INTERNATIONAL STANDARD

ISO 3082

Fifth edition
2017-07

Iron ores — Sampling and sample preparation procedures

Minerais de fer — Procédures d'échantillonnage et de préparation des échantillons

Reference number
ISO 3082:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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 on 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 the following URL: 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 3082:2009), which has been technically revised. It also incorporates the Technical Corrigendum ISO 3082:2009/Cor.1:2009. The main changes compared to the previous edition are as follows:

— expansion of the definition of test sample;

— insertion of a new paragraph in 4.1 indicating that sampling from the top of a moving conveyor belt using cross-belt (hammer) samplers is not permitted;

— deletion of reference to increasing the cutter aperture above three times nominal top size to avoid bridging of the cutter lips for wet sticky ore at the end of 5.1.4.2;

— expression of bulk density in kg/m^3 in 5.1.4.4 and corresponding amendment of Formula (3);

— insertion of an explanation in the first paragraph of 5.2 that better precision means a lower value of β_{SPM};

— inclusion of an extra column in Table 1 and extra rows in Tables 3 and 5 for mass of lot over 340 000 tonnes and updating of the overall precision values for phosphorus content in Table 1 based on international data collected on precisions achieved in practice;

— updating of the sampling precision values for phosphorus content in Table 3 based on international data collected on precisions achieved in practice as well as minor adjustments to the sizing precisions for sized ore and sinter feed;

— changing of "there will not be any oversize material remaining" in 7.7.2 to "no more than 5 % by mass oversize material is retained on the relevant sieve";

— changing of "sample division" to "division" throughout 10.1.5;

— clarification of the requirements for preparation of test samples for moisture determination and division of individual increments or partial samples in 10.1.6.1.1, 10.1.6.1.2 and 10.1.6.2.3;

— correction of the mass of sample for physical testing to 600 kg in the last sentence of 10.1.6.3;

— major revision of 10.2.4 to clarify the special procedure for moisture content, including a revision of Table 7;

— insertion of a new clause (10.4.3) describing the manual strip-division method as an acceptable alternative to manual increment division and riffle division;

— amendment of all particle size specifications in 10.5 to nominal top size, including Figure 11 and Figure 12;

— significant revision of 10.6 to clarify the procedure for preparation of test samples for moisture determination.

Iron ores — Sampling and sample preparation procedures

WARNING — This document can involve hazardous materials, operations and equipment, and does not purport to address all the safety issues associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices.

1 Scope

This document provides

a) the underlying theory,

b) the basic principles for sampling and preparation of samples, and

c) the basic requirements for the design, installation and operation of sampling systems

for mechanical sampling, manual sampling and preparation of samples taken from a lot under transfer. This is in order to determine the chemical composition, moisture content, size distribution and other physical and metallurgical properties of the lot, except bulk density obtained using ISO 3852 (Method 2).

The methods specified in this document are applicable to both the loading and discharging of a lot by means of belt conveyors and other ore-handling equipment to which a mechanical sampler can be installed or where manual sampling can safely be conducted.

The methods are applicable to all iron ores, whether natural or processed (e.g. concentrates and agglomerates, such as pellets or sinters).

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

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

ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling*

ISO 3087, *Iron ores — Determination of the moisture content of a lot*

ISO 3271, *Iron ores for blast furnace and direct reduction feedstocks — Determination of the tumble and abrasion indices*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 3852, *Iron ores for blast furnace and direct reduction feedstocks — Determination of bulk density*

ISO 4695, *Iron ores for blast furnace feedstocks — Determination of the reducibility by the rate of reduction index*

ISO 4696-1, *Iron ores for blast furnace feedstocks — Determination of low-temperature reduction-disintegration indices by static method — Part 1: Reduction with CO, CO_2, H_2 and N_2*

ISO 4696-2, *Iron ores for blast furnace feedstocks — Determination of low-temperature reduction-disintegration indices by static method — Part 2: Reduction with CO and N_2*

ISO 4698, *Iron ore pellets for blast furnace feedstocks — Determination of the free-swelling index*

ISO 4700, *Iron ore pellets for blast furnace and direct reduction feedstocks — Determination of the crushing strength*

ISO 4701, *Iron ores and direct reduced iron — Determination of size distribution by sieving*

ISO 7215, *Iron ores for blast furnace feedstocks — Determination of the reducibility by the final degree of reduction index*

