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Chromium ores — Increment sampling

Minerais de chrome — Échantillonnage par prélèvements



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6153 was prepared by Technical Committee ISO/TC 65, *Manganese and chromium ores*.

Annex A forms an integral part of this International Standard.

Chromium ores — Increment sampling

1 Scope

This International Standard specifies the methods for taking samples of chromium ores to be used at the places of dispatch and acceptance of ores for determining the chemical composition and moisture content of a consignment.

The methods are applicable to both the manual and mechanical sampling of all chromium ores, whether natural or processed.

Details of the hammer and shovel method for sampling ores containing lumps of +100 mm in size are given in annex A.

2 Normative references

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

ISO 565 : 1983, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

ISO 6154 : 1989, *Chromium ores — Preparation of samples.*

ISO 8541 : 1986, *Manganese and chromium ores — Experimental methods for checking the bias of sampling and sample preparation.*

ISO 8542 : 1986, *Manganese and chromium ores — Experimental methods for evaluation of quality variation and methods for checking the precision of sampling.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 lot : A definite quantity of an ore, processed or produced under conditions which are presumed uniform.

3.2 consignment : A quantity of an ore delivered at one time. The consignment may consist of one or more lots or parts of lots.

3.3 increment :

(1) A quantity of an ore taken by a sampling device at one time from a consignment.

(2) A quantity taken by the increment division method.

3.4 sub-sample :

(1) A quantity of an ore consisting of two or more increments taken from a consignment.

(2) An aggregation of two or more increments each of which has been optionally crushed and/or optionally divided as necessary.

3.5 gross sample :

- (1) The quantity of an ore consisting of all the increments taken from a consignment.
- (2) An aggregation of all the increments or all the sub-samples each of which has been optionally crushed and/or optionally divided as necessary.

3.6 test sample : Any sample, for the determination of moisture content or chemical composition, which is prepared from each increment, each sub-sample, or from the gross sample in accordance with the specified method for that type of sample.

3.7 nominal top size : The smallest sieve in the range included in the R 20 series (in table 1 of ISO 565, square hole) such that not more than 5 % of the ore is retained.

3.8 increment sampling : The process in sampling whereby a sample is obtained by combining a number of increments from a consignment and meant for representing the consignment.

3.9 manual sampling : The sampling by human effort with sampling devices, including mechanically assisted devices.

3.10 stratified sampling : For a consignment which can be divided into strata, sampling carried out in such a way that specified proportions of the sample are drawn from different strata.

NOTE — The stratum is a part of a consignment which is derived by division of the consignment according to specific criteria.

3.11 periodic systematic sampling : The sampling in which increments are taken from a consignment at regular intervals. When the mass interval is adopted, it is called "periodic systematic sampling on mass basis", and when the time interval is adopted, it is called "periodic systematic sampling on time basis".

3.12 two-stage sampling : The sampling by which primary sampling units are selected first from a consignment and then secondary sampling units are taken from those selected primary sampling units. In this International Standard, the method can be applied to sampling from wagons or containers in which a certain number of wagons or containers are selected first as primary sampling units, and then increments are taken from those selected wagons or containers as secondary sampling units.

4 General rules

The rules given below are general and obligatory for the parties concerned.

- a) Sampling should be carried out by qualified samplers or mechanical sampling devices authorized by the seller and/or purchaser.

b) Sampling shall preferably be carried out during handling immediately before or immediately after weighing.

c) Sampling shall be carried out by the systematic method with a random start. The sampling of ores from wagons shall be conducted by the two-stage method or stratified method.

d) The mass of the increment shall be determined in accordance with the nominal top size of the ore in order to avoid the introduction of bias at the time of collecting the sample.

e) The number of increments to be taken from a consignment shall be determined according to classification of the quality variation of the ore and the required precision of sampling.

f) Throughout the procedure of sampling, the samples shall be protected from any contamination.

g) All sampling methods shall be confirmed to have no bias in accordance with ISO 8541.

h) When the planned number of increments has been taken before the handling has been completed, the taking of increments should continue at the same interval until the handling operation of the consignment has been completed.

i) Sampling shall be carried out in conformity with national safety standards.

