International Standard

ISO 4296/1

Manganese ores — Sampling — Part 1: Increment sampling

Minerais de manganèse — Échantillonnage — Partie 1: Échantillonnage par prélèvements

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Foreword

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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.

International Standard ISO 4296/1 was developed by Technical Committee ISO/TC 65, *Manganese and chromium ores*, and was circulated to the member bodies in May 1983.

It has been approved by the member bodies of the following countries:

Austria	India	South Africa, Rep. of
Bulgaria	Italy	Thailand
China	Japan	United Kingdom
Czechoslovakia	Poland	USSR
France	Romania	

The member body of the following country expressed disapproval of the document on technical grounds:

Australia

Manganese ores — Sampling — Part 1: Increment sampling

0 Introduction

ISO 4296 consists of the following parts :

Part 1 : Increment sampling

Part 2 : Preparation of samples

1 Scope and field of application

This part of ISO 4296 specifies methods for taking samples of manganese 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 manganese ores, whether natural or processed.

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

2 References

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

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

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

ISO 4296/2, *Manganese ores — Sampling — Part 2 : Preparation of samples.*

3 Definitions

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 ore taken at one time from a consignment using a sampling device.

2) A quantity of ore taken in the increment division method.

3.4 subsample :

1) A quantity of an ore consisting of several increments taken from a part of a consignment.

2) A composite of several increments which have been crushed and divided individually.

3.5 gross sample :

1) The quantity of an ore consisting of all increments taken from a consignment.

2) An aggregation of all the increments or all the subsamples after they have been crushed and divided individually.

3.6 divided sample : A sample obtained by a method of division.

3.7 moisture sample : The sample taken for the determination of moisture content of the consignment or part of the consignment.

3.8 sample for chemical analysis : The sample taken for the determination of chemical composition of the consignment or part of the consignment.

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

3.10 maximum particle size : The aperture size of the sieve on which approximately 5 % of the mass of the manganese ore is retained.

3.11 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 strata are part of a consignment which is derived by division of the consignment according to specific criteria.

1

3.12 periodic systematic sampling : Sampling in which increments are taken from a consignment at regular intervals.

3.13 two-stage sampling : Sampling carried out by selecting the units to be sampled (wagons, trucks, large containers) and then taking increments at random from each unit selected.

4 General rules

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

4.1 Sampling shall be carried out by qualified samplers authorized by the seller and/or purchaser.

4.2 Sampling should preferably be carried out during handling immediately before or immediately after weighing.

4.3 Sampling shall be carried out by a systematic method with a random start. Sampling from wagons shall be conducted by the two-stage method or the stratified method.

4.4 The mass of the increment shall be determined in accordance with the maximum particle size of the ore in order to avoid the introduction of bias at the time of collecting the sample.

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

4.6 Throughout all the procedures of sampling, the samples shall be protected from any contamination.

4.7 All sampling methods shall be demonstrated to have no bias (see ISO 3086).

4.8 When the planned number of increments has been taken before the handling has been completed, additional increments shall be taken at the same interval until the handling operation is completed.

5 Methods of sampling in general

5.1 General sampling procedure

The general sampling procedure is as follows :

a) identify the consignment or part of the consignment to be sampled;

b) ascertain the maximum particle size;

c) determine the mass of increment;

d) ascertain the quality variation of the consignment (see ISO 3084);

e) determine the number of increments;

f) determine the point of sampling and the method of taking and combining the increments;

g) constitute a gross sample or subsample.

NOTE — If a consignment contains ores of different quality, it shall be divided into an appropriate number of lots and sampling and quality variation assessment shall be carried out on the lot basis.

5.2 Sampling plan

The gross sample or subsamples shall be constituted according to 5.7. (See figure 1.)

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.

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 must 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 absolute value of the arithmetic mean, $\bar{m}_1$, of the masses of the increments, expressed as a percentage :

$$\frac{\sigma}{\bar{m}_1} \times 100 \qquad \qquad \ldots (1)$$

Table 1 — Mass of increment in manual sampling

Maximum particle size	Minimum mass of increment
mm	kg
≥ 150	25
≥ 100 to 150	13
≥ 50 to 100	4
≥ 40 to 50	3
≥ 30 to 40	2
≥ 20 to 30	1
≥ 10 to 20	0,5
≥ 2,8 (3,0) to 10	0,2