ISO 7992, *Iron ores for blast furnace feedstocks — Determination of reduction under load*

ISO 8371, *Iron ores for blast furnace feedstocks — Determination of the decrepitation index*

ISO 11256, *Iron ore pellets for shaft direct-reduction feedstocks — Determination of the clustering index*

ISO 11257, *Iron ores for shaft direct-reduction feedstocks — Determination of the low-temperature reduction-disintegration index and degree of metallization*

ISO 11258, *Iron ores for shaft direct-reduction feedstocks — Determination of the reducibility index, final degree of reduction and degree of metallization*

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

ISO 13930, *Iron ores for blast furnace feedstocks — Determination of low-temperature reduction-disintegration indices by dynamic method*

3 Terms and definitions

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

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

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

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

3.1
lot
discrete and defined quantity of iron ore or direct reduced iron for which quality characteristics are to be assessed

3.2
increment
quantity of iron ore or direct reduced iron collected in a single operation of a device for sampling or sample division

3.3
sample
relatively small quantity of iron ore or direct reduced iron, so taken from a lot as to be representative in respect of the quality characteristics to be assessed

3.4
partial sample
sample comprising of less than the complete number of increments needed for a gross sample

3.5
gross sample
sample comprising all increments, entirely representative of all quality characteristics of a lot

3.6
test sample
sample prepared from an increment, a partial sample or a gross sample to meet all specific conditions for a test

3.7
test portion
part of a test sample that is actually and entirely subjected to the specific test

3.8
stratified sampling
sampling of a lot carried out by taking increments from systematically specified positions and in appropriate proportions from strata

Note 1 to entry: Examples of strata include production periods (e.g. 5 min), production masses (e.g. 1 000 t), holds in vessels, wagons in a train, or containers and trucks representing a lot.

3.9
systematic sampling
sampling carried out by taking increments from a lot at regular intervals

3.10
mass-basis sampling
sampling carried out so that increments are taken at equal mass intervals, increments being as near as possible of uniform mass

3.11
time-basis sampling
sampling carried out so that increments are taken from free falling streams, or from conveyors, at uniform time intervals, the mass of each increment being proportional to the mass flow rate at the instant of taking the increment

3.12
proportional mass division
division of samples or increments such that the mass of each retained divided portion is a fixed proportion of the mass being divided

3.13
constant mass division
division of samples or increments such that the retained divided portions are of almost uniform mass, irrespective of variations in mass of the samples or increments being divided

Note 1 to entry: This method is required for sampling on a mass basis.

Note 2 to entry: “Almost uniform” means that variations in mass are less than 20 % in terms of the coefficient of variation.

3.14
split use of sample
separate use of parts of a sample, as test samples for separate determinations of quality characteristics

3.15
multiple use of sample
use of a sample in its entirety for the determination of one quality characteristic, followed by the use of the same sample in its entirety for the determination of one or more other quality characteristics

3.16
nominal top size
particle size expressed by the smallest aperture size of the test sieve (from a square opening complying with the R20 or R40/3 series in ISO 565), such that no more than 5 % by mass of iron ore is retained on the sieve

4 General considerations for sampling and sample preparation

4.1 Basic requirements

The basic requirement for a correct sampling scheme is that all parts of the ore in the lot have an equal opportunity of being selected and becoming part of the sample for analysis (Gy[1]; Pitard[2]). Any deviation from this basic requirement can result in an unacceptable loss of trueness and precision. An incorrect sampling scheme cannot be relied on to provide representative samples.

The best sampling location to satisfy the above requirement is at a transfer point between conveyor belts. Here the full cross-section of the ore stream can be conveniently intercepted at regular intervals, enabling representative samples to be obtained.

Sampling from the top of a moving conveyor belt using cross-belt (hammer) samplers is not permitted, because it is impossible to extract a complete cross-section of the ore stream. Consequently, all parts of the lot do not have an equal opportunity of being sampled. All attempts to validate hammer samplers show significant bias compared to falling-stream and stopped-belt sampling.

In situ sampling of ships, stockpiles, containers and bunkers is not permitted, because it is impossible to drive the sampling device down to the bottom and extract the full column of ore. Consequently, all parts of the lot do not have an equal opportunity of being sampled. The only effective procedure is sampling from a transfer point at the end of or between conveyor belts when ore is being conveyed to or from the ship, stockpile, container or bunker.

