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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3082 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 1, *Sampling*.

This third edition cancels and replaces the second edition (ISO 3082:1998), together with ISO 3081:1986 and ISO 3083:1986, of which it constitutes a collation and technical revision.

Annexes B, D and E form a normative part of this International Standard. Annexes A and C are for information only.

Iron ores — Sampling and sample preparation procedures

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

1 Scope

This International Standard gives

a) the underlying theory,

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

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

for mechanical sampling, manual sampling and preparation of samples taken from a lot under transfer to determine the chemical composition, moisture content and size distribution of the lot. Sampling and sample preparation procedures for physical testing are specified in ISO 10836.

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

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

ISO 3085:—[1)], *Iron ores — Experimental methods for checking the precision of sampling and sample preparation and mesasurement.*

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

1) To be published. (Revision of ISO 3085:1996)

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

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

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

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

3 Terms and definitions

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

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

3.2
increment
quantity of ore collected in a single operation of a sampling device

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

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

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

3.6
test sample
sample, prepared to meet all specific conditions for a test

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

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

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

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

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

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

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

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

NOTE This method is required for sampling on a mass basis. “Almost uniform” means that variations in mass are less than 20 % in terms of the coefficient of variation.

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

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

3.16
nominal top size
smallest aperture size, within the range of the R20 Series (in ISO 565:1990, square opening), such that no more than 5 % by mass of an ore is retained on the sieve

4 General considerations for sampling and sample preparation

4.1 Basic requirements

The basic requirement for a correct sampling scheme is that all parts of the ore in the lot have an equal opportunity of being selected and becoming part of the partial sample or gross sample for analysis. Any deviation from this basic requirement can result in an unacceptable loss of accuracy and precision. An incorrect sampling scheme cannot be relied on to provide representative samples.

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

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

In-situ sampling from stationary situations such as wagons is permitted only for fine iron ore concentrates, provided the sampling device, e.g., a spear or an auger, penetrate to the full depth of the concentrate at the point selected for sampling and the full column of concentrate is extracted.

Sampling shall be carried out by systematic sampling either on a mass basis (see 6.1) or on a time basis (see 6.2), provided no bias is introduced by periodic variation in quality or quantity. If this is not the case, stratified random sampling within fixed mass or time intervals shall be carried out (see 6.3).

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

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

4.2 Establishing a sampling scheme

The procedure for establishing a sampling scheme is as follows:

a) identify the lot to be sampled;

b) ascertain the nominal top size;

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

d) specify the precision required;

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

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

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

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

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

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

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

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

m) crush the sample, if necessary, except for the size sample;

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

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

p) prepare the test sample.

4.3 System verification

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

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

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

5 Fundamentals of sampling and sample preparation

5.1 Minimization of bias

5.1.1 General

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

5.1.2 Minimization of particle size degradation

Minimization of particle size degradation of samples used for determination of size distribution is vital 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 or density of individual particles. If this requirement is not respected, bias is easily introduced. This results in the following design requirements for sampling and sample preparation systems:

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

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

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

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

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

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

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

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

i) a complete column of concentrate shall be extracted when sampling iron ore concentrate in a wagon (see 8.2).

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

5.1.4 Increment mass

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

5.1.4.1 Increment mass for falling stream sampling

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

$$m_I = \frac{q l_1}{3{,}6\, v_C} \qquad (1)$$

where

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

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

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

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

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

5.1.4.2 Increment mass for stopped-belt sampling

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

$$m_I = \frac{q l_2}{3\,600\, v_B} \qquad (2)$$

where

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

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

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

5.1.4.3 Increment mass for manual sampling using spear or auger

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

$$m_I = \frac{\pi \rho l_3^2 L}{4\ 000} \tag{3}$$

where

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

L is the depth of concentrate in the wagon, in metres.

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

This method of extracting increments is only applicable to sampling fine iron ore concentrates.

5.2 Overall precision

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

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

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

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

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

where

σ_S is the sampling standard deviation;

σ_P is the sample preparation standard deviation;

σ_M is the measurement standard deviation;

σ_W is the quality variation of the ore;

n_1 is the number of primary increments.

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

Quality characteristics		Approximate overall precision β_{SPM} Mass of lot t								
		Over 270 000	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		0,34	0,35	0,37	0,38	0,40	0,42	0,45	0,49	0,55
Silica content		0,34	0,35	0,37	0,38	0,40	0,42	0,45	0,49	0,55
Alumina content		0,11	0,12	0,12	0,13	0,14	0,15	0,16	0,18	0,20
Phosphorus content		0,003 4	0,003 5	0,003 6	0,003 7	0,003 8	0,004 0	0,004 2	0,004 5	0,004 8
Moisture content		0,34	0,35	0,37	0,38	0,40	0,42	0,45	0,49	0,55
Size –200 mm ore	–10 mm fraction mean 20 %	3,4	3,5	3,6	3,7	3,9	4,0	4,2	4,4	5,0
Size –50 mm ore										
Size –31,5+6,3 mm ore	–6,3 mm fraction mean 10 %	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 %	0,68	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.

Equations (4), (5) and (6) are based on the theory of stratified sampling (see annex B for details). The number of primary increments to be taken for a lot is dependent on the sampling precision required and on the quality variation of the ore to be sampled. Thus, before the number of primary increments can be determined, it is necessary to define:

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

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

When on-line sample preparation takes place within the sample plant away from the preparation laboratory, the distinction between the terms sampling and sample preparation becomes unclear. The precision of on-line sample preparation may be included in either the sampling precision or in the sample preparation precision. The choice depends on how easy it is to separate the precision of secondary and tertiary sampling from that of primary sampling. In any event, sample preparation also constitutes a sampling operation, because a representative part of the sample is selected for subsequent processing.

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

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

where

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

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

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

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

5.3 Quality variation

The quality variation, σ_W, is a measure of the heterogeneity of the lot and is the standard deviation of the quality characteristics of increments within strata for mass-basis systematic sampling. The characteristics to be selected for determining quality variation include the iron, silica, alumina, phosphorus and moisture contents and the percentage of a given size fraction.

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

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

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

Any ore whose quality variation is unknown shall be considered to have “large” quality variation. In this case, measurements shall be conducted at the earliest possible opportunity in accordance with ISO 3084 in order to determine the quality variation.

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

5.4 Sampling precision and number of primary increments

5.4.1 Mass basis sampling

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

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

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.

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		
>	⩽	Fe, SiO_2 or moisture content	Al_2O_3 content	P content	–200 mm or –50 mm ores, –10 mm fraction	31,5 mm ores, +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
270		0,31	0,09	0,002 3	1,55	0,77	0,47	260	130	65
210	270	0,32	0,09	0,002 4	1,61	0,80	0,48	240	120	60
150	210	0,34	0,10	0,002 5	1,69	0,84	0,51	220	110	55
100	150	0,35	0,10	0,002 6	1,77	0,88	0,53	200	100	50
70	100	0,37	0,11	0,002 7	1,86	0,92	0,56	180	90	45
45	70	0,39	0,11	0,002 9	1,98	0,98	0,59	160	80	40
30	45	0,42	0,12	0,003 1	2,11	1,05	0,63	140	70	35
15	30	0,45	0,13	0,003 4	2,28	1,13	0,68	120	60	30
0	15	0,50	0,14	0,003 7	2,50	1,24	0,75	100	50	25

NOTE The values of n_1 may 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 $\sqrt{2} = 1{,}4$.

5.4.2 Time basis sampling

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

5.5 Precision of sample preparation and overall precision

5.5.1 General

The precision of sample preparation depends on the choice of the 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 shall be better than that specified in Table 3 for each ore type.

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:

$$\sigma^2_{SPM} = \sigma^2_S + \sigma^2_P + \frac{\sigma^2_M}{n_2} \tag{9}$$

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:

$$\sigma^2_{SPM} = \sigma^2_S + \frac{\sigma^2_P + \frac{\sigma^2_M}{n_2}}{n_3} \tag{10}$$

where σ_P is the precision of preparing a test sample from each partial sample.

Further, when the above n_3 partial samples are combined into a gross sample at an appropriate (–10 mm or less) stage after individual sample preparation, and n_2 measurements are carried out on the gross sample, the overall precision will be:

$$\sigma^2_{SPM} = \sigma^2_S + \frac{\sigma^2_{P1}}{n_3} + \sigma^2_{P2} + \frac{\sigma^2_M}{n_2} \tag{11}$$

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:

$$\sigma^2_{SPM} = \sigma^2_S + \frac{\sigma^2_P + \frac{\sigma^2_M}{n_2}}{n_1} \quad (12)$$

where

σ_P is the precision of preparing a test sample from each increment;

n_1 is the number of primary increments.