5 Methods of sampling in general

5.1 General sampling procedure

The procedure shall be as follows.

- a) Identify the consignment to be sampled.
- b) Ascertain the nominal top size of the consignment.
- c) Determine the mass of increment.
- d) Ascertain the classification of quality variation of the consignment.
- e) Determine the minimum number of increments to be taken from the consignment in the case of systematic and stratified sampling. In the case of two-stage sampling, allocate the wagons or containers to be selected from the consignment and the increments to be taken to the points of the wagons or containers selected.
- f) Determine the interval of taking increments in the case of systematic sampling and stratified sampling or the interval of selecting the wagons or containers on a mass basis.

- g) Determine the point of sampling and the method of taking increments.
- h) The gross sample or sub-sample shall be constituted according to ISO 6154. An example is given in figure 1.

5.2 Sampling precision and overall precision

This International Standard is designed to obtain the sampling precision (β_s) given in table 3. The sampling precision is one component of the overall precision (β_{SDM}), which shall be based on the fact that sample preparation has been undertaken in accordance with ISO 6154 and that measurement has been carried out in accordance with methods given in the appropriate International Standards.

The overall precision defines with 95 % probability the average value of the quality characteristics of a consignment. The sampling precision means that on average, in 95 % of cases the chromium oxide content of the gross sample shall not differ from that of the analysed consignment by more than β_s %.

β_s is a measure of the precision of sampling and is equal to twice the standard deviation of sampling, expressed as an absolute percentage.

β_{SDM} is a measure of the overall precision of sampling, sample division and measurement, and is equal to twice the standard deviation of the overall process of sampling, sample division and measurement, expressed as an absolute percentage.

The sampling precision shall be checked in accordance with ISO 8542.

5.3 Mass of increment

5.3.1 The minimum mass of increment in manual sampling, depending on maximum particle size is specified in table 1.

Table 1 – Mass of increment in manual sampling

Nominal top size mm	Minimum mass of increment kg
150 and over	30
100	15
50,0	5
22,4 (20,0)	2
10,0	0,5
2,8 (3,0)	0,2

The increments shall be taken in such a manner as to ensure that they have an almost uniform mass.

NOTE – "Almost uniform mass" means that the variation in mass shall be less than 20 % in terms of coefficient of variation.

The coefficient of variation (CV) is defined as the ratio of the standard deviation (σ) to the mean value (\bar{x}) of the mass of increments, expressed as a percentage.

