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Iron ores — Sampling and sample preparation procedures

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National foreword

This British Standard is the UK implementation of ISO 3082:2017. It supersedes BS ISO 3082:2009, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Iron ores — Sampling and sample preparation procedures

Minerais de fer — Procédures d'échantillonnage et de 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.

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;

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— 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.

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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*

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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

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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

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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;

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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.

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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.

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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. $3d$, 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

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ρ_b is the bulk density of the ore with nominal top size less than 1 mm, in kilograms per cubic metre;

l_4 is the depth of ore with nominal top size less than 1 mm in the truck or wagon, in metres.

The minimum increment mass that can be taken, while still avoiding bias, is determined by the minimum diameter of the spear or auger, i.e. 30 mm.

This method of extracting increments is only applicable to sampling ore with nominal top size less than 1 mm.

5.2 Overall precision

This document is designed to attain the overall precision, β_{SPM}, at a probability level of 95 %, given in Table 1 for the total iron, silica, alumina, phosphorus, and moisture contents and the percent size fraction of the lot. Better precision, i.e. a lower value of β_{SPM}, may be adopted if required. The precision shall be determined in accordance with ISO 3085.

NOTE The overall precision for other physical characteristics and metallurgical properties is not specified in this document, because they are used to qualitatively compare the behaviour of iron ores during handling and reduction processes.

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.

$$\sigma_{SPM} = \sqrt{\sigma_S^2 + \sigma_P^2 + \sigma_M^2} \tag{4}$$

$$\beta_{SPM} = 2\sigma_{SPM} = 2\sqrt{\sigma_S^2 + \sigma_P^2 + \sigma_M^2} \tag{5}$$

$$\sigma_S = \frac{\sigma_W}{\sqrt{n_1}} \tag{6}$$

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 ore;

n_1 is the number of primary increments.

Formulae (4), (5) and (6) are based on the theory of stratified sampling (see Annex B for 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 ore 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, and

b) the quality variation, σ_W, of the ore to be sampled.

When on-line sample preparation takes place within the sample plant away from the preparation laboratory, the distinction between the terms sampling and sample preparation becomes unclear. The precision of on-line sample preparation may be included in either the sampling precision or in the sample preparation precision. The choice depends on how easy it is to separate the precision of secondary and

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tertiary sampling from that of primary sampling. In any event, sample preparation also constitutes a sampling operation, because a representative part of the sample is selected for subsequent processing.

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

Quality characteristics		Approximate overall precision β_{SPM}									
		Mass of lot t									
		Over 340 000	270 000 to 340 000	210 000 to 270 000	150 000 to 210 000	100 000 to 150 000	70 000 to 100 000	45 000 to 70 000	30 000 to 45 000	15 000 to 30 000	Less than 15 000
Iron content		0,33	0,34	0,35	0,37	0,38	0,40	0,42	0,45	0,49	0,55
Silica content		0,33	0,34	0,35	0,37	0,38	0,40	0,42	0,45	0,49	0,55
Alumina content		0,10	0,11	0,12	0,12	0,13	0,14	0,15	0,16	0,18	0,20
Phosphorus content		0,005 7	0,005 8	0,005 9	0,006 3	0,006 5	0,006 8	0,007 2	0,007 7	0,008 4	0,009 4
Moisture content		0,33	0,34	0,35	0,37	0,38	0,40	0,42	0,45	0,49	0,55
Size of 200 mm nominal top size ore	−10 mm fraction, mean 20 %	3,3	3,4	3,5	3,6	3,7	3,9	4,0	4,2	4,4	5,0
Size of 50 mm nominal top size ore											
Size of −31,5 + 6,3 mm sized ore	−6,3 mm fraction, mean 10 %	1,65	1,7	1,75	1,8	1,85	1,95	2,0	2,1	2,2	2,5
Size of sinter feed	+6,3 mm fraction, mean 10 %										
Size of pellet feed	−45 µm fraction, mean 70 %										
Size of pellets	−6,3 mm fraction, mean 5 %	0,66	0,68	0,70	0,72	0,74	0,78	0,80	0,84	0,88	1,00
NOTE The values of β_{SPM} for silica, alumina and phosphorus content are indicative and subject to confirmation through international testwork.											