5.3.2 The mass of increment, m_1, in kilograms, taken by means of mechanical samplers from the ore stream, is directly proportional to the flow rate of the belt conveyor and to the cutting aperture of the sampler, and inversely proportional to the cutter speed of the sampler; it may be calculated by the equation

$$m_1 = \frac{q_m b}{3,6 \, v} \qquad \qquad \ldots (2)$$

where

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

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

v is the cutter speed, expressed in metres per second, of the sampler.
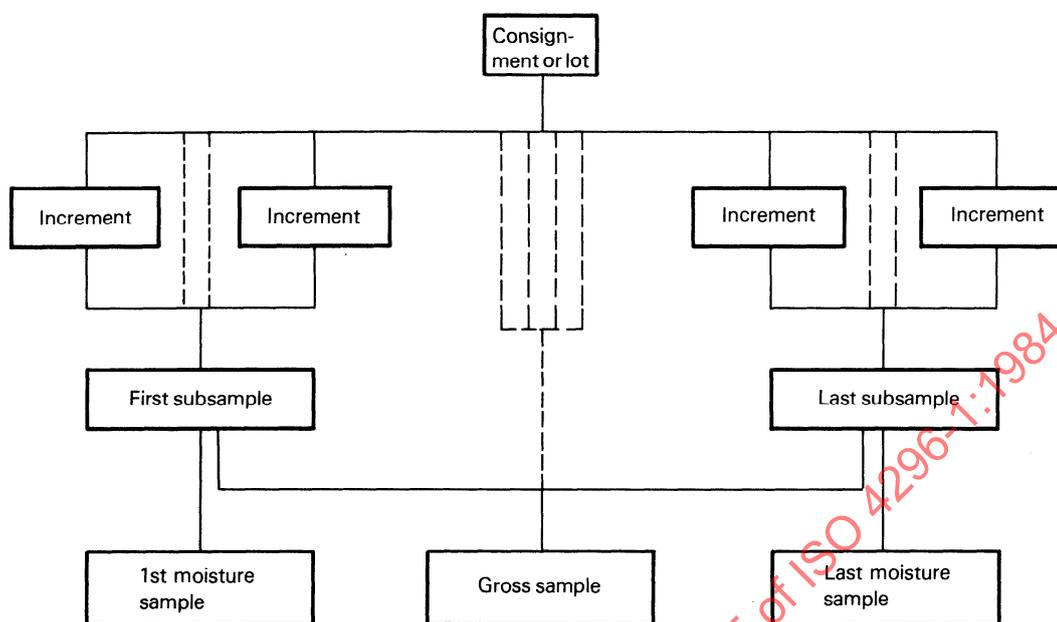
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Figure 1 — Sampling plan

5.3.3 The mass of increment taken either by means of mechanical samplers or manually may be increased by agreement between the interested parties.

5.4 Classification of quality variation

The quality variation is a measure of the heterogeneity of the consignment in respect of a particular characteristic and is determined by the standard deviation of the values measured on individual increments within the strata of the consignment, σ_w, expressed as a percentage.

When the consignment is supplied in wagons (trucks or large containers) two types of variation are possible : variation within a wagon (truck or container), σ_w; and variation between wagons (trucks or containers), σ_b. In such a case the larger of the values σ_b and σ_w shall be taken as the basis for classification. The classification of quality variation is given in table 2.

Table 2 — Classification of quality variation

Quality variation	Standard deviation of manganese content
	%
Large	$\sigma_w > 2,0$ or $\sigma_b > 2,0$
Medium	$2,0 > \sigma_w \geqslant 1,0$ or $2,0 > \sigma_b \geqslant 1,0$
Small	$\sigma_w < 1,0$ or $\sigma_b < 1,0$

Quality variations shall be determined experimentally over several consignments. All ore, the quality variation of which is not known, shall be considered as having "large" quality variation. The quality variation shall be determined in accordance with ISO 3084.

5.5 Sampling precision and overall precision

This part of ISO 4296 is designed to obtain a sampling precision, β_S, as shown 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 taken in accordance with ISO 4296/2 and that measurement has been carried out in accordance with the 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 manganese 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 sample precision shall be checked in accordance with ISO 3085.

5.6 Number of increments

5.6.1 In the case of systematic sampling, under the theory of stratified sampling, the number of increments, n_1, is given by the equation

$$n_1 = \left(\frac{2\,\sigma_w}{\beta_S} \right)^2 \qquad \ldots (3)$$

where

σ_w is the standard deviation within strata, expressed as an absolute percentage;

β_S is the two-sigma precision of sampling, expressed as an absolute percentage;

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

5.6.2 The minimum number of increments, n_1, to be taken from a consignment in order to attain the desired precision of sampling determined by equation (3) is specified in table 3 according to the mass of the consignment and the classification of quality variation.

