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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;

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

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

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

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

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

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

Iron ores — Sampling and sample preparation procedures

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

1 Scope

This document provides

a) the underlying theory,

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

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

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

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

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3 Terms and definitions

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

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3.16

nominal top size

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

4 General considerations for sampling and sample preparation

4.1 Basic requirements

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

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

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

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

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

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

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

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

4.2 Establishing a sampling scheme

The procedure for establishing a sampling scheme is as follows:

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

b) ascertain the nominal top size;

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

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

e) specify the precision required;

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

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

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

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

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

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

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

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

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

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

p) prepare the test sample.

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

4.3 System verification

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

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

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

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

5 Fundamentals of sampling and sample preparation

5.1 Minimization of bias

5.1.1 General

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

5.1.2 Minimization of particle size degradation

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

5.1.3 Extraction of increments

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

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

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

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

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

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

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

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

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

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

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

5.1.4 Increment mass

5.1.4.1 General

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

5.1.4.2 Increment mass for falling stream sampling

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

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

where

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

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

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

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

5.1.4.3 Increment mass for stopped-belt sampling

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

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

where

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

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

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

The minimum increment mass that can be taken, while still avoiding bias, is determined by the minimum length of ore removed from the conveyor, i.e. 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

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.

$$\sigma_{\mathrm{SPM}} = \sqrt{\sigma_{\mathrm{S}}^2 + \sigma_{\mathrm{P}}^2 + \sigma_{\mathrm{M}}^2} \tag{4}$$

$$\beta_{\mathrm{SPM}} = 2\sigma_{\mathrm{SPM}} = 2\sqrt{\sigma_{\mathrm{S}}^2 + \sigma_{\mathrm{P}}^2 + \sigma_{\mathrm{M}}^2} \tag{5}$$

$$\sigma_{\mathrm{S}} = \frac{\sigma_{\mathrm{W}}}{\sqrt{n_1}} \tag{6}$$

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 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

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.

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.

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 Sinter feed, + 6,3 mm fraction	Pellet feed, – 45 µm fraction Pellets, – 6,3 mm fraction	Quality variation Large (L), Medium (M) or Small (S)		
								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.

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):

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

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):

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

where

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

n_1 is the number of primary increments.

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.

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):

$$\Delta m \leq \frac{m_L}{n_1} \qquad (15)$$

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.

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.

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.

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).

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;

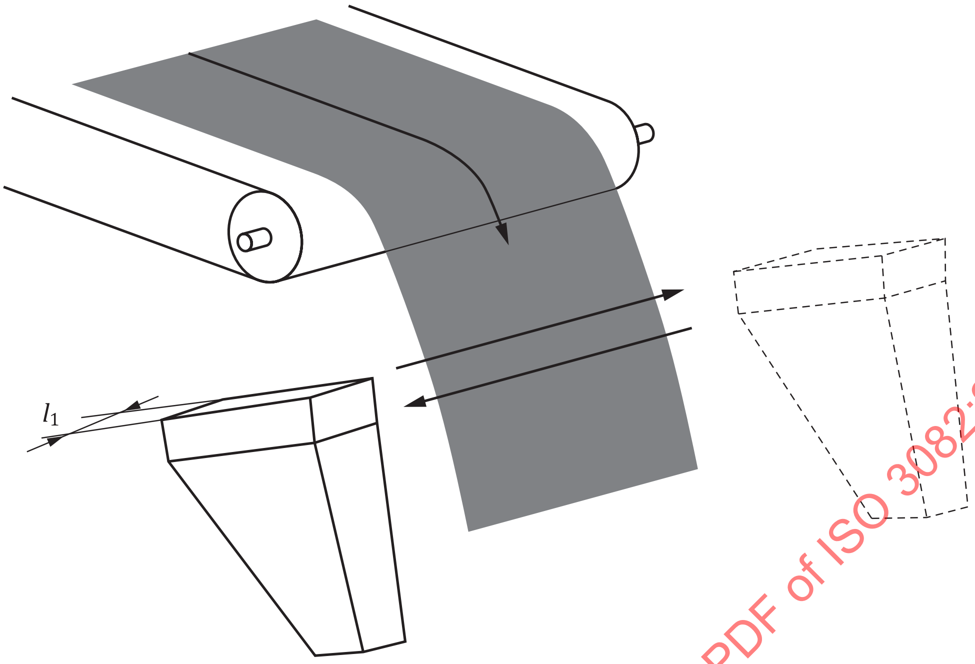
h) the sample cutter shall intersect the ore stream either in a plane perpendicular to or along an arc normal to the mean trajectory of the stream;

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

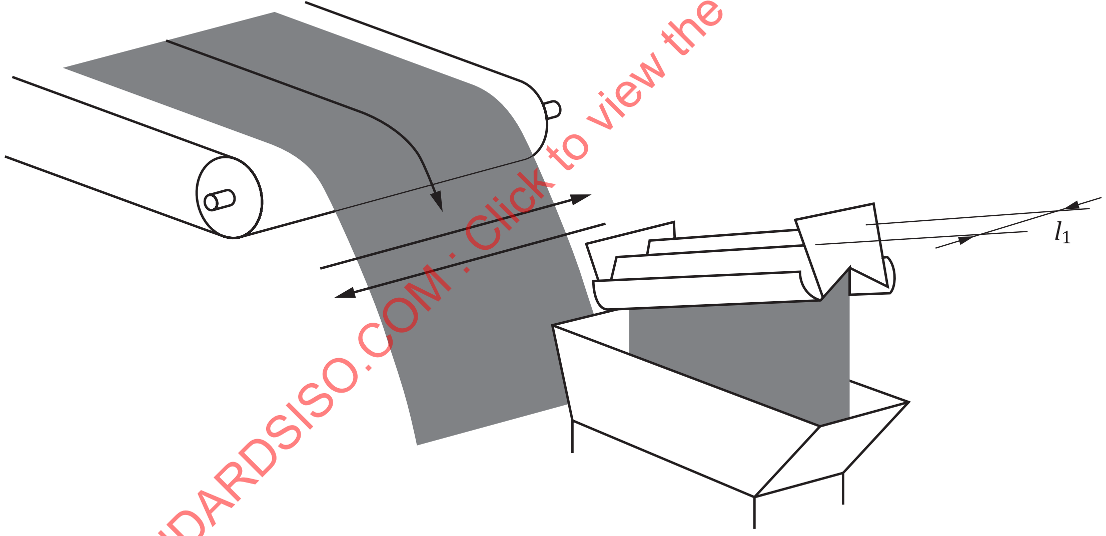
j) the geometry of the cutter aperture shall be such that the cutting time at each point in the stream is equal, not deviating by more than ± 5 %, e.g. straight-path cutters shall have parallel cutter lips and rotary cutters shall have radial cutter lips;

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

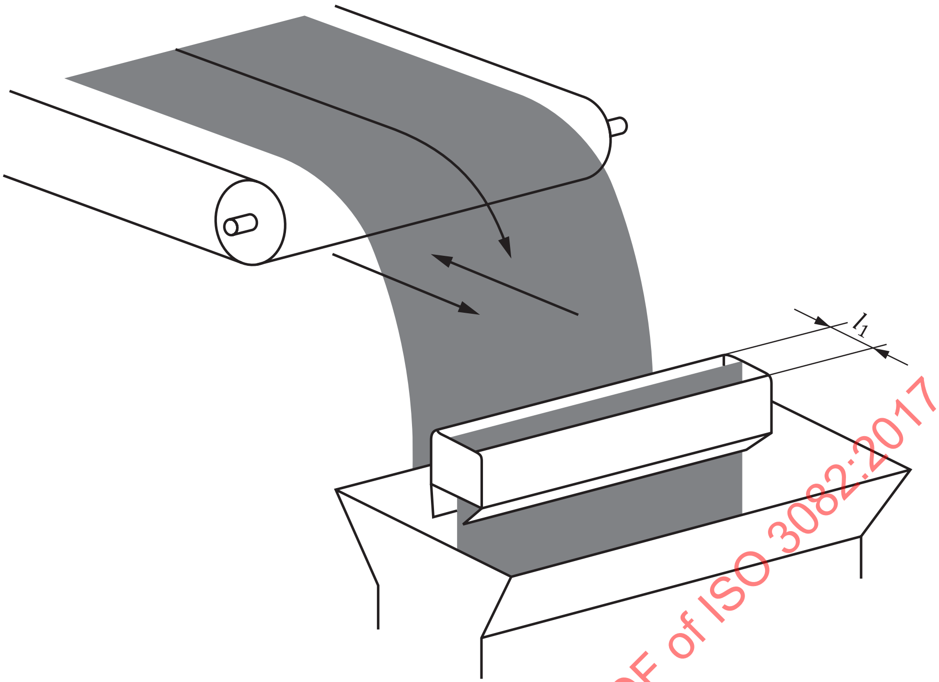
An example of a checklist for mechanical sampling systems is given in Annex A.



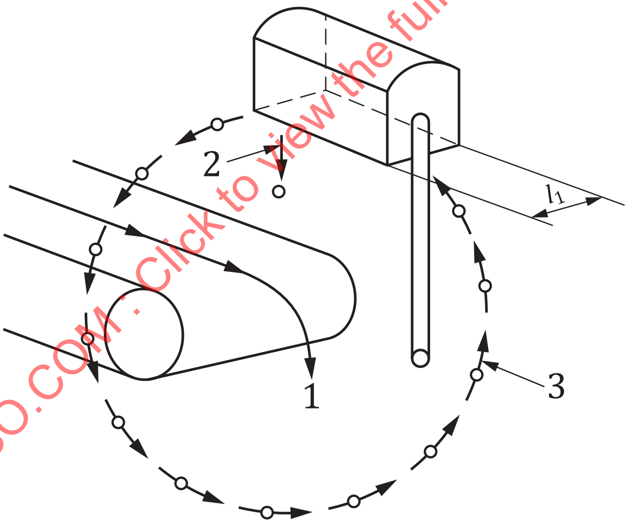
a) Cutter-chute type

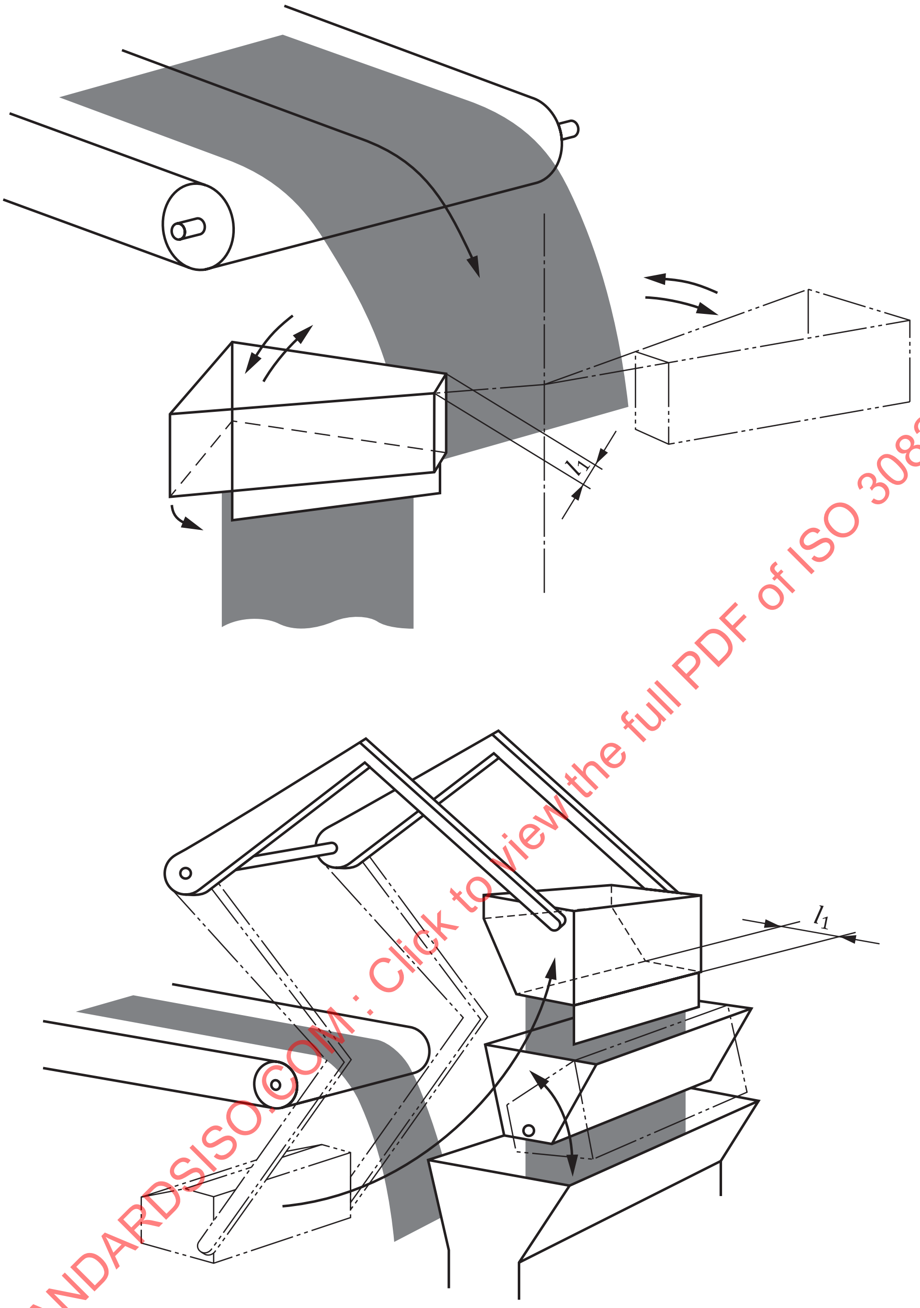


b) Cutter bucket type (i)



c) Cutter bucket type (ii)





d) Swing arm types

Key

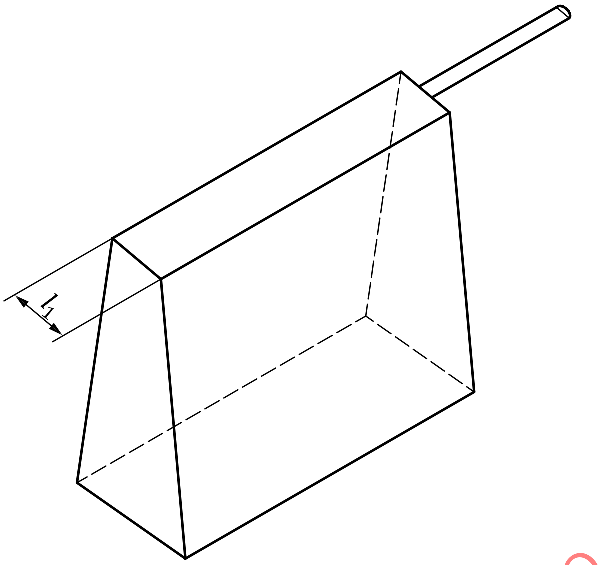
1 main ore flow

2 increment

3 travelling pass of primary sampler

l_1 Cutting aperture of primary sampler.

Figure 1 — Examples of cutter-type samplers



Key

l_1 Cutting aperture of primary sampler.

Figure 2 — Example of a manual sample cutter

7.5.4 Cutter aperture of primary sampler

The cutting aperture of the primary sampler (dimension l_1 in Figure 1 and Figure 2) shall be at least three times the nominal top size of the ore, or 30 mm, whichever is the greater. However, with certain ores, e.g. sticky ores, bridging and consequent bias may occur for a cutter aperture of three times the nominal top size. In these instances, larger cutter apertures shall be used to prevent the introduction of significant bias.

7.5.5 Cutter speed of primary sampler

For either of the two kinds of primary samplers mentioned in 6.1.5 or 6.2.5, the cutter shall be designed to travel at a uniform speed, not deviating by more than ± 5 %, during the course of taking each increment.

The cutter speed is one of the most important design parameters in designing a mechanical sampling system. Too high a cutter velocity will lead to

a) biasing of the sample due to deflection of the larger particles,

b) biasing of the sample by rebounding particles and dust caused by excessive turbulence, and

c) shock load problems and difficulties in maintaining constant speed while cutting the ore stream.