Further, when all the increments are combined into a gross sample at an appropriate stage (~10 mm or less) after individual sample preparation, and n_2 measurements are carried out on the gross sample, the overall precision will be:

$$\sigma^2_{SPM} = \sigma^2_S + \frac{\sigma^2_{P1}}{n_1} + \sigma^2_{P2} + \frac{\sigma^2_M}{n_2} \quad (13)$$

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, C_V, is defined as the ratio of standard deviation, σ_{mass}, to the mean value, $\bar{m}$, of the mass of the increments, expressed as a percentage as follows:

$$C_V = \frac{100\sigma_{mass}}{\bar{m}} \quad (14)$$

For example, if the average mass of increment is to be 100 kg, the increments shall 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 must 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 uniform mass, one or more of the following measures shall be taken:

a) installation of a variable-speed cutter;

b) control of the ore flow on the conveyor belt ahead of the sampling point;

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 (according to 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.

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 the equation:

$$\Delta m \leqslant \frac{m_{\mathrm{L}}}{n_1} \tag{15}$$

where

m_{L} is the mass, in tonnes, of the lot;

n_1 is the number of primary increments determined in 5.4.1.

The mass interval selected shall be smaller than that calculated above to ensure that the minimum number of primary increments is greater than 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 may involve the sampler taking a forward and return cut through the ore stream.

NOTE 2 Stopped-belt sampling may 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 cutter may be used 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 the equation:

$$\Delta t \leqslant \frac{60 m_{\mathrm{L}}}{q_{\max} n_1} \qquad (16)$$

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 than that calculated in order to ensure that the minimum number of primary increments is greater than 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 may take a forward and return cut through the ore stream in a complete cycle.

NOTE 2 Stopped-belt sampling may 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 used 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 International Standard deals only with sample cutters which 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 upon 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 according to 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.

NOTE 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;

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 which 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

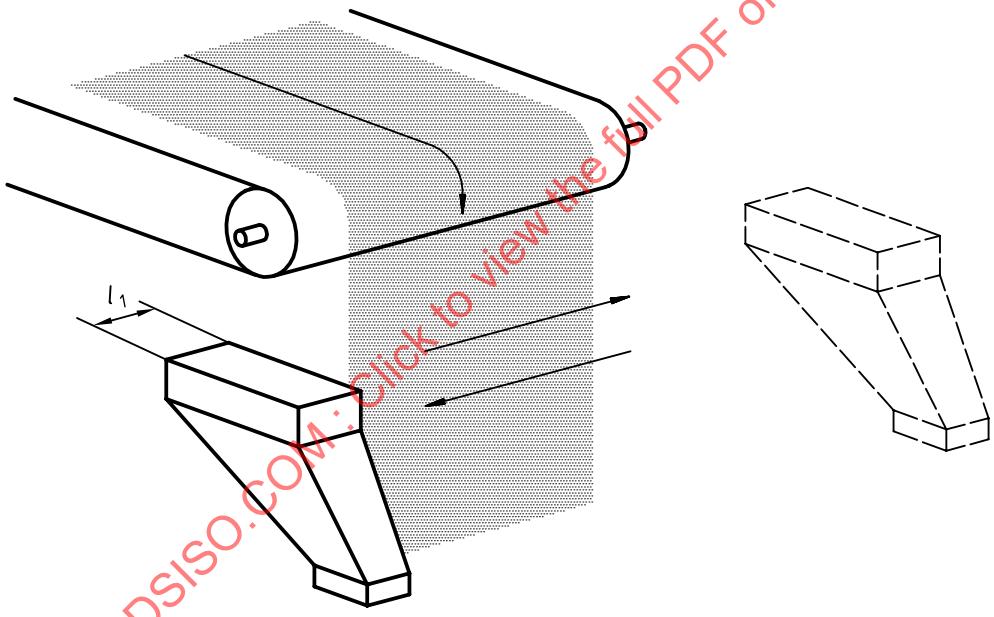
7.5.1 Location

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.

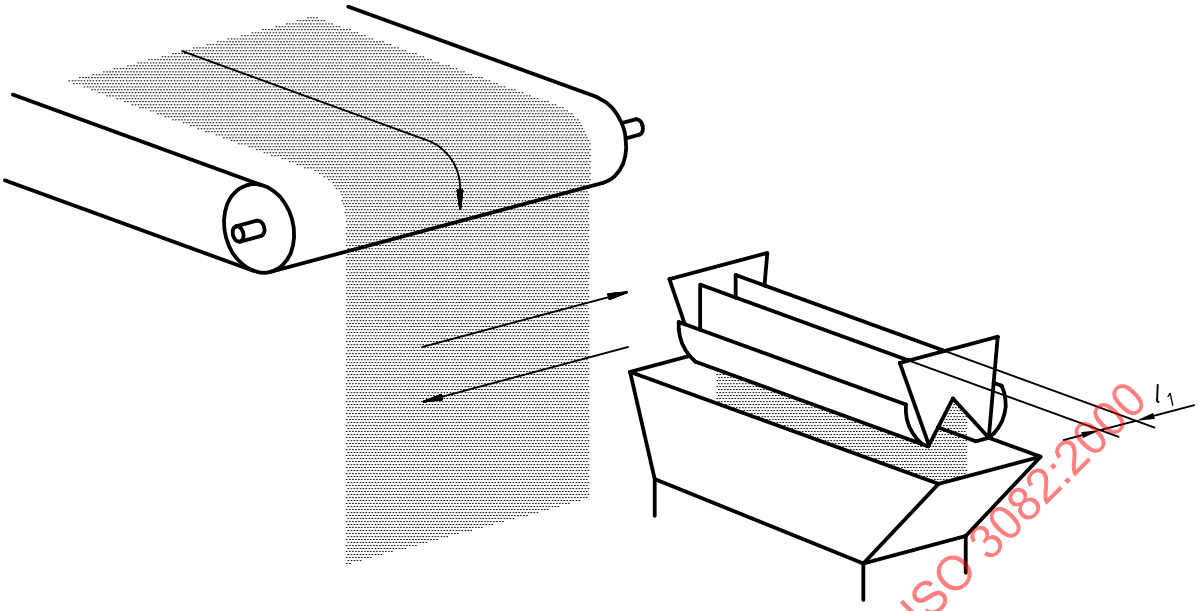
7.5.2 Types of primary sampler

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, travelling 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).

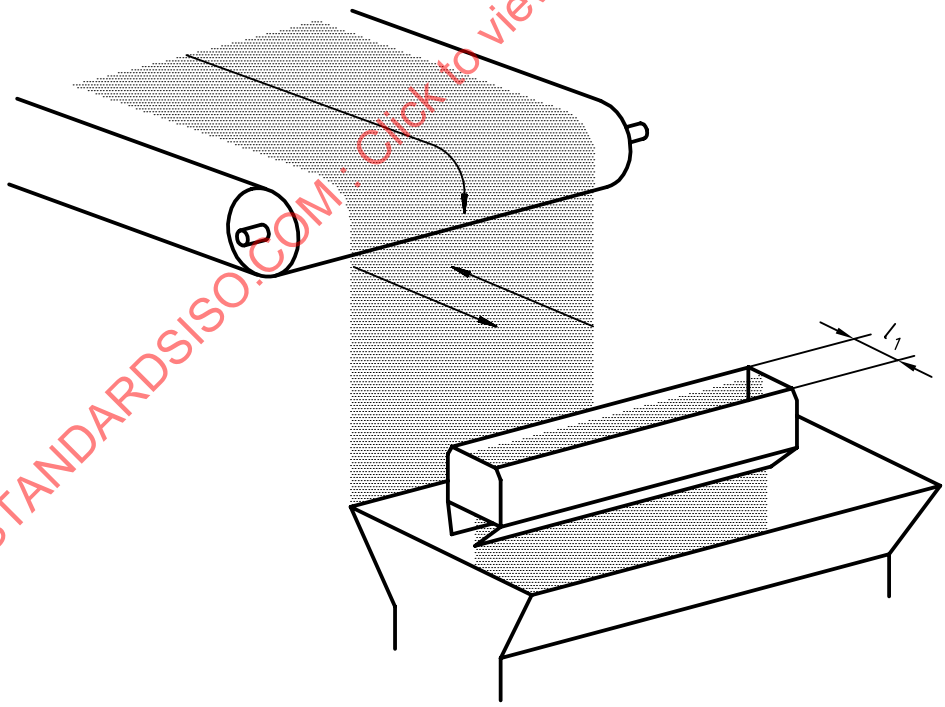
Examples of mechanical cutter-type samplers are shown diagrammatically in Figure 1. An example of a manual sample cutter is shown in Figure 2.



