
**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.

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.

International Standard ISO 3082 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 1, *Sampling*.

This second edition cancels and replaces the first edition (ISO 3082:1987), together with ISO 3081:1986 and ISO 3083:1986, of which it constitutes a collation and technical revision.

Annexes B, D and E form an integral part of this International Standard. Annexes A, C and F are for information only.

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Iron ores — Sampling and sample preparation procedures

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;
- 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 size distribution of the lot. Sampling and sample preparation procedures for physical testing are specified in ISO 10836.

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

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All Standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 3084:—¹⁾, *Iron ores — Experimental methods for evaluation of quality variation*.

ISO 3085:1996, *Iron ores — Experimental methods for checking the precision of sampling*.

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

1) To be published. (Revision of ISO 3084:1986)

ISO 3087:—²⁾, *Iron ores — Determination of moisture content of a lot.*

ISO 4701:1985, *Iron ores — Determination of size distribution by sieving.*

ISO 10836:1994, *Iron ores — Method of sampling and sample preparation for physical testing.*

ISO 11323:1996, *Iron ores — Vocabulary.*

3 Definitions

For the purposes of this International Standard the definitions contained in ISO 11323 as well as those given below apply.

3.1

lot

discrete and defined quantity of ore for which quality characteristics are to be assessed

3.2

increment

quantity of ore collected in a single operation of a sampling device

3.3

sample

relatively small quantity of ore, so taken from a lot as to be representative in respect of the quality characteristics to be assessed

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3.4

partial sample

sample, consisting 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

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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

2) To be published. (Revision of ISO 3087:1987)

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

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3.16**nominal top size**

smallest aperture size, within the range of the R20 Series (in ISO 565, square opening), such that no more than 5 % (m/m) of an 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 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 ore stream can be conveniently intercepted at regular intervals, enabling representative samples to be obtained.

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 conveyor belt when ore is being conveyed to or from the ship, stockpile, container or bunker.

In-situ sampling from stationary situations such as wagons is permitted only for fine iron ore concentrates, provided the sampling device, e.g., a spear or an auger, penetrate 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 variation in quality or quantity. If this is not the case, 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 impervious air-tight 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;
- b) ascertain the nominal top size;
- c) determine the mass of increment considering the nominal top size, the ore 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 "large" quality variation as specified in 5.3;
- f) determine the minimum number of primary increments, n , 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 ore stream at the time of sampling for time basis sampling. Increments are to be taken at the intervals determined in f) 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;
- 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.

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 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. 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 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.

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 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 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 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 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 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 concentrate 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 7, 8, 9 and 10.

5.1.4 Increment mass

The increment mass required to obtain an unbiased sample can be calculated for typical sampling situations (see 5.1.4.1, 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.

5.1.4.1 Increment mass for falling stream sampling

The mass of increment, m_1 , in kilograms, to be taken (mechanically or manually) by a cutter-type primary sampler from the ore stream at the discharge end of a conveyor belt is given by:

$$m_1 = \frac{q l_1}{3,6 v_c} \quad \dots(1)$$

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 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.

For practical reasons, e.g. in the case of lumpy ore, it may be necessary for the cutter aperture to exceed three times the nominal top size of the ore.

5.1.4.2 Increment mass for stopped-belt sampling

The mass of increment, m_1 , in kilograms to be taken manually from a stopped-belt is equal to the mass of a complete cross-section (of length l_2) of the ore on the conveyor. It is given by the equation:

$$m_1 = \frac{q l_2}{3\,600 v_B} \quad \dots(2)$$

where

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

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 10 mm.

5.1.4.3 Increment mass for manual sampling using spear or auger

The mass of increment, m_1 , in kilograms to be taken from a wagon in a lot using a spear or an auger of diameter, l_3 , in millimetres, is given by:

$$m_1 = \frac{\pi \rho l_3^2 L}{4\ 000} \quad \dots(3)$$

where

ρ is the bulk density of the fine ore (particle size < 1 mm), in tonnes per cubic metre;

L is the depth of concentrate in the 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 fine iron ore concentrates.

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 total iron, silica, alumina, phosphorus, and moisture contents and the percent size fraction of the lot. The overall precision for lots of intermediate mass to those shown in table 1 may be obtained by linear interpolation. Higher precision 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.

$$\sigma_{\text{SPM}} = \sqrt{\sigma_{\text{S}}^2 + \sigma_{\text{P}}^2 + \sigma_{\text{M}}^2} \quad \dots(4)$$

$$\beta_{\text{SPM}} = 2\sigma_{\text{SPM}} = 2\sqrt{\sigma_{\text{S}}^2 + \sigma_{\text{P}}^2 + \sigma_{\text{M}}^2} \quad \dots(5)$$

$$\sigma_{\text{S}} = \frac{\sigma_{\text{W}}}{\sqrt{n_1}} \quad \dots(6)$$

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.