Experimental work undertaken by Gy[1] for falling stream cutters shows that, when sampling heterogeneous ore streams at low belt loading where the particle size distribution is very narrow, significant bias may be introduced when the cutter speed exceeds 0,6 m/s or the cutter aperture is less than three times the nominal top size of the ore.

Based on this evidence, cutters that have a cutter aperture (l_1) equal to three times the nominal top size of the ore shall not exceed a cutter speed of 0,6 m/s, so that significant bias is not introduced.

For cutters where the effective aperture (l_1) is in excess of three times the nominal top size (d), the maximum cutter speed allowed (v_C) can be increased in accordance with Formula (17), subject to a maximum of 1,5 m/s:

$$v_C = 0{,}3\left(1+\frac{l_1}{3d}\right) \quad (17)$$

Cutter speeds in excess of the values specified above shall not be used, unless a bias test conducted in accordance with ISO 3086 proves that no significant bias is introduced.

7.6 Secondary and subsequent samplers

The requirements for design and operation of secondary and subsequent samplers are identical to those for primary samplers specified in 7.5.2 to 7.5.5.

The aperture of the sample cutter shall be at least three times the nominal top size of the ore, or 10 mm, whichever is the greater.

7.7 Online sample preparation

7.7.1 Arrangement for sample preparation

The sample preparation plant shall be designed to carry out preparation of individual increments, individual partial samples or gross samples in accordance with the requirements given in Clause 10. The system for handling primary increments, from the primary sampling station to that stage of the sample preparation system where size testing is undertaken, or where size and other physical test samples are taken, shall be carefully designed to avoid severe handling that could cause size degradation of the ore sample. The number of transfer points, and the height of fall at each transfer point, shall be kept to a minimum.

Sampling and sample preparation installations may be either integrated or separate. For an integrated layout, the sample preparation installation shall be capable of completely processing each increment within the time interval between two consecutive increments taken for the same purpose.

The sample preparation equipment shall be capable of crushing, grinding and pulverizing the sample to the desired particle size and then dividing the sample to the desired mass without bias. The crushing and dividing equipment shall be appropriately sealed to protect the samples from excessive air flow. The circulation of air through the equipment shall also be reduced to a minimum in order to prevent loss of fine materials and moisture. If it is difficult to incorporate equipment for grinding to 160 µm or 100 µm nominal top size into the sample preparation system, the grinding operation may be carried out separately.

7.7.2 Crushers

To obtain the desired nominal top size of the sample at each stage of crushing, grinding or pulverizing, the equipment for these processes shall be adjusted so that no more than 5 % by mass oversize material is retained on the relevant sieve.

7.7.3 Dividers

The following are examples of dividers:

a) cutter-chute divider [same design as the primary sampler given in Figure 1 a)];

b) slotted belt divider [see Figure 3 a)];

c) chain bucket divider [see Figure 3 b)];

d) rotary sample divider [see Figure 3 c)];

e) rotary plate divider [see Figure 3 d)];

f) rotary cutter divider [see Figure 3 e)];

g) chute type divider [see Figure 3 f)].

To avoid bias, the divider shall have a random start. The operation of the cutter should be interlocked with the operation of the feeder via a random timer. The time range of operation of the random selector in the timer shall be adjusted to equal the computed cutting interval, so that there is equal probability of the first cut being taken at any point of time within the duration of the first interval. Special design precautions are required for the random timer used for constant mass division. Because the cutting interval may be different for each increment or partial sample to be divided, the time range of operation of the random selector in the timer should be manually or automatically adjusted for each successive sample division, to match the computed cutting interval.

If the installation is such that the above requirements cannot be met, then a considerably larger number of cuts than the specified minimum is required to minimize bias.

It is recommended that a uniform feed be provided to the divider at each stage of division. The cutter aperture shall be as specified in 7.5.4, and the cutter speed shall be constant (see 7.5.3 and 7.5.5).

7.7.4 Dryers

After the moisture sample has been extracted, a dryer may be used to dry the chemical analysis sample so that subsequent sample preparation can be carried out without difficulty. Drying shall be conducted at or below 105 °C, because above this temperature there is likely to be a change in the chemical composition of the sample. Care shall also be taken not to introduce other sources of bias, e.g. loss of fines during drying.

7.8 Checking precision and bias

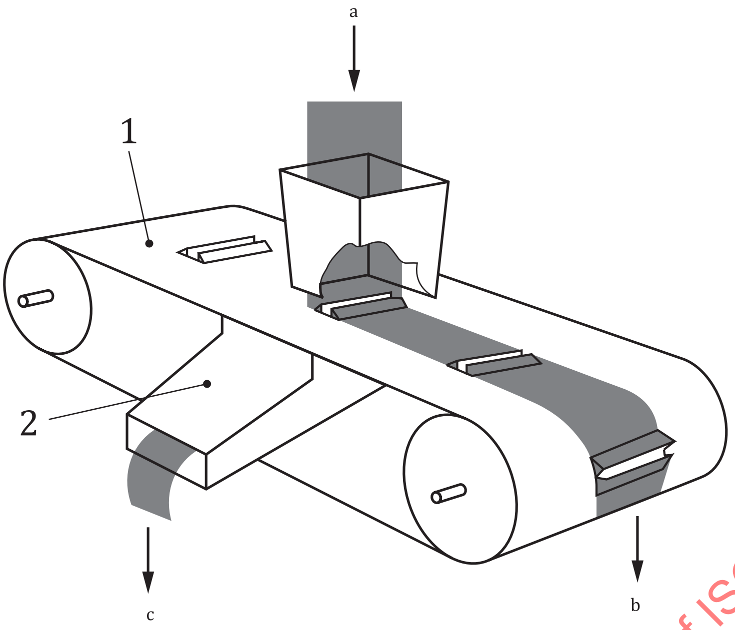
When a sampling installation is newly constructed, when principal parts of the installation are modified, or when a new ore is being sampled, check experiments for precision (ISO 3085) and bias (ISO 3086) shall be carried out for the installation as a whole, and for each stage when possible. Visual checks shall also be conducted at regular intervals during routine operation to identify any irregularities in equipment performance. Bias tests should be carried out when these visual checks indicate that there is a problem or some other change is suspected. The installation shall be capable of attaining sampling and sample preparation precision better than those specified in 5.4 and 5.5.

The bias of a sampling installation shall be checked by comparison with “stopped-belt” sampling as specified in Clause 9, preferably using size determination as the criterion.

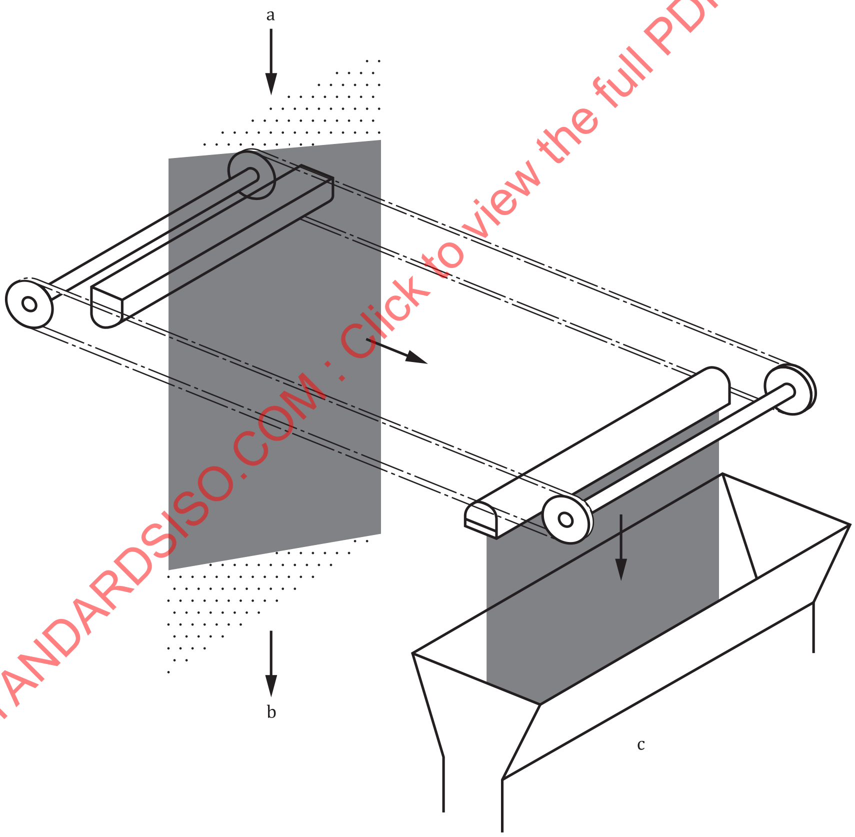
7.9 Cleaning and maintenance

The sampling system should be readily accessible to facilitate inspection, thorough cleaning, repairs or check experiments.

Upon completion of sampling a lot, the major units of the installation should be cleaned either by applying fresh water, by using dry and oil-free compressed air, or by using a vacuum system. When a change is made in the type of ore being sampled, a quantity of material taken from the lot to be sampled should be passed through the entire installation to remove any possible contaminants.



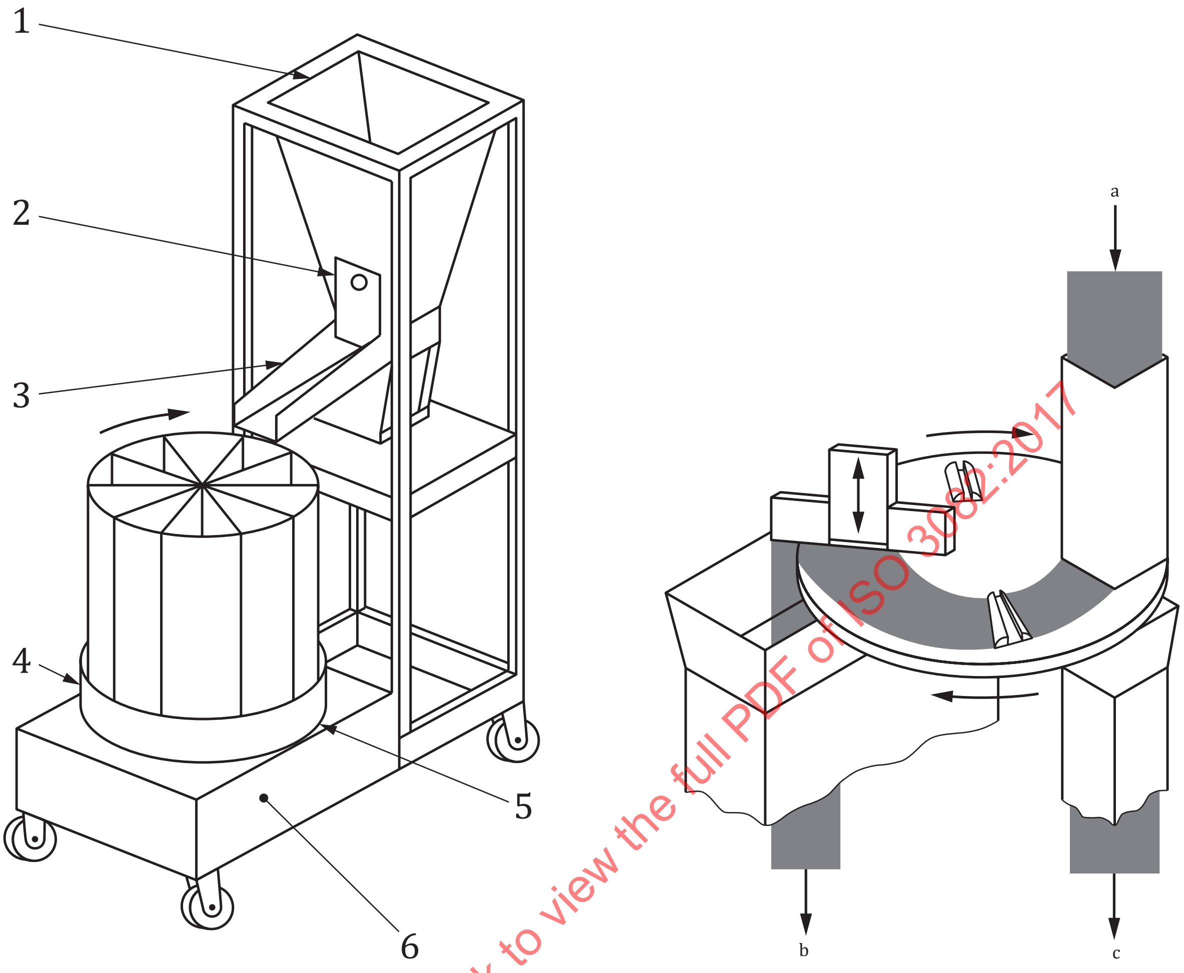
a) Slotted belt type divider



b) Chain bucket type divider

Key

1	slotted belt	a	Feed.
2	inclined chute	b	Reject.

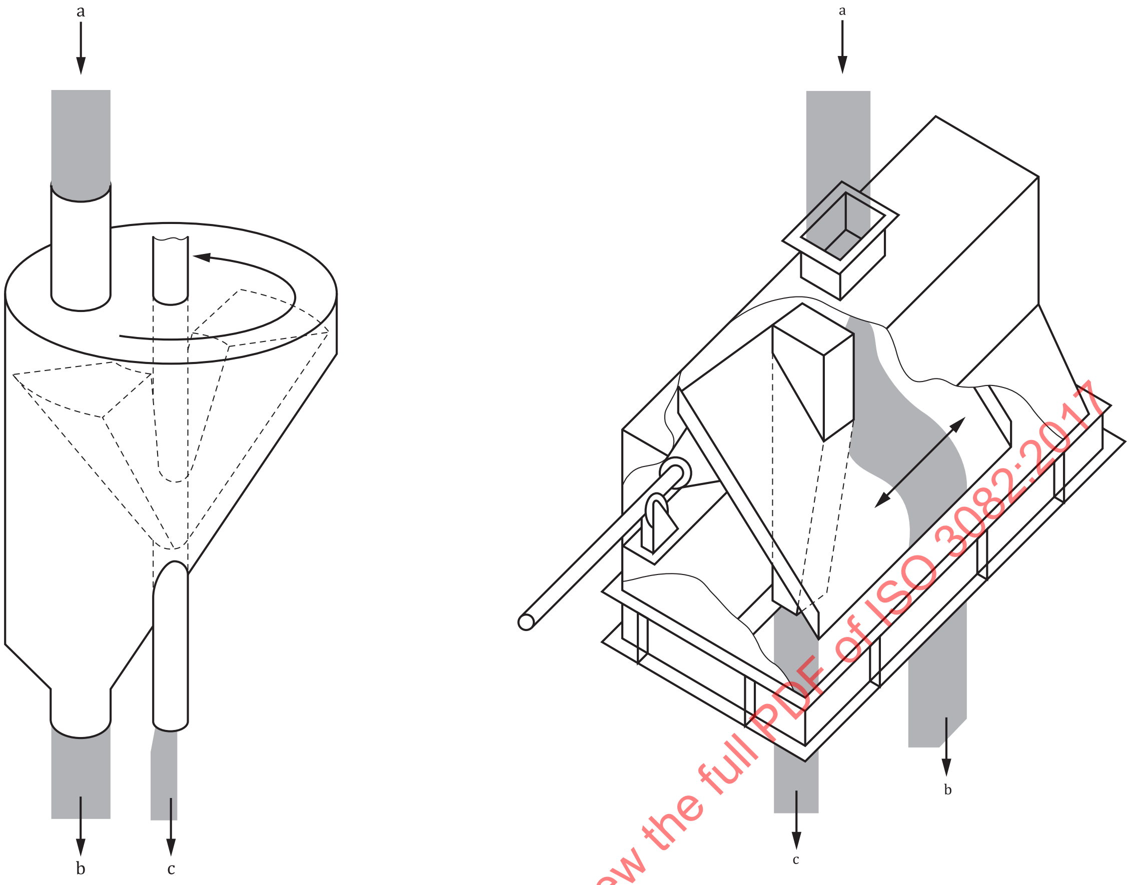


c) Rotary sample divider

d) Rotary plate type divider

Key

1	feed hopper
2	slide gate
3	vibratory feeder
4	removable canisters
5	turntable
6	drive (enclosed)
a	Feed.
b	Reject.
c	Divided sample.



e) Rotary cutter divider

f) Chute type divider

Key

a	Feed.	c	Divided sample.
b	Reject.		