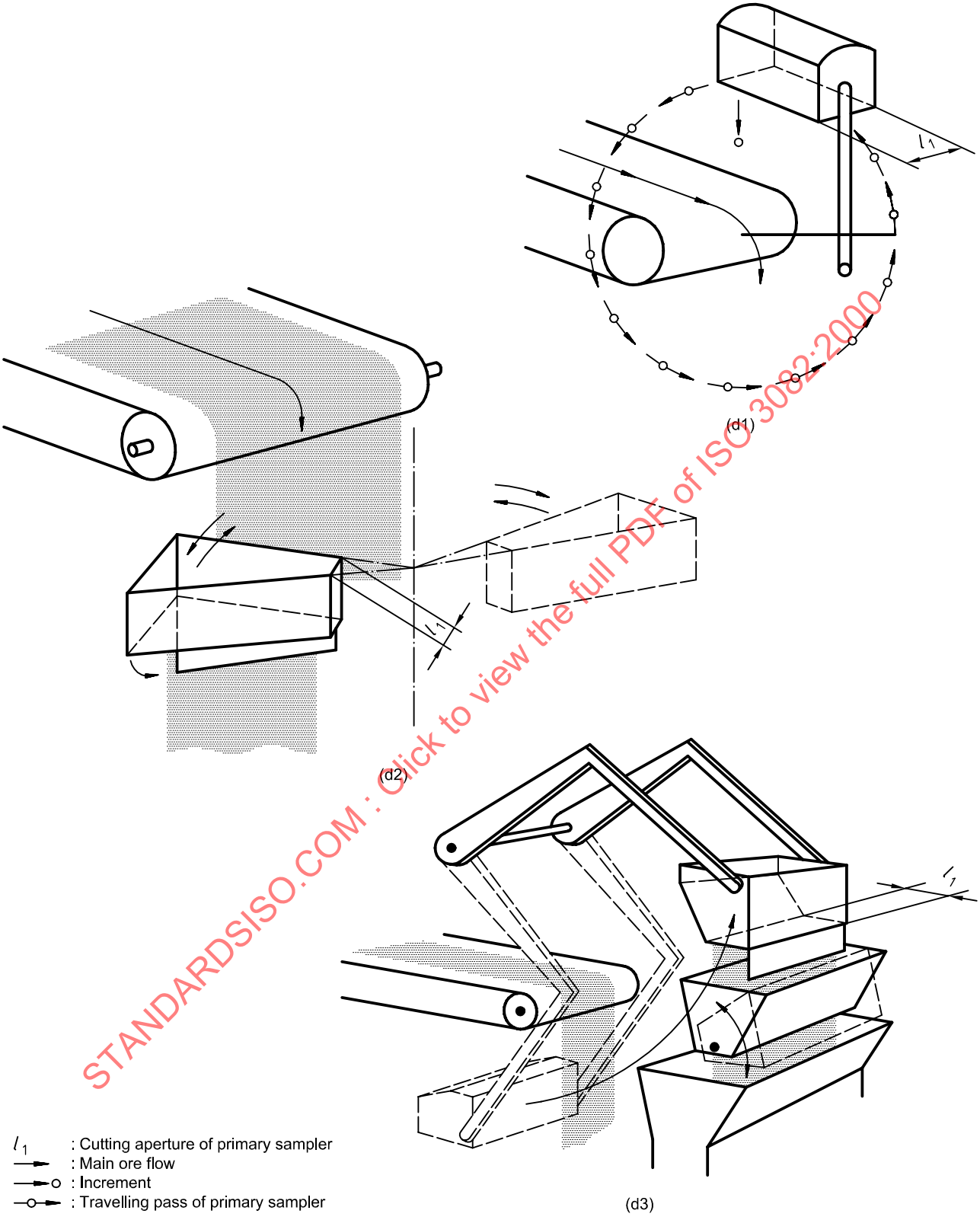
a) cutter-chute type



b) cutter bucket type (i)



c) cutter bucket type (ii)



d) swing arm types

Figure 1 — Examples of cutter-type samplers

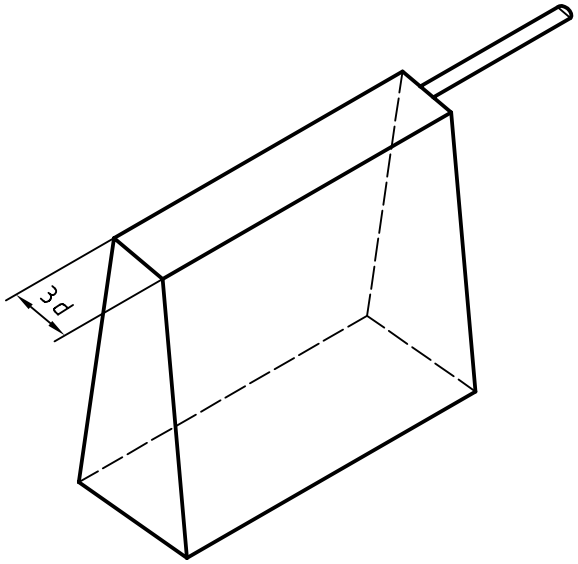


Figure 2 — Example of a manual sample cutter

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.

7.5.4 Cutter aperture of primary sampler

The cutting aperture of the primary sampler (dimension l_1 in Figure 1) 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 sampler 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. Cutter speeds that are too high 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;

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

Experimental work undertaken by Gy[2] 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 the following equation, subject to a maximum of 1,5 m/s:

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

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 On-line 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 mm or −100 mm 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 there will not be any oversize material remaining.

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)];

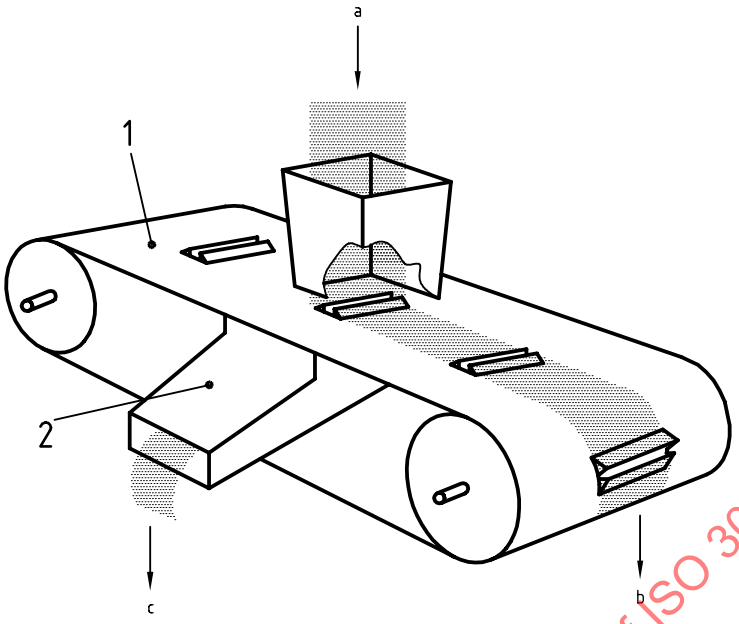
e) rotary plate divider [see Figure 3 d)];

f) rotary cutter-chute divider [see Figure 3 e)].

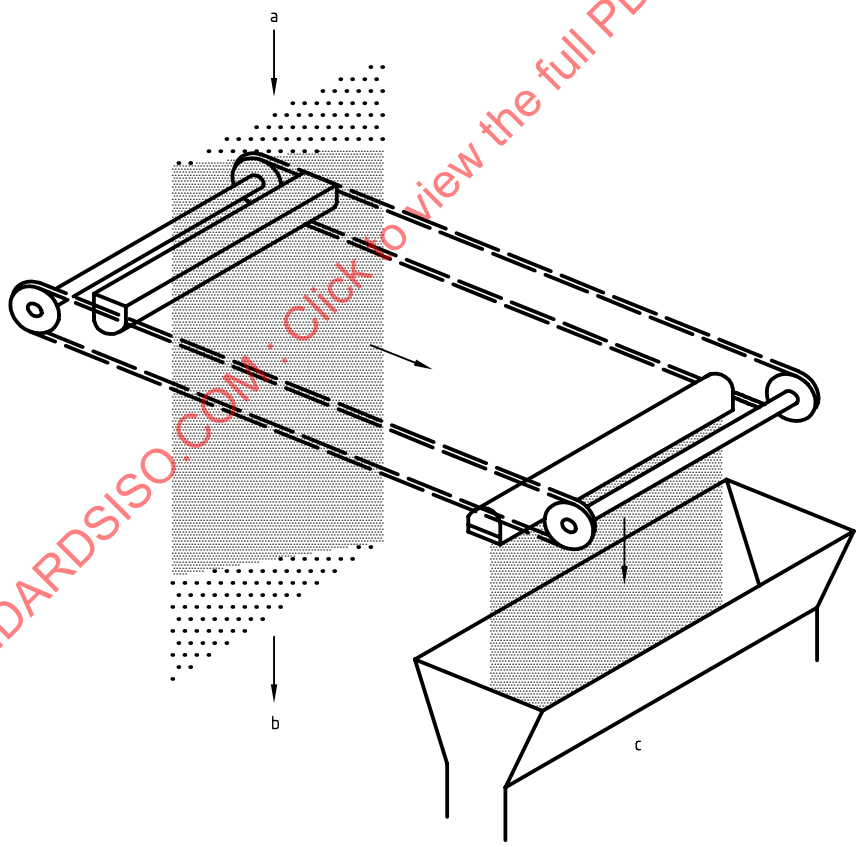
To avoid bias, the divider shall have a random start. The operation of the cutter should be interlocked with the operation of the feeder via a random timer. The time range of operation of the random selector in the timer shall be adjusted to equal the computed cutting interval, so that there is equal probability of the first cut being taken at any 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, in order 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).



