bar{x}}_{1..} + \bar{\bar{x}}_{2..}\right) \tag{10}$$

$$R_3 = \left|\bar{\bar{x}}_{1..} - \bar{\bar{x}}_{2..}\right| \tag{11}$$

7.2.6 Calculate the overall mean, $\bar{\bar{\bar{\bar{x}}}}$, and the variances, $\hat{\sigma}_1{}^2$, $\hat{\sigma}_2{}^2$ and $\hat{\sigma}_3{}^2$ using Formulae (12) to (15):

$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{n}\sum \bar{\bar{\bar{x}}} \tag{12}$$

$$\hat{\sigma}_1{}^2 = \frac{1}{8n}\sum R_1^2 \tag{13}$$

$$\hat{\sigma}_2{}^2 = \frac{1}{4n}\sum R_2^2 \tag{14}$$

$$\hat{\sigma}_3{}^2 = \frac{1}{2n}\sum R_3^2 \tag{15}$$

where n is the number of lots.

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

The upper control limits for the R-charts are 3,64 $\hat{\sigma}_1$ for R_1, 3,64 $\hat{\sigma}_2$ for R_2 and 3,64 $\hat{\sigma}_3$ for R_3.

The factor 3,64 (i.e. 2,576 × $\sqrt{2}$) gives the 99 % limit for the difference between two independent measurements from the same normal distribution, because the difference has twice the original variance.

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 may not be in a state of statistical control and should be checked in order to detect assignable causes. If these assignable causes can be identified, then the set of measurements should be repeated after the assignable causes have been corrected. Otherwise, the computation should proceed without eliminating the outliers.

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 Formulae (16) to (18), respectively:

$$\hat{\sigma}_M^2 = \hat{\sigma}_1^2 \quad (16)$$

$$\hat{\sigma}_P^2 = \hat{\sigma}_2^2 - \frac{1}{2}\hat{\sigma}_1^2 \quad (17)$$

$$\hat{\sigma}_S^2 = \hat{\sigma}_3^2 - \frac{1}{2}\hat{\sigma}_2^2 \quad (18)$$

If $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ as calculated from Formulae (17) and (18) is found to be negative, $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ shall be replaced by zero. When n_1 increments are taken in accordance with 5.1.3, the estimated value of the standard deviation of sampling, $\hat{\sigma}_S$ from Formula (18) 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 Formulae (16) and (17).

7.2.9 Calculate the estimated values of the precision of sampling $\left(\beta_S = 2\hat{\sigma}_S\right)$, 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 $\left(\beta_{SPM} = 2\hat{\sigma}_{SPM}\right)$, using Formula (19):

$$\hat{\sigma}_{SPM} = \sqrt{\hat{\sigma}_S^2 + \hat{\sigma}_P^2 + \hat{\sigma}_M^2} \quad (19)$$

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 Formulae (20) and (21):

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \quad (20)$$

$$R_1 = |x_1 - x_2| \tag{21}$$

7.3.4 Calculate the mean, $\bar{\bar{x}}$, and the range, R_2, using Formulae (22) and (23):

$$\bar{\bar{x}} = \frac{1}{2}(\bar{x} + x_3) \tag{22}$$

$$R_2 = |\bar{x} - x_3| \tag{23}$$

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 Formulae (24) and (25):

$$\bar{\bar{\bar{x}}} = \frac{1}{2}\left(\bar{\bar{x}} + x_4\right) \tag{24}$$

$$R_3 = \left|\bar{\bar{x}} - x_4\right| \tag{25}$$

7.3.6 Calculate the overall mean, $\bar{\bar{\bar{\bar{x}}}}$, and the variances, $\hat{\sigma}_1{}^2$, $\hat{\sigma}_2{}^2$ and $\hat{\sigma}_3{}^2$ using Formulae (26), (27), (28) and (29), respectively:

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

$$\hat{\sigma}_1^2 = \frac{1}{2n}\sum R_1^2 \tag{27}$$

$$\hat{\sigma}_2^2 = \frac{1}{2n}\sum R_2^2 \tag{28}$$

$$\hat{\sigma}_3^2 = \frac{1}{2n}\sum R_3^2 \tag{29}$$

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 may not be in a state of statistical control and should be checked in order to detect assignable causes. If these assignable causes can be identified, then the set of measurements should be repeated after the assignable causes have been corrected. Otherwise, the computation should proceed without eliminating the outliers.