$$CV(\%) = \frac{\sigma}{\bar{x}} \times 100 < 20 \% \quad \dots (1)$$

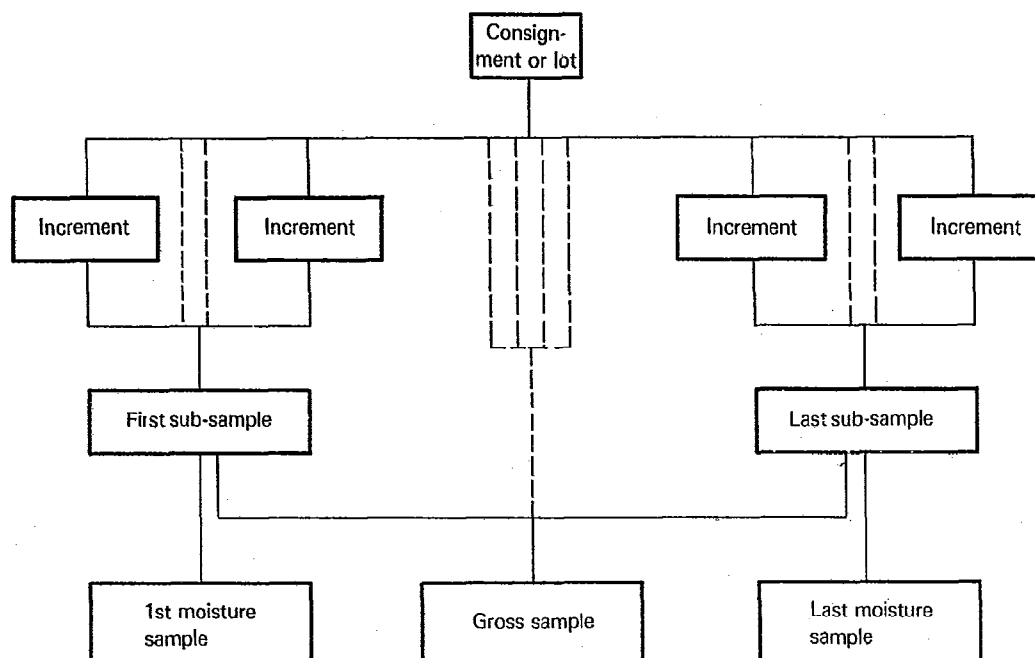


Figure 1 – Sampling plan

5.3.2 The mass of increment taken by means of mechanical samplers from the ore stream is directly proportional to the flow rate of the belt conveyor and to the opening aperture of the sampler, and inversely proportional to the cutter speed of the sampler; it may be calculated from the equation

$$m = \frac{q_m \cdot l}{3,6 \cdot v} \quad \dots (2)$$

where

m is the mass of an increment, in kilograms;

q_m is the flow rate of the belt conveyor, in tonnes per hour;

l is the cutting aperture of the sampler, in metres;

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

5.4 Classification of quality variation

The quality variation is a measure of the heterogeneity of a consignment.

5.4.1 In the case of systematic sampling and stratified sampling the quality variation, σ_w , is the standard deviation of a quality characteristic between increments taken from within the strata of the consignment.

In the case of two-stage sampling, the quality variation is expressed by σ_b and σ_w . σ_b is the quality variation between wagons or containers selected from the consignment in terms of standard deviation. σ_w is the quality variation in terms of standard deviation between increments taken from within wagons or containers selected.

5.4.2 The values of σ_w and σ_b should be estimated for each type or each brand of chromium ore and for each handling system, under normal operating conditions in accordance with ISO 8542, and the chromium ore should be classified with respect to the magnitude of quality variation as specified in table 2.

Table 2 — Classification of quality variation

Quality variation	Standard deviation of chromium oxide standard (Cr ₂ O ₃)
Large	σ_w or $\sigma_b > 1,0$
Small	σ_w or $\sigma_b < 1,0$

5.4.3 Any type and/or brand of chromium ore whose estimated value of quality variation is unknown shall be considered as having "large" quality variation. In this case, the experiment shall be conducted at the earliest possible opportunity in accordance with ISO 8542, and the classification of quality variation shall be determined.

5.5 Number of increments

5.5.1 In the case of systematic sampling under the theory of stratified sampling, the number of increments is calculated from the equation

$$n = \left(\frac{2 \sigma_w}{\beta_s} \right)^2 \quad \dots (3)$$

where

n is the number of increments;

2 is the factor related to two-sigma (approximately 95 %) probability level;

σ_w is the standard deviation within strata, % absolute;

β_s is the two-sigma precision of sampling, % absolute.

Table 3 — Minimum number of increments and sampling precision depending on quality variation (% Cr₂O₃)

Mass of consignment t		Precision of sampling	Number of increments at quality variation	
over	up to and including		large $\sigma_w = 1,5$	small $\sigma_w = 0,7$
30 000	45 000	0,33	85	20
15 000	30 000	0,37	65	15
5 000	15 000	0,39	60	15
2 000	5 000	0,42	50	10
1 000	2 000	0,55	30	7
500	1 000	0,60	25	6
	500	0,65	20	5

NOTE — The number of increments may be increased by agreement between the parties concerned, for example if a greater precision is required.

5.5.2 In the case of two-stage sampling the number of increments shall be calculated according to 7.1.2.3.

5.6 Method of taking increments

Each increment shall be taken at one time by a single motion of a sampling device, but, if it is difficult, it may be taken by several motions of the sampling device from a point selected at random (with equal probability). The latter shall be proven to have no bias with each type of ore before being applied.

6 Equipment

6.1 Manual sampling shall be conducted using the following tools :

- a) shovels (see figure 2 and table 4);
- b) hammer, mass 400 g to 900 g;
- c) probe (see figure 3);
- d) sampling frame.

