102

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Iron ores — Preparation of samples

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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.

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It has been approved by the Member Bodies of the following countries :

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Brazil
Canada
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India

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Iron ores — Preparation of samples

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the method of preparing samples of iron ore for the determination of size distribution, moisture content and chemical composition. The prescribed methods are applicable to all iron ores, whether natural or processed (pellets, concentrates and agglomerates).[1)]

2 REFERENCES

This document should be read in conjunction with the following International Standards :

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

ISO 3082, *Iron ores — Increment sampling — Mechanical method.*[2)]

ISO 3084, *Iron ores — Experimental methods for evaluation of quality variation.*[2)]

ISO 3085, *Iron ores — Experimental methods for checking the precision of sampling.*[2)]

ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling.*[3)]

ISO 3087, *Iron ores — Determination of moisture content.*[3)]

ISO . . ., *Iron ores — Determination of size distribution by sieving.*[2)]

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 : A quantity of an ore obtained by a sampling device at one time from a consignment; also a quantity taken in the increment division method.

3.4 sub-sample : A quantity of an ore consisting of several increments taken from a part of the consignment; also a composite of several increments after having been crushed and divided individually.

3.5 gross sample : The quantity of an ore consisting of all of the increments taken from a consignment; also the composite of all of the increments or all of the sub-samples after having been crushed and divided individually.

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

3.7 final sample : Any sample for determination of size distribution, moisture content, chemical composition or other physical characteristics, 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.

3.8 size sample : The sample taken for the determination of size distribution of the consignment or part of the consignment.

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

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

3.11 sample for physical testing : The sample taken for the determination of physical characteristics of the consignment or part of the consignment.

1) The preparation of samples for physical testing will be specified if necessary.
2) In preparation.
3) At present at the stage of draft.

4 GENERAL RULES

4.1 The gross sample, sub-samples or increments for the required determinations shall be taken according to ISO 3081 or ISO 3082.

4.2 Samples for moisture determination and chemical analysis shall be prepared separately. Moisture content should be determined immediately.

4.3 Care shall be taken not to change the quality of the sample during crushing. The crusher shall be cleaned before crushing the ores.

4.4 The division shall be conducted by the method which satisfies the planned precision without bias within the specified limits of division in section 6.

4.5 When the sample is very wet or sticky and the sample preparation cannot be conducted, pre-drying may be carried out in accordance with section 9. If necessary, the pre-drying moisture content shall be determined.

4.6 When a sample contains a considerable amount of metallic iron, due care shall be given to the handling of the sample at the time of sample preparation.

4.7 Throughout all the procedures of sample preparation, the sample shall be protected from any alteration.

5 SAMPLE PREPARATION IN GENERAL

5.1 Procedure of sample preparation

5.1.1 *Preparation of size sample*

Each increment, each sub-sample or the gross sample taken for size determination shall be used and the size determination shall be carried out in accordance with the method specified in ISO . . ..

5.1.2 *Preparation of moisture sample*

1) When a gross sample is taken for a consignment, at least four final moisture samples shall be prepared.

NOTES

1 When a consignment is very large, it is recommended that a moisture sub-sample be prepared and subjected to moisture determination for each part of the consignment divided into the number of parts indicated in Table 1. This is in order to obtain not only a better precision (including sampling, preparation and moisture determination) but also a result that has no bias.

2 When it takes a long time for loading or unloading of a consignment, the consignment shall be divided for each 8 h handling, and a moisture sub-sample shall be constituted and subjected to moisture determination for each part thus obtained. Such division shall be subject to the condition of weather, for example heavy rain, high temperature, etc. and/or to the conditions or circumstances at the time of loading or unloading, and shall be decided by agreement between the parties concerned.

TABLE 1 — **Minimum number of parts per consignment**

Mass of consignment tonnes		**Minimum number of parts per consignment**
Over	Up to and including	
70 000	150 000	10
30 000	70 000	5
15 000	30 000	3
5 000	15 000	2
	5 000	1

2) The moisture sample shall always be stored in an air-tight container in order to avoid any change of moisture when sample preparation is not carried out immediately.

3) after crushing each increment, each sub-sample or the gross sample to minus 22,4 mm or minus 10 mm, the final moisture sample of 5 kg or 1 kg or more shall be obtained immediately. The remainder of the sample shall be divided and prepared for chemical analysis.

NOTE — Some moisture may be lost from the sample in the course of crushing from minus 22,4 mm to minus 10 mm in the case of a moderately wet sample. However, if no bias is confirmed by check experiments on a specific ore, the moisture sample of 1 kg or more may be obtained by crushing the sample to minus 10 mm.