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

Quality characteristics	Approximate overall precision			
	(β_{SPM})			
	Mass of lot			
	(t)			
	210 000 to 270 000	45 000 to 70 000	0 to 15 000	
Iron content	0,35	0,4	0,5	
Silica content	0,35	0,4	0,5	
Alumina content	0,1	0,15	0,2	
Phosphorus content	0,002	0,003	0,004	
Moisture content	0,35	0,4	0,5	
Size -200 mm ore	-10 mm fraction mean 20 %	3,5	4,0	5,0
Size -50 mm ore				
Size -31,5 +6,3 mm ore	-6,3 mm fraction mean 10 %			
Size - sinter feed	+6,3 mm fraction mean 10 %	1,75	2,0	2,5
Size - pellet feed	-45 μ m fraction mean 70 %			
Size - pellets	-6,3 mm fraction mean 5 %	0,7	0,8	1,0

Equations (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;
- 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 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.

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

$$\sigma_{\text{SPM}} = \sqrt{\sigma_{\text{S1}}^2 + \sigma_{\text{S2}}^2 + \sigma_{\text{S3}}^2 + \sigma_{\text{P}}^2 + \sigma_{\text{M}}^2} \quad \text{..(7)}$$

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 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.

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		$\sigma_w \geq 2,0$	$2,0 > \sigma_w \geq 1,5$	$\sigma_w < 1,0$
Alumina content		$\sigma_w \geq 0,6$	$0,6 > \sigma_w \geq 0,4$	$\sigma_w < 0,5$
Phosphorus content		$\sigma_w \geq 0,012$	$0,012 > \sigma_w \geq 0,009$	$\sigma_w < 0,01$
Moisture content		$\sigma_w \geq 2,0$	$2,0 > \sigma_w \geq 1,5$	$\sigma_w < 1,5$
Size of -200 mm ore	-10 mm fraction mean 20 %	$\sigma_w \geq 10$	$10 > \sigma_w \geq 7,5$	$\sigma_w < 7,5$
Size of -50 mm ore				
Size of -31,5 +6,3 mm 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				
Size of pellet feed	-45 μ m 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 %			

Any ore whose quality variation is unknown shall be considered to have "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, moisture content, size distribution, etc., 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:

$$n_1 = \left(\frac{2\sigma_w}{\beta_s} \right)^2 \tag{8}$$

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 the table. The theoretical background is given in annex B. 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 value of the lot.

Table 3 — Example of minimum number of increments required, n_i , for desired sampling precision, β_s

Mass of lot (1 000 t)		Sampling precision (β_s)						Number of primary increments (n_i)		
Over	Up to	Fe, SiO ₂ or moisture content	Al ₂ O ₃ content	P content	−200 mm or −50 mm ores, −10 mm fraction	−31,5 mm ores, −6,3 mm fraction	Pellet feed, −45 μm fraction	Quality variation large (L), medium (M) or small (S)		
								L	M	S
						Sinter feed, +6,3 mm fraction	Pellets, −6,3 mm fraction			
270		0,31	0,09	0,001 8	1,55	0,77	0,47	260	130	65
210	270	0,32	0,09	0,001 9	1,61	0,80	0,48	240	120	60
150	210	0,34	0,10	0,002 0	1,69	0,84	0,51	220	110	55
100	150	0,35	0,10	0,002 1	1,77	0,88	0,53	200	100	50
70	100	0,37	0,11	0,002 2	1,86	0,92	0,56	180	90	45
45	70	0,39	0,11	0,002 3	1,98	0,98	0,59	160	80	40
30	45	0,42	0,12	0,002 5	2,11	1,05	0,63	140	70	35
15	30	0,45	0,13	0,002 7	2,28	1,13	0,68	120	60	30
0	15	0,50	0,14	0,003 0	2,50	1,24	0,75	100	50	25

NOTE — The values of n_i may be increased or decreased to alter the sampling precision. For example, if the number of increments is $2n_i$, then β_s will be improved by a factor of $1/\sqrt{2} = 0,71$; and if it is $n_i/2$, 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 (8), but table 3 may also be used, as specified in 5.4.1.