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

5.6.3 In the case of two-stage sampling, the number of increments shall be calculated according to 7.3.2.1.

Table 3 — Minimum number of increments, n_1, and sampling precision depending on quality variation (Mn %)

Mass of consignment (t)	Precision of sampling, β_S	Number of increments[1] according to quality variation		
		Large	Medium	Small
> 30 000 to 45 000	0,35	170	100	19
> 15 000 to 30 000	0,37	150	90	17
> 5 000 to 15 000	0,39	135	85	15
> 2 000 to 5 000	0,42	115	70	13
> 1 000 to 2 000	0,46	100	60	11
> 500 to 1 000	0,55	70	45	8
≤ 500	0,70	40	25	5

1) The number of increments is calculated as follows : large variation, σ_w = 2,25 %; medium variation, σ_w = 1,75 %; small variation, σ_w = 0,75 %.

5.7 Method of constituting a gross sample

5.7.1 All the individual increments or subsamples shall be combined into a gross sample.

If greater precision is required, each increment shall be tested separately by agreement between the interested parties.

5.7.2 When a consignment is very large and a long time is required for loading and unloading it, the consignment shall be divided into parts and subsamples shall be taken for each part obtained. The minimum number of parts per consignment, depending on the mass of the consignment, shall be determined in accordance with table 4.

Table 4 — Minimum number of parts per consignment

Mass of consignment (t)	Minimum number of parts per consignment
> 30 000 to 45 000	5
> 15 000 to 30 000	3
> 5 000 to 15 000	2
≤ 5 000	1

6 Equipment

6.1 Mechanical sampling

Mechanical sampling devices (rotary-arc, bucket, cutter-chute sampler, etc.) shall meet the following requirements :

a) the 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 a complete increment at one time and shall be filled to no more than two-thirds of its volume;

c) the effective opening of the sampling device shall have a minimum dimension of 3 times the maximum particle size of the ore;

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

6.2 Manual sampling

The following tools shall be used :

a) shovels (see figure 2 and table 5);

b) hammer, of mass 400 to 900 g;

c) probe (see figure 3);

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.

d) sampling frame.

7 Sampling methods

7.1 Mechanical sampling

As a rule, sampling shall be carried out by mechanical methods.

Table 5 — Dimensions of increment shovel

Maximum particle size (mm)	Shovel No.	Dimensions of shovel (mm)				
		a	b	c	d	e
> 50 to 100	100	250	110	250	220	100
> 40 to 50	50	150	75	150	130	65
> 31,5 (30) to 40	40	110	65	110	95	50
> 22,4 (20) to 31,5 (30)	30	90	50	90	80	40
> 10 to 22,4 (30)	20	80	45	80	70	35
> 2,8 (3,0) to 10	10	60	35	60	50	25
< 2,8 (3,0)	3	40	25	40	30	15

NOTE — Other sampling devices, including mechanically assisted devices, may be used to take increments. These devices should have a minimum opening equivalent to c and in the case of over 100 mm at least three times the maximum particle 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.
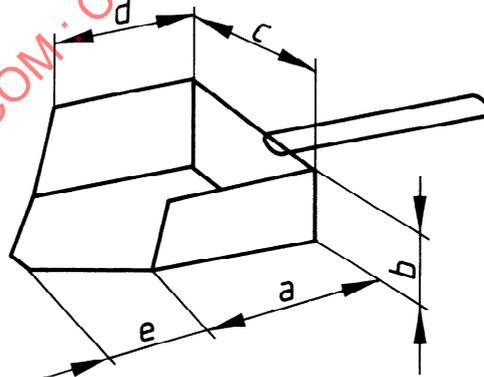