NOTE — The probe is a piece of pipe 250 mm long attached to a wooden handle. The pipe may be whole or have two slots. In the latter, a locking ring is fitted on the pipe. At 140 mm from the end of the pipe, the angle is welded for knocking the sample out of the probe. The scoop probe may be made from a pipe which is cut into two equal parts. The sharp end, which is inserted into the ore, is cone-shaped and is separated from the main cavity by a partition welded inside the pipe.

6.2 Mechanical sampling shall be conducted using mechanical sampling devices (rotary-arc, bucket, cutter-chute sampler etc.), which meet the following requirements :

- a) a sampling device shall travel at a uniform speed during the course of cutting a complete cross-section of the stream;
- b) the capacity of the sampling device shall be sufficient for taking the complete increment at one time and shall be filled to no more than 2/3 of its volume;

c) the effective opening of the sampling device shall have a minimum dimension of three times the nominal top size of the ore and shall not be less than 10 mm;

d) the sampler shall be designed so as to permit its cleaning and checking.

NOTE — Other sampling devices, including mechanical assisted devices, may be used to take increments. These devices should have a minimum opening equivalent to column (c) in table 4 and, in the case of over 100 mm, at least three times the nominal top size.

The volume of the device in the effective collection area should be sufficient to contain at least twice the minimum mass of increment in table 1.

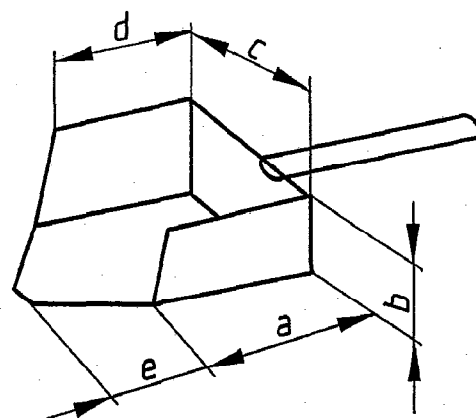


Figure 2 — Increment shovel

Table 4 — Dimensions of increment shovels

Nominal top size, mm		Shovel No.	Dimensions of shovel, mm				
over	up to and including		a	b	c	d	e
50	100	100	300	110	300	220	100
40	50	50	150	75	150	130	65
31,5 (30)	40	40	110	65	110	95	50
22,4 (20)	31,5 (30)	30	90	50	90	80	40
10	22,4 (20)	20	80	45	80	70	35
2,8 (3,0)	10	10	60	35	60	50	25
	< 2,8 (3,0)	3	40	25	40	30	15

