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International Standard ISO 3083

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Iron ores — Preparation of samples — Manual method

Minerais de fer — Préparation des échantillons — Méthode manuelle

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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.

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 3083 was prepared by Technical Committee ISO/TC 102, *Iron ores.*

This second edition cancels and replaces the first edition (ISO 3083-1973), of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Contents

Annexes

Iron ores — Preparation of samples — Manual method

1 Scope

This International Standard specifies

a) the underlying theory,

b) the basic principles, and

c) the basic requirements for the devices and their operation

for the manual method of preparation of samples taken from a consignment of iron ore in accordance with ISO 3081 or ISO 3082, for the purpose of determining the quality characteristics of the consignment.

NOTE — The theory and basic principles given herein are similar to those given in ISO 3081 and ISO 3082.

2 Field of application

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

Samples are prepared for the determination of size distribution, moisture content, and chemical composition.

3 References

ISO 3081, *Iron ores — Increment sampling — Manual method.*

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method.*[1]

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 3087, *Iron ores — Determination of moisture content of a consignment.*

4 Definitions

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

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

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

4.3 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.

4.4 subsample:

(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 individually has been optionally crushed and/or optionally divided as necessary.

4.5 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 subsamples each of which individually has been optionally crushed and/or optionally divided as necessary.

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

4.7 test sample: Any sample, for the determination of size distribution, moisture content, 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.

A representative part of a test sample which is actually subjected to the test is designated the **test portion.** If the entire quantity of a test sample is subjected to the test, the test sample may also be called "test portion".

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[1] At present at the stage of draft.

4.8 size sample: The sample taken for the determination of size distribution of the consignment.

4.9 moisture sample: The sample taken for the determination of moisture content of the consignment.

4.10 sample for chemical analysis: The sample taken for the determination of chemical composition of the consignment.

4.11 maximum particle size: Particle size expressed in terms of the size of openings of the sieve on which approximately 5 % (*m*/*m*) of iron ore is retained.

NOTE — The maximum particle size of the consignment may be ascertained either by past experience or by experiment. However, if no information is available, visual estimation is acceptable.

4.12 whole-through sieve size: Particle size expressed by the size of the smallest sieve aperture size through which all of a sample passes.

4.13 sample preparation: The process of making the sample ready for the determination of quality characteristics. It covers sample division, crushing, mixing and sometimes predrying and may be performed in several stages.

4.14 sample division: The process in sample preparation whereby the mass of a sample is reduced by partition or extraction without crushing.

4.15 constant-mass division: A type of division for obtaining divided samples having almost uniform mass regardless of the variation in mass of samples to be divided.

NOTE — Almost uniform mass means that the variation in mass shall be less than 20 % in terms of the coefficient of variation (CV).

4.16 fixed-rate division: A type of division for obtaining divided samples having such masses as to be proportional to the varied masses of samples to be divided.

4.17 split use of sample: The sample is split into two or more parts, which are used individually for the determination of their two or more quality characteristics.

4.18 multiple use of sample: The sample in its entirety is used for the determination of one quality characteristic, then the same sample in its entirety or part is used for the determination of other quality characteristics.

4.19 mass-basis sampling: The taking of increments in uniform mass intervals throughout the mass sampled.

4.20 time-basis sampling: The taking of increments in uniform time intervals throughout the mass sampled.

5 General procedures for sample preparation

The increments taken in accordance with ISO 3081 or ISO 3082 for the required determinations shall be prepared into test samples according to the following general procedures:

a) determine whether the test sample is to be prepared from each increment, from each subsample or from the gross sample according to the requirements for the determination of quality characteristics;

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

c) select the method and type of sample division at each stage;

d) establish the flow of sample preparation including the processes of division, crushing, mixing and pre-drying (if necessary);

e) prepare the test sample according to the procedures mentioned in a) to d).

6 Fundamentals of sample preparation

6.1 Precision of sample preparation and overall precision

The precision of sample preparation, β_D, shall be within ± 0,3 % in total iron or moisture content with a 95 % probability. However, if sample preparation is carried out first on individual increments or subsamples at an appropriate stage of the sample preparation and then those divided increments or subsamples are combined into the gross sample, the precision of sample preparation will be further improved (see 6.1.2 and 6.1.3).

The precision of sample preparation and measurement, β_{DM}, for size determination shall be within the values specified for the type of ore (see table 8).

Variations from the tables in this International Standard may be made, provided it can be demonstrated that the specified precision of sample preparation can be met. The precision should be determined in accordance with ISO 3085.

The overall precision in terms of standard deviation, σ_{SDM}, for the cases where division and measurement are carried out on the gross sample, on each of the subsamples or on each of the increments may be expressed as follows.

6.1.1 When the gross sample is prepared for a consignment and n_7 determinations (chemical analyses) are carried out on the gross sample, the overall precision will be

$$\sigma_{SDM}^2 = \sigma_S^2 + \sigma_D^2 + \frac{\sigma_M^2}{n_7}$$

where

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

σ_D is the precision of sample preparation in terms of standard deviation comprising the processes from the gross sample to the test sample;

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

6.1.2 When n_8 subsamples, each subsample consisting of an equal number of increments, are prepared and n_7 determinations are carried out on each subsample, the overall precision will be

$$\sigma^2_{\mathrm{SDM}} = \sigma^2_{\mathrm{S}} + \frac{\sigma^2_{\mathrm{D}} + (\sigma^2_{\mathrm{M}}/n_7)}{n_8}$$

where σ_{D} is the precision of sample preparation in terms of standard deviation comprising the processes from the subsample to the test sample.

Further, when n_8 subsamples are combined into the gross sample at an appropriate stage after individual sample preparation, and n_7 determinations are carried out on the gross sample, the overall precision will be

$$\sigma^2_{\mathrm{SDM}} = \sigma^2_{\mathrm{S}} + \frac{\sigma^2_{\mathrm{D_1}}}{n_8} + \sigma^2_{\mathrm{D_2}} + \frac{\sigma^2_{\mathrm{M}}}{n_7}$$

where

$\sigma_{\mathrm{D_1}}$ is the precision of sample preparation in terms of standard deviation comprising the processes from the subsample to the divided subsample at an appropriate stage;

$\sigma_{\mathrm{D_2}}$ is the precision of sample preparation in terms of standard deviation comprising the processes from the gross sample at an appropriate stage to the test sample.