Figure 3 — Examples of mechanical dividers

7.10 Example of a flowsheet

The wide variation in mechanical installations for sampling and sample preparation makes it impracticable to describe a standardized flowsheet. Consequently, only guidelines for constructing a new mechanical installation can be provided.

An example of a flowsheet is given in Figure 4 illustrating the following:

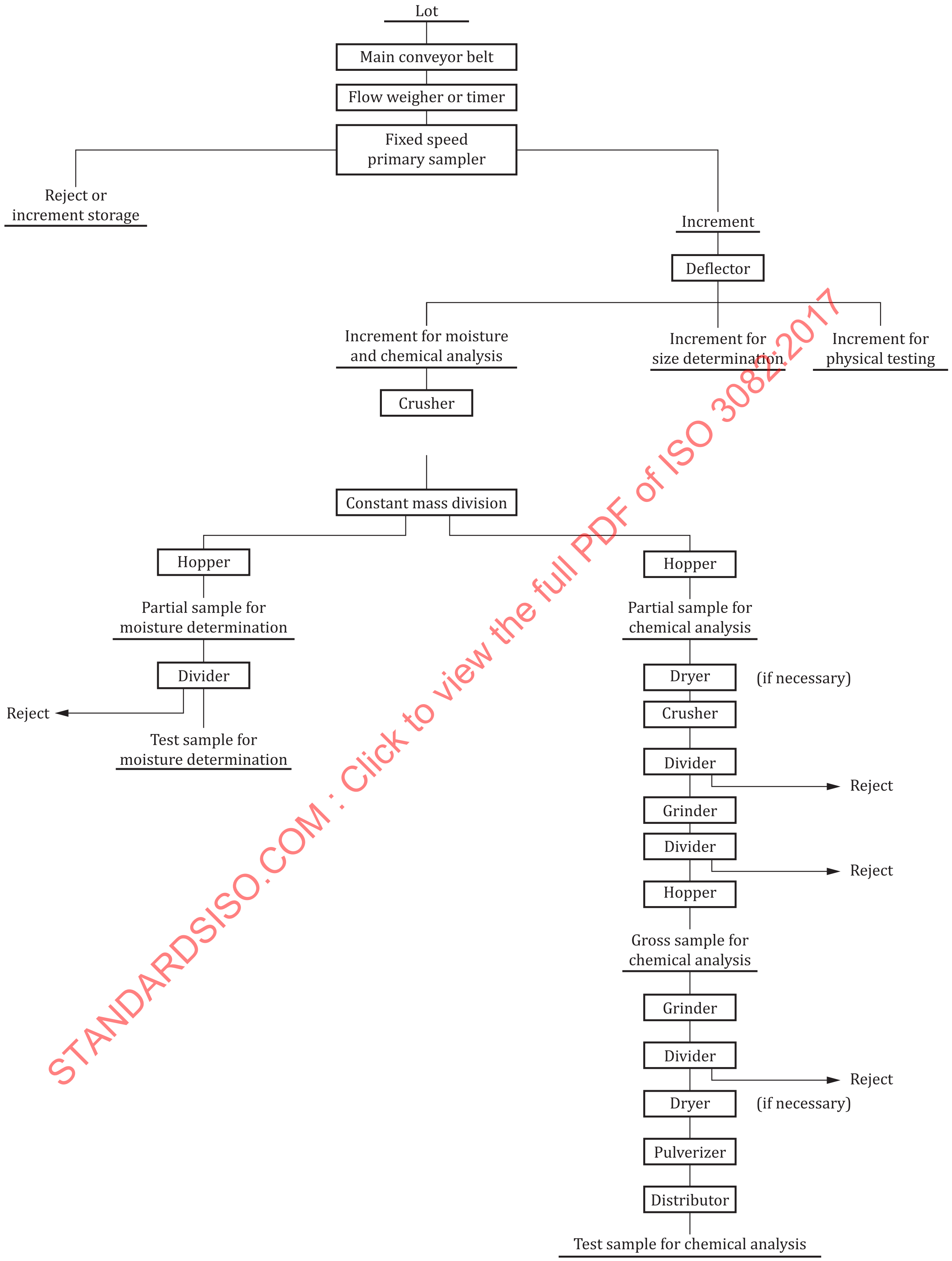
a) mass-basis sampling;

b) fixed-speed primary sampler;

c) coefficient of variation of mass of increments > 20 %;

d) constant-mass division of increments;

e) separate preparation of size sample, physical testing sample, moisture sample and chemical analysis sample.



NOTE It is permissible to use a screen before the crusher, provided that there is a facility for mixing the crushed oversize and the undersize material prior to the division stage.

Figure 4 — Example of a sampling and sample preparation flowsheet

8 Sampling from stationary situations

8.1 General

To avoid bias, it is essential that increments are extracted from the lot so that all parts of the ore have an equal opportunity of being selected and becoming part of the final sample for analysis. Consequently, sampling from moving streams is the preferred method of obtaining representative samples for determining the quality characteristics of the lot, because equal access is available to the entire lot. Sampling of stationary lots can be carried out only when access to the full depth of the ore is available and the complete increment can be extracted. This is sometimes possible with a spear sampler or auger. However, it is not possible with sampling shovels, which therefore are not recommended.

8.2 Sampling from trucks and wagons

8.2.1 General

In situ sampling of ores with nominal top size less than 1 mm from trucks or wagons is permitted using a spear sampler or an auger, but only if the sampling device penetrates to the full depth of the ore at the point selected for sampling and the full column of ore is extracted. However, a more reliable procedure is to sample from a conveyor belt during transfer of the ore to or from the truck or wagon. Sampling of coarser ore from trucks or wagons using a spear or an auger is not permitted.

8.2.2 Sampling devices

The spear sampler (see Figure 5) or auger for extracting increments shall have minimum internal dimensions of 30 mm.

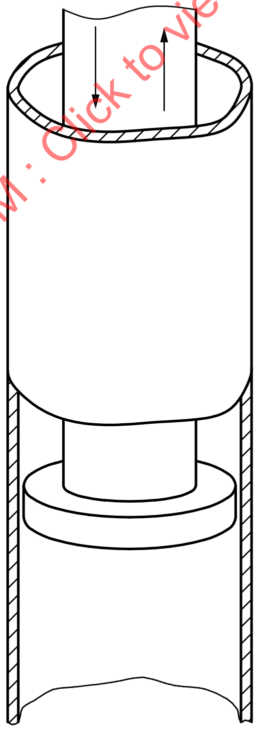


Figure 5 — Example of a spear sampler for sampling ores with nominal top size less than 1 mm

8.2.3 Number of primary increments

The number of primary increments shall be in accordance with 5.4. The number of increments, n_W, to be taken from each truck or wagon constituting the lot is given by Formula (18):

$$n_W \geq \frac{n_1}{N_T} \tag{18}$$

where

n_1 is the total number of primary increments to be taken in accordance with 5.4;

N_T is the number of trucks or wagons constituting the lot.

The value of n_W obtained from Formula (18) shall be rounded up to the next higher whole number.

8.2.4 Method of sampling

The increments to be taken from each truck or wagon shall be taken from locations spaced as evenly as possible over the surface of the ore in the truck or wagon using a spear sampler or an auger so that increments represent almost uniform masses of ore. It is essential that each increment is taken from the full depth of ore in the truck or wagon, and that the full vertical column of ore is extracted for the increment to be representative. If these conditions are not met, the sampling procedure does not conform to this document.

Care shall be taken when using spear samplers, because internal friction within the probe can prevent the full vertical column from being collected.

8.3 Sampling from ships, stockpiles and bunkers

In situ sampling of ships, stockpiles 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. A sample taken from the top or sides only cannot be regarded as representative of the whole ship, stockpile or bunker, particularly when the lot is composed of ore from more than one source, e.g. if increments are taken from a 10 m high stockpile using a spear sampler that penetrates to a depth of 2 m only, the resultant sample can only be representative of ore down to that depth, i.e. a 2 m thick shell on the surface of the stockpile. The only effective procedure is sampling from a conveyor belt during transfer of the ore to or from the ship, stockpile or bunker using the procedures described in Clause 7.

9 Stopped-belt reference sampling

Stopped-belt sampling is the accepted method for obtaining a reference sample against which other sampling procedures may be compared. However, special care is required if the samples are for moisture determination, because moisture may be lost while the reference sample is being removed from the conveyor.

The procedure for sampling from a stopped-belt is as follows:

a) determine the parameters for sampling in accordance with 4.2;

b) stop the belt at the mass or time intervals determined in accordance with 6.1.4 or 6.2.4;

c) at each stoppage, place a suitably profiled sampling frame (see Figure 6) with minimum internal dimensions of 3 times the nominal top size of the ore or 30 mm, whichever is the greater, across the width of the stationary belt, and insert it through the ore so that it is in contact with the belt across its full width;

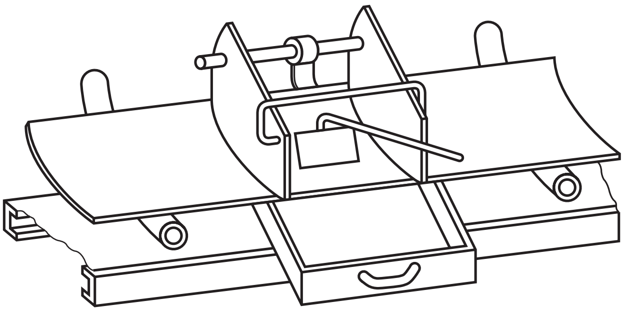


Figure 6 — Example of a sampling frame for use on stopped belts

d) should any ore particles obstruct insertion of the sampling frame, push those at the left-hand edge of the frame into the increment and those at the right-hand edge of the frame out of the increment;

e) remove the ore within the sampling frame in the shortest possible time to minimize moisture loss, ensuring that all ore particles are collected by sweeping the belt clean, and deposit each increment into a suitable container;

f) if paired comparisons are required on an increment-by-increment basis, keep the increments separate;

g) if the quality of the lot is required, combine the increments into partial samples or a gross sample in accordance with 10.2;

h) store the increments, partial samples or gross samples in labelled containers as specified in Clause 11.

If it is not practicable to use the stopped-belt method, one of the schemes described in Annex C may be used.

10 Sample preparation

10.1 Fundamentals

10.1.1 General

Sample preparation is carried out in a number of stages, each stage consisting of a series of drying (if necessary), crushing, mixing and division operations.

Sample preparation shall be carried out in such a manner that there will be no contamination or introduction of materials other than the sample and no change in its quality. In particular, the moisture sample shall be kept in an airtight, non-absorbent container in order to avoid any change in its moisture content.

Checking experiments for precision and bias shall be carried out regularly in the sample preparation process, so that any significant errors in the procedure may be detected.

Sample preparation prior to the test sample stage may be conducted on each increment, on each partial sample constituted from increments, or on the gross sample constituted from partial samples or increments.

The gross sample is constituted from all the increments or partial samples, either as-taken or after having been prepared individually to an appropriate stage of division.

Partial samples are constituted from two or more increments, either as-taken or after having been prepared individually to an appropriate stage of division.

An example of a sample preparation scheme for constituting partial samples from increments and a gross sample from partial samples is given in Figure 7.

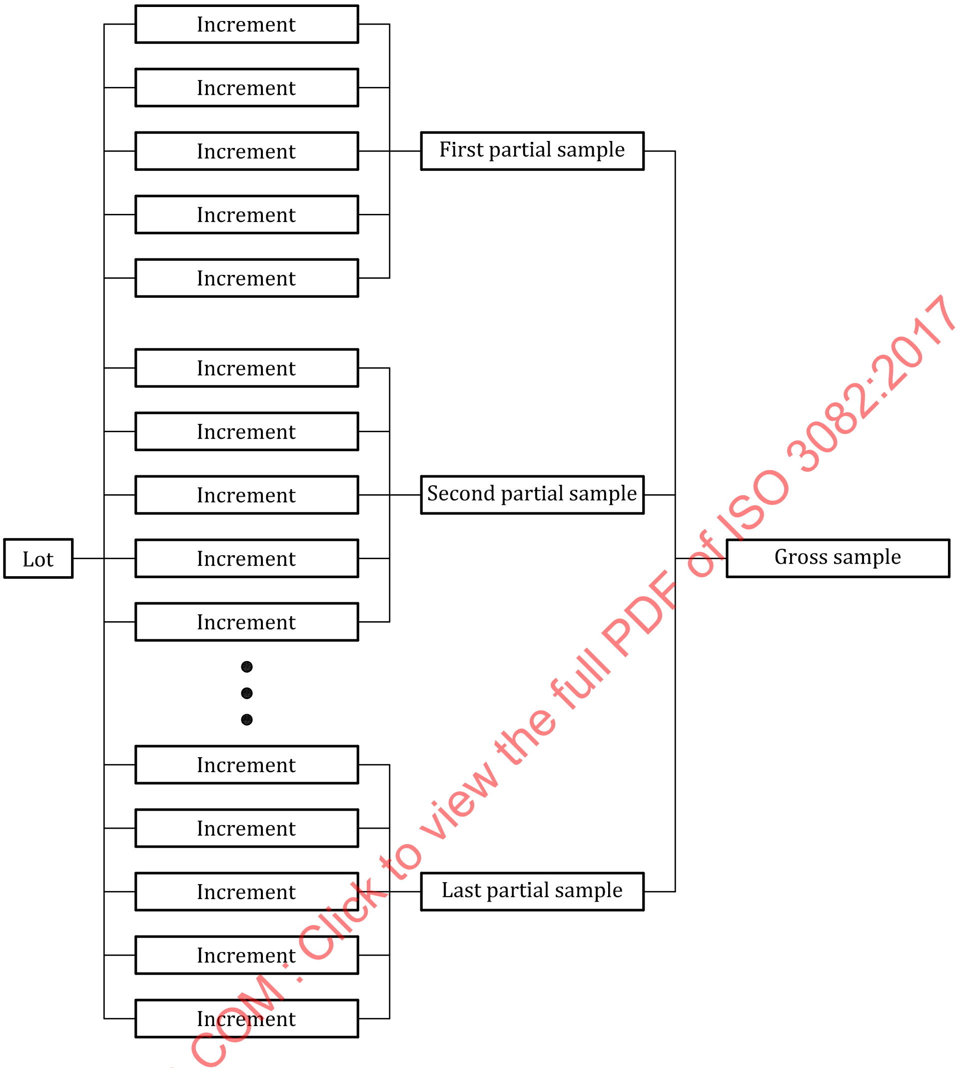


Figure 7 — Example of a sample preparation scheme showing constitution of partial samples from increments and a gross sample from partial samples

10.1.2 Drying

When the sample is very wet or sticky and sample preparation cannot be carried out, the sample shall be dried at or below a temperature of 105 °C so that sample preparation may then be carried out without difficulty. For moisture samples, see 10.6.

10.1.3 Crushing and grinding

Crushing and grinding shall be conducted with equipment that is suitable for the size and hardness of the ore particles. The crusher and grinder shall be purged with ore from the same source.

10.1.4 Mixing

Depending on the method of mixing and the ore characteristics, mixing the sample may make it more homogeneous and consequently reduce the errors in sample division. The need for mixing is particularly important when samples from more than one source are combined. Where possible, the sample processing scheme should be designed so that the need for mixing is minimized, because it may have the opposite effect to that intended and may lead to increased segregation.

Mixing of moisture samples may result in moisture loss and hence bias, so moisture samples shall not be mixed prior to division.

Examples of suitable mixing methods include

a) mechanical mixers such as a V-mixer, and

b) passing the sample through a riffle or preferably a rotary sample divider at least three times in succession, recombining the portions after each pass. Dust losses shall be minimized.

NOTE Some methods of hand mixing, for example forming and reforming a conical pile, can have the opposite effect to that intended, and can lead to increased segregation.

10.1.5 Division

10.1.5.1 General

Division shall be carried out on the sample, crushed if necessary to an appropriate particle size, to reduce the sample mass.

To obtain the specified precision of sample preparation, the following aspects of division shall be considered:

a) nominal top size of the sample to be divided;

b) minimum mass of the sample after division, specified for each quality characteristic to be determined (see 10.1.6).

10.1.5.2 Method of division

One or more of the following methods of division shall be conducted individually or jointly:

a) mechanical increment division (see 10.3.1);

b) other mechanical division methods (e.g. mechanically charged riffle divider, see 10.3.2);

c) manual division (see 10.4).