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 Formulae (30), (31) and (32), respectively:

$$\hat{\sigma}_M^2 = \hat{\sigma}_1{}^2 \tag{30}$$

$$\hat{\sigma}_P^2 = \hat{\sigma}_2{}^2 - \frac{3}{4}\hat{\sigma}_1^2 \tag{31}$$

$$\hat{\sigma}_S^2 = \hat{\sigma}_3^2 - \frac{3}{4}\hat{\sigma}_2^2 - \frac{1}{8}\hat{\sigma}_1^2 \tag{32}$$

If $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ as calculated from Formulae (31) and (32) is found to be negative, $\hat{\sigma}_P^2$ or $\hat{\sigma}_S^2$ shall be replaced by zero.

When n_1 increments are taken in accordance with 5.1.3, the estimated value of the standard deviation of sampling, $\hat{\sigma}_S$ from Formula (32) 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 Formulae (30) and (31).

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

7.3.10 Calculate the estimated value of the overall precision of sampling, sample preparation and measurement $\left(\beta_{SPM} = 2\hat{\sigma}_{SPM}\right)$, using Formula (33):

$$\hat{\sigma}_{SPM} = \sqrt{\hat{\sigma}_S{}^2 + \hat{\sigma}_P{}^2 + \hat{\sigma}_M{}^2} \tag{33}$$

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 shown by Formula (34):

$$\hat{\sigma}_{SPM}^2 = \hat{\sigma}_S^2 + \hat{\sigma}_P^2 + \hat{\sigma}_M^2 \tag{34}$$

The estimated value of precision shall be calculated in accordance with 7.4.2 to 7.4.5.

7.4.2 Calculate the mean, $\bar{x}$ and the range, R_1, for each pair of measurements using Formulae (35) and (36):

$$\bar{x} = \frac{1}{2}\left(x_1 + x_2\right) \tag{35}$$

$$R_1 = |x_1 - x_2| \tag{36}$$

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

Calculate the overall mean, $\bar{\bar{x}}$, and the estimated value of the overall standard deviation, $\hat{\sigma}_{SPM}$, using Formula (37):

$$\bar{\bar{x}} = \frac{1}{n}\sum \bar{x} \tag{37}$$

When $2n_1$ increments are taken, calculate the estimated value of the overall standard deviation, $\hat{\sigma}_{SPM}$, using Formula (38):

$$\hat{\sigma}_{SPM}^2 = \frac{1}{2n}\sum R^2 \tag{38}$$

where n is the number of lots.

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

The upper control limit for the R-chart is 3,64 $\hat{\sigma}_{SPM}$ (see 7.2.6).

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 may not be in a state of statistical control and should be checked in order to detect assignable causes. If these assignable causes can be identified, then the set of measurements should be repeated after the assignable causes have been corrected. Otherwise, the computation should proceed without eliminating the outliers.

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

When n_1 increments are taken in accordance with 5.1.3, 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 ISO 3082:2017, Table 1. 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 shall be changed in accordance with the revised quality variation using ISO 3082:2017, Table 2.

8.2.2 Increasing number of increments

If a greater number, n_1', of increments is 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 systematic sampling by method 1

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

Sampling	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 $\hat{\sigma}_M, \hat{\sigma}_P, \hat{\sigma}_S$ and $\hat{\sigma}_{SPM}$.

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 as follows:

— standard deviation and precision of sampling:

$$\hat{\sigma}_S = 0{,}24\left(\%\text{Fe}\right) \qquad \beta_S = 2\hat{\sigma}_S = 0{,}48\left(\%\text{Fe}\right)$$

— standard deviation and precision of sample preparation:

$$\hat{\sigma}_\text{P} = 0{,}15\left(\%\text{Fe}\right) \qquad \beta_\text{P} = 2\hat{\sigma}_\text{P} = 0{,}30\left(\%\text{Fe}\right)$$

— standard deviation and precision of measurement:

$$\hat{\sigma}_\text{M} = 0{,}078\left(\%\text{Fe}\right) \qquad \beta_\text{M} = 2\hat{\sigma}_M = 0{,}16\left(\%\text{Fe}\right)$$

The overall standard deviation of sampling, sample preparation and measurement, calculated by Formula (19)/(33), is:

$$\hat{\sigma}_\text{SPM} = 0{,}29\left(\%\text{Fe}\right) \qquad \text{and } \beta_\text{SPM} = 0{,}58(\%\ \text{Fe})$$

This value of β_{SPM} does not satisfy the overall precision shown in ISO 3082:2017, Table 1, and therefore action needs to be taken on the sampling, sample preparation and measurement procedures.