The most rigorous approach is to break up the sampling standard deviation into its components for each sampling stage, in which case Formula (4) becomes Formula (7):

$$\sigma_{SPM} = \sqrt{\sigma_{S1}^2 + \sigma_{S2}^2 + \sigma_{S3}^2 + \sigma_P^2 + \sigma_M^2} \tag{7}$$

where

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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 the iron, silica, alumina, phosphorus, and moisture contents and the percentage of a given size fraction.

The value of σ_W shall be measured experimentally for each type or brand of iron ore and for each handling plant under normal operating conditions, in accordance with ISO 3084. The quality variation of the iron ore 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 ore is uniform on the belt, then time-basis sampling is the same as mass-basis sampling and ISO 3084 can be applied.

For ores whose quality variation is unknown, measurements shall be conducted at the earliest opportunity in accordance with ISO 3084 to determine the quality variation. Prior to the determination of quality variation, the classification adopted shall be as follows:

a) when no prior information exists on the quality variation of the ore or similar ores, the ore shall be considered to have “large” quality variation;

b) when prior information exists on the quality variation of a similar ore, the quality variation classification of that ore shall be adopted as the starting point.

When separate samples are taken for the determination of chemical composition, moisture content and size distribution, the quality variation for the individual characteristics shall be adopted. When separate samples are taken for the determination of other physical characteristics or metallurgical properties not specified in Table 2, large quality variation should be used. When the sample is used for the determination of more than one quality characteristic, the largest classification category for quality variation in Table 2 shall be adopted.

For small lots, it may not be possible to take the number of increments specified by Formula (8) for large quality variation. In this case, the maximum number of increments possible shall be taken, but in an attempt to compensate for the poorer sampling precision, the precision of sample preparation shall be improved to achieve the required overall precision β_{SPM}, e.g. by preparing and analysing more partial samples. The procedure adopted should be recorded in the sampling report.

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Table 2 — Classification of quality variation σ_W (values as absolute percentages)

Quality characteristics		Classification of quality variation σ_W		
		Large	Medium	Small
Iron content		$\sigma_W \geq 2{,}0$	$2{,}0 > \sigma_W \geq 1{,}5$	$\sigma_W < 1{,}5$
Silica content		$\sigma_W \geq 2{,}0$	$2{,}0 > \sigma_W \geq 1{,}5$	$\sigma_W < 1{,}5$
Alumina content		$\sigma_W \geq 0{,}6$	$0{,}6 > \sigma_W \geq 0{,}4$	$\sigma_W < 0{,}4$
Phosphorus content		$\sigma_W \geq 0{,}015$	$0{,}015 > \sigma_W \geq 0{,}011$	$\sigma_W < 0{,}011$
Moisture content		$\sigma_W \geq 2{,}0$	$2{,}0 > \sigma_W \geq 1{,}5$	$\sigma_W < 1{,}5$
Size of 200 mm nominal top size ore	−10 mm fraction, mean 20 %	$\sigma_W \geq 10$	$10 > \sigma_W \geq 7{,}5$	$\sigma_W < 7{,}5$
Size of 50 mm nominal top size ore				
Size of − 31,5 + 6,3 mm sized ore	−6,3 mm fraction, mean 10 %	$\sigma_W \geq 5$	$5 > \sigma_W \geq 3{,}75$	$\sigma_W < 3{,}75$
Size of sinter feed	+6,3 mm fraction, mean 10 %			
Size of pellet feed	−45 µm fraction, mean 70 %	$\sigma_W \geq 3$	$3 > \sigma_W \geq 2{,}25$	$\sigma_W < 2{,}25$
Size of pellets	−6,3 mm fraction, mean 5 %			

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 shown by Formula (8):

$$n_1 = \left(\frac{2\sigma_W}{\beta_S} \right)^2 \tag{8}$$

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 Table 3. The theoretical background is given in Annex B. In Table 3, the levels of sampling precision have been increased slightly for smaller lot sizes as a trade-off between sampling cost and the uncertainty in the value of the lot.

5.4.2 Time-basis sampling

The minimum number of primary increments shall preferably be determined using Formula (8), but Table 3 may also be used, as specified in 5.4.1.