6.1.3 When n_7 determinations are carried out on each increment, the overall precision will be

$$\sigma^2_{\mathrm{SDM}} = \sigma^2_{\mathrm{S}} + \frac{\sigma^2_{\mathrm{D}} + (\sigma^2_{\mathrm{M}}/n_7)}{n_1}$$

where

σ_{D} is the precision of sample preparation in terms of standard deviation covering the processes from the increment to the test sample;

n_1 is the number of increments.

Further, when all the increments are combined into the gross sample at an appropriate stage after individual sample preparation, and n_7 determinations are carried out on the gross sample, the overall precision will be

$$\sigma^2_{\mathrm{SDM}} = \sigma^2_{\mathrm{S}} + \frac{\sigma^2_{\mathrm{D}}}{n_1} + \frac{\sigma^2_{\mathrm{M}}}{n_7}$$

where σ_{D} is the precision of sample preparation in terms of standard deviation covering the processes from the increment to the divided increment at an appropriate stage.

6.2 Composition of samples

When samples are to be composed of the increments, the following shall be taken into consideration:

a) quality characteristics to be determined;

b) overall precision required;

c) coefficient of variation (CV) in mass of increments taken by mass-basis sampling.

6.3 Division rules

In order to obtain the specified precision of sample preparation the following aspects of division shall be considered:

a) minimum mass of the sample after division, specified for each quality characteristic to be determined;

b) method and type of division to be adopted;

c) whole-through sieve size of the sample to be divided.

6.4 Method and type of division

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

a) manual increment division method (see 9.1);

b) manual riffle division method (see 9.2);

c) coning and quartering method (see 9.3);

d) mechanical division method (see ISO 3082).

This International Standard specifies three methods of manual division a), b) and c), which shall be applied to increments or subsamples on the basis of taking increments and division type as shown in table 1.

Combining of increments taken on time-basis sampling and mass-basis sampling shall incorporate the procedures specified in clause 8.

6.5 Split use and multiple use of sample

When a sample taken from the consignment meets the respective requirements for the determination of quality characteristics, the sample may be either in split use or in multiple use for obtaining the test samples for moisture determination, size determination and chemical analysis.

6.6 Crushing and grinding

The crushing and grinding shall be conducted with such a crusher and a grinder as are suitable for the size and hardness of the ore particles.

The crusher and grinder shall be purged with material from the same source.

Table 1 — Application of manual division method[1]

Division of	Conditions of increment				Manual division method to be applied[2]	
	Sampling		Number of increments composed	CV (%)	Constant-mass division	Fixed-rate division
	Method	Type			Increment division method	Riffle division and C/Q[3]
Increment	Manual	Mass-basis		< 20	×	×
				> 20	×	–
	Mechanical			< 20	×	×
				> 20	×	–
		Time-basis			–	×
Subsample	Manual	Mass-basis			×	×
	Mechanical		Equal		×	×
			Unequal		–	×
		Time-basis			–	×

1) Any manual division method applies to the gross sample.

2) × denotes applicable; – denotes not applicable

3) C/Q denotes coning and quartering method.

6.7 Mixing

By mixing the sample thoroughly, it may be made homogeneous and consequently the errors in sample division can be lessened.

The mixing may be conducted either by a mechanical mixer or by hand. The mixer shall be selected to suit the sample and its particle size.

6.8 Pre-drying

When the sample is very wet or sticky and sample preparation cannot be carried out, the sample may be pre-dried in air or in an oven or similar device, below the temperature at which there is likely to be a change in quality, so that sample preparation may then be carried out without difficulty. If necessary, the pre-drying should be carried out according to the method in annex A to ISO 3087.

6.9 Requirements for sample preparation

6.9.1 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.

6.9.2 Check experiments for precision and bias shall be carried out from time to time on the sample preparation process, so that significant errors in the results caused by the process may be detected.

7 Apparatus

The following apparatus, which shall be thoroughly cleaned and examined before and after use, shall be provided for sample preparation.

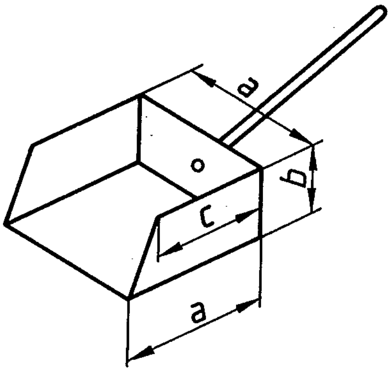
7.1 Crushers and **grinders.**

7.2 Drying ovens, capable of regulating the temperature at any point in the oven to within ±5 °C of the desired temperature.

7.3 Mixers.

7.4 Riffle dividers, details of which are given in annex A.

7.5 Scoop, for increment division, details of which are given in figure 1.



Scoop number	Dimensions of scoop (mm)			Thickness of metal sheet	Volume (approx.)
	a	*b*	*c*	(mm)	(ml)
31,5 D	90	60	80	2	450
22,4 D	80	45	70	2	270
16 D	70	40	60	2	180
10 D	60	35	50	1	110
5 D	50	30	40	1	65
2,8 D	40	25	30	0,5	35
1 D	30	15	25	0,5	10
0,5 D	20	10	20	0,5	4
0,25 D	15	10	12	0,3	2
0,1 D	10	10	10	0,3	1

Figure 1 — Scoop for increment division and its dimensions

8 Combining increments for sample preparation

The method of combining increments shall be selected according to the types of sampling employed for taking increments, viz. whether the increments have been taken on mass-basis sampling or on time-basis sampling. Systematic sampling is classified into two types, viz. mass basis and time basis. Stratified and two-stage samplings are performed on the mass basis.