a) slotted belt type divider

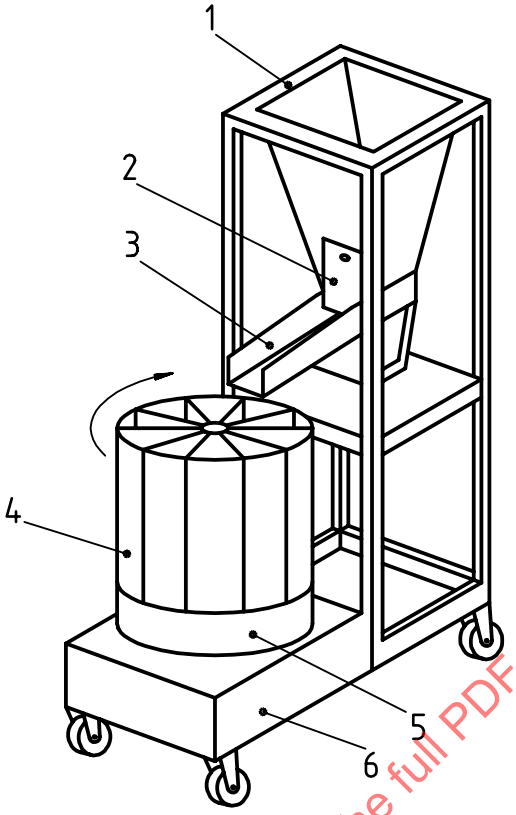


b) bucket type divider

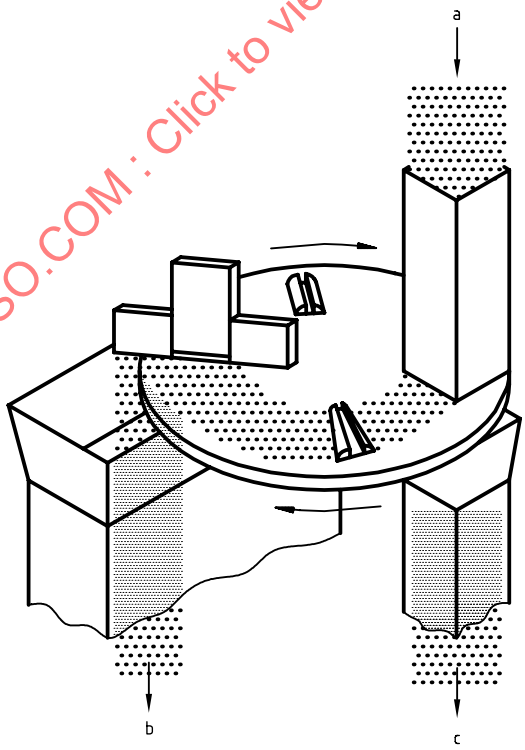
Key

1 Slotted belt
2 Inclined chute

a Feed.
b Reject.
c Divided sample.



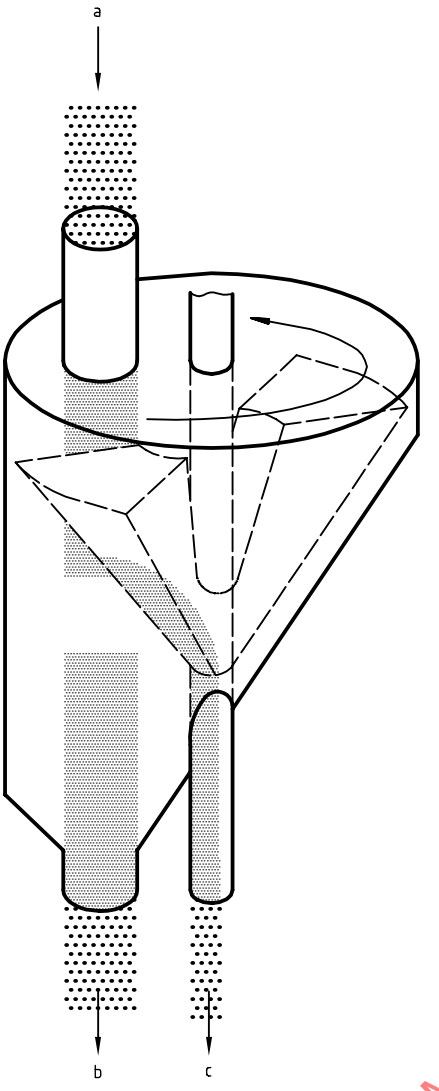
c) rotary sample divider



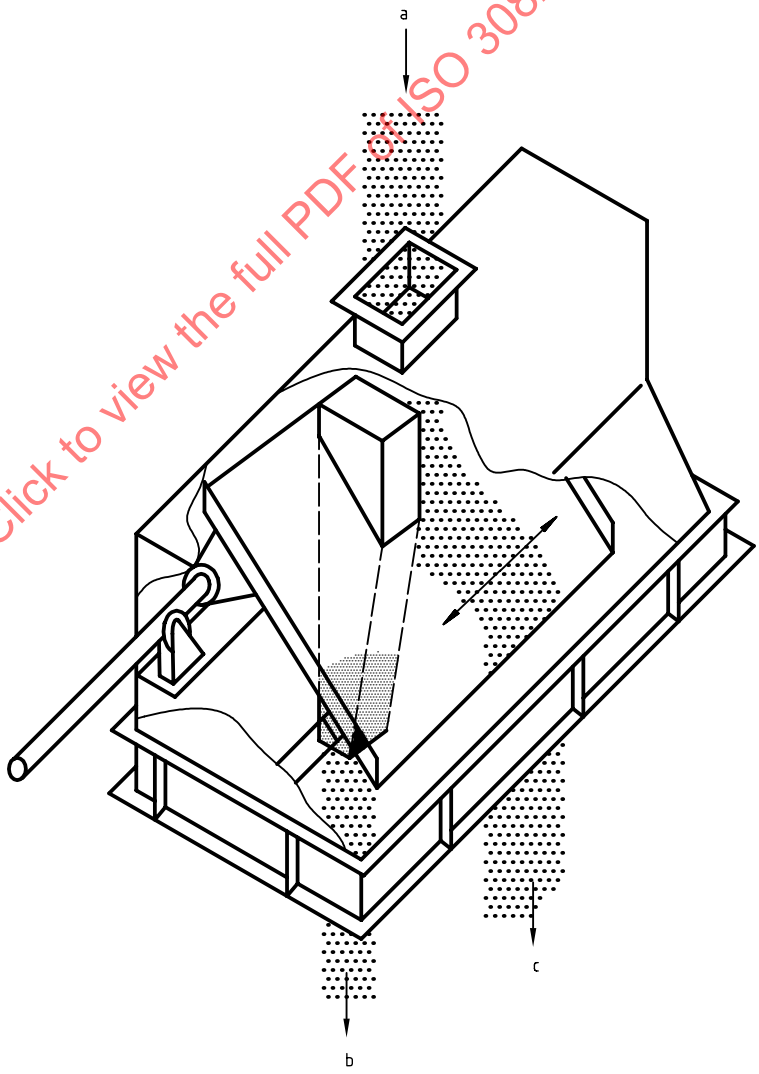
d) rotary plate type divider

Key

1	Feed hopper	3	Vibratory feeder	5	Turntable
2	Slide gate	4	Removable canisters	6	Drive (enclosed)
a	Feed.	b	Reject.	c	Divided sample.



(e1)



(e2)

e) rotary cutter-chute type dividers

a Feed.
b Reject.
c Divided sample.

Figure 3 — Examples of mechanical dividers

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 quality 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 needed. 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 shall be readily accessible to facilitate inspection, thorough cleaning, repairs or check experiments.

Upon completion of sampling a lot, the major units of the installation shall 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 shall be passed through the entire installation to remove any possible contaminants.