4) It is recommended that moisture samples be prepared by the increment division method, in order to avoid moisture evaporation. Any other method may be used, if it can be confirmed that there is no bias in the result obtained by the method.

5) The mass of the final moisture sample shall be determined immediately. When the immediate determination of the mass is not possible, the sample shall be packed tightly in a moisture-proof container and kept in a place in which temperature and humidity have the least effect.

6) It shall be indicated which part in mass of the consignment is represented by which increments or sub-samples.

5.1.3 *Preparation of sample for chemical analysis*

1) Each increment, each sub-sample or the gross sample shall be crushed to minus 500 μm in size by repeating crushing and division according to section 6.

When the division is conducted on an individual increment or sub-sample before the formation of a gross sample, the gross sample shall be obtained, at some stage of the division, by combining quantities proportional to the mass of the individual increment or sub-sample.

2) The sample of minus 500 μm in size shall be ground to minus 160 μm in size.

NOTE — If such grinding equipment is not available, the sample may be ground to minus 250 μm and a sample of more than 500 g shall be taken by the increment division method. This sample shall be ground to minus 160 μm in size.

3) From this sample, not less than four sets of final analysis samples, each of 100 g or more, shall be prepared by the increment division method. Three of the samples are intended for the seller, the purchaser, and arbitration, and one is to be held in reserve.

The final reserved sample shall be retained for 6 months.

4) The final analysis samples shall be placed in suitable containers, sealed, and clearly marked in accordance with 5.2.

An example of the sample preparation process for moisture samples and samples for chemical analysis and physical testing is shown in Figure 1.

NOTES

1 The flow chart is an example of sample preparation of lumpy ore, when a sub-sample comprises three increments and several sub-samples compose a gross sample.

2 When the mass of a consignment and handling speed of the consignment are very large and each increment is subject to mechanical sampling plant, the sample for various determinations may be prepared from the size sample, upon confirmation that no bias will be introduced into each quality characteristic by the procedure.

