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Direct reduced iron and hot briquetted iron — Sampling and sample preparation

Minerais de fer préréduits et fer briqueté à chaud — Échantillonnage et préparation des échantillons

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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 2.

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

ISO 10835 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 1, *Sampling*.

This second edition cancels and replaces the first edition (ISO 10835:1995), which has been technically revised.

Direct reduced iron and hot briquetted iron — Sampling and sample preparation

WARNING — This International Standard may involve hazardous materials, operations and equipment, and does not purport to address all of the safety issues associated with its use. It is the responsibility of the user of this standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard gives

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, to determine the chemical composition, moisture content and physical properties of the lot.

The methods specified in this International Standard are applicable to both the loading and discharging of direct reduced iron (DRI) and hot briquetted iron (HBI), by means of belt conveyors and other ore handling equipment to which a mechanical sampler may be installed or where stopped-belt sampling may safely be conducted. In this International Standard, DRI includes both reduced pellets and reduced lump ores.

CAUTION — Direct reduced iron (DRI) and, in some cases, hot briquetted iron (HBI), for example, with low density or high fines content, may react with water and air to produce hydrogen and heat. The heat produced may cause ignition. Therefore, due consideration shall be given to the safety of operators by respecting applicable regulations or international codes.

2 Normative references

The following referenced documents are indispensable for the application 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 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

ISO 3085:2002, *Iron ores — Experimental methods for checking the precision of sampling, sample preparation and mesasurement*

ISO 3086:1998, *Iron ores — Experimental methods for checking the bias of sampling*

ISO 3087:1998, *Iron ores — Determination of moisture content of a lot*

ISO 3534-1:2006, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

ISO 4701:1999, *Iron ores — Determination of size distribution by sieving*

ISO 11323:2002, *Iron ores and direct reduced iron — Vocabulary*

3 Terms and definitions

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

3.1
lot
discrete and defined quantity of DRI or HBI for which quality characteristics are to be assessed

3.2
increment
quantity of DRI or HBI collected in a single operation of a sampling device

3.3
sample
relatively small quantity of DRI or HBI, taken from a lot so as to be representative in respect of the quality characteristics to be assessed

3.4
partial sample
sample consisting 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 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 identified parts called strata

NOTE Examples of strata, based on time, mass or space, include production periods (e.g. 5 min), production masses (e.g. 1 000 t), holds in vessels, wagons in a train, or containers.

3.9
systematic sampling
selection of increments at regular intervals from a lot

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 sample 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 This method is required for sampling on a mass basis. “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 of DRI
smallest aperture size, within the range of the R20 Series (in ISO 565, square opening), such that no more than 5 % by mass of the DRI is retained on the sieve

3.17
nominal top size of HBI
prior to crushing, the largest average dimension of HBI briquettes, or, after crushing, the smallest aperture size, within the range of the R20 Series (in ISO 565, square opening), such that no more than 5 % by mass of the HBI 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 of the DRI or HBI in the lot has an equal opportunity of being selected and becoming part of the partial sample or gross sample for analysis. Any deviation from this basic requirement can result in an unacceptable loss of accuracy 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 DRI or HBI stream can be conveniently intercepted at regular intervals, enabling representative samples to be obtained. Alternatively, samples may be taken from a stopped conveyor belt, provided a full cross-section of DRI or HBI of adequate length is taken from the conveyor (see Clause 9).

In situ sampling of ships, stockpiles, wagons, containers and bunkers is not permitted, because there is no suitable sampling device that can be driven down to the bottom and then extract the full column of DRI or HBI. Consequently, all parts of the lot do not have an equal opportunity of being sampled. The only effective procedure is sampling from a conveyor belt when the DRI or HBI is being conveyed to or from the ship, stockpile, container or bunker.

Sampling shall be carried out by systematic sampling or stratified random sampling either on a mass basis (see 6.1 or 6.3.2) or on a time basis (see 6.2 or 6.3.3). However, if periodic variations in quality or quantity are present, sampling shall be restricted to stratified random sampling within fixed mass or time intervals (see 6.3.2 or 6.3.3).

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 impervious 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 mass of increment considering the nominal top size, the DRI- or HBI-handling equipment and the device for taking increments;

d) specify the precision required;

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

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

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

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

i) take increments having almost uniform mass for mass-basis sampling or having a mass proportional to the flow rate of the stream at the time of sampling for time-basis sampling; Increments are to be taken at the intervals determined in item g) 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) dry the samples, if necessary, except for the moisture sample;

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

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.