8.1 Combining increments taken by mass-basis sampling

8.1.1 Composition of subsamples or gross sample from increments

8.1.1.1 When the variation in mass of individual increments is under 20 % (CV < 20 %), the increments either as taken or after having been prepared individually by the constant-mass or fixed-rate division at an appropriate stage shall be combined into subsamples or the gross sample.

8.1.1.2 When the variation in mass of individual increments is 20 % or over (CV > 20 %), the increments as they are taken shall not be combined into subsamples or the gross sample.

The increments prepared after having been divided individually by the constant-mass division at a practical stage should be combined into subsamples or the gross sample at an appropriate stage (see table 1). Otherwise, each increment should be prepared into a test sample to be subjected to the determination of quality characteristics.

8.1.2 Composition of gross sample from subsamples

The subsamples composed according to 8.1.1 should, with or without division, be combined into the gross sample.

When division is carried out on each subsample to compose the gross sample, the division shall be carried out as follows:

a) if the subsamples consist of an equal number of increments, constant-mass or fixed-rate division may be applied;

b) if the subsamples consist of different numbers of increments, only the fixed-rate division shall be applied.

8.2 Combining increments taken by time-basis sampling

8.2.1 Composition of subsamples or gross sample from increments

8.2.1.1 The increments as they are taken should be combined into subsamples or the gross sample, irrespective of the variation in mass of increments.

8.2.1.2 When division is carried out on each increment and the divided increments are combined into subsamples or the gross sample, the division shall be carried out on each increment by the fixed-rate division at any stage (see table 1).

8.2.2 Composition of gross sample from subsamples

8.2.2.1 The subsamples composed according to 8.2.1 should, with or without division, be combined into the gross sample, irrespective of the variation in mass of subsamples.

8.2.2.2 When division is carried out on each subsample and the divided subsamples are combined into the gross sample, the division shall be carried out on each subsample by the fixed-rate division at any stage (see table 1).

9 Manual method of division

The manual method of division shall be applied to ores of minus 31,5 mm in whole-through sieve size. However, the coning and quartering method is not applicable, except on ores of minus 10 mm in whole-through sieve size. In that case 9.3 shall apply.

9.1 Manual increment division method

The manual increment division method shall be carried out using a scoop for increment division according to the stipulations of 9.1.1, 9.1.2 and 9.1.3.

This method will provide the specified precision in spite of the high division ratio. However, this method should not be applied to certain samples such as pellets and sized ores, which roll freely and/or segregate easily (see 9.2). When the pellets have been crushed to a sufficiently small particle size, this method may be applied satisfactorily.

9.1.1 Mass of increment

The mass of each increment shall be as specified in table 2.

Table 2 — Whole-through sieve size of sample and minimum mass of each increment

Whole-through sieve size		Minimum mass of each increment (g)
Over	Up to and including	
22,4 mm	31,5 mm	1 000
16,0 mm	22,4 mm	600
10,0 mm	16,0 mm	400
5,00 mm	10,0 mm	250
2,80 mm	5,00 mm	150
1,00 mm	2,80 mm	80
500 µm	1,00 mm	25
250 µm	500 µm	10
100 µm	250 µm	5
	100 µm	2

9.1.2 Number of increments

The number of increments as given in table 3 shall be taken by the manual increment division method.

Table 3 — Number of increments to be taken by manual increment division method

Division of	Minimum number of increments
Gross sample	20
Subsample	12
Increment (primary)	4

A lesser number may be taken provided it has been demonstrated that no bias and/or no lack of precision is introduced (see ISO 3085 and ISO 3086).

9.1.3 Procedure

Sample division by the manual increment division method shall be carried out as follows.

9.1.3.1 Form the sample to be divided (minus 31,5 mm) on a smooth and flat plate (non-moisture absorbing) into a flat rectangle with a uniform thickness of the sample layer as specified in table 4.

9.1.3.2 Arrange the rectangle in the same number of parts as the minimum number of increments specified in table 3.

9.1.3.3 Select an appropriate scoop in figure 1, according to the whole-through sieve size. Take a scoop of sample from each of the parts (the place of taking such an increment being selected at random in each part), and combine these scoops of sample.

The scoop shall be thrust in to the bottom of the sample layer in the above procedure. It is recommended that a bumper plate be fixed vertically in front of the scoop, which shall be thrust in to the bottom of that sample layer in order to take an increment without any bias.

Table 4 — Whole-through sieve size and thickness of spread sample and scoop for increment division

Whole-through sieve size		Thickness of spread sample for increment division	Scoop for increment division	
Over	Up to and including	(mm)	Scoop number	Volume (approx.) ml
22,4 mm	31,5 mm	60 to 80	31,5 D	450
16,0 mm	22,4 mm	50 to 60	22,4 D	270
10,0 mm	16,0 mm	40 to 50	16 D	180
5,00 mm	10,0 mm	30 to 40	10 D	110
2,80 mm	5,00 mm	25 to 35	5 D	65
1,00 mm	2,80 mm	20 to 30	2,8 D	35
500 µm	1,00 mm	10 to 20	1 D	10
250 µm	500 µm	5 to 10	0,5 D	4
100 µm	250 µm	5 to 10	0,25 D	2
	100 µm	5 to 10	0,1 D	1

9.1.3.4 When the mass of the divided sample might become smaller than that required for subsequent testing purposes, the *mass of the increment and/or the number of increments shall* be increased.

Figure 2 illustrates an example of sample division for the gross sample by the manual increment division method.

9.2 Manual riffle division method

The manual riffle division method shall be carried out using a riffle divider according to the stipulations of 9.2.1, 9.2.2, 9.2.3 and 9.2.4.

The riffle divider is the most satisfactory type of manual divider for pellets or sized ores.

9.2.1 Selection of riffle divider

Dependent on the whole-through sieve size of the sample, an appropriate riffle divider specified in table 5 shall be selected.