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:

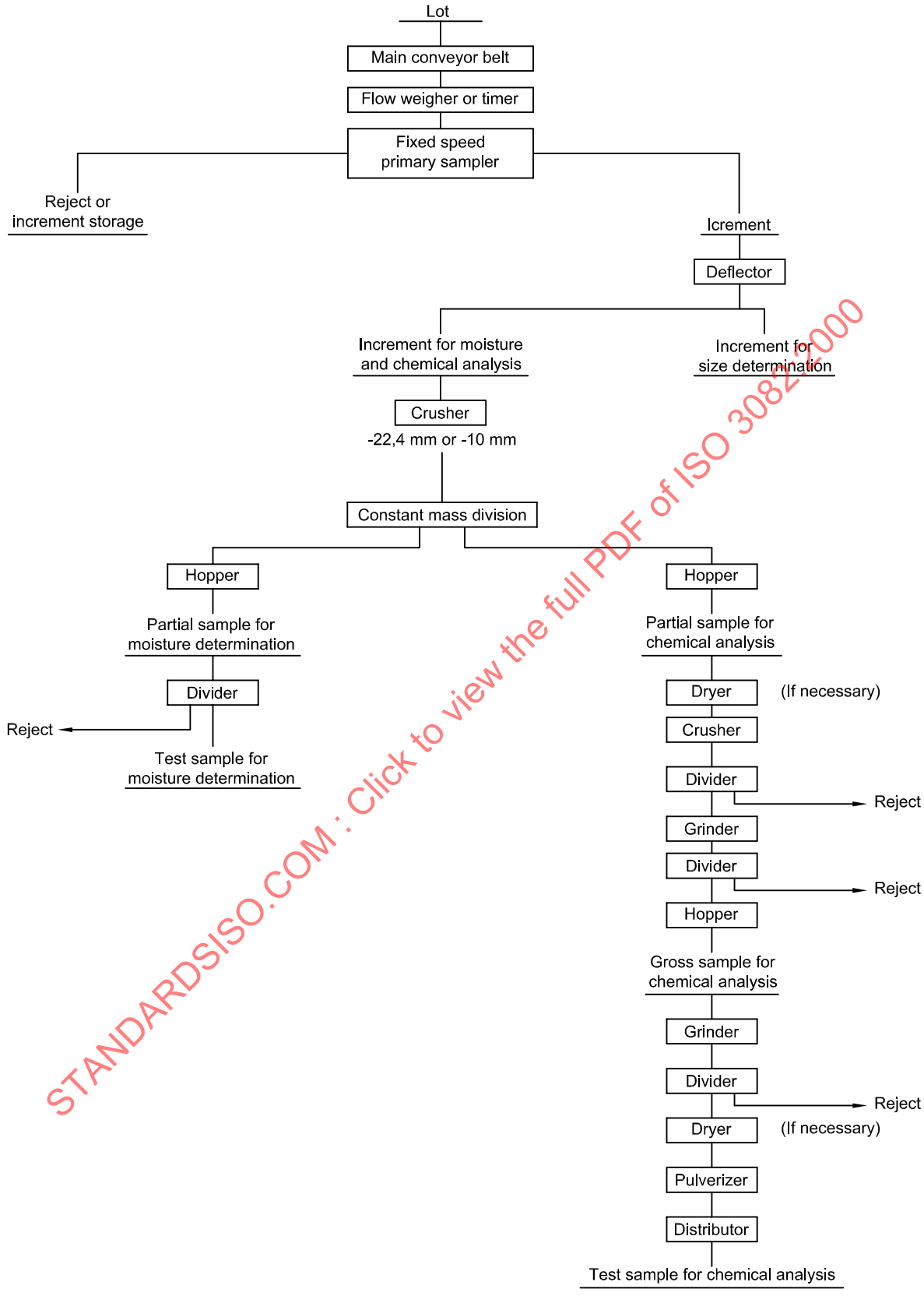
a) mass basis sampling;

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, 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 be 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 wagons

8.2.1 General

In-situ sampling of fine ore from wagons is permitted using a spear sampler or an auger, but only if the sampling device penetrates to the full depth of the fine ore at the point selected for sampling and the full column of fine ore is extracted. However, a more reliable procedure is to sample from a conveyor belt during transfer of the fine ore to or from the wagon. Sampling of coarse ore from 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.

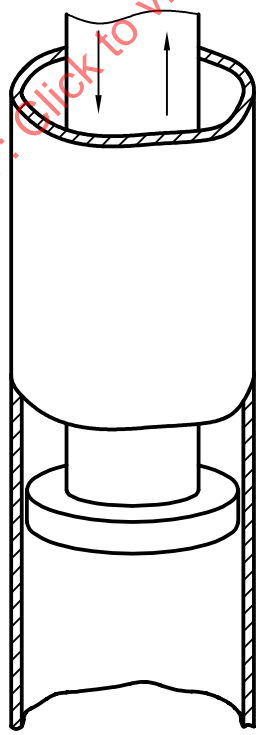


Figure 5 — Example of a spear sampler for sampling fine ores

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 wagon constituting the lot is given by:

$$n_W \geqslant \frac{n_1}{N_T} \quad (18)$$

where

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

N_T is the number of wagons constituting the lot.

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

8.2.4 Method of sampling

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

NOTE Care needs to 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 time or mass 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;

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 samples 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.

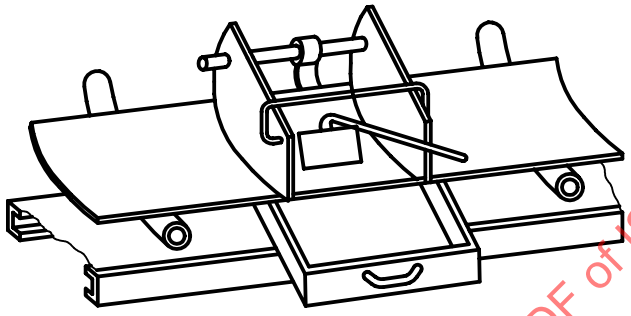


Figure 6 — Example of a sampling frame for use on stopped belts

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 of 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.

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

Sample preparation 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.

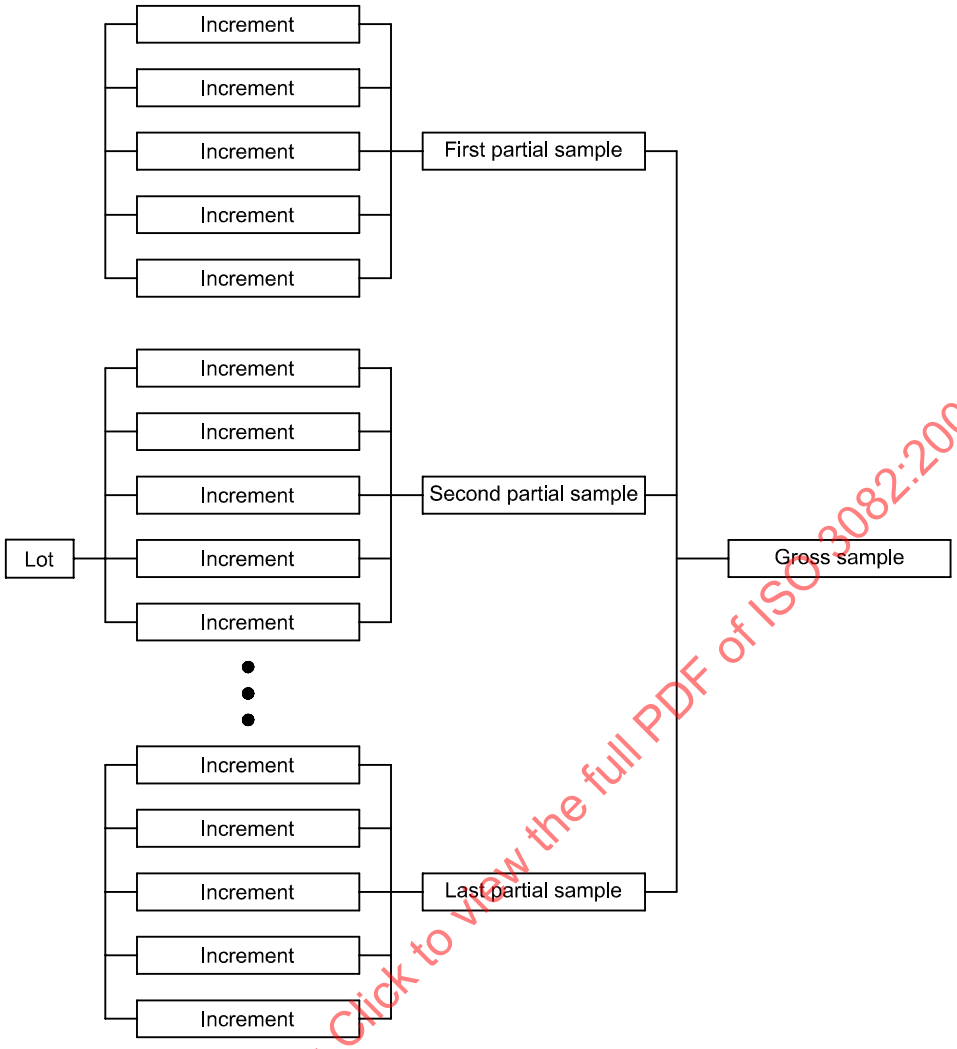


Figure 7 — Example of a sample preparation scheme showing constitution of partial samples from increments and a gross sample from partial samples

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.