Dimensions in millimetres

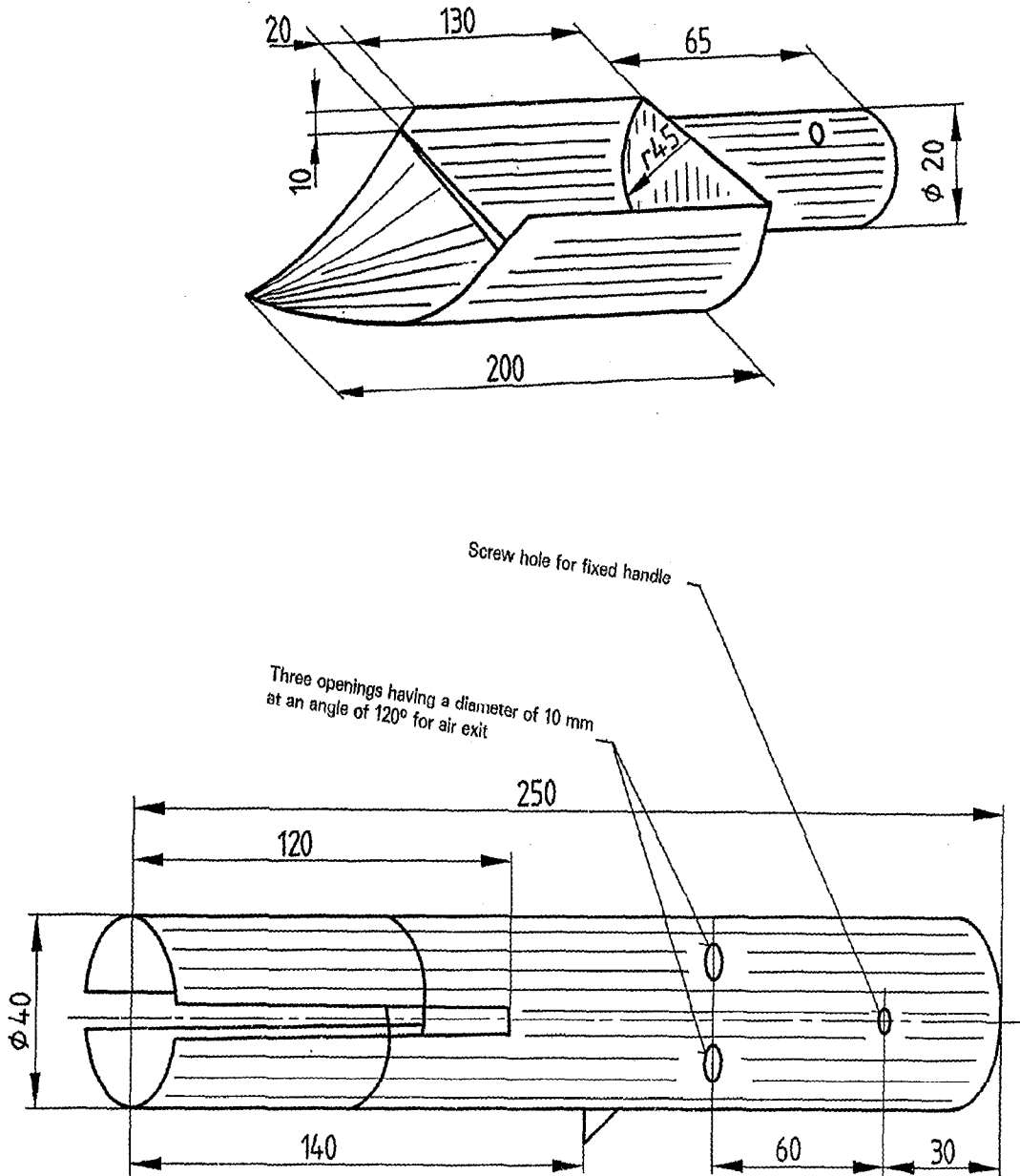


Figure 3 – Increment probes

7 Sampling methods

The sampling of ores from conveyors, wagons or containers, ships, stockpiles etc. shall be carried out at the time of loading or unloading of a consignment so that manual or mechanical sampling can be carried out safely.

7.1 Manual sampling

Sampling shall be carried out on a mass basis while a consignment is being transferred.

7.1.1 Sampling from conveyors

7.1.1.1 The increment shall be taken after stopping the conveyor; a section of the adequate length and full width and thickness of the chromium ore stream shall be taken from the specified position in the direction of the stream.

This adequate length shall be sufficient to ensure that a minimum mass of increment as specified in table 1 can be taken and shall be more than three times the nominal top size.

NOTE — During stopped-belt sampling, the sampling frame shall be placed so that it is in contact with the belt across its full width, and all ore particles lying inside the sampling frame shall be swept off into the container. Any ore particles obstructing the insertion of the sampling frame shall be either taken, at the left side of the sampling frame, into the increment, or removed, at the right side of the sampling frame, from the increment.

7.1.1.2 When the increment is taken from the moving conveyor, the full width and thickness of the chromium ore stream shall be taken by a mechanically assisted device from the falling stream.

7.1.1.3 When the effect of segregation in particle size is known to be not significant at the point of sampling and there is no pulsation in the stream, the individual increments may be taken from points selected at random either on the stopped conveyor or within the falling stream.

7.1.1.4 The interval of taking increments shall be uniform, on a mass basis, throughout the whole consignment and shall not be changed during the course of sampling.