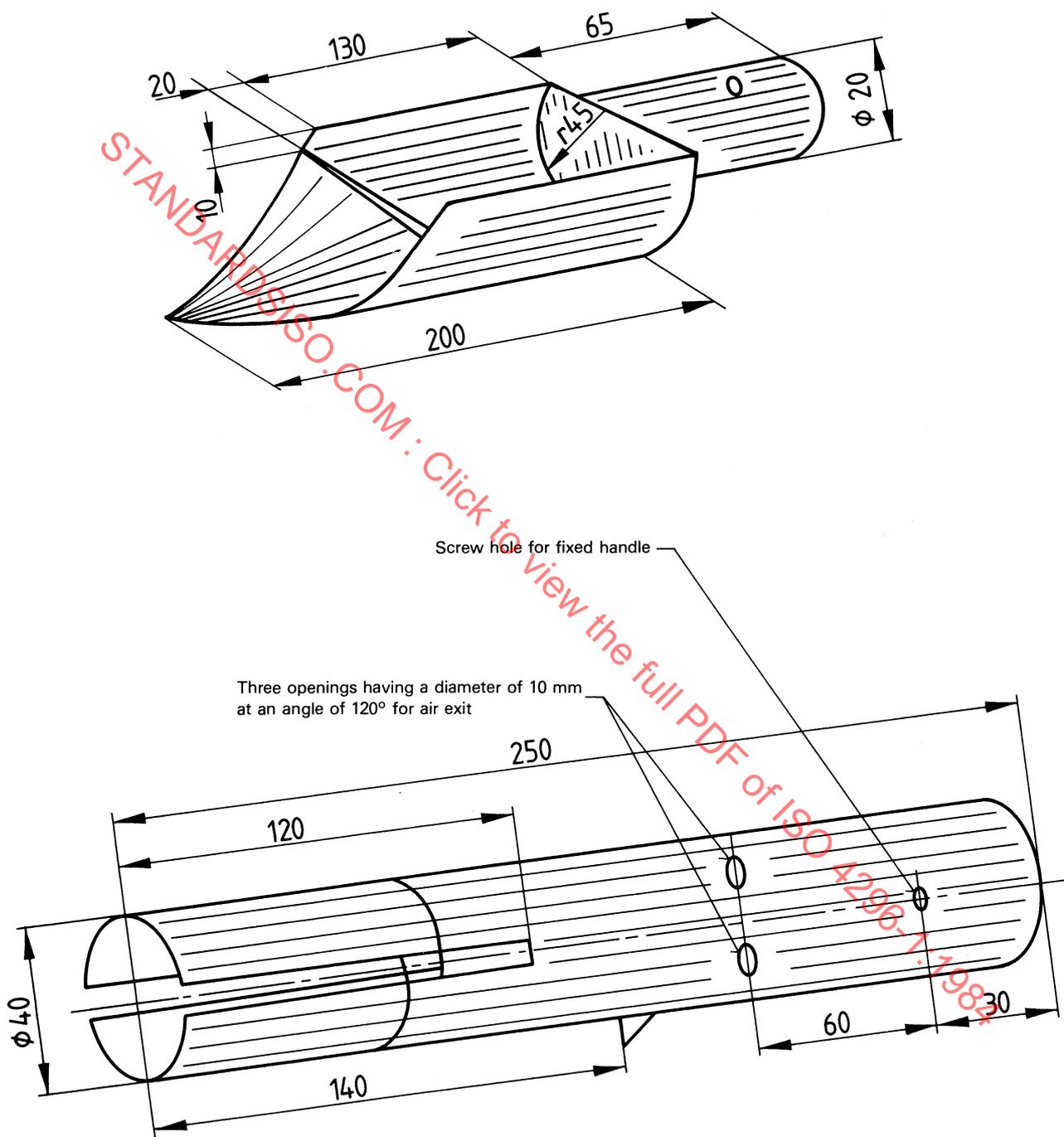Figure 2 — Increment shovel

Dimensions in millimetres

Screw hole for fixed handle

Three openings having a diameter of 10 mm
at an angle of 120° for air exit

Figure 3 — Increment probes

7.1.1 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.

7.1.2 The number of cuttings during the taking of a gross sample by mechanical sampling devices shall not be less than the required number of increments.

7.1.3 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.

7.1.4 The mass interval between taking increments, Δm, in tonnes, is given by the condition

$$\Delta m \leqslant \frac{m_2}{n_1} \qquad \ldots (4)$$

where

m_2 is the mass, in tonnes, of the consignment;

n_1 is the number of increments required.

7.1.5 The time interval between taking increments, Δt, in minutes, is given by the equation

$$\Delta t = \frac{60 \, m_2}{q_{m,\,max} \, n_1} \qquad \ldots (5)$$

where

m_2 is the mass, in tonnes, of the consignment;

$q_{m,\,max}$ is the maximum flow rate, expressed in tonnes per hour, of the belt conveyor;

n_1 is the number of increments required.

7.2 Stopped-belt sampling

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 a 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.