In situ sampling from stationary situations such as trucks or wagons is permitted only for ores with nominal top size less than 1 mm, provided the sampling device, e.g. a spear or an auger, penetrates to the full depth of the concentrate at the point selected for sampling and the full column of concentrate is extracted.

Sampling shall be carried out by systematic sampling either on a mass basis (see 6.1) or on a time basis (see 6.2), provided no bias is introduced by periodic variations in quality or quantity. However, if periodic variations that could introduce bias are present, stratified random sampling within fixed mass or time intervals shall be carried out (see 6.3).

The methods used for sampling and sample preparation depend on the final choice of the sampling scheme and on the steps necessary to minimize possible biases and obtain acceptable overall precision.

Moisture samples shall be processed as soon as possible and test portions weighed immediately. If this is not possible, samples shall be stored in non-absorbent airtight containers with a minimum of free air space to minimize any change in moisture content, but should be prepared without delay.

4.2 Establishing a sampling scheme

The procedure for establishing a sampling scheme is as follows:

a) identify the lot to be sampled and the quality characteristics to be determined;

b) ascertain the nominal top size;

c) determine the sampling location and the method of taking increments;

d) determine the mass of increment considering the nominal top size, the ore-handling equipment and the device for taking increments;

e) specify the precision required;

f) ascertain the quality variation, σ_W, of the lot in accordance with ISO 3084, or, if this is not possible, assume “large” quality variation as specified in 5.3;

g) determine the minimum number of primary increments, n_1, to be taken from the lot for systematic or stratified random sampling;

h) determine the sampling interval in tonnes for mass-basis sampling or in minutes for time-basis sampling;

i) take increments having almost uniform mass for mass-basis sampling or having a mass proportional to the flow rate of the ore stream at the time of sampling for time-basis sampling. Increments are to be taken at the intervals determined in (h) during the entire period of handling the lot;

j) determine whether the sample is for split use or multiple use;

k) establish the method of combining increments into a gross sample or partial samples;

l) establish the sample preparation procedure, including division, crushing, mixing and drying;

m) crush the sample, if necessary, except for the size sample and some physical testing samples;

n) dry the sample, if necessary, except for the moisture sample;

o) divide samples according to the minimum mass of divided sample for a given nominal top size, employing constant mass or proportional division for mass-basis sampling, or proportional division for time-basis sampling;

p) prepare the test sample.

Special attention shall be given to the total mass of test sample required for physical tests (see 10.1.6.3). When the mass of the gross sample or partial samples is expected to be less than that required for preparation of test samples for physical testing, the number and/or mass of increments to be taken shall be increased to give the required mass. It is preferable that the number of increments be increased, rather than the increment mass.

4.3 System verification

Stopped-belt sampling is the reference method for collecting samples against which mechanical and manual sampling procedures may be compared to establish that they are unbiased in accordance with procedures specified in ISO 3086. However, before any bias tests are conducted, sampling and sample preparation systems shall first be inspected to confirm that they conform to the correct design principles specified in this document. Inspections shall also include an examination of whether any loading, unloading or reclaiming procedures could produce periodic variations in quality in phase with the taking of increments. These periodic variations could include characteristics such as particle size distribution and moisture content. When such cyclic variations occur, the source of the variations shall be investigated to determine the practicability of eliminating the variations. If this is not possible, stratified random sampling shall be carried out (see 6.3).

An example of a suitable inspection procedure and checklist is provided in Annex A. This will quickly reveal any serious deficiencies in the sampling or sample preparation system and may avoid the need for expensive bias testing. Consequently, sampling systems shall be designed and constructed in a manner that facilitates regular verification of correct operation.

NOTE Further details can be found in Reference [3].

Regular checks of quality variation and precision shall also be carried out in accordance with ISO 3084 and ISO 3085 to monitor variations in quality variation and to verify the precision of sampling, sample preparation and measurement. This is particularly important for new products or new sampling systems or when significant changes are made to existing systems.

5 Fundamentals of sampling and sample preparation

5.1 Minimization of bias

5.1.1 General

Minimization of bias in sampling and sample preparation is vitally important. Unlike precision, which can be improved by collecting more increments or repeating measurements, bias cannot be reduced by replicating measurements. Consequently, the minimization or preferably elimination of possible biases should be regarded as more important than improvement of precision. Sources of bias that can be completely eliminated at the outset by correct design of the sampling and sample preparation system include sample spillage, sample contamination and incorrect delineation and extraction of increments, while sources that can be minimized but not completely eliminated include change in moisture content, loss of dust and particle degradation (for size determination).