The mass interval of taking increments shall be calculated from the formula

$$T < \frac{Q}{n} \quad \dots (4)$$

where

T is the mass interval, in tonnes, between taking increments;

Q is the mass, in tonnes, of consignment;

n is the number of increments required.

The mass interval of taking increments shall be determined to be smaller than the calculated mass interval (T) in taking the convenience of operation into account.

If the stream of ore is regular, the mass interval may be converted into an equivalent time interval.

7.1.1.5 The first increment shall be taken after a randomly selected tonnage has been handled within the first mass interval after the start of the handling operation.

7.1.2 Sampling from wagons or containers

7.1.2.1 Method of taking increments

The increment shall be taken at random from the new surface of chromium ore exposed during the loading or the unloading of wagons or containers [hereinafter called wagon(s)].

When it seems possible that there is some bias in the chromium ore in the wagon between the top and bottom, between the front and the rear, or between the left and the right, it is advisable to take the increments from each divided stratum or from different places in each of the selected wagons.

NOTE — The point of sampling shall be located at a distance of not less than 0,5 m from the wagon board. Examples of sampling point location are shown in figure 4.

There is a danger of introducing some bias in sampling when the sampling is conducted with a sampling probe or a boring sampler from the top surface of chromium ore loaded on wagons and accordingly, the method shall be applied only after having ascertained, by check experiments, that there is no bias present.

NOTES

1 The sampling of ore of less than 22,4 mm in size may be carried out by taking increments with a shovel, or thrusting a probe into the ore to its full length, provided that this is practical and that a full core may be drawn without bias. While taking the sample, the ore shall not spill out from the probe. The sample shall be poured out from the pipe probe by tapping on the brim of the container with the angle welded to the pipe.

2 In the sampling of ore of less than 100 mm in size increments shall be taken with a shovel. If necessary, at the points at which increments are taken, hollows of depth 0,2 m to 0,5 m shall be made and the increments shall be taken by motion along the walls of the hollows vertically up from below. The ore shall not be taken from the bottom of the hollow. Care shall be taken to prevent the ore from spilling over the edges of the shovel.

3 For lumps of greater than 100 mm in size in addition to taking the increments with a shovel (table 4), the hammer and shovel method may be used (see annex A).

7.1.2.2 Sampling from all wagons (stratified sampling)

When the number of wagons forming one consignment is not more than the number of increments specified in table 3, the sampling shall be conducted by taking increments from each

wagon. The number of increments which are to be taken from each wagon of the consignment shall be calculated from the formula

$$n_w = \frac{n}{M} \quad \dots (5)$$

where

n_w is the number of increments to be taken from each wagon;

n is the number of increments in table 3 according to the mass of the consignment;

M is the number of wagons in the consignment.

The result obtained shall be rounded up to the nearest whole number.

In the case of wagons having different load capacities, the number of increments shall be determined in proportion to their load capacity.

7.1.2.3 Sampling from selected wagons (two-stage sampling)

When the number of wagons forming one consignment is more than the number of increments specified in table 3, the sampling shall be conducted by selecting the wagons to be sampled and then by taking increments at random from each wagon selected.

The number of wagons to be selected (m) is determined by the equation

$$m = \frac{M\sigma_b^2 + \frac{(M-1)\sigma_w^2}{\bar{n}_w}}{(M-1)(\beta_s/2)^2 + \sigma_b^2} \quad \dots (6)$$

where

M is the number of wagons constituting a consignment;

m is the number of wagons to be selected in the first stage of sampling;

\bar{n}_w is the number of increments to be taken from each of the selected wagons in the second stage of sampling;

σ_b is the standard deviation between wagons, % absolute;

σ_w is the standard deviation within wagons, % absolute;

2 is the factor related to two-sigma (approximately 95 %) probability level;

β_s is the two-sigma precision of sampling, % absolute.

When the values of σ_w and σ_b are classified in terms of large or small quality variation in accordance with table 2, table 5 shall be used to determine the minimum number of wagons to be selected for a particular consignment.

