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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

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Foreword

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

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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.

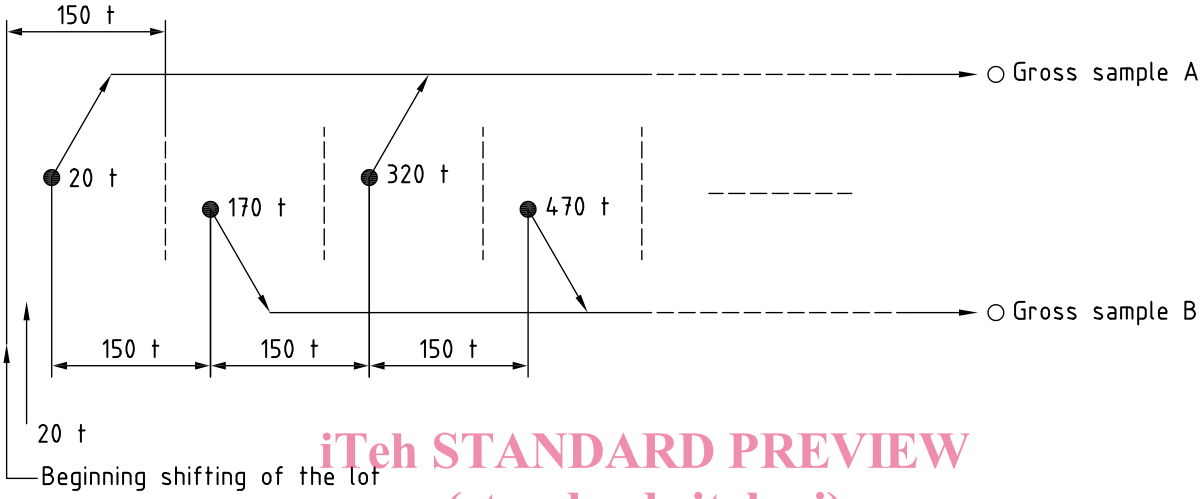
A lot of 19 000 t is transferred by belt conveyors and the number of increments determined in accordance with ISO 3082 for routine mass basis sampling, n_1, is 60.

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):

$$n_3 = \frac{n_1}{n_4} \tag{5}$$

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.

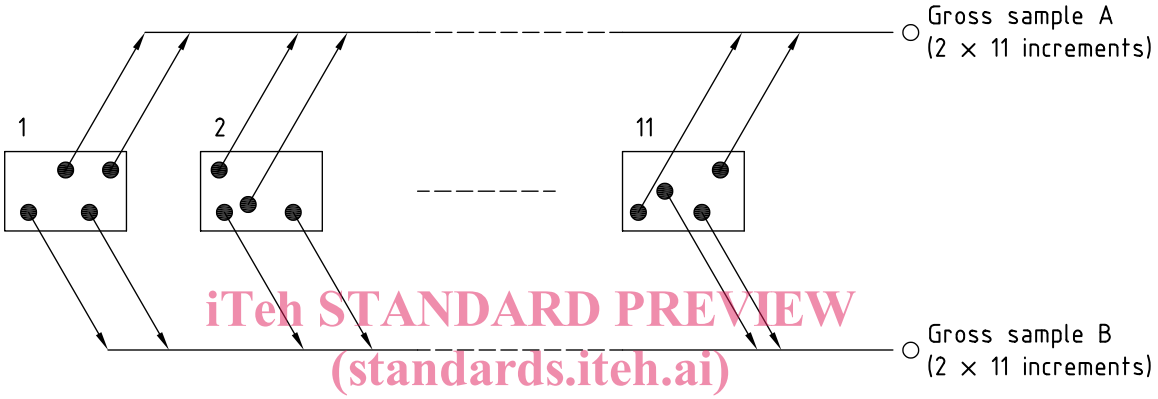
6.1.2.3 The two partial samples from each stratum shall be combined into two gross samples, A and B, respectively.

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.