Table 5 — Whole-through sieve size of sample and size of riffle divider

Whole-through sieve size mm		Riffle divider number	Opening width of riffle (mm)
Over	Up to and including		
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

9.2.2 Procedure

9.2.2.1 Place the sample to be divided (minus 31,5 mm) into a container after mixing and divide it into two by dropping the sample uniformly with a light shaking of the container into the middle of the riffles (at a right angle to the riffle). One of the two divided samples should be selected at random in order to avoid introducing any bias.

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

9.2.3 Division limit for moisture sample and sample for chemical analysis

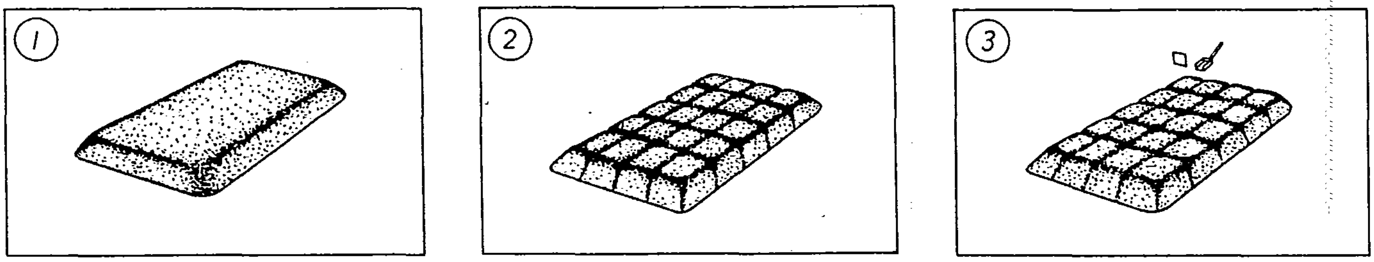
9.2.3.1 Gross sample

When the gross sample is divided, the division shall be carried out according to table 6.

The gross sample shall not be divided further than the specified mass corresponding to the whole-through sieve size.

Table 6 — Minimum mass of divided gross sample for moisture determination and/or chemical analysis by manual riffle division method

Whole-through sieve size		Minimum mass of divided sample (kg)
Over	Up to and including	
22,4 mm	31,5 mm	750
16,0 mm	22,4 mm	250
10,0 mm	16,0 mm	150
5,00 mm	10,0 mm	50
2,80 mm	5,00 mm	25
1,00 mm	2,80 mm	15
500 µm	1,00 mm	10
250 µm	500 µm	5
	250 µm	0,5



1) Form the sample into a flat rectangle with a uniform thickness.

2) Arrange in 20 equal parts, for example five equal parts lengthwise and four equal parts breadthwise.

3) Take a scoop of sample from each of the 20 parts by thrusting in the scoop to the bottom of the sample layer and combine the 20 scoops of sample.

Figure 2 — Example of manual increment division method for gross sample

9.2.3.2 Increment or subsample

When an individual increment or subsample is divided, the division shall be carried out according to table 7. The sample shall not be divided further than the specified mass corresponding to the whole-through sieve size.

Table 7 — Minimum mass of divided individual increment or subsample for moisture determination and/or chemical analysis by manual riffle division method

Whole-through sieve size		Minimum mass of divided sample
Over	Up to and including	(kg)
22,4 mm	31,5 mm	150
16,0 mm	22,4 mm	50
10,0 mm	16,0 mm	30
5,00 mm	10,0 mm	10
2,80 mm	5,00 mm	5
1,00 mm	2,80 mm	3
500 µm	1,00 mm	2
250 µm	500 µm	1
	250 µm	0,1

9.2.4 Division limit for size sample

The division of the size sample shall be carried out according to table 8. According to variations from table 8 on the percentage of the size fraction, the minimum mass specified in the table shall be modified using the formula specified in 9.2.4.2.

In the case of variations from table 8 with respect to the type of the iron ore and specification size fraction, annex B shall be applied instead of the table.

9.2.4.1 Gross sample

When the gross sample is divided, the mass of the divided gross sample shall not be less than that specified in table 8.

NOTES

1 The recalculation procedure referred to in 9.2.4.2 was performed assuming an apparent density of 5 000 kg/m^3.

2 For the whole-through sieve size, 1 mm for pellet feed and 20 mm for pellets are used.

Table 8 — Minimum mass of divided size gross sample by manual riffle division method and precision of sample preparation

Type of iron ore		−31,5+6,3mm sized ore		Sinter feed		Pellet feed		Pellets	
Typical specification size fraction		−6,3 mm		+6,3 mm		+45 µm		−5 mm	
Average percentage of size fraction, P_0 (%)		10		10		30		5	
Mass of consignment, m_1 (t)		Minimum mass of divided gross sample, m_3 (kg)							
Over	Up to and including		β_{DM} (%)		β_{DM} (%)		β_{DM} (%)		β_{DM} (%)
270 000	—	120	1,6	8,0	1,5	0,5	0,15	250	0,44
210 000	270 000	110	1,7	7,0	1,6	0,5	0,15	240	0,44
150 000	210 000	110	1,7	7,0	1,6	0,5	0,15	240	0,44
100 000	150 000	110	1,7	7,0	1,6	0,5	0,15	230	0,45
70 000	100 000	95	1,8	6,0	1,7	0,5	0,15	215	0,47
45 000	70 000	95	1,8	6,0	1,7	0,5	0,15	215	0,47
30 000	45 000	85	1,9	5,0	1,9	0,5	0,15	210	0,48
15 000	30 000	75	2,0	5,0	1,9	0,5	0,15	210	0,48
5 000	15 000	60	2,3	4,0	2,1	0,5	0,15	145	0,57
2 000	5 000	40	2,8	2,5	2,7	0,5	0,15	85	0,75
1 000	2 000	30	3,2	2,0	3,0	0,5	0,15	60	0,89
500	1 000	25	3,5	1,5	3,5	0,5	0,15	50	0,97
	500	15	4,6	1,0	4,2	0,5	0,15	50	0,97

9.2.4.2 Modification

When the actual percentage of the size fraction is considerably different from that specified in table 8, the minimum mass specified in the tables shall be modified using the following equation derived from the binomial rule:

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

where

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

m_3 is the minimum mass of the divided gross sample specified in table 8;

P is the actual percentage of the size fraction, which is considerably different from that specified in table 8;

P_0 is the average percentage of the size fraction specified in table 8.