10.1.5.3 Types of division

When several increments or partial samples are prepared individually and constituted into partial samples or a gross sample, the division of increments or partial samples shall be conducted either by constant-mass division or by proportional division subject to the conditions set out in 10.2.2 and 10.2.3.

10.1.5.4 Types of divider

Acceptable types of mechanical dividers include cutter-chute, slotted belt, chain bucket, rotary sample, rotary plate, rotary cutter, chute type and mechanically charged riffle (see 10.3.2).

10.1.6 Mass of divided sample

10.1.6.1 Division of chemical analysis and moisture samples

10.1.6.1.1 Division of gross sample

When a gross sample is divided for subsequent analysis, the minimum mass of the divided sample, m_S, in kilograms, is given by Formula (19), which is based on the minimum mass formula derived in Reference [1]:

$$m_\mathrm{S} = \frac{0{,}000\,32 d^{2{,}5}}{\sigma_\mathrm{D}^2} \qquad (19)$$

where

d is the nominal top size of the sample, in millimetres;

σ_D is the desired standard deviation of sample division, in % Fe, which is the major component of the standard deviation of sample preparation, σ_P, for a given sample preparation stage.

The gross sample shall not be divided further than the mass given by Formula (19) for the nominal top size of the sample until it is crushed to a smaller particle size, subject to an absolute minimum of 500 g to satisfy the requirements for preparation of test samples for chemical analysis (see 10.5). Preparation of test samples for moisture determination is set out in 10.6.

Examples of minimum divided-gross-sample masses given by Formula (19), for a division standard deviation of 0,1 % Fe and 0,05 % Fe, and typical nominal top sizes are given in Table 4. The standard deviation of division σ_D applies to each sample division stage, and the variances are additive for the sample division scheme selected.

Table 4 — Examples of minimum mass of divided gross sample for moisture determination and/or chemical analysis

Nominal top size mm	Minimum mass of divided gross sample kg	
	σ_D = 0,1 % Fe	σ_D = 0,05 % Fe
40	325	1 300
31,5	180	710
22,4	75	300
10	10	40
6,3	3,2	13
2,8	0,5	1,7
≤ 1,4	0,5	0,5

NOTE Additional sample mass is required if gross samples are used for both chemical analysis and moisture determination.

10.1.6.1.2 Division of individual increments or partial samples

When increments or partial samples are divided, the division shall be carried out ensuring that the cumulative mass shall be not less than the minimum divided-gross-sample mass given by Formula (19) or Table 4. However, the overall precision of analysis of individual increments or individual partial samples will be poorer than the overall precision of analysis of the correctly constituted gross sample, because the number of increments constituting the samples is less than for the gross sample and the sample masses are less than the minimum mass of divided gross sample. The standard deviation of division σ_D applies to each sample division stage, and the variances are additive for the sample division scheme selected.

10.1.6.2 Division of size samples

10.1.6.2.1 General

The division of the size sample shall be carried out in accordance with Table 5. If the percentage of the size fraction varies from that specified in Table 5, the minimum mass specified in Table 5 shall be modified in accordance with Formula (20).

When the type of iron ore and specification-size fraction vary from that given in Table 5, Annex D shall be used to determine the minimum sample mass.

10.1.6.2.2 Division of gross sample

When the gross sample is divided, the mass of the divided gross sample shall not be less than the minimum specified in Table 5.

Bias is easily introduced in the division of a size sample and hence particular care shall be taken when dividing a size sample. When 200 mm nominal top size ore is divided, manual increment division should not be used, because segregation is likely to be a serious problem.

Table 5 — Examples of minimum mass of divided gross sample for size determination using other mechanical division methods (see 10.3.2.1)

Type of iron ore		200 mm nominal top size ore		50 mm nominal top size ore		−31,5 + 6,3 mm sized ore		Sinter feed		Pellet feed		Pellets	
Typical specification-size fraction		−10 mm		−10 mm		−6,3 mm		+6,3 mm		+45 µm		−6,3 mm	
Average percentage of the size fraction		20		20		10		10		30		5	
Mass of lot t		Minimum mass of divided gross sample, m_3, and sample preparation and measurement precision. β_{PM}											
Over	Up to	m_3 kg	β_{PM} %	m_3 kg	β_{PM} %	m_3 kg	β_{PM} %	m_3 kg	β_{PM} %	m_3 kg	β_{PM} %	m_3 kg	β_{PM} %
340 000		1 100	3,0	270	2,9	120	1,5	8,0	1,5	0,5	1,6	250	0,50
270 000	340 000	1 080	3,0	250	3,0	120	1,5	8,0	1,5	0,5	1,6	250	0,50
210 000	270 000	1 010	3,1	230	3,1	110	1,6	7,0	1,6	0,5	1,7	240	0,51
150 000	210 000	950	3,2	220	3,2	110	1,6	7,0	1,6	0,5	1,7	240	0,51
100 000	150 000	890	3,3	210	3,3	110	1,6	7,0	1,6	0,5	1,8	230	0,52
70 000	100 000	840	3,4	190	3,4	95	1,7	6,0	1,7	0,5	1,9	215	0,54
45 000	70 000	790	3,5	180	3,5	95	1,7	6,0	1,7	0,5	1,9	215	0,54
30 000	45 000	750	3,6	170	3,6	85	1,8	5,0	1,8	0,5	2,0	210	0,55
15 000	30 000	670	3,8	150	3,8	75	1,9	5,0	1,9	0,5	2,1	210	0,55
	15 000	530	4,3	120	4,3	60	2,2	4,0	2,2	0,5	2,4	145	0,66

When the actual percentage of the size fraction is considerably higher than that specified in Table 5, the minimum mass, m_3, specified in Table 5 shall be revised using Formula (20) based on the binomial rule:

$$m_4 = m_3 \times \frac{P(100-P)}{P_0(100-P_0)} \tag{20}$$

where

m_4 is the revised minimum mass, in kilograms, of the divided gross sample;

m_3 is the minimum mass, in kilograms, of the divided gross sample specified in Table 5;

P is the actual percentage of the size fraction, which is considerably higher than that specified in Table 5;

P_0 is the percentage of the size fraction specified in Table 5.

The greater of m_3 and m_4 shall be used.

For example, for a lot of 40 000 t of 200 mm nominal top size ore, if the percentage of the −10 mm fraction is about 50 %, the minimum mass of the divided gross sample shall be revised as follows:

$$m_4 = 750 \times \frac{50(100-50)}{20(100-20)} \approx 1\ 175\ \text{kg}$$

10.1.6.2.3 Division of increment or partial sample

When increments or partial samples are divided, the division shall be carried out ensuring that the cumulative mass shall not be less than the minimum divided sample mass given by Formula (20) or Table 5. However, the overall precision of size determination of individual increments or individual partial samples will be poorer than the overall precision of size determination of the correctly constituted gross sample, because the number of increments constituting the samples is less than for the gross sample and the sample masses are less than the minimum mass of divided gross sample. If the percentage of the size fraction varies from that specified in Table 5, the minimum mass specified in Table 5 shall be modified in accordance with Formula (20).

10.1.6.3 Division of samples for physical testing

The minimum mass of the sample for physical testing shall be determined from the test requirements and the required physical and metallurgical properties to be determined. In general, except for the sample for the bulk density test, samples for physical testing shall weigh at least 500 kg. When the bulk density test is carried out in accordance with Method 1 of ISO 3852, the mass of the sample for physical testing shall be equal to at least the test sample mass of 600 kg.

When determining the physical and metallurgical properties of a lot in accordance with the relevant International Standards, the precision of sample preparation and measurement, β_{PM}, shall attain the values specified in Table 6.

10.1.7 Split use and multiple use of sample

A sample taken from a lot and meeting the specific requirements for the determination of several quality characteristics may be subjected to split or multiple use to obtain test samples for chemical analysis, moisture determination, size determination and physical testing (see Figure 12 for further details).

Table 6 — Precision of sample preparation and measurement for physical tests

Type of test	International Standard	Precision, β_{PM}		
		Pellets	**Sinter**	**Lump ore**
Tumble and abrasion indices	ISO 3271			
AI – Abrasion index (%)		0,4	0,5	0,5
TI – Tumble index (%)		0,5	0,5	0,6
Bulk density — Method 1	ISO 3852			
ρ_b – (kg/m^3)		0,1	0,2	0,2
Reducibility by rate of reduction index	ISO 4695			
Rt – Degree of reduction		0,05	0,1	0,1
Low-temperature reduction-disintegration indices by static method — Part 1: Reaction with CO, CO_2, H_2 and N_2	ISO 4696-1			
RDI-1$_{-3,15}$ (%)		3,0	4,0	4,0
Low-temperature reduction-disintegration indices by static method — Part 2: Reaction with CO and N_2	ISO 4696-2			
RDI-2$_{-2,8}$ (%)		3,0	4,0	4,0
Pellets for BF feedstocks — Free-swelling index	ISO 4698			
V_{FS} (%)		3,0	NA	NA
Pellets for BF and DR feedstocks — Crushing strength	ISO 4700			
CS (daN/pellet)		27,0	NA	NA
Reducibility by final degree of reduction index	ISO 7215			
Rf – Final degree of reduction (%)		3,0	3,0	5,0
Reduction under load	ISO 7992			
Δp 80 (kPa)		6,0	NA	6,0
Decrepitation index	ISO 8371			
DI$_{-6,3}$ index (%)		NA	NA	5,0
Pellets for DR feedstocks — Clustering index	ISO 11256			
CI – cluster index (%)		3,0	NA	NA
DR feedstocks — Low-temperature reduction-disintegration index and degree of metallization	ISO 11257			
RDI$_{DR}$ – Reduction-disintegration index (%)		1,5	NA	3,0
M – Degree of metallization (%)		2,0	NA	2,5
DR feedstocks — Reducibility index, final degree of reduction and degree of metallization	ISO 11258			
R$_{90}$ – Final degree of reduction (%)		2,5	NA	4,0
Low-temperature reduction-disintegration indices by dynamic method	ISO 13930			
LTD$_{+6,3}$ (%)		3,0	NA	3,5
LTD$_{-0,5}$ (%)		1,0	NA	2,0
NA = Not applicable				

10.2 Method of constituting partial samples or a gross sample

10.2.1 General

According to measurement requirements, a gross sample may be constituted for a lot or partial samples may be constituted for individual parts of the lot. Furthermore, in some cases, in accordance with sample preparation requirements, it may be necessary to constitute partial samples first and then constitute a gross sample.

10.2.2 Method of constitution for mass-basis sampling

10.2.2.1 Constitution of partial samples or a gross sample from increments

When the coefficient of variation of increment masses is less than 20 %, the increments, either as-taken or after having been prepared individually by constant-mass or proportional division to an appropriate stage, may be combined into partial samples or a gross sample.

However, when the coefficient of variation of increments masses is 20 % or over, the increments as-taken shall not be combined into partial samples or a gross sample. Individual increments shall first be divided by constant-mass division at a practical stage. The prepared increments may then be combined into partial samples or a gross sample at an appropriate stage. Alternatively, each increment may be prepared prior to the test sample stage and subjected to quality determination.

10.2.2.2 Constitution of a gross sample from partial samples

Partial samples constituted in accordance with 10.2.2.1 may be combined into a gross sample.

When division is carried out on each partial sample to constitute a gross sample, division shall be carried out as follows:

a) if the partial samples consist of an equal number of increments, constant-mass or proportional division shall be used;

b) if the partial samples consist of different numbers of increments, only proportional division shall be used.

10.2.3 Method of constitution for time-basis sampling

10.2.3.1 Constitution of partial samples or a gross sample from increments

Increments as-taken may be combined into partial samples or a gross sample, irrespective of the variation of masses of increments. When division is carried out on each increment and the divided increments are combined to constitute partial samples or a gross sample, proportional division shall be used.

10.2.3.2 Constitution of a gross sample from partial samples

Partial samples constituted in accordance with 10.2.3.1 may be combined into a gross sample irrespective of the variation of masses of partial samples.

However, when division is carried out on each partial sample and the divided partial samples are combined to constitute a gross sample, proportional division shall be used.

10.2.4 Special procedure for moisture content

Constitution and collection of moisture test samples for moisture determination shall be such that changes in moisture content are minimised, including measures to minimise the effects of weather (heat, humidity and rainfall) and time (evaporation), and may include use of special sample containers and storage conditions. This will minimise bias and result in better overall precision (including sampling, sample preparation and moisture determination).

When it takes a long time for loading or unloading a lot, the lot shall be divided into parts corresponding to not more than 8 h periods. A moisture partial sample shall be constituted representing each part and a moisture determination shall be carried out. The division into parts shall be subject to the weather conditions, e.g. heavy rain or high temperature, and other conditions or circumstances at the time of loading or unloading. However, if the moisture sample containers and the storage conditions prevent a change in the moisture content of the moisture partial samples, a moisture gross sample may be prepared for the whole lot.

Alternatively, for very large lots it is recommended that the lot be divided into the number of parts shown in Table 7 and a separate moisture partial sample be prepared representing each part for moisture determination. This will ensure that each moisture partial sample is constituted over a short period of time, thereby minimizing moisture evaporation from the sample.

Partial samples or a gross sample for moisture determination shall be constituted by the procedure specified in 10.2.2 or 10.2.3.

Table 7 — Minimum number of parts per lot for constituting partial samples and number of test portions per partial sample for moisture determination

Mass of lot t		Minimum number of parts per lot	Number of test portions per partial sample to be tested	Minimum number of tests
Over	Up to			
340 000		20	1	20
270 000	340 000	16	1	16
210 000	270 000	10	1	10
150 000	210 000	8	1	8
100 000	150 000	4	2	8
70 000	100 000	4	2	8
	70000	2	4	8

10.3 Mechanical methods of division

10.3.1 Mechanical increment division

10.3.1.1 General

The samples for chemical analysis, moisture determination, size determination and physical testing may be divided by mechanical increment division using a cutter-type divider in accordance with the following conditions.

10.3.1.2 Mass of increment (cut)

The mass of each cut shall be uniform. In order to achieve this, the flow of sample to be divided shall be uniform and the cutting aperture and speed of the cutter shall be constant.

NOTE Alternatively, a combination of the variable feed rate of the sample and the variable cutter speed can be considered for taking a uniform cut.

The cutting aperture shall be at least 3 times the nominal top size of the sample to be divided or 10 mm, whichever is greater.

10.3.1.3 Number of increments (cuts)

The number of cuts, n_i, for division of increments, partial samples and gross samples should be determined experimentally from the quality variation, σ_{Wi}, of the stream to be divided and the required sampling precision, β_{Si}, for the particular sampling stage, i, using Formula (21):

$$n_i = \left(\frac{2\sigma_{\mathrm{W_i}}}{\beta_{\mathrm{S_i}}} \right)^2 \tag{21}$$

However, if no information is available on the quality variation for the particular sampling stage, the following number of cuts may be used as a starting point:

- division of a gross sample:
  - a minimum of 20;
- division of individual partial samples:
  - for constant-mass division, a minimum of 10;
  - for proportional division, a minimum of 10 for the average mass of partial sample;
- division of individual increment:
  - for constant-mass division, a minimum of four;
  - for proportional division, a minimum of five for the average mass of increment.

10.3.1.4 Interval between cuts

When constant mass division is used, the interval between cuts shall be varied according to the mass of the sample to be divided.

When proportional division is applied, the interval between cuts shall be constant irrespective of the variation of masses of samples to be divided.

10.3.1.5 Avoiding bias

To avoid bias, the first cut for each sample to be divided shall be taken at a random position within the first interval.

10.3.1.6 Mass of divided sample

The minimum mass of divided sample shall conform to the requirements of 10.1.6, 10.5 and 10.6.

10.3.2 Other mechanical division methods

10.3.2.1 General

The samples for chemical analysis, moisture determination, size determination and physical testing may be divided using mechanical dividers other than cutter-type dividers (see 10.1.5.4), in accordance with the following procedures and division limits.

10.3.2.2 Mass of divided sample

The minimum mass of divided sample shall conform to the requirements of 10.1.6, 10.5 and 10.6.

10.4 Manual methods of division

10.4.1 General

Manual division of samples for chemical analysis, moisture determination, size determination and physical testing can only be applied to ores of less than 40 mm nominal top size.