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Table 3 — Example of minimum number of increments required, n_1, for desired sampling precision, β_S

Mass of lot 1 000 t		Sampling precision β_S						Number of primary increments n_1		
Over	Up to	Fe, SiO_2 or moisture content	Al_2O_3 content	P content	200 mm or 50 mm nominal top size ores, – 10 mm fraction	– 31,5 mm + 6,3 mm sized ore, – 6,3 mm fraction	Pellet feed, – 45 µm fraction	Quality variation Large (L), Medium (M) or Small (S)		
						Sinter feed, + 6,3 mm fraction	Pellets, – 6,3 mm fraction	L	M	S
340		0,30	0,08	0,003 7	1,49	0,75	0,45	280	140	70
270	340	0,31	0,09	0,003 9	1,55	0,78	0,47	260	130	65
210	270	0,32	0,09	0,004 1	1,61	0,81	0,48	240	120	60
150	210	0,34	0,10	0,004 2	1,69	0,84	0,51	220	110	55
100	150	0,35	0,10	0,004 4	1,77	0,88	0,53	200	100	50
70	100	0,37	0,10	0,004 5	1,86	0,93	0,56	180	90	45
45	70	0,39	0,11	0,004 9	1,98	0,99	0,59	160	80	40
30	45	0,42	0,12	0,005 2	2,11	1,06	0,63	140	70	35
15	30	0,45	0,13	0,005 7	2,28	1,14	0,68	120	60	30
0	15	0,50	0,14	0,006 2	2,50	1,25	0,75	100	50	25

NOTE The values of n_1 can be increased or decreased to alter the sampling precision; e.g. 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 $\sqrt{2} = 1{,}4$.

5.5 Precision of sample preparation and overall precision

5.5.1 General

The precision of sample preparation depends on the choice of the sample preparation scheme. It can be improved if sample preparation is carried out first on individual increments or partial samples at an appropriate stage of sample preparation and then the divided increments or partial samples are combined into a gross sample.

The precision of sample preparation and measurement, β_{PM}, for size determination and other physical tests shall be better than that specified in Tables 5 and 6 respectively for each ore type. For checking β_{PM}, methods 1 and 2 of ISO 3085 shall be used.

The overall precision in terms of the standard deviation, σ_{SPM}, where sample preparation and measurement are carried out on the gross sample, on each of the partial samples or on each of the increments is specified below.

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5.5.2 Preparation and measurement of gross sample

When a gross sample for a lot is constituted by combining all increments and n_2 measurements are carried out on the gross sample, the overall precision will be as shown by Formula (9):

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \sigma_{\mathrm{P}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_2} \tag{9}$$

where σ_{P} is the precision of preparing a test sample from the gross sample.

5.5.3 Preparation and measurement of partial samples

When n_3 partial samples consisting of an equal number of increments are constituted and n_2 measurements are carried out on each partial sample, the overall precision will be as shown by Formula (10):

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \frac{\sigma_{\mathrm{P}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_2}}{n_3} \tag{10}$$

where σ_{P} is the precision of preparing a test sample from each partial sample.

Furthermore, when the above n_3 partial samples are combined into a gross sample at an appropriate (10 mm nominal top size or less) stage after individual sample preparation, and n_2 measurements are carried out on the gross sample, the overall precision will be as shown by Formula (11):

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \frac{\sigma_{\mathrm{P1}}^2}{n_3} + \sigma_{\mathrm{P2}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_2} \tag{11}$$

where

σ_{P1} is the precision of preparing each partial sample prior to constituting the gross sample;

σ_{P2} is the precision of preparing a test sample from the gross sample.

5.5.4 Preparation and measurement of each increment

When n_2 measurements are carried out on each increment, the overall precision will be as shown by Formula (12):

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \frac{\sigma_{\mathrm{P}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_2}}{n_1} \tag{12}$$

where

σ_{P} is the precision of preparing a test sample from each increment;

n_1 is the number of primary increments.

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Furthermore, when all the increments are combined into a gross sample at an appropriate stage (10 mm nominal top size or less) after individual sample preparation, and n_2 measurements are carried out on the gross sample, the overall precision will be as shown by Formula (13):

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \frac{\sigma_{\mathrm{P1}}^2}{n_1} + \sigma_{\mathrm{P2}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_2} \tag{13}$$

where

σ_{P1} is the precision of preparing each increment prior to constituting the gross sample;

σ_{P2} is the precision of preparing a test sample from the gross sample.