For example, for a consignment of 40 000 t of minus 31,5 mm sized ore in table 8, if the percentage of minus 6,3 mm fraction is about 20 %, the minimum mass of the divided gross sample to be applied shall be modified as follows:

$$m_4 = 85 \times \frac{20\,(100 - 20)}{10\,(100 - 10)} \approx 151 \text{ kg}$$

9.2.4.3 Increment or subsample

When each increment or each subsample is divided, the mass of divided increment or subsample shall not be less than that calculated according to annex B.

9.3 Coning and quartering method

When the sample to be divided is minus 10,0 mm in whole-through sieve size, the coning and quartering method shall be carried out according to the stipulations of 9.3.1 and 9.3.2.

9.3.1 Division limit

In dividing the moisture sample and sample for chemical analysis, the division shall be carried out according to tables 6 and 7, and in dividing the size sample, according to table 8.

9.3.2 Procedure

9.3.2.1 Mix the sample to be divided (minus 10,0 mm) on a flat, clean steel plate, by heaping it into a cone and turning over onto a different place three times. Form a conical heap by depositing each shovelful on top of the preceding one, taking care to place it on the apex of the cone; distribute the portions which slide down the sides as evenly as possible, taking care not to displace the centre of the cone.

Form a new cone twice in a similar way and take care to work steadily around the previous one until it is all transferred.

9.3.2.2 Spread the third cone in a flattened heap by repeated vertical insertions of the edge of a shovel or board, commencing at the centre and working radially around the cone, lifting the shovel or board clear of the sample after each insertion. Form a flattened heap having a uniform thickness and diameter, its centre coinciding with that of the original cone.

NOTE — The thickness of the flattened heap shall be such as to allow for a neat separation of the quarters, without the danger of remixing the ore in adjacent quarters.

9.3.2.3 Cut the flattened heap into quarters by two lines which intersect at right angles at the centre of the heap. Use a quartering iron, if available. Remove two diagonally opposite sectors and reject them. Take the remainder as a divided sample. If necessary, crush the remainder, mix, and heap into a cone after turning over three times. Select at random the first two opposite sectors that are rejected each time to avoid introducing any bias.

9.3.2.4 Repeat the procedures specified in 9.3.2.2 and 9.3.2.3.

10 Preparation of test samples

10.1 Preparation of test sample for size determination

The test sample for size determination shall be prepared from each increment, each subsample or the gross sample without crushing.

10.2 Preparation of test sample for moisture determination

10.2.1 The test sample for moisture determination shall be prepared from each increment, each subsample or the gross sample.

When increments are taken by time-basis sampling, the test sample shall be prepared specially from each subsample or the gross sample. In this case, each subsample and corresponding mass of its part of a consignment should be clearly shown.

10.2.2 The test sample for moisture determination may be prepared by splitting the sample for chemical analysis at the stage permitted such as minus 31,5 mm, minus 22,4 mm or minus 10,0 mm in particle size.

10.2.3 The moisture sample having a whole-through sieve size of greater than 31,5 mm shall be crushed to minus 31,5 mm, minus 22,4 mm or minus 10,0 mm in particle size.

A test portion of 10 kg minimum for minus 31,5 mm, 5 kg minimum for minus 22,4 mm, or 1 kg minimum for minus 10,0 mm shall be obtained from the test sample by any of the methods specified in 6.4, irrespective of the division limit.

NOTE — It is recommended that the test portion of minus 10,0 mm be checked for biased results (lower moisture content) against the test portion of minus 22,4 mm or that of 31,5 mm.

10.2.4 Moisture determination shall be conducted immediately after the sample preparation. When immediate determination is impossible, the test sample shall be packed tightly in a moisture-proof container and kept in an environment which has approximately constant temperature and humidity.

10.2.5 When the gross sample is to be subjected to the determination of moisture content, one test sample for moisture determination shall be prepared from the gross sample and then four test portions taken from that test sample.

When each subsample is to be subjected to moisture determination, one test sample shall be prepared from each subsample and then a number of test portions as given in table 9 taken from each test sample, according to the number of subsamples per consignment.

Table 9 — Number of test portions per test sample

Number of subsamples per consignment	Number of test portions to be taken per test sample
2	4
3 to 7	2 minimum
8 and over	1 minimum

When moisture measurement is to be carried out on each increment, at least one test portion per increment shall be prepared.

10.2.6 When a consignment is comparatively large, the increments consecutively taken from the consignment should be combined into a number of subsamples for moisture determination as indicated in table 10.

When it takes a long time for loading or unloading of a consignment, the said increments should be combined into a subsample every 8 h at longest and such a process should be continued until the handling operation is completed.

Table 10 — Minimum number of subsamples per consignment for moisture determination

Mass of consignment, m_1 (t)		Minimum number of subsamples per consignment	Number of test portions	Number of tests
Over	Up to and including			
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
5 000	15 000	2	4	8
	5 000	1	4	4

10.3 Preparation of test sample for chemical analysis

The test sample for chemical analysis should be prepared from each increment, each subsample or the gross sample (see figure 4).

10.3.1 Mass and particle size of test sample for chemical analysis

A test sample of 50 g minimum at minus 100 µm in particle size shall be prepared for ordinary ores.

However, for ores containing significant amounts of combined water and/or oxidizable compounds, a test sample of 100 g minimum at minus 160 µm in particle size shall be prepared.

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

Precautions may 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 to satisfy the attainment of whole-through sieve size.

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

10.3.2 Preparation of test sample for chemical analysis

The procedures for preparation of a test sample for chemical analysis shall be as follows.

10.3.2.1 Prepare a sample of 500 g minimum at minus 250 µm in particle size from each increment, each subsample or the gross sample according to the division methods specified in clause 9.