Sample containers for DRI and crushed HBI shall be suitable for storing and transporting the material in very well-protected conditions. Samples shall be stored in airtight containers and shall not be left unprotected from the atmosphere at any stage.

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 the 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 International Standard. 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, e.g. size distribution. 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 a regular verification of correct operation.

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 analysis. This is particularly important for new products or new sampling systems, or when significant changes are made to existing systems. Sampling systems should therefore be designed at the outset to enable constitution of duplicate samples for determining quality variation and for checking precision.

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, minimizing or preferably eliminating possible biases is more important than improving 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 extraction of increments, while sources that can be minimized but not completely eliminated include loss of dust and particle-size degradation (for size determination). Samples should be as dry as possible before crushing.

5.1.2 Minimization of particle-size degradation

Minimization of particle-size degradation of samples used for determination of size distribution is vital 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 the DRI or HBI has an equal opportunity of being selected and becoming part of the final sample for analysis, irrespective of the size, mass 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 DRI or HBI stream shall be taken when sampling from a moving stream (see 7.5) or a stopped belt (see Clause 9);

b) the aperture of the sample cutter shall be at least three times the nominal top size of the DRI or HBI for primary sampling, or 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 stream at uniform speed (see 7.5.3), both the leading and trailing edges of the cutter clearing the 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 DRI or HBI 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;

Sampling systems shall be designed to accommodate the maximum nominal top size and flow rate of the DRI or HBI 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 5.1.4.2 and 5.1.4.3). 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 primary sampler from the DRI or HBI stream at the discharge end of a conveyor belt is given by:

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

where

q is the flow rate, in tonnes per hour, of DRI or HBI on the conveyor belt;

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

v_c is the cutter speed, in metres per second, of the primary 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 DRI or HBI on the conveyor. It is given by the equation:

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

where

q is the flow rate, in tonnes per hour, of DRI or HBI on the conveyor belt;

l_2 is the length of the section of DRI or HBI removed from the conveyor, in metres;

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 the section of DRI or HBI removed from the conveyor, i.e. 3d, where d is the nominal top size of the DRI or HBI in metres, subject to a minimum of 0,01 m for DRI and crushed HBI. In practice, the section of HBI briquettes removed from a conveyor is usually 1 m.

5.2 Overall precision

This International Standard is designed to attain the overall precision, β_{SPM}, at a probability level of 95 %, given in Table 1 for the chemical (total iron, metallic iron, carbon, silica, alumina, phosphorus, sulfur and moisture content) and physical (percent size fraction, apparent density, bulk density, tumble index and abrasion index) characteristics of the lot. Higher precision values may be adopted if required. The precision shall be determined in accordance with ISO 3085.

The overall precision, β_{SPM}, is a measure of the combined precision of sampling, sample preparation and measurement, and is twice the standard deviation of sampling, sample preparation and measurement, σ_{SPM}, expressed as an absolute percentage, i.e.:

$$\sigma_{SPM} = \sqrt{\sigma_S^2 + \sigma_P^2 + \sigma_M^2} \quad (3)$$

$$\beta_{SPM} = 2\sigma_{SPM} = 2\sqrt{\sigma_S^2 + \sigma_P^2 + \sigma_M^2} \quad (4)$$

$$\sigma_S = \frac{\sigma_W}{\sqrt{n_1}} \quad (5)$$

where

σ_S is the sampling standard deviation;

σ_P is the sample-preparation standard deviation;

σ_M is the measurement standard deviation;

σ_W is the quality variation of the DRI or HBI;

n_1 is the number of primary increments.

Equations (3), (4) and (5) are based on the theory of stratified sampling (see Annex B for more details). The number of primary increments to be taken for a lot is dependent on the sampling precision required and on the quality variation of the DRI or HBI to be sampled. Thus, before the number of primary increments can be determined, it is necessary to define:

a) the sampling precision, β_S , to be attained;

b) the quality variation, σ_W, of the DRI or HBI to be sampled.