NOTE Each sample preparation stage has its own variance, so the total variance will be greater than that for a single stage. It is desirable to use larger samples for those stages of sample preparation for which this does not greatly increase costs. This needs to be taken into account when optimizing sample preparation schemes.

6 Methods of sampling

6.1 Mass-basis sampling

6.1.1 Mass of increment

The mass of increment shall be determined in accordance with 5.1.4.

Increments shall be taken so that they are of "almost uniform mass", i.e. the coefficient of variation of increment masses shall be less than 20 %. The coefficient of variation, CV, is defined as the ratio of the standard deviation, σ_{mass}, to the mean value, $\bar{m}$, of the mass of the increments, expressed as a percentage as shown by Formula (14):

$$\mathrm{CV} = \frac{100\sigma_{\mathrm{mass}}}{\bar{m}} \tag{14}$$

For example, if the average mass of increment is to be 100 kg, the increments should be taken in such a manner that 95 % of the increments vary between 60 kg and 140 kg, with an average of 100 kg. Provision shall therefore be made, either in the manner in which the increments are taken or by subsequent weighing and division of each increment, to ensure that they have almost uniform mass.

To obtain increments of almost uniform mass, one or more of the following measures shall be taken:

a) control of the ore flow on the conveyor belt ahead of the sampling point to reduce variations in flow rate;

b) installation of a variable-speed cutter which varies its speed increment-by-increment so that the speed is proportional to the flow rate of ore on the conveyor belt at the time of taking each increment;

c) installation of equipment which rejects increments of non-uniform mass and immediately restarts the primary sampler.

If the coefficient of variation of increment masses is 20 % or greater, each increment may be subjected to division (in accordance with the rules of division) and the quality characteristics determined. Alternatively, divided increments of "almost uniform mass" may be combined at an appropriate stage of division into a partial sample or a gross sample (see 10.2.2.1).

6.1.2 Quality variation

The quality variation shall be determined experimentally in accordance with ISO 3084.

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6.1.3 Number of primary increments

The number of primary increments shall be determined in accordance with 5.4.1.

6.1.4 Sampling interval

The mass interval, Δm, in tonnes, between increments shall be calculated from Formula (15):

$$\Delta m \leq \frac{m_L}{n_1} \tag{15}$$

where

m_L is the mass, in tonnes, of the lot;

n_1 is the number of primary increments determined in 5.4.1.

The mass interval selected shall be smaller or equal to that calculated above to ensure that the minimum number of primary increments is at least that determined in accordance with 5.4.1.

6.1.5 Methods of taking increments

Each increment shall be taken at one time by a single motion or by a complete cycle of the sampling device so that a full cross-section of the ore stream is taken. Free-fall drops of increments shall be kept to a minimum to reduce size degradation of the ore and hence minimize bias in size distribution.

NOTE 1 A complete cycle can involve the sampler taking a forward and return cut through the ore stream.

NOTE 2 Stopped-belt sampling can also be used to take a full cross-section of the ore stream.

The first increment shall be taken after a randomly selected tonnage has been handled within the first mass interval after commencing the handling operation. Subsequent increments shall be taken at the fixed mass interval determined in 6.1.4 until handling of the lot has been completed. When the calculated mass of the sample is less than that required for testing (size determination, physical testing, etc.), the number and/or mass of the increments shall be increased.

Either of the following two kinds of cutters may be employed for the primary sampler:

a) a fixed-speed cutter whose cutting speed is constant during the course of handling the entire lot;

b) a variable-speed cutter whose cutting speed is constant while cutting the stream but can be regulated, increment by increment, according to the flow rate of ore on the conveyor belt.

Sampling shall be carried out at the nearest possible point to the loading or discharging facilities, preferably immediately before or after the point of weighing.

6.2 Time-basis sampling

6.2.1 Mass of increment

The mass of increment shall be proportional to the flow rate of the ore stream at the time of sampling. When a test sample is prepared from each increment or partial sample, the mass of each increment or partial sample shall be determined in order to obtain the weighted mean of the quality characteristics for the lot. Alternatively, the tonnage of ore that the sample represents may be used to obtain the weighted mean.

6.2.2 Quality variation

When the variation in the ore flow rate is less than 20 % in terms of the coefficient of variation, ISO 3084 shall be used to obtain an approximate value for the quality variation.

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6.2.3 Number of increments

The number of primary increments shall be determined in accordance with 5.4.2.