10.4.2 Manual increment-division method

10.4.2.1 General

Manual increment division is applicable to ore not exceeding 40 mm nominal top size. It shall be carried out using an increment-division scoop of the type and dimensions shown in Figure 8 and Table 8. The scoop shall be flat bottomed and a curved base is not acceptable. However, manual increment division should not be applied to certain samples such as pellets and sized ores, which roll freely and/or segregate easily. When the pellets have been crushed to a sufficiently small particle size, manual increment division may be applied satisfactorily.

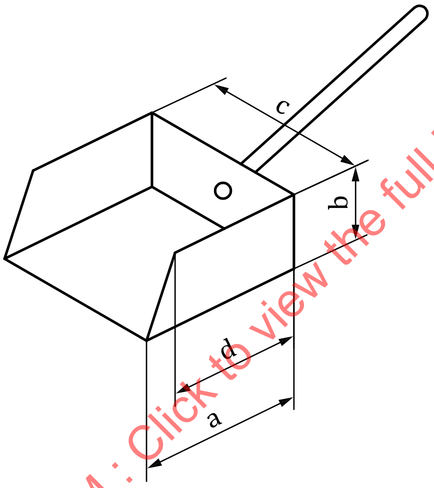


Figure 8 — Example of an increment scoop

10.4.2.2 Mass of increment

The mass of each increment shall be as specified in Table 8.

10.4.2.3 Number of increments

The number of increments for manual increment division shall be as specified in Table 9.

Table 8 — Nominal top size, thickness of spread sample, scoop dimensions and increment mass for manual increment division

Nominal top size mm		Thickness of spread sample mm	Scoop number	Dimensions of increment scoop mm				Increment mass kg
Over	Up to			a	b	c	d	
31,5	40	80	40D	220	160	220	200	16,3
22,4	31,5	65	31,5D	180	120	180	150	9,0
10	22,4	50	22,4D	120	100	120	100	3,6
6,3	10	30	10D	75	40	75	60	0,5
2,8	6,3	20	6,3D	50	30	50	40	0,16
1	2,8	15	2,8D	40	25	40	30	0,10
0,5	1	10	1D	25	20	25	20	0,03
0,1	0,5	8	0,5D	15	10	15	12	0,006
0	0,1	5	0,1D	10	6	10	8	0,0015

Table 9 — Number of increments for manual increment and manual strip division

Sample	Number of manual increments
Gross sample	20
Partial sample	10
Primary increment	4

10.4.2.4 Mass of divided sample

The minimum mass of divided sample shall conform to the requirements of 10.1.6, 10.5 and 10.6.

10.4.2.5 Procedure

Manual increment division shall be carried out as follows:

a) spread the sample to be divided on a smooth and flat plate (non-moisture-absorbing) in the form of a rectangle with uniform sample thickness as specified in Table 8;

b) mark a matrix on the spread sample, dividing it into the number of parts corresponding to the minimum number of increments specified in Table 9;

c) select an appropriate scoop from Table 8, according to the nominal top size of the ore to be divided, and collect one increment of approximately equal mass from each part of the matrix (the location being selected at random in each part);

d) insert a flat bump plate vertically through the spread sample until it comes into contact with the mixing surface. Then thrust the scoop down to the bottom of the sample layer, and take the increment by moving the scoop horizontally until its open end comes into contact with the bump plate, ensuring that all ore particles are collected from the top of the mixing surface;

e) lift the scoop and bump plate together to ensure that no sample is lost from the scoop, thereby minimizing bias.

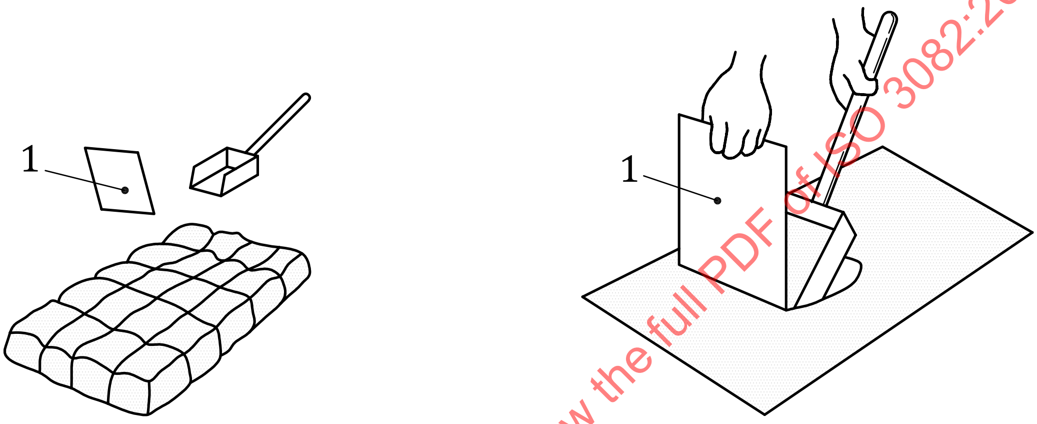
When the mass of the divided sample is likely to be smaller than that required for subsequent testing purposes, the mass of the increment and/or the number of increments shall be increased.

Figure 9 illustrates the division of a gross sample by the manual increment-division method.

NOTE Manual increment division is suitable for the division of moisture samples.

a) Spread the crushed gross sample into a rectangle with a thickness as specified in Table 8

b) Arrange in 20 equal parts e.g. into 5 equal parts lengthwise and 4 equal parts breadthwise



c) Take a scoopful of sample at random from each of the 20 parts by inserting the scoop to the bottom of the sample layer, and combine the 20 scoopfuls of sample into a divided sample

d) Outline of taking an increment by using a bump plate as shown in c)

Key

1 bump plate

Figure 9 — Example of manual increment division of a gross sample (20 parts)

10.4.3 Manual strip-division method

10.4.3.1 General

Manual strip division is applicable to ore not exceeding 40 mm nominal top size. It shall be carried out using a manual cutter with a cutter aperture not less than three times the nominal top size of the ore to be divided or 10 mm, whichever is the greater, as shown in Figure 10. However, manual strip division should not be applied to certain samples such as pellets and sized ores, which roll freely and/or segregate easily. When the pellets have been crushed to a sufficiently small particle size, manual strip division may be applied satisfactorily.

10.4.3.2 Mass of increment

The mass of each increment shall be as specified in Table 10.

Table 10 — Nominal top size, height of strip, width of strip, width of manual cutter and increment mass for manual strip division

Nominal top size mm		Height of strip mm	Width of strip mm	Minimum width of manual cutter mm	Minimum increment mass kg
Over	Up to				
31,5	40	195	540	120	16,4
22,4	31,5	160	460	94,5	9,0
10	22,4	120	340	67,2	3,6
6,3	10	70	200	30	0,5
2,8	6,3	50	135	18,9	0,16
1	2,8	25	75	10	0,025
0,5	1	25	75	10	0,025
0,1	0,5	25	75	10	0,025
0	0,1	8	25	10	0,0025

NOTE The strip width and minimum increment mass are based on the assumption that the angle of repose of the sample is approximately 36° and the bulk density is about 2 600 kg/m³.

10.4.3.3 Number of increments

The number of increments for manual strip division shall be as specified in Table 9.

10.4.3.4 Mass of divided sample

The minimum mass of divided sample shall conform to the requirements of 10.1.6, 10.5 and 10.6.

10.4.3.5 Procedure

Manual strip division shall be carried out as follows.

a) Spread the sample to be divided on a smooth and flat plate (non-moisture-absorbing) in the form of a strip by distributing the sample along the length of the strip as evenly as possible, working randomly from end to end and from both sides of the strip. The height and width of the strip are specified in Table 10, the length of the strip, l_5, in metres, being determined by the mass of the sample to be divided as given by Formula (22):

$$l_5 = \frac{2m \times 10^6}{\rho_b l_6 l_7} \quad (22)$$

where

m is the mass of sample to be divided, in kilograms;

ρ_b is the bulk density of the sample, in kilograms per cubic metre;

l_6 is the width of the strip, in millimetres;

l_7 is the height of the strip, in millimetres.

End plates may be used to ensure that size segregation only occurs laterally.

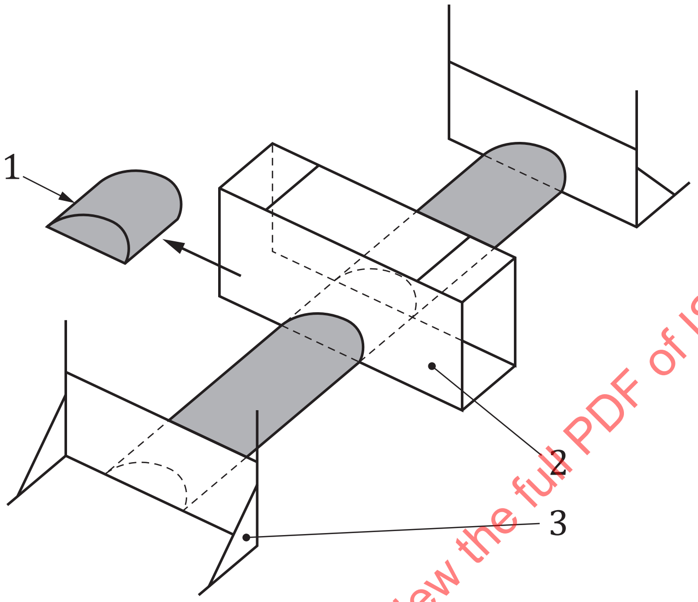
b) Increments shall be taken as a complete section across the strip using a manual cutter, ensuring that no sample is lost from the cutter, thereby minimizing bias. The width of each cross-section shall be not less than 3 times the nominal top size of the ore or 10 mm, whichever is the greater, as specified in Table 10.

c) Take the number of increments specified in Table 9.

When the mass of the divided sample is likely to be smaller than that required to conform to the requirements of 10.1.6 or for subsequent testing purposes, the mass of the increments and/or the number of increments shall be increased.

Figure 10 illustrates the division of a gross sample by the manual strip-division method.

NOTE Manual strip division is not suitable for division of moisture samples.



Key

1	increment	3	end plates
2	manual cutter		

Figure 10 — Strip-division method

10.4.4 Manual riffle-division method

10.4.4.1 General

Manual riffle division is applicable to ore not exceeding 40 mm nominal top size. It shall be carried out in accordance with the procedures specified below. Riffle dividers are the most satisfactory type of manual divider for pellets and sized ore.

10.4.4.2 Selection of riffle divider

An appropriate riffle divider as specified in Table 11 shall be selected to match the nominal top size of the ore. Details on the dimensions and design of riffles may be found in Annex E.

10.4.4.3 Mass of divided sample

The minimum mass of divided sample shall conform to the requirements of 10.1.6, 10.5 and 10.6.

Table 11 — Nominal top size of sample and size of riffle divider

Nominal top size mm		Riffle divider number	Riffle opening mm
Over	Up to		
31,5	40,0	90	90 ± 1
22,4	31,5	60	60 ± 1
16,0	22,4	50	50 ± 1
10,0	16,0	30	30 ± 1
5,00	10,0	20	20 ± 1
2,80	5,00	10	10 ± 0,5
	2,80	6	6 ± 0,5

10.4.4.4 Procedure

After mixing, place the sample to be divided in a container and divide it into two parts by dropping the sample uniformly with light shaking of the container into the middle of the riffles (at right angles to the riffle). One of the two divided samples should be selected at random in order to avoid introducing any bias.

Care shall be taken not to leave any material remaining in the slots of the riffle divider.

NOTE Manual riffle division is suitable for division of moisture samples, provided that there is no mixing prior to division and the number of division steps is minimized.

10.5 Preparation of test samples for chemical analysis

10.5.1 Mass and particle size

An example of a sample preparation scheme suitable for preparation of chemical analysis samples is shown in Figure 11. The particle size of the test sample for chemical analysis shall be either 100 µm or 160 µm nominal top size. The preferred method is to prepare a test sample for chemical analysis of 50 g minimum and 100 µm nominal top size from the divided gross sample of 250 µm nominal top size. However, if an appropriate grinder is utilized, a test sample for chemical analysis of either 100 µm or 160 µm nominal top size can be prepared directly from a sample coarser than 250 µm nominal top size, e.g. 2,8 mm nominal size (see Figure 11), provided the sample masses conform to Table 4.

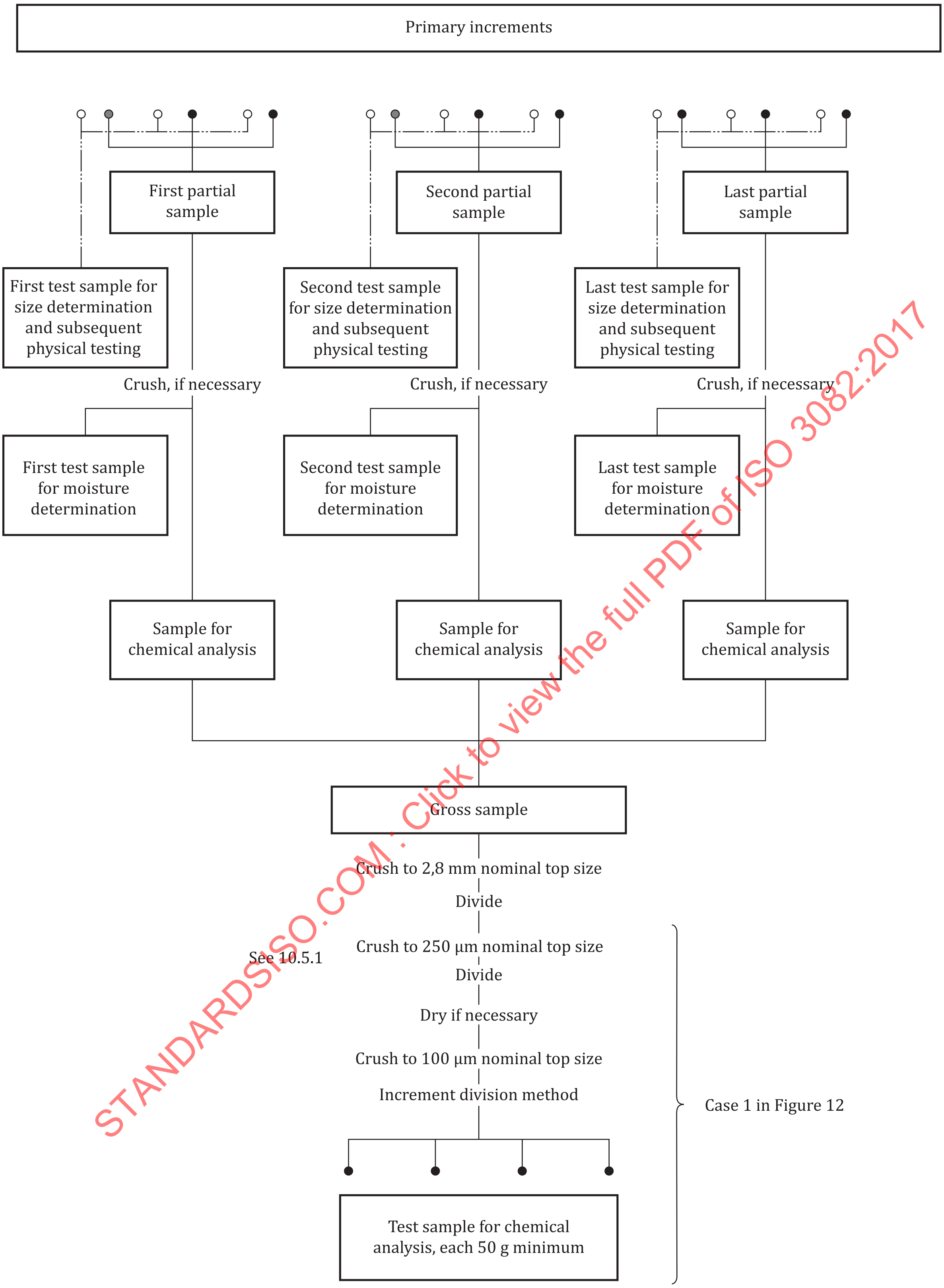


Figure 11 — Example of a sample preparation scheme for chemical analysis, moisture, size and physical testing samples where size analysis samples are subsequently used for physical testing

However, for ores containing more than 2,5 % combined water and/or oxidizable compounds, where excessive grinding would affect the result, the test sample for chemical analysis shall be 160 µm nominal top size and 100 g minimum in mass.