Table 1 — Overall precision, β_{SPM} (values as absolute percentages)

Quality characteristics		Approximate overall precision β_{SPM}		
		Mass of lot (t)		
		45 000 to 70 000	15 000 to 45 000	0 to 15 000
Total iron content		0,3	0,4	0,5
Metallic iron content		1,0	1,2	1,5
Carbon content		0,10	0,12	0,15
Silica content		0,10	0,12	0,15
Alumina content		0,10	0,12	0,15
Phosphorus content		0,002 0	0,002 4	0,003 0
Sulfur content		0,002 0	0,002 4	0,003 0
Moisture content		0,10	0,12	0,15
Size (– 31,5 + 6,3 mm DRI lump)	– 6,3 mm fraction mean 10 %	2,0	2,2	2,5
Size (DRI pellets)	– 6,3 mm fraction mean 5 %	0,8	0,9	1,0
Size (– 100 mm HBI)	– 25 + 6,3 mm fraction mean 10 %	0,3	0,4	0,5
	– 6,3 mm fraction mean 10 %	0,3	0,4	0,5
Apparent density (HBI only)		0,10	0,12	0,15
Bulk density		0,10	0,12	0,15
Tumble index		0,5	0,6	0,7
Abrasion index		0,5	0,6	0,7
NOTE The values of β_{SPM} are indicative and subject to confirmation through international tests.				

NOTE When on-line sample preparation takes place within the sample plant away from the preparation laboratory, the distinction between sampling and sample preparation becomes less clear. In this case, the precision of on-line sample preparation may be included in either the sampling precision or in the sample-preparation precision. Sample preparation is strictly a sampling operation, because a representative part of the sample is selected for subsequent processing. Hence, the most rigorous approach is to break up the sampling standard deviation into its components for each sampling stage, in which case Equation (3) becomes:

$$\sigma_{SPM} = \sqrt{\sigma_{S1}^2 + \sigma_{S2}^2 + \sigma_{S3}^2 + \sigma_P^2 + \sigma_M^2}$$

where

σ_{S1} is the sampling standard deviation for primary sampling;

σ_{S2} is the sampling standard deviation for secondary sampling;

σ_{S3} is the sampling standard deviation for tertiary sampling.

Using this approach, the precision of each sampling stage can be separately determined and optimized, resulting in a fully optimized sampling and sample-preparation regime.

5.3 Quality variation

The quality variation, σ_W, is a measure of the heterogeneity of the lot and is the standard deviation of the quality characteristics of increments within strata for mass-basis systematic sampling. The characteristics to be selected for determining quality variation include all the chemical and physical characteristics of the DRI or HBI being sampled.

The value of σ_W shall be measured experimentally for each type or brand of DRI or HBI and for each handling plant under normal operating conditions, in accordance with ISO 3084. The quality variation of the DRI or HBI may then be classified into three categories according to its magnitude as specified in Table 2. In the case of time-basis sampling, if the flow rate of the DRI or HBI is uniform on the belt, then time-basis sampling is the same as mass-basis sampling and ISO 3084 can be applied.

All DRI or HBI, of which the quality variation is unknown, shall be considered to have a “large” quality variation. In this case, measurements shall be conducted at the earliest possible opportunity in accordance with ISO 3084 to determine the quality variation.

When separate samples are taken for the determination of chemical composition and physical characteristics, the quality variation for the individual characteristics shall be adopted. When the sample is used for the determination of more than one quality characteristic, the largest classification category for quality variation shall be adopted.

5.4 Sampling precision and number of primary increments

5.4.1 Mass-basis sampling

When the value of σ_W is known, the number of primary increments, n_1, can be calculated for the desired sampling precision, β_S, as follows:

$$n_1 = \left(\frac{2\sigma_W}{\beta_S} \right)^2 \quad (6)$$

This is the preferable method of determining the number of primary increments. However, when the value of σ_W is classified in terms of large, medium or small quality variation in accordance with Table 2, Table 3 may be used to determine the minimum number of primary increments required for the sampling precision, β_S, specified in the table. In Table 3, the sampling precisions have been increased slightly for smaller lot sizes as a trade-off between sampling cost and the uncertainty in the commercial value of the lot.

NOTE 1 The values of β_S are indicative and subject to confirmation through international test work.

NOTE 2 The values of n_1 may be increased or decreased to alter the sampling precision. For example, if the number of increments is $2n_1$, then β_S will be improved by a factor of $1/\sqrt{2} = 0{,}71$; and if it is $n_1/2$, then β_S will be worsened by a factor of $\sqrt{2} = 1{,}4$.

5.4.2 Time-basis sampling

The minimum number of primary increments shall preferably be determined using Equation (6), but Table 3 may also be used, as specified in 5.4.1.