5.1.2 Minimization of particle size degradation

Minimization of particle size degradation of samples used for determination of size distribution is vital in order to reduce bias in the measured size distribution. To prevent particle size degradation, it is essential to keep free-fall drops to a minimum.

5.1.3 Extraction of increments

It is essential that increments be extracted from the lot in such a manner that all parts of the ore have an equal opportunity of being selected and becoming part of the final sample for analysis, irrespective of the size, mass, shape or density of individual particles. If this requirement is not respected, bias is easily introduced. This results in the following design requirements for sampling and sample preparation systems:

a) a complete cross-section of the ore stream shall be taken when sampling from a moving stream (see 7.5);

b) the aperture of the sample cutter shall be at least three times the nominal top size of the ore, or 30 mm for the primary sampling and 10 mm for subsequent stages, whichever is the greater (see 7.5.4);

c) the speed of the sample cutter shall not exceed 0,6 m/s, unless the cutter aperture is correspondingly increased (see 7.5.5);

d) the sample cutter shall travel through the ore stream at uniform speed (see 7.5.3), both the leading and trailing edges of the cutter clearing the ore stream at the end of its traverse;

e) the lips on the sample cutter shall be parallel for straight-path samplers and radial for rotary cutters (see 7.5.3), and these conditions shall be maintained as the cutter lips wear;

f) changes in moisture content, dust losses and sample contamination shall be avoided;

g) free-fall drops shall be kept to a minimum to reduce size degradation of the ore and hence minimize bias in size distribution;

h) primary cutters shall be located as near as possible to the loading or discharging point to further minimize the effects of size degradation;

i) a complete column of ore with nominal top size less than 1 mm shall be extracted when sampling iron ore concentrate in a wagon (see 8.2).

Sampling systems shall be designed to accommodate the maximum nominal top size and flow rate of the ore being sampled. Detailed design requirements for sampling and sample preparation systems are provided in Clauses 7, 8, 9 and 10.

5.1.4 Increment mass

5.1.4.1 General

The increment mass required to obtain an unbiased sample can be calculated for typical sampling situations [see Formulae (1), (2) and (3)]. Comparing the calculated masses with the actual increment masses is useful for checking the design and operation of sampling systems. If the difference is significant, the cause shall be identified and corrective action taken to rectify the problem.

5.1.4.2 Increment mass for falling stream sampling

The mass of increment, m_I, in kilograms, to be taken (mechanically or manually) by a cutter-type sampler from the ore stream at the discharge end of a conveyor belt is given by Formula (1):

$$m_I = \frac{q l_1}{3{,}6 v_C} \tag{1}$$

where

q is the flow rate, in tonnes per hour, of ore on the conveyor belt;

l_1 is the cutter aperture, in metres, of the sampler;

v_C is the cutter speed, in metres per second, of the sampler.

The minimum increment mass that can be taken, while still avoiding bias, is determined by the minimum cutter aperture specified in 7.5.4 and the maximum cutter speed specified in 7.5.5.

5.1.4.3 Increment mass for stopped-belt sampling

The mass of increment, m_I, in kilograms, to be taken manually from a stopped-belt is equal to the mass of a complete cross-section of the ore on the conveyor. It is given by Formula (2):

$$m_I = \frac{q l_2}{3{,}6 v_B} \tag{2}$$

where

q is the flow rate, in tonnes per hour, of ore on the conveyor belt;

l_2 is the length, in metres, of the complete cross-section of ore removed from the conveyor;

v_B is the speed of the conveyor belt, in metres per second.

The minimum increment mass that can be taken, while still avoiding bias, is determined by the minimum length of ore removed from the conveyor, i.e. 3*d*, where *d* is the nominal top size of the ore, in millimetres, subject to a minimum of 30 mm for primary sampling and 10 mm for subsequent stages.

5.1.4.4 Increment mass for manual sampling using spear or auger

The mass of increment, m_I, in kilograms to be taken from a truck or wagon in a lot using a spear or an auger of diameter, l_3, in millimetres, is given by Formula (3):

$$m_I = \frac{\pi \rho_b l_3^2 l_4}{4 \times 10^6} \tag{3}$$

where