The preparation of test samples for chemical analysis can be carried out in one of three ways as shown in Figure 12.

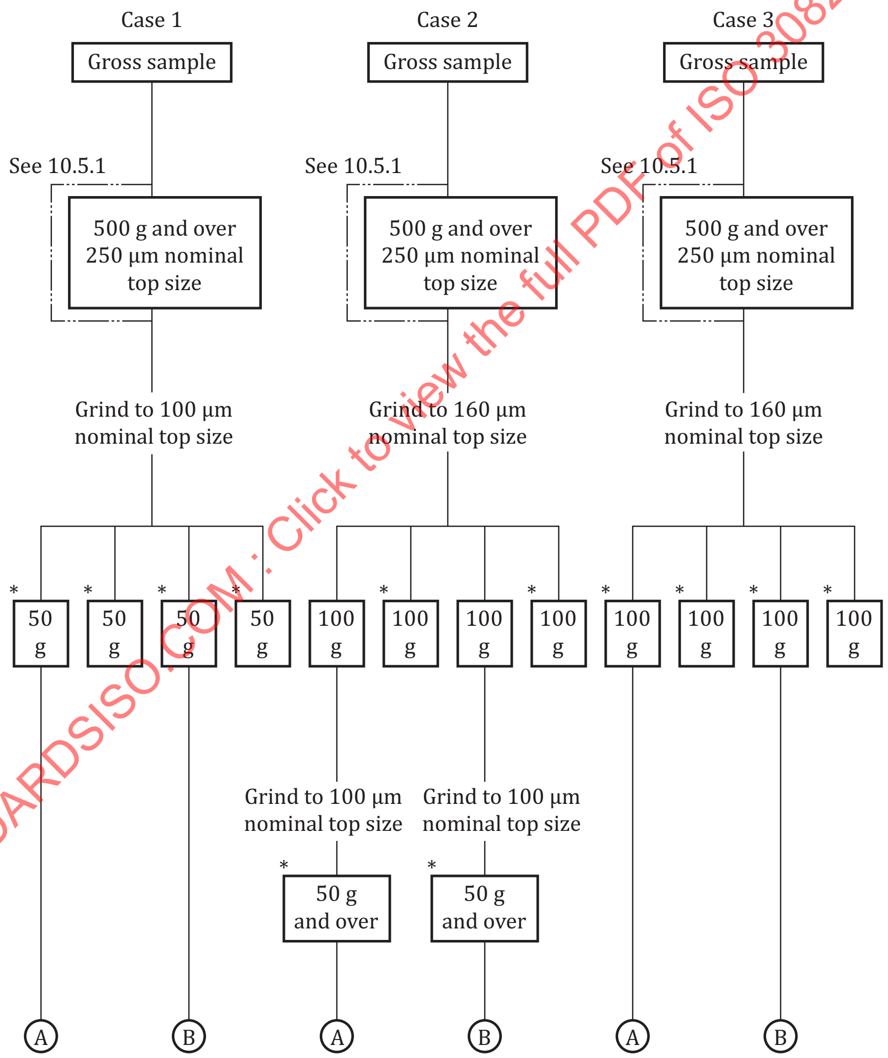
During preparation of ores which contain significant amounts of combined water and/or oxidizable compounds, special precautions should be taken to ensure that the grinding process does not generate excessive heat which could significantly change the chemical composition of the ore. Special precautions include

a) reducing the grinding time by grinding smaller charges,

b) use of a single-pass straight-through type of grinder, and

c) grinding for the minimum time to obtain the required nominal top size.

Grinding with an agate pestle and mortar or other suitable manual techniques should be used for reference purposes.



Key

* sealed samples

Figure 12 — Preparation of test samples for chemical analysis

10.5.2 Preparation to 250 µm nominal top size

If each increment, each partial sample or the gross sample is ground to 250 µm nominal top size, this shall be carried out by repeated crushing, grinding and division in accordance with 10.3 or 10.4. When the division is conducted on individual increments or partial samples before constitution of a gross sample, the gross sample shall be obtained, at a certain stage of the division, by combining quantities proportional to the mass of the individual increment or partial sample. After drying if necessary, the sample of 250 µm nominal top size shall be ground to 160 µm or 100 µm nominal top size.

The mass of the 250 µm nominal top size sample shall be sufficient to generate the required number of exchange samples.

10.5.3 Final preparation

10.5.3.1 Case 1

The 250 µm nominal top size sample, or alternatively the sample of coarser nominal top size as specified in 10.5.1, shall be ground to 100 µm nominal top size. A set of not less than four test samples, each of 50 g minimum, shall be prepared from the 100 µm nominal top size sample by an appropriate division method.

10.5.3.2 Case 2

The 250 µm nominal top size sample, or alternatively the sample of coarser nominal top size as specified in 10.5.1, shall be ground to 160 µm nominal top size. A set of not less than four test samples, each of 100 g minimum, shall be prepared from the 160 µm nominal top size sample by an appropriate division method. The test samples for chemical analysis sent to the laboratory shall be ground to 100 µm nominal top size.

10.5.3.3 Case 3

The 250 µm nominal top size sample, or alternatively the sample of coarser nominal top size as specified in 10.5.1, shall be ground to 160 µm nominal top size. A set of not less than four test samples, each of 100 g minimum, shall be prepared from the 160 µm nominal top size sample by an appropriate division method. The test samples for chemical analysis sent to the laboratory shall not be ground to any finer particle size.

10.5.4 Grinding to 100 µm or 160 µm nominal top size

10.5.4.1 General

When the 250 µm nominal top size sample or sample of coarser nominal top size is ground to 100 µm or 160 µm nominal top size, the procedure described below shall be used.

10.5.4.2 Type of grinder

Several types of grinders may be used to grind the sample for chemical analysis to 160 µm or 100 µm nominal top size, such as a top grinder, a pot mill or a ring mill.

10.5.4.3 Selection of material of construction for grinder

The selection of material for the grinder is one of the most important considerations to be given, so that the chemical composition of the sample does not change during the grinding operation.

It is recommended that an experiment be carried out, in accordance with ISO 3086, to check whether bias in chemical composition has been introduced by the grinding operation.

10.5.4.4 Dry grinding

The whole of the 250 µm nominal top size sample or sample of coarser nominal top size for chemical analysis should be ground at one time to 100 µm or 160 µm nominal top size using an appropriate grinder. When the grinding of the sample cannot be carried out at one time, the sample may be divided into a number of parts for separate grinding. After all the divided parts have been ground to 100 µm or 160 µm nominal top size, they shall be mixed thoroughly in a suitable mixer. Samples for finer grinding shall not be screened into oversize and undersize fractions, for example plus and minus 100 µm fractions, to carry out the grinding on the oversize fraction only.

Impact-type mills should be avoided for ore containing materials which have an extraordinarily different grindability to the constituent iron minerals, such as grains of quartz and fragments of shale, because of the tendency for selective grinding.

10.5.4.5 Wet grinding

When the sample for chemical analysis is cohesive in the vibrating mill during finer grinding, and when a shorter grinding time is preferable to avoid oxidation of the samples, wet grinding in a vibrating mill with a chemical medium of *n*-hexane is permissible.

10.5.5 Distribution of samples for chemical analysis

A set of not less than four test samples for chemical analysis shall be prepared in accordance with 10.5.3. The test samples to be distributed, e.g. to the seller, purchaser, referee and if required held in reserve, shall be placed in suitable containers, tightly sealed, and clearly marked in accordance with Clause 11.

10.6 Preparation of test samples for moisture determination

In mass-basis sampling, the test sample for moisture determination may be taken from each increment, each partial sample or the gross sample. When it is difficult to conduct crushing and dividing owing to a sample being adhesive or excessively wet, the sample may be predried in accordance with ISO 3087. In time-basis sampling, the test sample may also be taken from each increment, partial sample or the gross sample provided that in each case sufficient mass is obtained to constitute the required number of test portions. An example of a sample preparation scheme for moisture samples is shown in Figure 11.

Moisture samples shall be kept in airtight, non-absorbent containers to avoid any change in moisture prior to the determination of moisture content in accordance with ISO 3087.

The nominal top size of the moisture test sample shall be 31,5 mm or less, as specified in ISO 3087. Samples with a nominal top size greater than 31,5 mm shall be crushed prior to extraction of test samples for moisture determination. When it is difficult to conduct crushing and dividing owing to a sample being adhesive or excessively wet, the sample may be predried in accordance with ISO 3087.

A check is recommended to determine whether the 10 mm nominal top size test sample is biased with respect to the 22,4 mm or 31,5 mm nominal top size test sample.

The first stage of division shall be carried out in accordance with the rules of division specified in 10.3 or 10.4. Then to obtain a test portion of 10 kg minimum for 31,5 mm nominal top size, 5 kg minimum for 22,4 mm nominal top size or 1 kg minimum for 10 mm nominal top size, an appropriate method of division specified in 10.3 or 10.4 shall be used. When extracting test portions for moisture determination, the minimum mass of divided sample given in Table 4 and calculated by Formula (19) no longer applies. However, the test portions thus obtained shall not be used for preparation of samples for chemical analysis.

Preparation of test samples for moisture determination shall be carried out carefully, but quickly, to avoid moisture evaporation. The remainder of the sample may be used for preparation of a sample for chemical analysis.

NOTE Instead of preparing one test sample of 10 kg minimum at 31,5 mm nominal top size, two test portions of 5 kg minimum, each, can be prepared by dividing the test sample of 10 kg minimum into two parts.

It is recommended that moisture test samples be prepared by mechanical increment division, manual increment division or riffle division as specified in 10.3, 10.4.2 and 10.4.4 to minimize moisture evaporation. A scoop number, one or two ranks smaller than that specified in Table 8, may be used for this purpose for ores of nominal top size 31,5 mm, 22,4 mm, 10 mm or under. However, the test sample thus obtained shall not be used for preparation of a sample for chemical analysis.

The mass of the test portion shall be determined immediately. When the immediate determination of mass is not possible, the test portion shall be packed tightly in a moisture-proof container and kept in an environment that has approximately constant temperature and humidity.

The relationship between each increment or partial sample and each part (by mass) of the lot shall be recorded.

The number of test portions for moisture determination should be as specified in Table 12.

Table 12 — Number of test portions for moisture determination

Preparation of test sample	Number of partial samples per lot	Number of test portions to be tested
From gross sample	—	4
From partial sample	2	4
	3 to 7	2
	≥ 8	1
From increment	—	1

10.7 Preparation of test samples for size determination

Each increment, each partial sample or the gross sample taken for size determination, or the divided sample obtained by division of the size sample without crushing, shall be used and the size determination shall be carried out in accordance with the method specified in ISO 4701.

10.8 Preparation of test samples for physical testing

10.8.1 Selection of sample preparation procedure

10.8.1.1 General

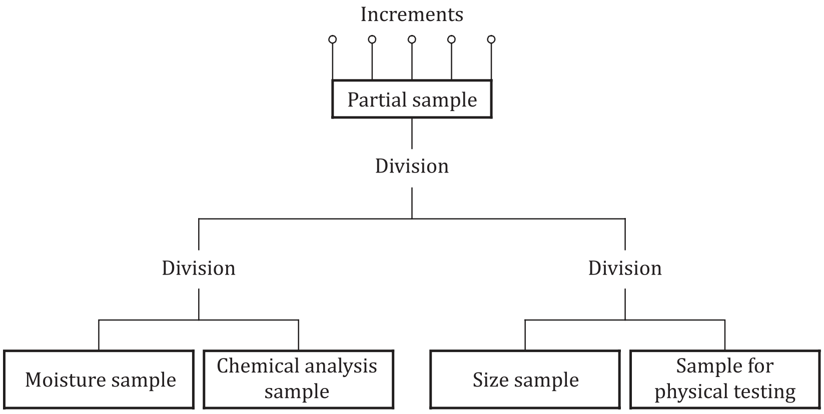
Selection of the sample preparation procedure for physical testing shall take into consideration the sample preparation apparatus and sample requirements. Test sieves shall have square openings and shall conform to the requirements of ISO 3310-1 and ISO 3310-2. Examples of suitable schemes for extraction of samples for physical testing involving both split use and multiple use of samples are described in 10.8.1.2 and 10.8.1.3.

10.8.1.2 Split use of sample

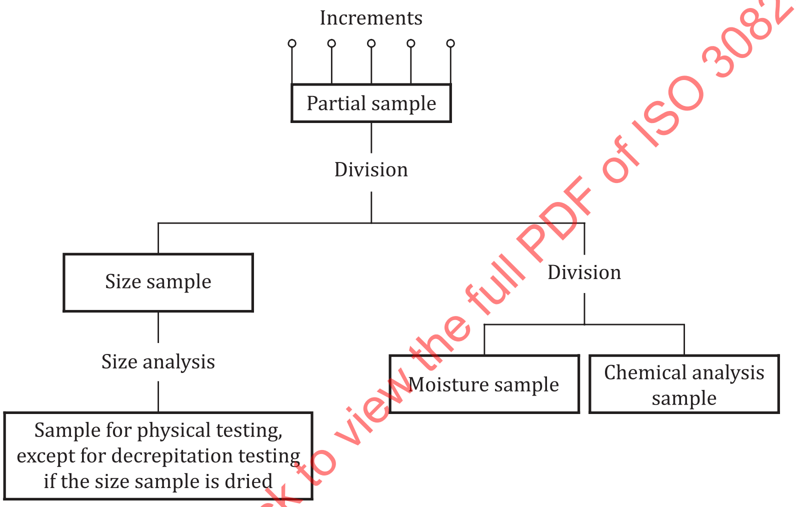
Split each partial sample into four parts and use one part for physical testing and the other three parts for determination of chemical composition, moisture content and size distribution [see Figure 13 a)].

10.8.1.3 Combination of split use and multiple use of sample

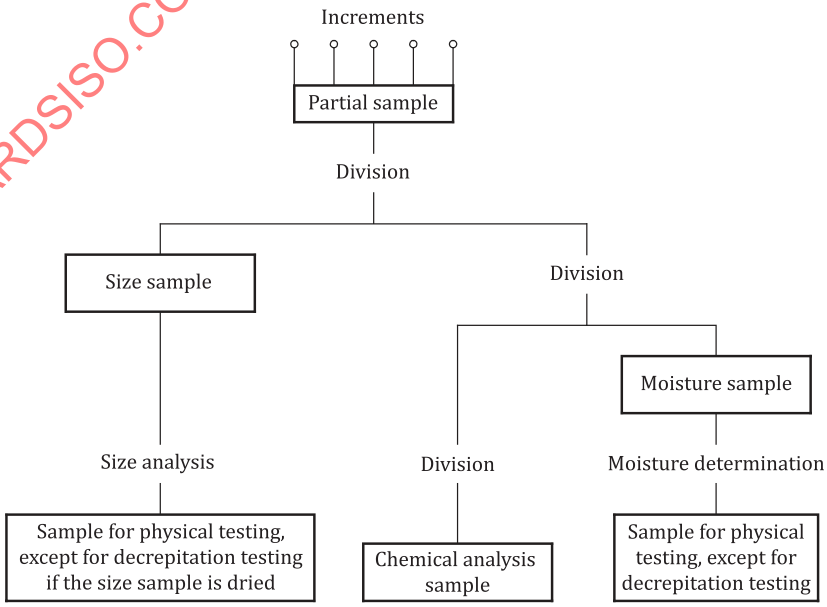
Split each partial sample into two parts and use one part for size analysis and subsequent physical testing, and the other part for preparation of moisture and chemical analysis samples [see Figure 13 b)]. If the size analysis requires drying before sieving, this sample shall not be used for decrepitation testing, which involves sieving before drying. The moisture sample may also be used subsequently for physical testing, except for decrepitation tests [see Figure 13 c)].



a) Split use of partial sample



b) Multiple use of one part of the partial sample (for size distribution and physical testing) and split use of the other part of the partial sample (for moisture determination and chemical analysis)



c) Preparation of two samples for physical testing

Figure 13 — Examples of schemes for preparation of samples for physical testing

10.8.2 Extraction of test samples

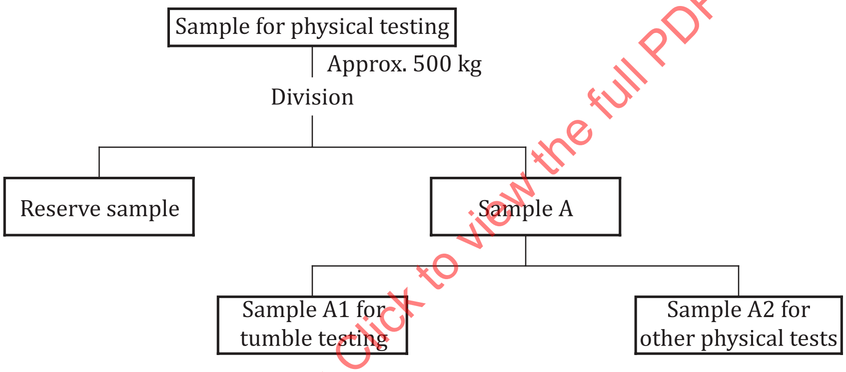
10.8.2.1 General

The sample for physical testing shall be divided in accordance with 10.3 or 10.4 to prepare test samples for determination of physical properties, irrespective of the minimum mass rules specified in 10.1.6. However, whatever division method is used, it should be demonstrated that the precision detailed in Table 6 is obtained using the procedures outlined in ISO 3085.