NOTE — The increment shall be taken on a complete cross-section of the ore for a length which is at least 3 times the maximum particle size of an ore.

7.3 Manual sampling

Manual sampling shall be used only when mechanical sampling is impracticable.

7.3.1 Method of taking increments

7.3.1.1 In the manual 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 0,5 to 1 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.

7.3.1.2 The sampling of ore of less than 20 mm in size may be carried out by taking the 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.

7.3.1.3 For lumps of greater than 100 mm in size, alongside with taking the increments with a shovel (table 5), the hammer and shovel method may be used (see the annex).

7.3.2 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 to be taken from each wagon of the consignment, n_2, is given by the equation

$$n_2 = \frac{n_1}{n_3} \qquad \ldots (6)$$

where

n_1 is the number of increments in table 3 according to the mass of the consignment;

n_3 is the number of wagons in the consignment.

The result obtained shall be rounded to the next higher whole number.

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

7.3.3 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.

7.3.3.1 Minimum number of wagons to selected

The number of wagons to be selected in the first stage of sampling, n_4, is given by the equation

$$n_4 = \frac{n_3\sigma_b^2 + [(n_3 - 1)\sigma_w^2]/n_2}{(n_3 - 1)(\beta_S/2)^2 + \sigma_b^2} \qquad \ldots (7)$$

where

n_2 is the number of increments to be taken from each of the selected wagons in the second stage of sampling;

n_3 is the number of wagons in the consignment;

σ_b is the standard deviation between wagons, expressed as an absolute percentage;

σ_w is the standard deviation within wagons, expressed as an absolute percentage;

β_S is the two-sigma precision of sampling, expressed as an absolute percentage;

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

When the values of σ_w and σ_b are classified in terms of large, medium or small quality variation in accordance with table 2, then table 6 shall be used to determine the minimum number of wagons to be selected for a particular consignment.

When the capacity of the wagon is not 60 t, the number of wagons to be selected, n_5, is given by the equation

$$n_5 = n_4 \sqrt{\frac{60}{m_3}} \qquad \ldots (8)$$

where m_3 is the capacity, in tonnes, of the wagon.

7.3.3.2 Number of increments to be taken from each selected wagon

Four increments shall be taken from each wagon selected. When the capacity of the wagon is not 60 t, the number of increments to be taken from each wagon, n_6, is given by the equation

$$n_6 = 4 \sqrt{\frac{m_3}{60}} \qquad \ldots (9)$$

where m_3 is the capacity, in tonnes, of the wagon.

The result obtained shall be rounded to the next lower whole number.

7.3.4 Where to take increments fom wagons

The points 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.

7.3.5 Sampling from stockpiles

Sampling from stockpiles shall be conducted in accordance with the height of the stratum. Sampling of ore in stockpiles less than 1,5 m high shall be carried out as follows. The entire surface of the stockpile to be sampled or its part shall be divided into squares the number of which shall be equal to the required number of increments.

Sampling of ore in stockpiles more than 1,5 m high shall be carried out by shovelling using a grab or by layer removing.

7.3.6 Number of containers to be sampled

During the handling of ore by means of periodically filled and emptied containers (grab, bucket, skip, etc.), increments shall be taken manually either in the new surface exposed during taking of the ore by the handling device or from ore poured out by the device on to 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 of sampling, expressed as a number of containers (grabs, etc.), Δn, is given by the equation

$$\Delta n = \frac{m_2}{m_4\, n_1} \qquad \ldots (10)$$

where

m_2 is the mass, in tonnes, of the consignment;

m_4 is the mass, in tonnes, of ore taken by the handling container at one time;

n_1 is the required number of increments.

7.3.7 Safety

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

8

Table 6 — Minimum required number of wagons to be selected, n_4, for manganese ore

Mass of consignment (t)	Number of wagons consti- tuting a consignment	Classification of quality control σ_b \ σ_w	Minimum required number of wagons to be selected, n_4			Precision of sampling, β_S
			Large	Medium	Small	
> 30 000 to 45 000	750	Large	170	157	140	0,35
		Medium	125	111	95	
		Small	59	43	23	
> 15 000 to 30 000	500	Large	143	132	118	0,37
		Medium	108	95	80	
		Small	52	38	20	
> 5 000 to 15 000	250	Large	109	101	90	0,39
		Medium	87	77	64	
		Small	46	33	18	
> 2 000 to 5 000	84	Large	61	56	50	0,42
		Medium	54	48	40	
		Small	36	27	14	
> 1 000 to 2 000	34	Large	32	29	26	0,46
		Medium	31	27	23	
		Small	27	20	11	
> 500 to 1 000	17	Large	17	16	14	0,55
		Medium	17	15	13	
		Small	17	13	7	
< 500	9	Large	9	9	8	0,70
		Medium	9	9	7	
		Small	9	8	4	

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

a) quality variation with respect to manganese content (σ_b or σ_w): large, 2,25 %; medium, 1,75 %; small, 0,75 %

b) capacity of wagon, $m_3 = 60$ t

c) number of increments to be taken from each selected wagon, $n_2 = 4$.