6.2.4 Sampling interval

The time interval, Δt, in minutes, between increments shall be calculated from Formula (16):

$$\Delta t \leq \frac{60 m_L}{q_{max} n_1} \tag{16}$$

where

m_L is the mass, in tonnes, of the lot;

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

n_1 is the number of primary increments determined in 5.4.2.

The selected time interval between taking increments shall be smaller or equal to that calculated to ensure that the minimum number of primary increments is at least that determined in accordance with 5.4.2.

6.2.5 Methods of taking increments

Each increment shall be taken at one time by a single motion or by a complete cycle of the sampling device so that a full cross-section of the ore stream is taken. Free-fall drops of increments shall be kept to a minimum to reduce size degradation of the ore and hence minimize bias in size distribution.

NOTE 1 A sampler can take a forward and return cut through the ore stream in a complete cycle.

NOTE 2 Stopped-belt sampling can also be used to take a full cross-section of the ore stream.

The first increment shall be taken at random within the first time interval from the start of the handling operation. Subsequent increments shall be taken at the fixed time interval determined in 6.2.4 until handling of the lot is completed. When the calculated mass of the sample is less than that required for testing (size determination, physical testing, etc.), the sampling interval shall be shortened.

A fixed-speed cutter whose cutting speed is constant during the course of handling the entire lot shall be employed for the primary sampler.

Sampling shall be carried out at the nearest possible point to the loading or discharging facilities, preferably immediately before or after the point of weighing.

6.3 Stratified random sampling within fixed mass or time intervals

6.3.1 General

Sampling shall preferably be carried out by systematic sampling either on a mass basis (6.1) or on a time basis (6.2). However, when periodic variations in quality or quantity occur with a period approximately equal to any multiple of the proposed sampling interval, stratified random sampling within fixed mass or time intervals should be used.

Due to the nature of stratified random sampling, successive increments may be collected close together in space or time. Consequently, the sampling system shall be designed to handle two increments in quick succession.

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6.3.2 Fixed mass intervals

For stratified random sampling within fixed mass intervals, the procedure is as specified in 6.1 except that, when the mass interval has been set, the sample cutter is programmed to take one primary increment at random within this mass interval. This is achieved by using a random number generator, capable of giving a random mass number within the mass interval (determined in 6.1.4), which activates the sample cutter at the mass corresponding to the mass number generated.

6.3.3 Fixed time intervals

For stratified random sampling within fixed time intervals, the procedure is as specified in 6.2 except that, when the time interval has been set, the sample cutter is programmed to take one primary increment at random within this time interval. This is achieved by using a random number generator, capable of giving a random time number within the time interval (determined in 6.2.4), which activates the sample cutter at the time corresponding to the time number generated.

7 Sampling from moving streams

7.1 General

The basic requirements together with typical examples are described as a guide to the design and operation of sampling and sample preparation systems for moving streams. These requirements shall be taken into account from the early stages of design and engineering as well as during operation and maintenance of the systems.

This document deals only with sample cutters that take a complete cross-section of the ore stream. Sample cutters taking only part of the stream are incorrect in design, and cannot be relied on to provide representative samples, i.e. they may introduce significant bias.

It is not essential to construct or operate the sampling system as a single system. Any principal unit or combination of principal units may be operated mechanically and combined at any stage with manual operation to form a complete sampling and sample preparation system. A manual sample cutter may also be used, subject to the safety considerations mentioned in 7.2.

The sampling system shall be operated in accordance with the requirements of Clauses 5 and 6, which specify the mass of increment, number of increments, and sampling interval for mass-basis, time-basis and stratified random sampling. Operation of the system should be monitored at all times during sampling and sample preparation of a lot. In the event of a breakdown or failure of the installation, the mechanical operation shall be replaced immediately by a manual sampling procedure.

The samples taken manually should be processed separately from those taken mechanically.

Care shall be taken not to alter the quality of a lot after sampling at loading and prior to sampling at discharge. Where water is sprayed on a cargo for dust suppression or where water is removed from a lot, a correction for the water shall be made in accordance with ISO 3087.

7.2 Safety of operations

From the initial stage of design and construction of sampling systems, due consideration shall be given to the safety of operators. Local or national safety codes shall be respected.