Table 5 — Minimum required number of wagons to be selected (m)

Mass of consignment (tonnes)		Number of wagons constituting a consignment M	Classification of quality variation		Minimum required number of wagons to be selected (m)		Precision of sampling β_s
over	up to and including		σ_b	σ_w	Large	Small	
30 000	45 000	650	Large	Small	90	75	0,33
15 000	30 000	425	Large	Small	70	60	0,37
5 000	15 000	200	Large	Small	60	50	0,39
2 000	5 000	60	Large	Small	35	30	0,42
1 000	2 000	25	Large	Small	15	15	0,55
500	1 000	10	Large	Small	10	8	0,60
	500	10	Large	Small	9	7	0,65
			Small		7	5	

NOTE — The number of wagons to be selected is calculated under the following conditions :

- a) Quality variation with respect to : Large 1,5 %; small 0,7 %; Cr_2O_3 content (σ_b or σ_w);
- b) Capacity of wagon : 60 tonnes;
- c) Number of increments to be taken from each selected wagon (\bar{n}) : 4.

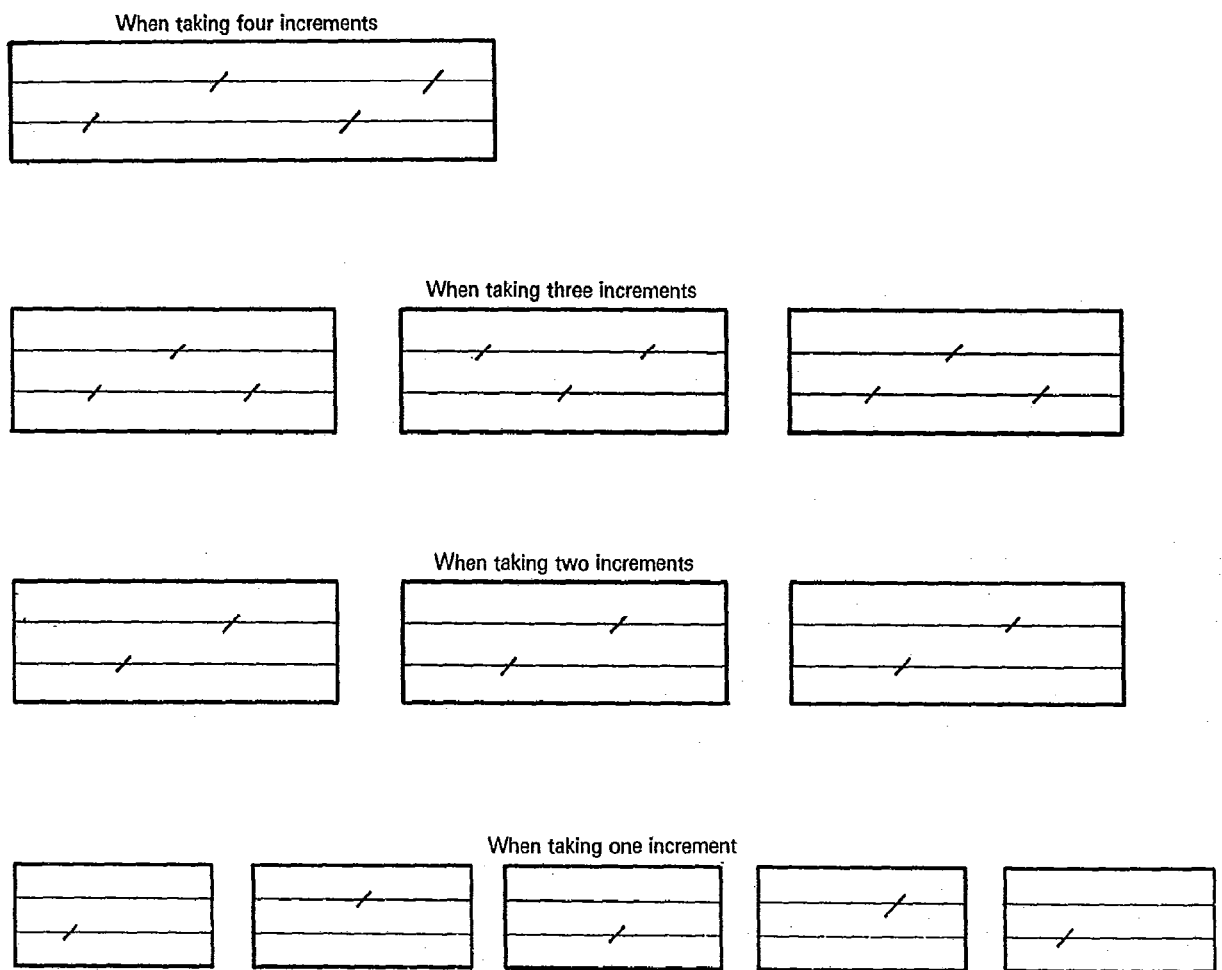


Figure 4 – Examples of location of points for taking increments from wagons

When the load of the wagon is not 60 tonnes, the minimum number of wagons to be selected shall be obtained from the following equation

$$m' = m \times \sqrt{\frac{60}{C}} \quad \dots (7)$$

where

m' is the minimum number of wagons to be selected;

m is the minimum number of wagons obtained by equation (6);

C is the load of the wagon, in tonnes.

The result obtained shall be rounded up to the nearest whole number to ensure adequate precision.

Four increments shall be taken from each wagon selected. When the load of the wagon is not 60 tonnes, the number of increments to be taken from each wagon shall be obtained from the equation

$$n'_w = 4 \sqrt{\frac{C}{60}} \quad \dots (8)$$

where

n'_w is the number of increments to be taken from wagons other than 60 tonnes wagons;

C is the load of the wagon, in tonnes.

The result obtained shall be rounded up to the nearest whole number, as little improvement will be obtained by going to a higher whole number.

7.1.3 The sampling of ore from stockpiles shall be conducted in accordance with the method specified in 7.1.1, during the formation of the stockpiles or during the reclaiming of the stockpiles for their transfer to other places.

7.1.4 During the handling of ore by means of periodically filled and emptied containers (grab, bucket, skip, etc.), increments shall be taken manually, either on the new surface exposed during taking of the ore by the handling device or from ore poured out by the device onto a special platform, without making hollows.