10.3.2.2 Then prepare the test sample for chemical analysis from the sample, prepared in 10.3.2.1, in any of the following three cases according to the characteristics of the ores and/or the facilities of the laboratories.

a) Case 1

Grind the sample in 10.3.2.1 to minus 100 µm in particle size, and from this sample, prepare a set of not less than four sealed test samples, each of 50 g minimum, by an appropriate division method as specified in clause 9.

This procedure is preferable to that in case 2.

b) Case 2

Grind the sample in 10.3.2.1 to minus 160 µm in particle size, and from this sample, prepare a set of not less than four sealed samples, each of 100 g minimum, by an appropriate division method. Then distribute each of the sealed samples to the laboratories concerned in 10.3.4 respectively, where the test sample of 50 g minimum for minus 100 µm in particle size shall be prepared from the sealed sample.

c) Case 3

Grind the sample in 10.3.2.1 to minus 160 µm in particle size, and from this sample, prepare a set of not less than four sealed test samples, each of 100 g minimum, by an appropriate division method. This applies to ores containing significant amounts of combined water and/or oxidizable compounds.

In the case where the preparation cannot be carried out without difficulty, the sample should be ground, after drying, to minus 100 µm or 160 µm in particle size.

The preparation of the test sample for chemical analysis is shown in figure 3.

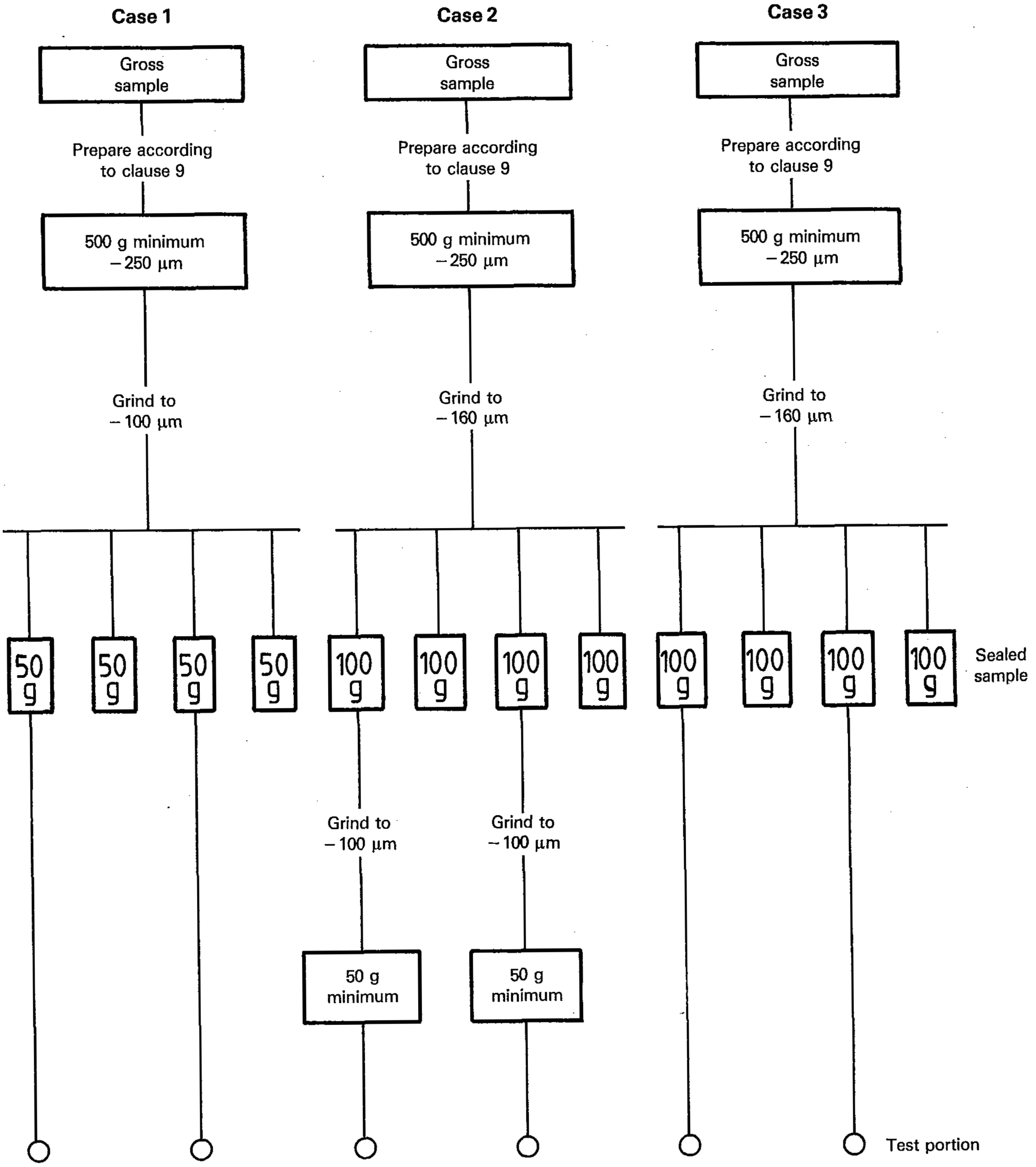


Figure 3 — Preparation of test samples for chemical analysis

10.3.3 Grinding to minus 100 µm or 160 µm

When a sample is ground to minus 100 µm or 160 µm from minus 250 µm in particle size, the following shall be taken into account.

10.3.3.1 Type of grinder

Several types of grinders may be used to grind the sample for chemical analysis from minus 250 µm to minus 160 µm or minus 100 µm, for example top grinder, disc grinder, pot mill, hammer mill, vibrating mill, and other suitable types of mill.

10.3.3.2 Selection of material of construction of grinder

The selection of material for the grinder is one of the most important considerations so that the chemical composition of the sample will 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.

10.3.3.3 Dry grinding

The whole mass of the sample for chemical analysis of 500 g minimum for minus 250 µm in particle size should be ground at one time to minus 100 µm or 160 µm, using an appropriate grinder. When the grinding of the sample cannot be carried out at one time, the grinding may be carried out on each part obtained, after dividing the sample into parts, and then after all the divided parts have been ground to minus 100 µm or 160 µm, they shall be mixed thoroughly by an appropriate mixer.