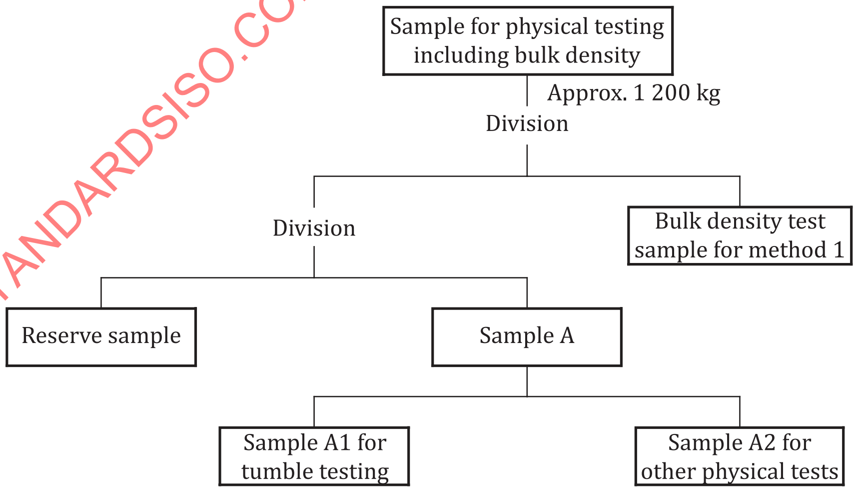
The sample for physical testing shall be divided into two parts; one for the preparation of test samples for the specified physical properties (Sample A) and the other for retention as a reserve sample [see Figure 14 a)].

When the bulk density test is carried out, the sample for physical testing (approximately 1 200 kg) shall be divided into two parts: one for preparation of the bulk density test sample (approximately 600 kg) and the other for further division into two parts, i.e. one for preparation of physical test samples other than for bulk density determination (approximately 300 kg, Sample A) and the other for retention as the reserve sample [see Figure 14 b)].

Sample A shall be divided into 2 parts: sample A1 for tumble testing and sample A2 for other physical tests [see Figure 14 b)].



a) Example of preparation of test samples for determination of physical properties



b) Example of preparation of test samples for determination of physical properties including bulk density

Figure 14 — Examples of schemes for preparation of test samples for determination of physical properties

10.8.2.2 Test sample for tumble test

10.8.2.2.1 General

Test samples for the tumble test specified in ISO 3271 for pellets, sinter and lump ores shall be prepared as specified in 10.8.2.2.2 and 10.8.2.2.3.

10.8.2.2.2 Pellets

Dry sample A1 at 105 °C ± 5 °C to constant mass and then allow to cool to room temperature. Sieve it using 40 mm and 6,3 mm sieves. Discard the +40 mm and −6,3 mm fractions. Divide the −40+6,3 mm fraction to obtain at least 60 kg for tumble testing [see Figure 15 a)].

10.8.2.2.3 Sinter and lump ores

Dry sample A1 at 105 °C ± 5 °C to constant mass and then allow to cool to room temperature. Sieve it using 40 mm and 10 mm sieves. Discard the +40 mm and −10 mm fractions. Divide the −40+10 mm fraction to obtain at least 60 kg for tumble testing [see Figure 15 b)].

a) Example of preparation of pellet samples for tumble testing

b) Example of preparation of sinter and lump ore samples for tumble testing

Figure 15 — Examples of schemes for preparation of pellet, sinter and lump ore test samples for tumble testing

10.8.2.3 Test sample for bulk density test

When a bulk density test is required using method 1 (small container), divide the sample for physical testing (approximately 1 200 kg) into two parts to obtain a test sample of at least 600 kg [see Figure 14 b)].

10.8.2.4 Test samples for physical tests other than tumble test and bulk density test

10.8.2.4.1 General

The size range and the approximate mass of test sample for each type of test and type of iron ore, required by the relevant International Standards, are detailed in Table 13.

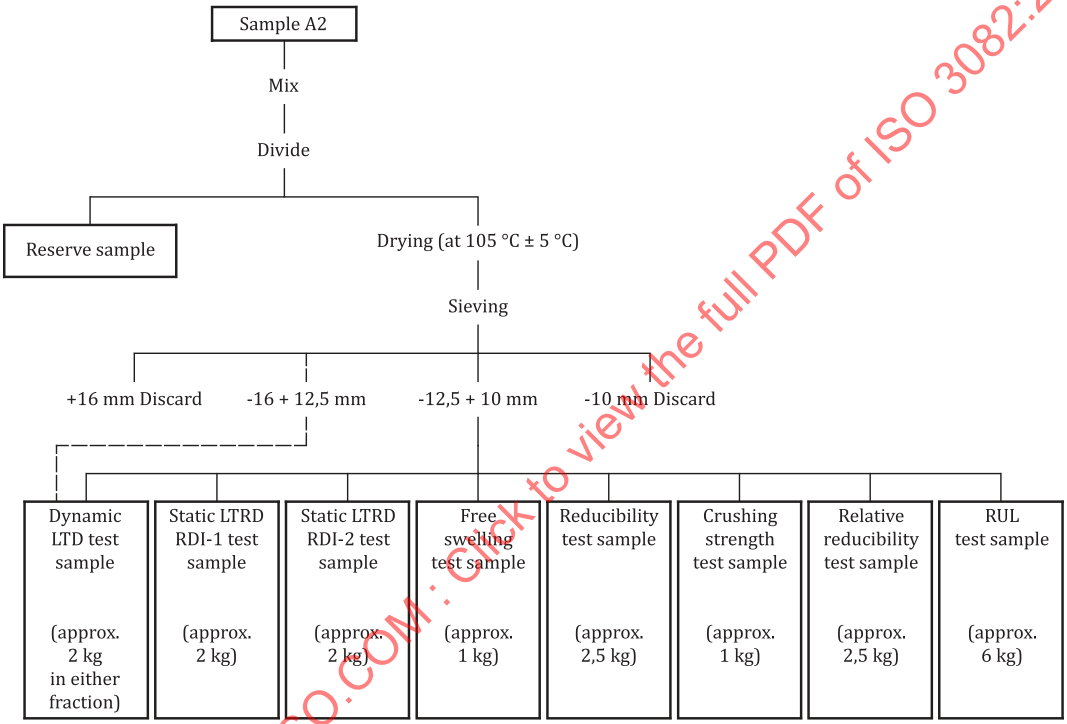
10.8.2.4.2 Blast-furnace pellets

Divide sample A2, and set one part aside in reserve. Dry the other sample at 105 °C ± 5 °C to constant mass and sieve it using 16 mm, 12,5 mm and 10 mm sieves. Discard the +16 mm and −10 mm fractions.

NOTE The 16 mm sieve is necessary only when the dynamic low-temperature disintegration (LTD) test is to be carried out using the −16+12,5 mm fraction.

Mix the −12,5+10 mm fraction and divide to obtain the required test samples for the physical tests. Use the −12,5+10 mm fraction to obtain the LTD dynamic test sample, or alternatively the −16+12,5 mm fraction if required (see Figure 16).

The requirements for each test are listed in Table 13.



Key

LTD	Low-temperature disintegration.
LTRD	Low-temperature reduction-disintegration.

Figure 16 — Example of a scheme for preparation of test samples for determination of physical properties of blast-furnace pellets

Table 13 — Size range and mass of test samples and test portions for physical tests other than tumble and bulk density tests

Test	International Standard	Application BF/DR	Type of iron ore	Size range of test sample and test portion	Min. mass of test sample	Number of test portions	Approx. mass of test portions
Reducibility	ISO 4695	BF	Pellets/ Sinters/ Lump ores	−12,5+10 mm	2,5 kg	4 for test 1 for chem.	0,5 kg
Static LTRD RDI-1	ISO 4696-1	BF	Pellets/ Sinters/ Lump ores	−12,5+10 mm	2,0 kg	4 for test	0,5 kg
Static LTRD RDI-2	ISO 4696-2	BF	Pellets	−12,5+10 mm	2,0 kg	4 for test	0,5 kg
			Sinters/ Lump ores	−20+16 mm			
Free-swelling	ISO 4698	BF	Pellets	−12,5+10 mm	1,0 kg	4 for test	18 pellets
Crushing strength	ISO 4700	BF/DR	Pellets	−12,5+10 mm	1,0 kg	1 for test	60 or more pellets
Relative reducibility	ISO 7215	BF	Pellets	−12,5+10 mm	2,5 kg	4 for test 1 for chem.	0,5 kg
			Sinters/ Lump ores	−20+18 mm			
RUL test	ISO 7992	BF	Pellets/ Lump ores	−12,5+10 mm	6,0 kg	4 for test 1 for chem.	1,2 kg
Decrepitation index	ISO 8371	BF/DR	Lump ores	−25+20 mm	5,0 kg	10 for test	0,5 kg
Clustering	ISO 11256	DR	Pellets	50 % −16+12,5 mm 50 % −12,5+10 mm	10,0 kg	4 for test 1 for chem.	2,0 kg
Disintegration and metallization	ISO 11257	DR	Pellets	50 % −16+12,5 mm 50 % −12,5+10 mm	2,0 kg	4 for test	0,5 kg
			Lump ores	50 % −20+16 mm 50 % −16+10 mm			
Reducibility and metallization	ISO 11258	DR	Pellets	50 % −16+12,5 mm 50 % −12,5+10 mm	2,5 kg	4 for test 1 for chem.	0,5 kg
			Lump ores	50 % −20+16 mm 50 % −16+10 mm			
Dynamic LTD test	ISO 13930	BF	Pellets	−16+12,5 mm or −12,5+10 mm	2,0 kg	4 for test	0,5 kg
			Lump ores	−12,5+10 mm			

LTRD = Low-temperature reduction-disintegration

RUL = Reduction under load

LTD = Low-temperature disintegration

BF = Blast furnace

DR = Direct reduction

Chem. = Chemical analysis

10.8.2.4.3 Direct-reduction pellets

Divide sample A2, and set one part aside in reserve. Dry the other sample at 105 °C ± 5 °C to constant mass and sieve using 16 mm, 12,5 mm and 10 mm sieves. Discard the +16 mm and − 10 mm fractions (see Figure 17).

Mix and divide the −16+12,5 mm and −12,5+10 mm fractions, separately. The test samples for clustering, disintegration and metallization, and reducibility and metallization tests, are constituted from the two fractions by mixing them on a 1:1 basis by mass. The test sample for crushing strength is constituted from the −12,5+10 mm fraction only.

The requirements for each test are listed in Table 13.

10.8.2.4.4 Sinter and blast-furnace lump ores

Divide sample A2 into three parts and prepare these parts as follows.

Part 1: Set aside in reserve.

Part 2: Dry at 105 °C ± 5 °C to constant mass. Divide into three portions, according to the physical tests to be carried out and prepare these portions as follows (see Figure 18).

> **Portion 1:** This portion is for the static LTRD RDI-1, dynamic LTD, reducibility and RUL tests. Sieve it using a 12,5 mm sieve. Crush the +12,5 mm fraction carefully and sieve it using a 16 mm sieve. Crush the +16 mm fraction to −16 mm. Mix the −12,5 mm and −16 mm fractions and sieve the mixture using 12,5 mm and 10 mm sieves. Discard the +12,5 mm and −10 mm fractions. Divide the −12,5+10 mm fraction to obtain the required test samples. Dynamic LTD and RUL tests are applicable only for lump ores.
>
> **Portion 2:** This portion is for the static LTRD RDI-2 test. Sieve it using a 20 mm sieve. Crush the +20 mm fraction carefully and sieve it using a 22,4 mm sieve. Crush the +22,4 mm fraction to −22,4 mm. Mix the −20 mm and −22,4 mm fractions and sieve the mixture using 20 mm and 16 mm sieves. Discard the +20 mm and −16 mm fractions. If necessary, divide the −20+16 mm fraction to obtain the test sample.
>
> **Portion 3:** This portion is for the relative reducibility test. Sieve it using 20 mm and 18 mm sieves. Discard the +20 mm and −18 mm fractions. If necessary, divide the −20+18 mm fraction to obtain the test sample.

Part 3: Use this part to obtain the decrepitation test sample. Sieve this part using 25 mm and 20 mm sieves. Discard the +25 mm and −20 mm fractions. Oven dry the −25+20 mm fraction at 105 °C ± 5 °C to constant mass. Screen the test sample again on a 20 mm sieve to remove any adhering fines and divide it to obtain the final test sample for the decrepitation test. Keep the test sample in a desiccator until testing takes place (see Figure 18).

The requirements for each test are listed in Table 13.

10.8.2.4.5 Direct-reduction lump ores

Divide sample A2 into three parts and prepare these parts as follows.

Part 1: Set aside in reserve.

Part 2: Dry this part at 105 °C ± 5 °C to constant mass. Sieve this part using 20 mm, 16 mm and 10 mm sieves. Discard the +20 mm and −10 mm fractions. Mix and divide the −20+16 mm and the −16+10 mm fractions separately. The test samples for disintegration and metallization tests and for reducibility and metallization tests are constituted from the two fractions by mixing them on a 1:1 basis by mass (see Figure 19).

Part 3: Use this part to obtain the decrepitation test sample. Sieve this part using 25 mm and 20 mm sieves. Discard the +25 mm and −20 mm fractions. Oven dry the −25+20 mm fraction at 105 °C ± 5 °C to constant mass. Screen the test sample again on a 20 mm sieve to remove any adhering fines and divide it to obtain the final test sample for the decrepitation test. Keep the test sample in a desiccator until testing takes place (see Figure 19).

The requirements for each test are listed in Table 13.

10.8.3 Reserve samples

The reserve samples shall be put into sealed containers and kept at least until the test report is accepted by the supplier and purchaser.