The number of containers to be sampled shall be not less than the required number of increments.

The interval equal to a number of containers (grabs, etc.) shall be determined using the equation

$$r = \frac{Q}{g \cdot n} \quad \dots (9)$$

where

r is the interval of sampling (expressed as a given number of containers);

Q is the mass of consignment, in tonnes;

g is the mass of ore taken by the handling container at one time, in tonnes;

n is the required number of increments.

7.2 Mechanical sampling

7.2.1 The sampling of ores during the loading or unloading of wagons, ships, stores, bunkers and in forming stockpiles by means of continuous action handling devices shall be carried out, using mechanical sampling devices, on the stream of ore falling from one handling device to another at a fixed interval of mass or time.

7.2.2 The number of cuttings by mechanical sampling devices shall be not less than the required number of increments.

7.2.3 The interval between taking increments from a consignment can be taken as a fixed interval of either mass or time and shall not be changed during the entire course of sampling of a consignment.

7.2.4 The mass interval between taking increments shall be determined using equation (4) in 7.1.1.4.

7.2.5 The time interval between taking increments shall be determined using the equation

$$t = \frac{60 \cdot Q}{G n} \quad \dots (10)$$

where

t is the time interval between taking increments, in minutes;

Q is the mass of consignment, in tonnes;

G is the maximum flow rate of the belt conveyor, in tonnes/hour;

n is the number of increments required.

Annex A **(normative)**

Hammer and shovel method

The hammer and shovel method shall be used in sampling of ores containing lumps of greater than 100 mm in size.

Increments shall be taken with a hammer and shovel by breaking off representative chippings. From obviously heterogeneous lumps, a greater number of smaller chippings shall be taken than from homogeneous lumps. The total mass

of chippings shall be 4 kg and be proportional to that of large lumps of ore being sampled. It shall be ascertained either visually or from past experience.

The ore taken from small and large lumps at one point shall constitute one increment. The increments thus sampled shall be placed in a bucket having a cover or in a container.

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