NOTES

1 The sample for finer grinding should not be screened into oversize and undersize fractions, for example plus and minus 100 µm fractions, in order to carry out the grinding on the oversize fraction only.

2 The impact-type mill should not be used for ore containing materials which have greatly different grindability from the iron ore constituent minerals, such as grains of quartz and fragments of shale, in order to avoid selective grinding.

10.3.3.4 Wet grinding

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

10.3.4 Distribution of samples for chemical analysis

The samples for chemical analysis to be distributed shall be placed in suitable containers, sealed, and clearly marked according to clause 11.

Of the samples prepared, one sample shall be provided for the seller, one for the purchaser, and one for the arbitrator and, if required, one to be held in reserve, or as agreed upon between the parties concerned. The reserved samples shall be retained for 6 months.

10.4 Example of sample preparation process

An example of the sample preparation process for moisture samples and samples for chemical analysis is shown in figure 4.

NOTE — The flow chart shown in figure 4 provides an example of sample preparation of lump ore, when a subsample comprises three increments and several subsamples compose the gross sample.

11 Packing and marking of sample

The samples for distribution shall be tightly sealed in airtight containers. The label and a card placed in the container shall contain the following particulars:

a) type of ore and name of consignment (name of ship, train, etc.);

b) mass of consignment;

c) sample number;

d) place, date and method of sampling;

e) moisture content of consignment;

f) place and date of sample preparation;

g) particle size of sample;

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

i) any other item (if necessary).

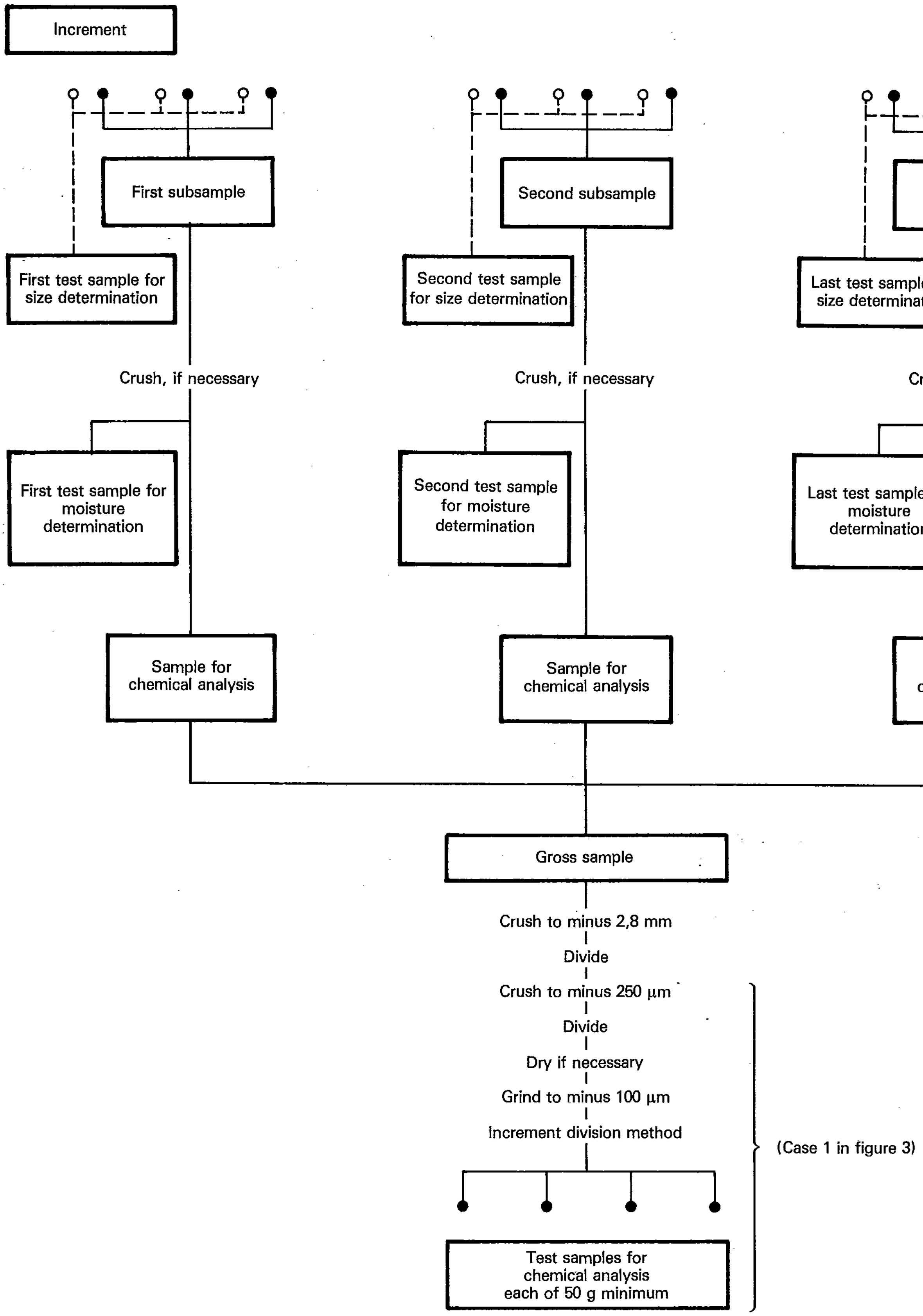


Figure 4 — Example of sample preparation

Annex A

Type of riffle divider

Table 11 — Dimensions of riffle divider

Riffle divider number			60	50	30	20	10	6
No. of riffles			12	12	12	16	16	16
Dimensions (mm)	Body	*A*	60 ± 1	50 ± 1	30 ± 1	20 ± 1	10 ± 0,5	6 ± 0,5
		B	760	630	380	346	171	112
		C	300	250	170	105	55	40
		D	600	500	340	210	110	80
		E	360	300	200	135	75	60
		F	60	50	30	30	20	20
		G	340	340	340	210	110	80
		H	230	200	140	85	45	30
		J	770	640	390	360	184	120
		K	240	220	220	140	65	55
	Receiver	*M*	240	220	220	140	65	55
		N	340	340	340	210	110	80
		P	300	250	170	105	55	40
		Q	80	75	55	35	20	15
		R	340	340	340	210	110	80
	Feeder	*S*	760	630	380	346	171	112
		T	400	400	300	200	120	80
		U	265	265	200	135	70	45
		V	200	200	150	105	50	35