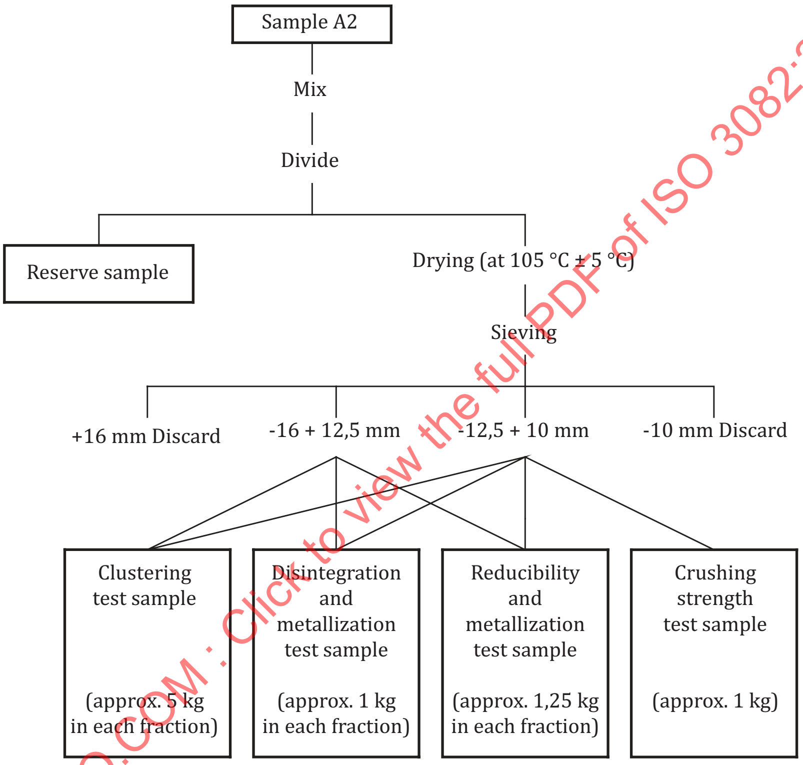


Figure 17 — Example of a scheme for preparation of test samples for determination of physical properties of direct-reduction pellets

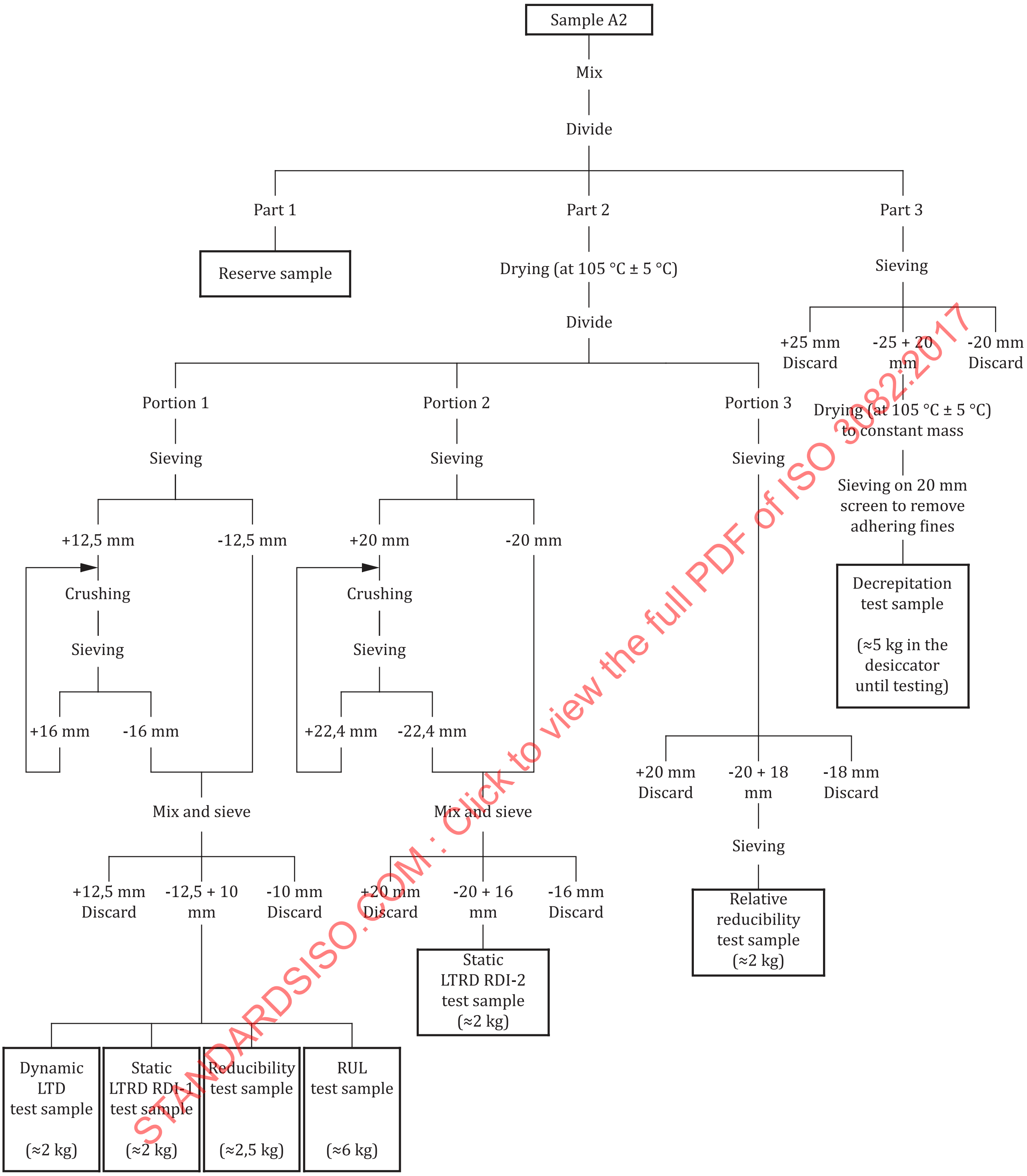


Figure 18 — Example of a scheme for preparation of test samples for determination of physical properties of sinter and blast-furnace lump ores

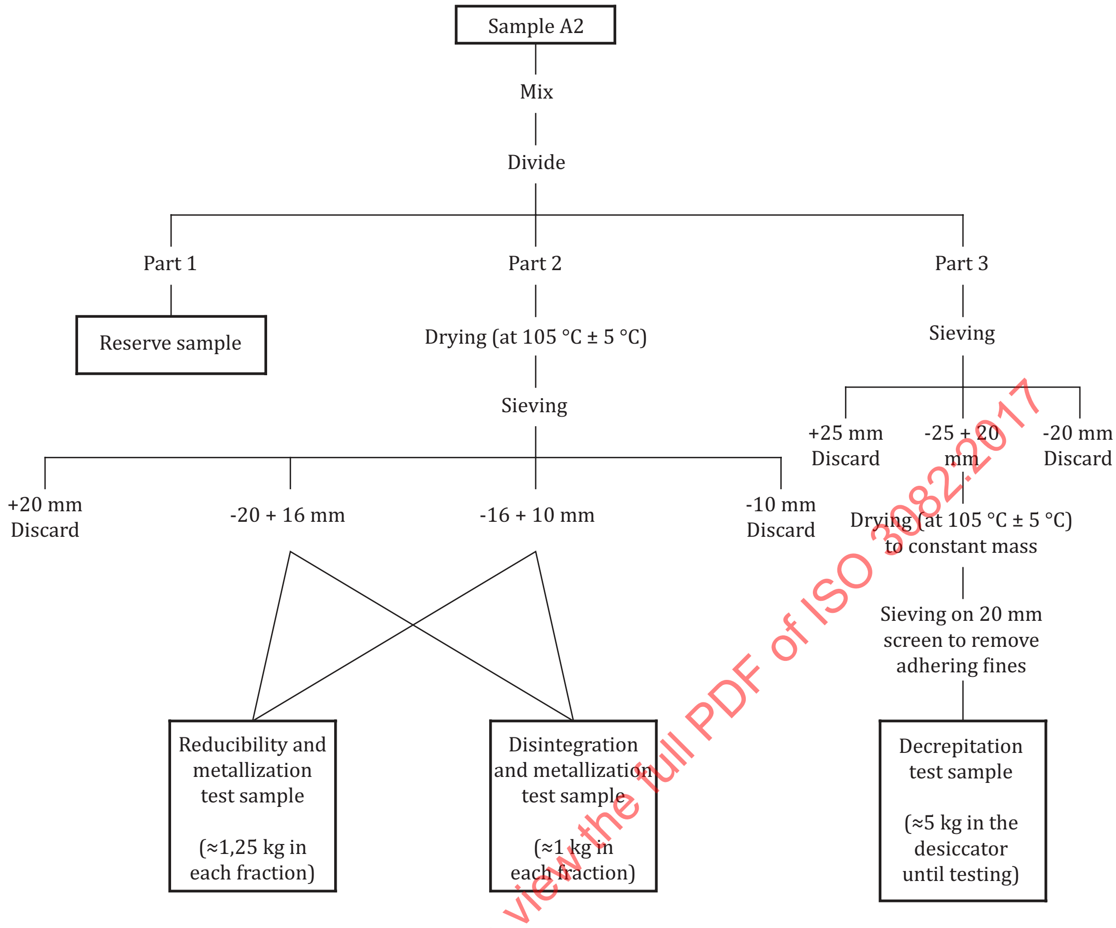


Figure 19 — Example of a scheme for preparation of test samples for determination of physical properties of direct-reduction lump ores

11 Packing and marking of samples

The samples for distribution shall be tightly sealed in airtight containers. The following information should be shown on the label and on a card placed in the container:

a) type and grade of the ore and name of the lot (name of ship or train, etc.);

b) mass of the lot;

c) sample number;

d) place, date and method of sampling;

e) moisture content of the lot;

f) place and date of sample preparation;

g) particle size of the sample;

h) purpose of sampling, e.g. bias test, shipping sample;

i) any other item as necessary.

Annex A
(informative)

Inspection of mechanical sampling systems

A.1 Purpose of inspections

To ensure reliable operation of mechanical sampling systems and conformance with this document, it is recommended that a sampling checklist be developed for each sampling installation with input from the following sources:

a) the design criteria specified in Clause 7;

b) sampling equipment operating and maintenance manuals;

c) management responsible for the system;

d) personnel operating or maintaining the system;

e) designers and commissioning personnel.

The frequency and detail of inspections are determined by such factors as, but not limited to, the reliability of the sampling system, the handling characteristics of the ore, the frequency of use of the system and the purpose of sampling. Operational procedures should be established and inspections carried out immediately before, during and immediately after using the sampling system for sampling a given lot.

A.2 Mechanical and operational inspections

It is recommended that mechanical and operational inspections commence at the primary cutter and follow through the system to the final *on-line* sample collection point, and that inspections be made both with and without ore running through the system. Inspection should include the following checks to ensure conformance with this document:

a) that operational settings are correct, taking into account the lot size, the number of primary increments and the sample collection interval;

b) that the cutter apertures are in good condition and comply with 7.5 and 7.6;

c) that the speed in both directions of all cutters complies with 7.5.5. The cutter speed can be determined by dividing the distance the cutter travels while in the ore stream by the time required to travel that distance. For time-based sampling, check that the speed is constant, while for mass-based sampling check the speed at several flow rates to ensure that the speed is proportional to the flow rate (variable speed cutter) or constant (fixed speed cutter);

d) that the speed of all cutters is uniform while in the ore stream;

e) that the correct number of increments is taken by all cutters. It should also be checked that the time or mass interval between primary cuts is correct to ensure that the minimum number of increments is collected for the lot based on the maximum attainable feed rate;

f) that all cutters are parked out of the ore stream in the parked position and that no ore enters the cutter in this position. There should be no holes in the baffle plates, dust doors or seals that may allow ore to enter the primary sample chute or hopper;

g) that increment masses conform to 5.1.4;

h) that cutter apertures and sample chutes are clear of ore build up or blockages and that they are clear of foreign material, such as wood, paper, rags, stones or metal;

i) that belt feeders (sample conveyors) and vibrating feeders are in good condition. The correct tracking of conveyor belts and the condition of conveyor belts, skirts and belt scrapers are especially important for sample integrity, so check the flow rate settings of vibrating belt feeders and that belt scrapers and skirts are adjusted properly to avoid spillages;

j) that crushers are in good condition and that the particle size and mass of crushed products conforms to design. Variations in product size over time can indicate that maintenance is required and ensure that there is no spillage from the crusher body and chutes;

k) that the final sample collection system is in good condition. Checks should be made to ensure that sample integrity is not being compromised through contamination, sample loss or moisture loss;

l) that final sample masses conform to Clause 10;

An example of a suitable checklist for a mechanical sampling system is given in Table A.1.

A.3 Control charts

A.3.1 General

Control charts are very useful for monitoring the performance of sampling systems on an ongoing basis, particularly for the sampling ratio and the extraction ratio as described in A.3.2 and A.3.3.

A.3.2 Sampling ratio

The sampling ratio (R_1) is the actual mass of sample in kilograms (m_A) divided by the mass of material that it represents in tonnes (m_{SL}), as expressed by Formula (A.1):

$$R_1 = \frac{1\,000\, m_A}{m_{SL}} \tag{A.1}$$

The sampling-ratio control chart is a plot of sampling ratio as a function of increments sampled.

Sampling-ratio comparisons should be made only for like system settings (same cutter apertures, timer settings, sub-lot size and mass flow rate through the system), so a separate control chart is required for each set of system settings used. Samples with a sampling ratio out of control are suspect and should be investigated for validity. When there is a significant variation in the sampling ratio, the reasons for this should also be investigated. An example of a sampling-ratio control chart is shown in Figure A.1.

A.3.3 Extraction ratio

The extraction ratio (R_2) is the actual mass of the sample in kilograms (m_A) divided by the mass of sample in kilograms (m_C) calculated from the material flow rate, the frequency of cuts, the cutter aperture and the cutter speed, as expressed by Formula (A.2):

$$R_2 = \frac{m_A}{m_C} \tag{A.2}$$

The results should be plotted over many lots so that long-term trends for a particular system can be monitored (see Figure A.1). When a sampling system is performing correctly, the extraction ratio should be the unity (1,0). However, when the extraction ratio differs significantly from 1,0, the sampling system should be investigated.

The extraction ratio is useful in determining whether there are long-term problems with a particular sampling system. For example, if a cutter speed reduces over several weeks due to a faulty drive, the mass of sample through the sampling system increases and the long-term extraction-ratio data would indicate a problem with the system. When system settings are changed, the extraction ratio is less likely to change than the sampling ratio, so it is more useful in comparing the performance of different sampling systems. An example of an extraction-ratio control chart is shown in Figure A.1.

Table A.1 — Example of a checklist for mechanical sampling systems

Company: ____________________ **Date:** ____________________
Sampler location and identification: __________ **Inspector:** ____________________

1 General information

		Observation	Specification (if applicable)	Tolerance (if applicable)
a)	Weather conditions			
b)	Ore type			
c)	Nominal top size of ore			
d)	Moisture content			
e)	Lot size			
f)	Flow rate (maximum and normal)			
g)	Purpose of sample			
h)	Source of ore			
i)	Number of transfer points between sampling point and point of loading/discharge		minimum	
j)	Total drop height between sampling point and point of loading/discharge		minimum	

2 Type of sampling system

Single stage ☐
Two stage ☐
Three stage ☐

3 Primary cutter

		Observation	Specification (if applicable)	Tolerance (if applicable)
a)	Type of cutter			
b)	Cutter drive			
c)	Nominal top size of ore			
d)	Drop height		minimize	
e)	Periodicity in ore stream		minimize	
f)	Cutter aperture (see 7.5.4)		3d minimum	
g)	Condition of cutter lips		straight and no significant wear	
h)	Angle between cutter aperture and stream		normal	
i)	Build-up in cutter aperture and throat		not significant	
j)	Unrestricted flow through cutter		no choking or reflux	
k)	Cutting full stream and belt scrapings		full-stream cut	
l)	Cutter speed (see 7.5.5)		0,6 m/s max.	5%
m)	Uniform cutter speed (see 7.6)		uniform speed	5%

Table A.1 *(continued)*

n)	Increment mass (see 6.1.1 and 6.2.1)			CV ≤ 20%
o)	Contamination or loss of sample, including hang-up in chutes and cutter buckets		not significant	
p)	Moisture loss		not significant	
q)	Cutter parks out of ore stream		out of stream	
r)	Mass or time-basis sampling			
s)	Interval between cuts			
t)	Number of cuts per lot			
u)	Extraction ratio (ratio of actual to calculated increment mass)		1.0	

4	**Primary sample feeder and chutes**	**Observation**	**Specification** (if applicable)	**Tolerance** (if applicable)
a)	Type of feeder			
b)	Feed rate			
c)	Tracking of feed conveyors			
d)	Chutes		no holes	
e)	Contamination or loss of sample		not significant	
f)	Moisture loss		not significant	
g)	Blockages		not significant	
h)	Crusher			
i)	Crusher product particle size			

5	**Secondary cutter**	**Observation**	**Specification** (if applicable)	**Tolerance** (if applicable)
a)	Type of cutter			
b)	Cutter drive			
c)	Nominal top size of ore			
d)	Drop height		minimize	
e)	Periodicity in ore stream		minimize	
f)	Cutter aperture (see 7.6)		3d minimum	
g)	Condition of cutter lips		straight and no significant wear	
h)	Angle between cutter aperture and stream		normal	
i)	Build-up in cutter aperture and throat		not significant	
j)	Unrestricted flow through cutter		no choking or reflux	
k)	Cutting full stream and belt scrapings		full-stream cut	
l)	Cutter speed (see 7.6)		0,6 m/s max.	5%
m)	Uniform cutter speed		uniform speed	5%
n)	Random start for first secondary cut relative to primary cutter			
o)	Increment mass			