When taking four increments

When taking three increments

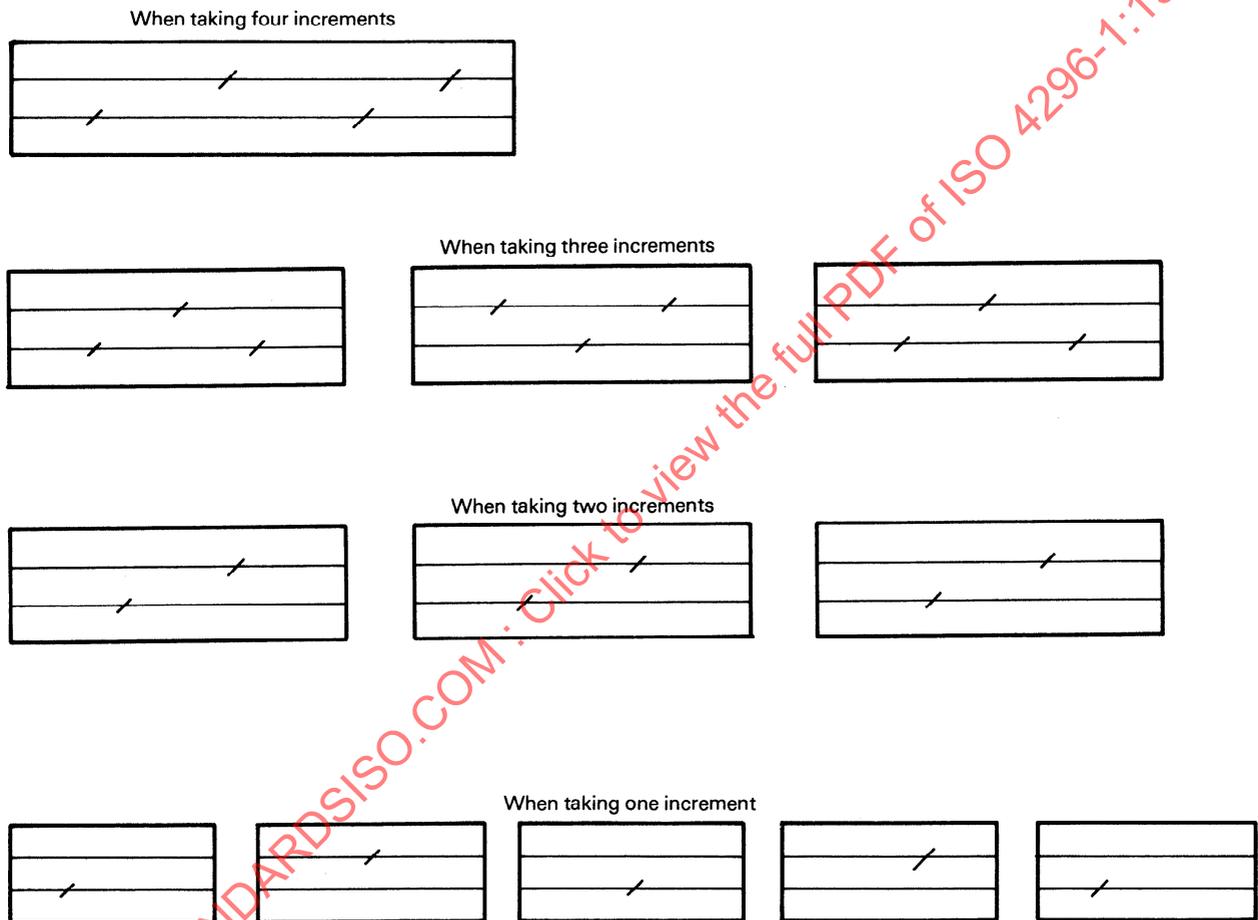When taking two increments

When taking one increment

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