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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)

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Foreword

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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.

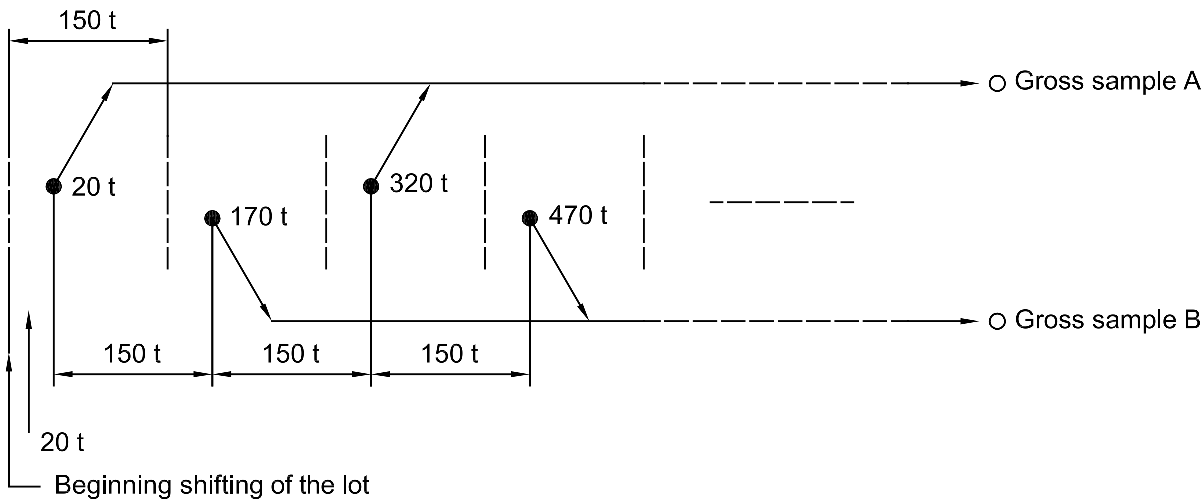
Suppose that a lot of 19 000 t is transferred by belt conveyors and the number of increments determined in accordance with ISO 3082 for routine sampling, n_1, is 60.

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.

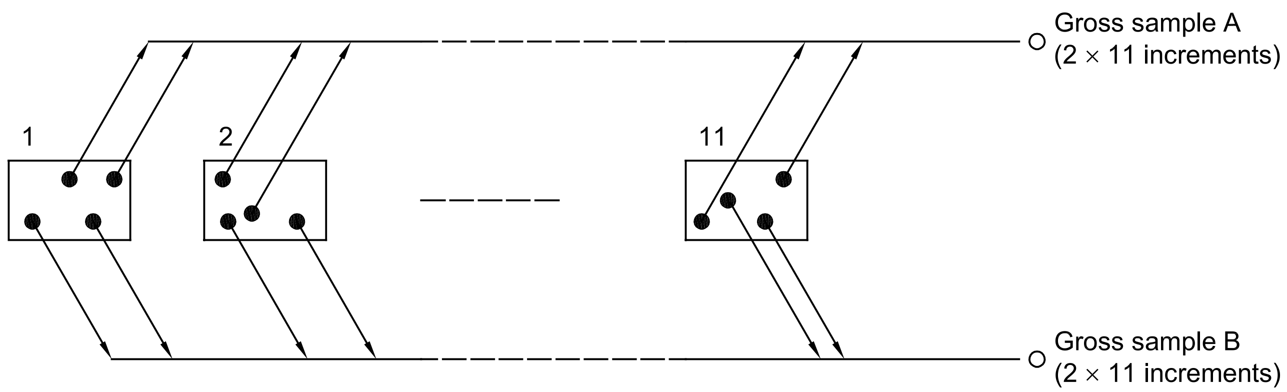
6.1.2.3 The two partial samples from each stratum shall be combined into two gross samples, A and B, respectively.

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.

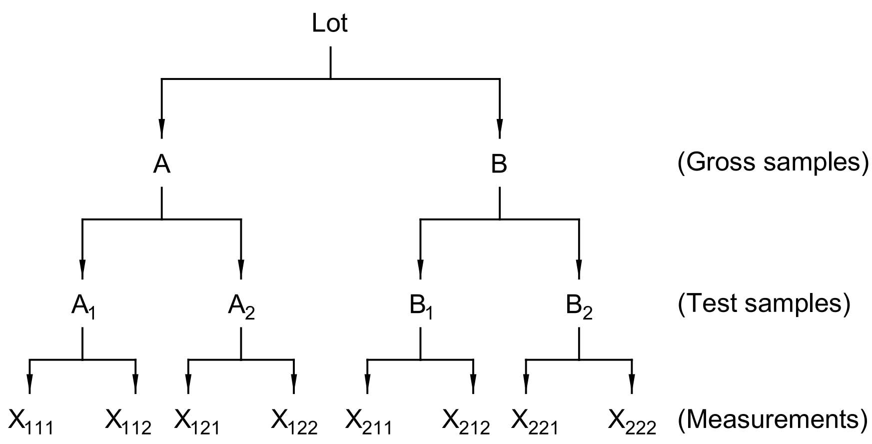


Figure 3 — Flowsheet for method 1

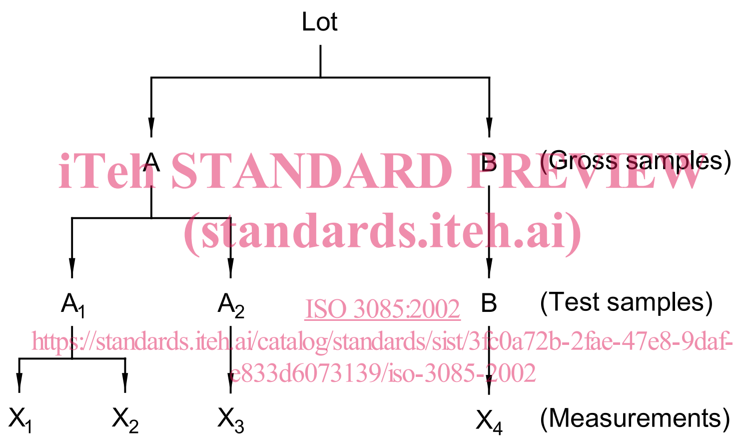


Figure 4 — Flowsheet for method 2

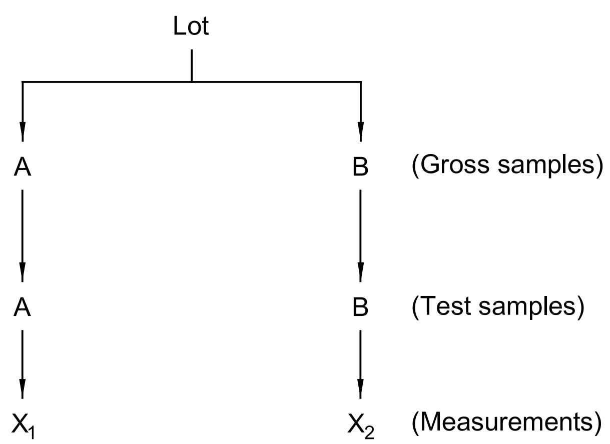


Figure 5 — Flowsheet for method 3

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

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