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

Mixing the sample will make it more homogeneous and consequently the errors in sample division will be reduced. 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.

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;

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

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

10.1.5 Sample division

10.1.5.1 General

Sample 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.

10.1.5.2 Method of division

One or more of the following methods of sample 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 10.2.1 and 10.2.2.

10.1.5.4 Types of divider

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

10.1.6 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 in order to obtain test samples for moisture determination, size determination and chemical analysis.

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. Further, in some cases, according to 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 increment 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 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 10.2.2.1 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

For very large lots it is recommended that the lot be divided into the number of parts shown in Table 4 and a separate moisture sample prepared for each part. This will ensure that each moisture sample is constituted over a short period of time, thereby minimizing moisture evaporation from the sample. This will minimize 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 eight hour periods. A moisture partial sample shall be constituted for each part and a moisture determination carried out. The division into parts may 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.

Alternatively, if the moisture sample containers and the storage conditions prevent a change in the moisture content of the moisture samples, a moisture gross sample may be prepared for the whole lot.

Table 4 — Minimum number of parts per lot for moisture determination

Mass of lot (t) >	Mass of lot (t) ⩽	Minimum number of parts per lot	Number of test portions per partial sample	Number of tests
270 000		15	1	15
70 000	270 000	10	1	10
30 000	70 000	5	2	10
15 000	30 000	4	2	8
	15 000	2	4	8

Partial samples or a gross sample for moisture determination shall be constituted by the procedure specified in 10.2.1 or 10.2.2.

NOTE The determination of the moisture content of individual increments is totally acceptable for both large and small lots.

10.3 Mechanical methods of division

10.3.1 Mechanical increment division

10.3.1.1 General

The size sample, moisture sample and sample for chemical analysis 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 variable feed rate of sample and variable cutter speed may 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.

10.3.1.3 Number of increments (cuts)

The number of cuts, n_i, for division of increments, partial samples and gross samples shall 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 the following equation:

$$n_i = \left(\frac{2\sigma_{Wi}}{\beta_{Si}} \right)^2 \qquad (19)$$

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:

a) division of a gross sample

— a minimum of 20

b) division of individual partial samples

— for constant mass division, minimum of 10;

— for proportional division, a minimum of 10 for the average mass of partial sample

c) 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.

10.3.2 Other mechanical division methods

10.3.2.1 General

The size sample, moisture sample and sample for chemical analysis may be divided using mechanical dividers other than cutter-type dividers, e.g., a mechanically charged riffle, in accordance with the following procedures and division limits.

10.3.2.2 Division of size sample

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 the tables shall be modified in accordance with equation (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.

Table 5 — Examples of minimum mass of divided gross sample for size determination using other mechanical division methods (e.g. a mechanically charged riffle divider)

Type of iron ore		–200 mm ore		–50 mm 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		70		5	
Mass of lot (t)		Minimum mass of divided gross sample, m_3 and sample preparation and measurement precision, 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} (%)	m_3 (kg)	β_{PM} (%)
270 000		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

10.3.2.2.1 Division of gross sample

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

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

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 the following equation based on the binomial rule:

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

where

m_4 is the revised minimum mass of the divided gross sample;

m_3 is the minimum mass 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.

E.g., for a lot of 40 000 t of –200 mm ore, if the percentage of –10 mm fraction is about 50 %, the minimum mass of the divided gross sample shall be revised as follows:

$$m_4 = 750 \times \frac{50(100 - 50)}{20(100 - 20)} \approx 1\,175 \text{ kg} \quad (21)$$

10.3.2.2.2 Division of increment or partial sample

When increments or partial samples are divided, the division shall be carried out ensuring that the mass of the gross sample for the lot obtained by combining divided increments or partial samples, shall be not less than the minimum specified in Table 5.

10.3.2.3 Division of moisture and chemical analysis sample

10.3.2.3.1 Division of gross sample

When a gross sample is divided, the minimum mass of the divided sample m_S, in kilograms, is given by equation 22[2)]:

$$m_S = \frac{0{,}000\,32 d^{\,2,5}}{\sigma_D^2} \quad (22)$$

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 equation (22) 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 10.7).

Examples of minimum divided gross sample masses given by equation (22) for division standard deviations of 0,1 % Fe and 0,05 % Fe and typical nominal top sizes are given in Table 6. The standard deviation of division σ_D applies to each sample division stage, and the variances are additive for the sample division scheme selected.

2) ISO/TC 102 Technical Committee Report No. 9, *Results of testwork on sample division for iron ores*, 1995.

Table 6 — 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
0,500	0,5	0,5
0,250	0,5	0,5

10.3.2.3.2 Division of individual increments or partial samples

When increments or partial samples are divided, the division shall be carried out ensuring that the mass of the gross sample for the lot obtained by combining divided increments or partial samples, shall be not less than the minimum gross sample mass given by equation (22) or Table 6. The standard deviation of division σ_D applies to each sample division stage, and the variances are additive for the sample division scheme selected.

10.4 Manual methods of division

10.4.1 General

Manual division can only be applied to ores of less than 40 mm nominal top size.

10.4.2 Manual increment division

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 7. However, it 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.

10.4.2.2 Mass of increment

The mass of each increment shall be as specified in Table 7.

10.4.2.3 Number of increments

The number of increments for manual increment division shall be as specified in Table 8.

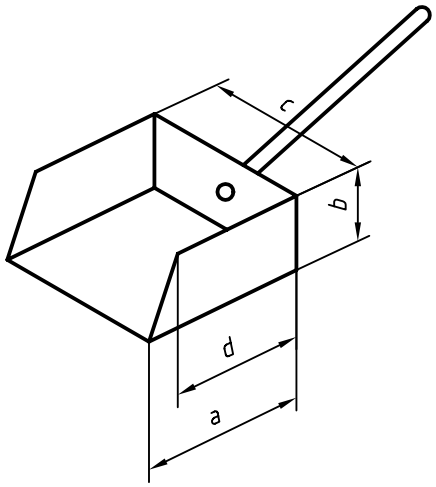


Figure 8 — Example of an increment scoop

Table 7 — 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)
>	⩽			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
	1	10	1D	25	20	25	20	0,03

Table 8 — Number of increments for manual increment division

Sample	Number of manual increments
Gross sample	20
Partial sample	12
Primary increment	4

10.4.2.4 Procedure

The manual increment division method 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 7;

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 8;

c) select an appropriate scoop from Table 7, 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 division of a gross sample by the manual increment division method.

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

10.4.3 Manual riffle division method

10.4.3.1 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 or sized ores.

10.4.3.2 Selection of riffle divider

An appropriate riffle divider specified in Table 9 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.

10.4.3.3 Procedure

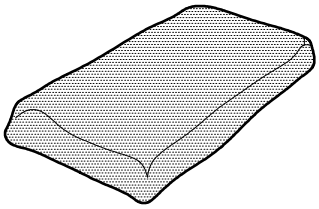
After mixing, place the sample to be divided in a container and divide it into two parts by dropping the sample uniformly with a light shaking of the container into the middle of the riffles (at right angles to the riffle). One of the two divided samples should be selected at random in order to avoid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

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

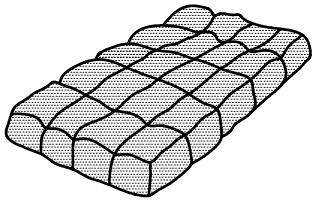
10.4.3.4 Division limit for moisture and chemical analysis sample

10.4.3.4.1 Gross sample

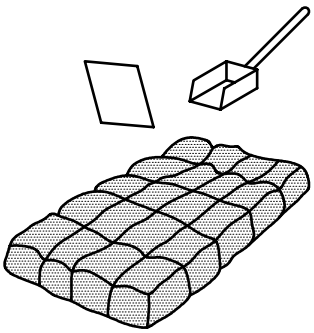
When the gross sample is divided, the division shall be carried out in accordance with equation (22) or Table 6. The gross sample shall not be divided further than the mass specified for the nominal top size of the ore. The standard deviation of division σ_D applies to each sample division stage, and the variances are additive for the sample division scheme selected.