It is recommended that mechanical sampling be used if the speed of the conveyor belt is high or the mass of the ore handled by the conveyor belt is large. Unless stopped-belt sampling is adopted, the use of manual sampling in such cases could be dangerous for the sampling staff.

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7.3 Robustness of sampling installation

Sampling and sample preparation systems shall be designed and constructed robustly to fulfil without failure their required function under given conditions at all times.

In the event of a breakdown of the installation or if the installation is unsuitable for a particular ore (e.g. an excessively sticky ore), an alternative sampling procedure should be available. For example, increments taken by the primary sampler may be bypassed through a preinstalled facility (e.g. a short conveyor, a concrete pad or a receiving truck) so that manual sample preparation can be performed.

It is recommended that mechanical sampling systems be arranged in such a way that the principal units can be operated individually to facilitate repair in the event of breakdown.

7.4 Versatility of sampling system

The design of sampling and sample preparation systems shall be

a) determined by the types of ore likely to be handled, the quality characteristics to be determined and the desired precision of sampling and sample preparation, and

b) such that bias is not introduced.

In all cases the minimum mass and number of increments comprising a sample shall comply with 5.1.4 and 5.4, respectively, in order to attain the specified precision and the required mass of sample for testing.

The size sample shall be taken before any crushing takes place. Multiple use of increments taken to constitute a sample is permissible, provided that the general procedures given in Clause 4 are fulfilled. If size determination is carried out on a sample that will subsequently be used for other purposes, care shall be taken to ensure that the size fractions are fully remixed before subsequent sample preparation is undertaken.

The installation shall be designed so that check experiments can be carried out in conjunction with routine sampling. Sampling systems should be capable of combining alternate increments to constitute pairs of interleaved samples, designated A and B, for determination of quality variation in accordance with ISO 3084 and for checking the precision of sampling in accordance with ISO 3085. To meet the sampling requirements of ISO 3085, the primary sampler should also be capable of taking at least twice the number of increments, n_1, specified in 5.4. When these design features are in place, it is recommended that the precision of sampling be determined routinely in accordance with ISO 3085 as part of normal sampling operations.

7.5 Primary samplers

7.5.1 Location

The primary sampler shall be installed at a point where the entire lot may be sampled. It should be installed at the nearest point to the loading or discharging facilities as close as possible to the point of weighing.

7.5.2 Types of primary sampler

There are several types of primary sampler, which vary in mode of operation and shape. The most widely accepted is a cutter-type primary sampler installed at the discharge end of a conveyor belt and designed to collect increments by cutting a complete cross-section of the ore stream, traveling through the ore stream at uniform speed. Increments should preferably be taken from a falling stream using a mechanical sample cutter, although a manual cutter may also be used if the ore flow rate is very low (see 7.2).

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Examples of mechanical cutter-type samplers are shown diagrammatically in Figure 1. An example of a manual sample cutter is shown in Figure 2.

7.5.3 General design criteria for primary cutters

To avoid bias, the primary sampler shall satisfy the following design criteria:

a) there shall be no overflow or spillage of sample or loss of ultra-fines;

b) there shall be no impedance to flow of sample material through the sample cutter at the maximum flow rate;

c) bucket-type cutters shall be of sufficient capacity to accommodate the increment mass obtained at the maximum flow rate of the ore;

d) there shall be no clogging or retention of residual material in the sample cutter, i.e. the cutter shall be self-clearing;

e) there shall be no contamination or introduction of material other than the sample into the sample cutter;

f) there shall be no significant change of the quality of the sample while taking increments, e.g. degradation of the constituent particles if the sample is taken for size determination or change in moisture content if the sample is taken for moisture determination;

g) the sample cutter shall take a complete cross-section of the ore stream, both the leading and trailing edges clearing the stream in one path;

h) the sample cutter shall intersect the ore stream either in a plane perpendicular to or along an arc normal to the mean trajectory of the stream;

i) the sample cutter shall travel through the ore stream at a uniform speed, not deviating by more than ± 5 % at any point;

j) the geometry of the cutter aperture shall be such that the cutting time at each point in the stream is equal, not deviating by more than ± 5 %, e.g. straight-path cutters shall have parallel cutter lips and rotary cutters shall have radial cutter lips;

k) the plane of the cutter aperture shall not be vertical or near-vertical.

An example of a checklist for mechanical sampling systems is given in Annex A.

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