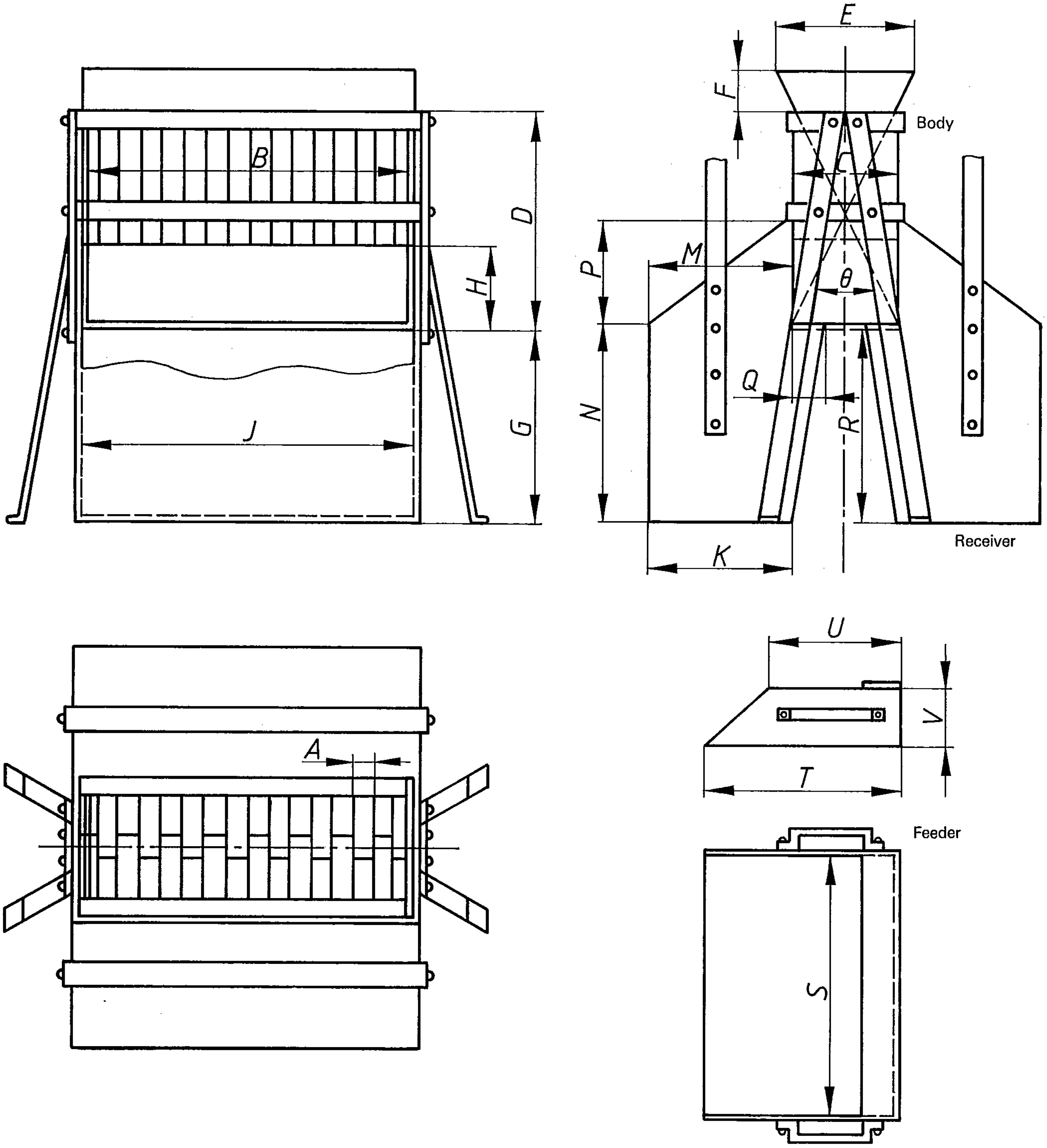
NOTES

1 *A* is the specified dimension. The other dimensions are shown as examples.

2 The number of riffles shall be even and not less than the number specified in the above table.

3 The sample receivers shall be fitted tightly to the opening of the divider to avoid scattering any fine particles.

4 The inside surface of the divider shall be smooth and free from rust.



NOTE — θ shall be 60° or less.

Figure 5 — Example of riffle divider

Annex B

Procedure for determining the minimum mass of divided size sample by manual riffle division method

This annex specifies the procedure for determining the minimum mass of the divided size sample by the manual riffle division method in the case of variations from table 8 in the type of iron ore and specification size fraction.

When the size sample is to be divided, the minimum mass, m_3, in kilograms of divided gross sample is given by equation (B1)

$$m_3 = \frac{k}{\beta_{DM}^2} \times \frac{\varrho}{5} \qquad \text{... (B1)}$$

where

β_{DM} is the precision, expressed as a percentage, for sample preparation and testing;

ϱ is the apparent density, expressed in tonnes per cubic metre, of the sample;

k is a value depending on the maximum particle size, the specification sieve size and the percentage of the size fraction, given by equation (B2):

$$k = 2{,}5 \times 10^{-5} P (100 - P) d^3 (l/d)^{0,5} \qquad \text{... (B2)}$$

in which

P is the percentage of the size fraction;

d is the maximum particle size, in millimetres, of the sample to be divided;

l is the specification sieve size, in millimetres.

When each increment or each subsample is to be divided, the minimum mass, m_5, in kilograms, of divided increment or subsample for size determination is given by equation (B3):

$$m_5 = \frac{m_3}{n_1} \qquad \text{...(B3)}$$

where n_1 is the number of increments or subsamples.