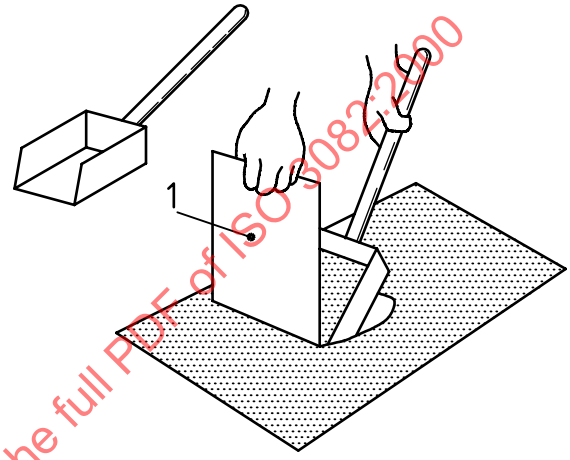
a) Spread the crushed gross sample into a rectangle with a thickness as specified in Table 7.



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 scoopsful of sample into a divided sample.



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

Key

1 Bump plate

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

Table 9 — Nominal top size of sample and size of riffle divider

Nominal top size (mm)		Riffle divider number	Riffle opening (mm)
>	⩽		
31,5	45,0	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

10.4.3.4.2 Increment or partial sample

When increments or partial samples are divided, the division shall be carried out ensuring that the mass of the gross sample for the lot obtained by combining divided increments or partial samples shall be not less than the minimum gross sample mass given by equation (22) or Table 6. The standard deviation of division σ_D applies to each sample division stage, and the variances are additive for the sample division scheme selected.

10.4.3.5 Division limit for size sample

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 the tables shall be modified in accordance with equation (20).

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

10.4.3.5.1 Gross sample

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

10.4.3.5.2 Increment or partial sample

When increments or partial samples are divided, the division shall be carried out ensuring that the mass of the gross sample for the lot obtained by combining divided increments or partial samples shall be not less than the minimum specified in Table 5.

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

10.5 Preparation of sample 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 4701:1999.

10.6 Preparation of sample 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 pre-dried in accordance with annex A of ISO 3087:1998. In time basis sampling, the test sample shall be taken from each partial sample or the gross sample to ensure that the specified mass is obtained.

The moisture sample shall be kept in an airtight, non-absorbent container to avoid any change in moisture prior to determination of moisture content in accordance with ISO 3087.

If necessary, the moisture sample shall be crushed to either –31,5 mm, –22,4 mm or –10 mm, as specified in ISO 3087. When it is difficult to conduct crushing and dividing owing to a sample being adhesive or excessively wet, the sample may be pre-dried in accordance with annex A of ISO 3087:1998. 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, 5 kg minimum for –22,4 m or 1 kg minimum for –10 mm particle size, one of the methods of division specified in 10.1.5.2 shall be used. As specified in ISO 3087, the minimum mass of divided gross sample given in Table 6 and by equation (22) no longer applies.

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 1 Instead of preparing one test portion of 10 kg minimum at –31,5 mm, two test portions of 5 kg minimum each may be prepared by dividing the test sample of 10 kg minimum into two parts.

NOTE 2 A check is recommended to determine whether the –10 mm test portion is biased with respect to the –22,4 mm or –31,5 mm test sample.

NOTE 3 It is recommended that any moisture test portion be prepared by manual increment division specified in 10.4.2 to minimize moisture evaporation. A scoop number, one or two ranks smaller than that specified in Table 7, 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 portion thus obtained should 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 sample shall be packed tightly in a moisture-proof container and kept in an environment which 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 10.

Table 10 — 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 3 to 7 ≥ 8	4 2 minimum 1 minimum
From increment	–	1 minimum

10.7 Preparation of test sample for chemical analysis

10.7.1 Mass and particle size

The particle size of the test sample for chemical analysis shall be either –100 μm or –160 μm. The preferred method is to prepare a test sample for chemical analysis of 50 g minimum and –100 μm particle size from the divided gross sample of –250 μm particle size. However, if an appropriate grinder is utilized, a test sample for chemical analysis of either –100 μm or –160 μm particle size can be prepared directly from samples coarser than –250 μm particle size, provided the sample masses conform to Table 6.

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 in particle size and 100 g minimum in mass.

The preparation of a sample for chemical analysis can be carried out in one of three ways as shown in Figure 10.

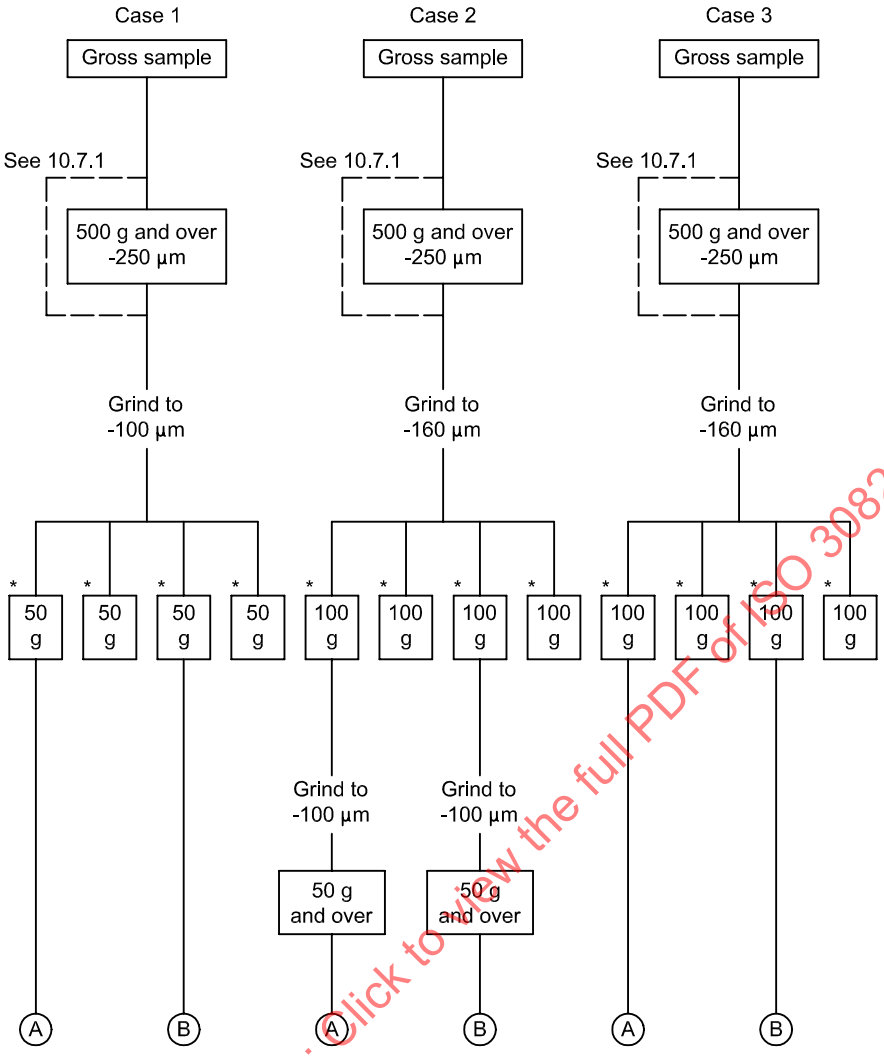
NOTE During preparation of ores that contain significant amounts of combined water and/or oxidizable compounds, special precautions should be taken to ensure that the grinding process does not generate excessive heat which could significantly change the chemical composition of the ore. Special precautions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

a) reducing the grinding time by grinding smaller charges;

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

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

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



* Sealed samples

Figure 10 — Sample preparation for chemical analysis

10.7.2 Preparation to –250 µm

If each increment, each partial sample or the gross sample is ground to –250 µm in particle size, it shall be carried out by repeating crushing and division according to 10.3 and 10.4. When the division is conducted on an individual increment or partial sample 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 in particle size shall be ground to –160 µm or –100 µm in particle size.

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

10.7.3 Final preparation

10.7.3.1 Case 1

If a –250 µm sample is prepared, it shall be ground to –100 µm in particle size. A set of not less than four test samples, each of 50 g minimum, shall be prepared from the –100 µm sample by an appropriate division method.

10.7.3.2 Case 2

If a –250 μm sample is prepared, it shall be ground to –160 μm in particle size. A set of not less than four test samples, each of 100 g minimum, shall be prepared from the –160 μm sample by an appropriate division method. The test sample for chemical analysis sent to the laboratory shall be ground to –100 μm in particle size.

10.7.3.3 Case 3

If a –250 μm sample is prepared, it shall be ground to –160 μm in particle size. A set of not less than four test samples, each of 100 g minimum, shall be prepared from the –160 μm 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.

10.7.4 Grinding to –100 μm or –160 μm

10.7.4.1 General

When the –250 μm sample is ground to –100 μm or –160 μm, the procedure described below shall be used.

10.7.4.2 Type of grinder

Several types of grinder may be used to grind the sample for chemical analysis from –250 μm to –160 μm or –100 μm, such as a top grinder, a disc grinder, a pot mill, a hammer mill or a vibrating mill.

10.7.4.3 Selection of material of construction of grinder

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

NOTE 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.

10.7.4.4 Dry grinding

The whole of the –250 μm sample for chemical analysis shall be ground at one time to –100 μm or –160 μm 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, they shall be mixed thoroughly in a suitable mixer.

NOTE 1 Samples for finer grinding should 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.

NOTE 2 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.

10.7.4.5 Wet grinding

When the sample for chemical analysis is cohesive in the vibrating mill during finer grinding, and when 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.

10.7.5 Distribution of samples for chemical analysis

A set of not less than four test samples for chemical analysis shall be prepared in accordance with 10.7.3. The test samples to be distributed shall be placed in suitable containers, sealed and clearly marked in accordance with clause 11.

One sample shall be provided for the distributor, one for the purchaser, one for the arbitrator and, if required, one is to be held in reserve. The reserve sample shall be retained for 6 months.

10.8 Example of sample preparation process

An example of the sample preparation process for moisture samples and samples for chemical analysis is shown in Figure 11.

NOTE The flow chart shown in Figure 11 provides an example of sample preparation of lump ore, where a partial sample comprises three increments and several partial samples compose a gross sample.

11 Packing and marking of sample

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.

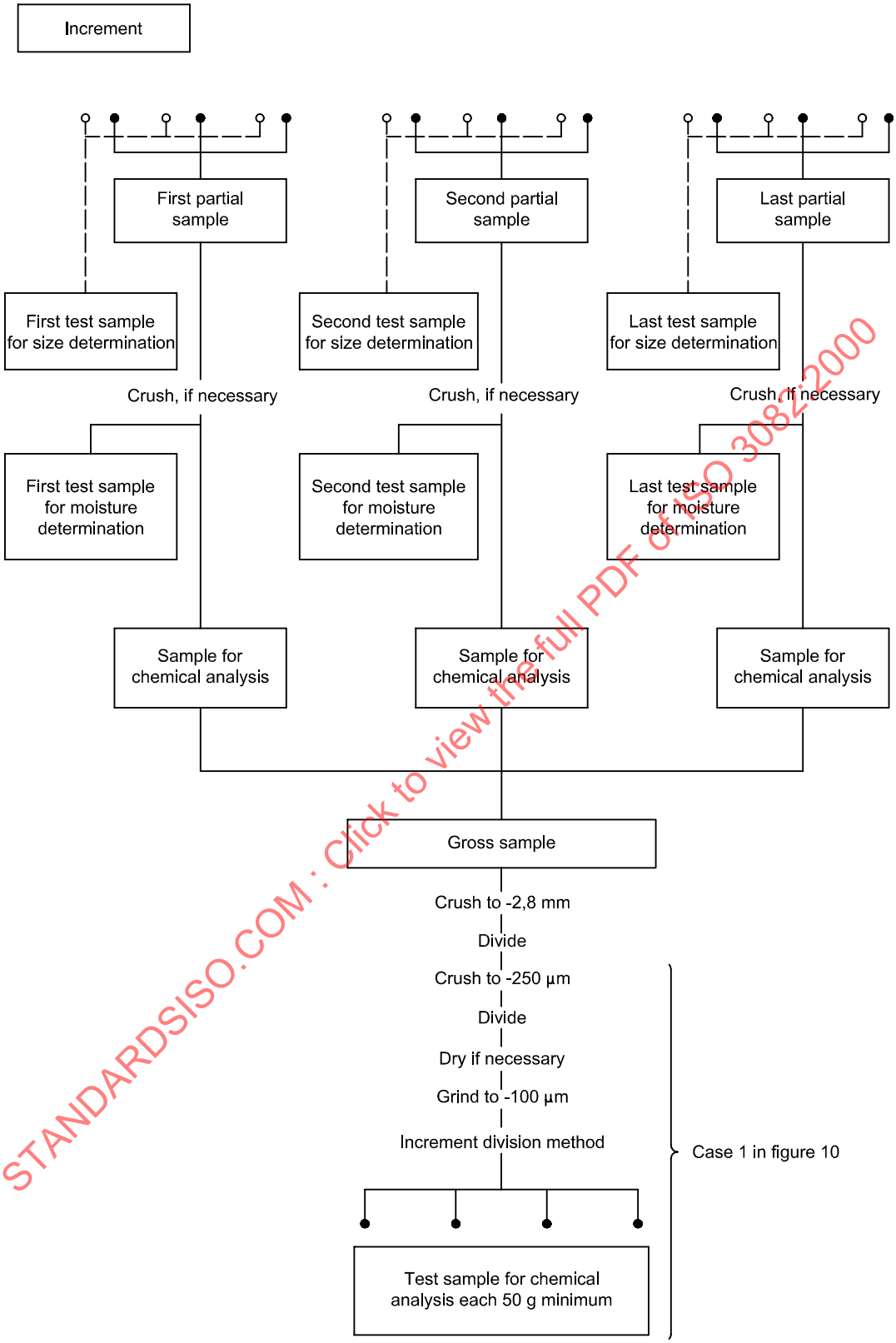


Figure 11 — Example of a sample preparation scheme

Annex A
(informative)

Checklist for mechanical sampling systems

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			
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/unloading		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			
f) Cutter aperture		3d minimum	
g) Condition of cutter lips		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		yes	
l) Cutter speed		0,6 m/s max.	5 %
m) Uniform cutter speed		yes	5 %
n) Increment mass			C_V < 20 %
o) Contamination or loss of sample		not significant	
p) Moisture loss		not significant	
q) Cutter parks out of ore stream		yes	
r) Mass or time basis sampling			
s) Interval between cuts			
t) Number of cuts per lot			

4 Primary sample feeder

	Observation	Specification (if applicable)	Tolerance (if applicable)
a) Type			
b) Feed rate			
c) Contamination or loss of sample		not significant	
d) Moisture loss		not significant	
e) Blockages		not significant	
f) Crusher			

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			
f) Cutter aperture		$3d$ minimum	
g) Condition of cutter lips		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		yes	
l) Cutter speed		0,6 m/s max.	5 %
m) Uniform cutter speed		yes	5 %
n) Increment mass			C_V < 20 %

o)	Contamination or loss of sample		not significant	
p)	Moisture loss		not significant	
q)	Cutter parks out of ore stream		yes	
r)	Interval between cuts			
s)	Number of cuts per primary increment			

6 Secondary sample feeder

		Observation	Specification (if applicable)	Tolerance (if applicable)
a)	Type			
b)	Feed rate			
c)	Contamination or loss of sample		not significant	
d)	Moisture loss		not significant	
e)	Blockages		not significant	
f)	Crusher			

7 Tertiary 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			
f)	Cutter aperture		3d minimum	
g)	Condition of cutter lips		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		yes	
l)	Cutter speed		0,6 m/s max	± 5 %
m)	Uniform cutter speed		yes	± 5 %
n)	Increment mass			C_V < 20 %
o)	Contamination or loss of sample		not significant	
p)	Moisture loss		not significant	
q)	Cutter parks out of ore stream		yes	
r)	Interval between cuts			
s)	Number of cuts per secondary increment			

8 Laboratory sample

		Observation	**Specification** (if applicable)	**Tolerance** (if applicable)
a)	Drop height to container		minimize	
b)	Blockages		not significant	
c)	Enclosed container		yes	
d)	Nominal top size			
e)	Sample mass			
f)	Moisture loss		no significant loss	

9 General comments

Annex B
(normative)

Equation for number of increments

B.1 Notation

n_1 is the minimum number of primary increments to be taken from a lot to attain the desired sampling precision

β is the precision at the 95 % probability level (or two-sigma probability level) and is twice the standard deviation

β_P is the 95 % probability precision of sample preparation

β_M is the 95 % probability precision of measurement

β_S is the 95 % probability precision of sampling

β_{SPM} is the overall precision, i.e., the aggregate 95 % probability precision of sampling, sample preparation and measurement

σ is the precision in terms of the standard deviation

σ_P is the precision of sample preparation in terms of the standard deviation

σ_M is the precision of measurement in terms of the standard deviation

σ_S is the precision of sampling in terms of the standard deviation

σ_W is the standard deviation of a quality characteristic within strata (or parts)

B.2 Derivation

The number of primary increments, n_1, to be taken from a single lot specified in Table 3 is derived from equation (B.7), the theoretical basis of which is stratified sampling.

From the definition of overall precision at a 95 % probability level, the relationship may be expressed mathematically as follows:

$$\beta_{SPM} = 2\sigma_{SPM} \quad \text{(B.1)}$$

or

$$\sigma_{SPM} = \frac{\beta_{SPM}}{2} \quad \text{(B.2)}$$

where

$$\sigma_{SPM} = \sqrt{\left(\sigma_S^2 + \sigma_P^2 + \sigma_M^2\right)} \quad \text{(B.3)}$$