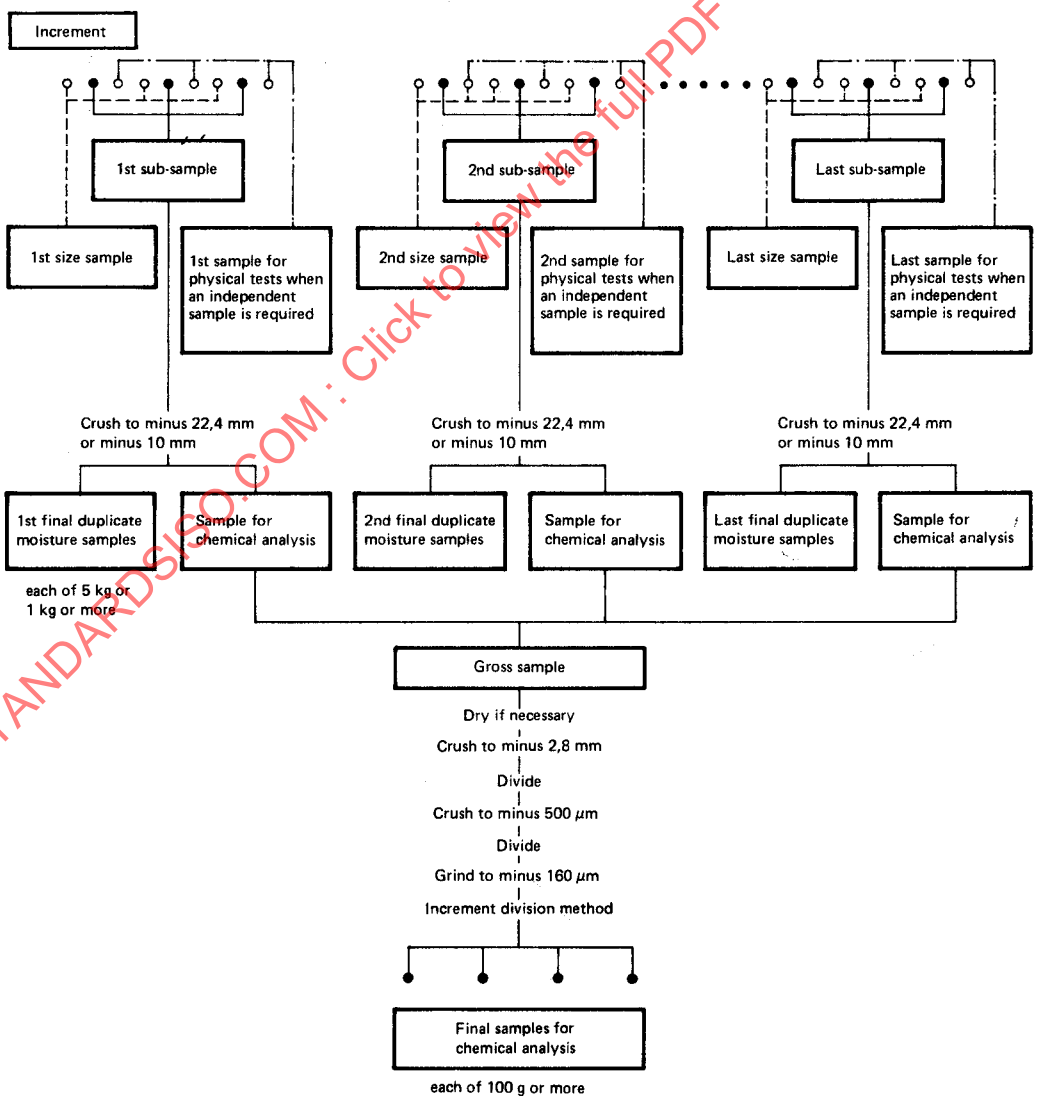


FIGURE 1 — **An example of sample preparation**

5.2 Packing and marking of sample for chemical analysis

The final samples shall be tightly sealed in air-tight containers. The following information shall be shown on the label and on a card placed in the container :

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

2) mass of the consignment;

3) sample number;

4) place and date of sampling;

5) moisture content of the consignment;

6) place and date of sample preparation;

7) particle size of the sample;

8) any other item (if necessary).

5.3 Precision of sample preparation

This International Standard is designed to obtain the precision of preparation within ± 0,4 % (absolute percent in iron or moisture content) with 95 % probability.

NOTE – If a better overall precision is required, the quality characteristics of each individual increment or each sub-sample shall be determined by agreement between the parties concerned.

6 SAMPLE DIVISION

6.1 Method of division

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

1) increment division method;

2) division by riffle divider;

3) coning and quartering method (for size determination, the method specified in ISO . . . shall be used);

4) division by mechanical dividing apparatus.

The sample division shall be conducted on the sample crushed to an appropriate particle size which is specified in 6.2, 6.3, 6.4 and 6.5.

6.2 Increment division method

This is a method of obtaining a specified precision in spite of the high division ratio. However, it is better to avoid this method on certain samples such as pellets, which segregate easily. When the pellets have been crushed to a sufficiently small particle size, this method may be applied satisfactorily.

6.2.1 *Size (volume) of increment*

The size of increment shall be as specified in Table 2.

TABLE 2 – **Whole-through sieve size of sample and size (volume) of increment**

Whole-through sieve size up to and including	Minimum size (volume) of increment ml	Thickness of layer mm
22,4 mm	250	50 to 60
10 mm	100	30 to 40
5 mm	60	25 to 35
2,8 mm	30	20 to 30
1 mm	10	10 to 20
250 μm	2	5 to 10

6.2.2 *Number of increments*

1) Division of one gross sample from one consignment : a minimum of 20.

2) Division of individual sub-sample : a minimum of 10 but 20 or more are advisable.

3) Division of individual increment : a minimum of 4.

6.2.3 *Procedure*

The division of the gross sample by the manual increment division method shall be carried out according to the following procedures (for example where 20 parts are required) :

1) Spread the crushed sample on a smooth and flat plate (non-moisture absorbing) into a uniform flat rectangle with the thickness of the layer as specified in Table 2.

2) Arrange the rectangle in five equal parts lengthwise and four equal parts breadthwise.

3) According to the whole-through sieve size, select the appropriate shovel with a flat bottom as specified in Table 3, take a shovelful of sample from each part arranged as in 2) (the place of taking such an increment being selected at random in each part), and combine the 20 shovelfuls of sample to form the divided sample.

The shovel shall be inserted to the bottom of the sample layer in the above procedure. It is recommended that a bumper plate be inserted vertically in front of the shovel, which shall be inserted to the bottom of that sample layer in order to scoop an increment without bias. The moisture and analysis samples shall be prepared by repetition of the above procedure.

Figure 3 illustrates an example of sample division by the manual increment division method.

NOTE – When the quantity of the divided sample may become smaller than that required by testing, the size of increment shall be made larger beforehand.

TABLE 3 — **Relation between whole-through sieve size of sample and specification of shovel for increment division**

Whole-through sieve size up to and including	Shovel number	Dimensions of shovel mm					a/c	b/c	Volume (approx.) ml
		a	*b*	*c*	*d*	Thickness of metal sheet			
22,4 mm	22,4 R	80	45	80	70	2	1,0	0,56	300
15 mm	16 R	70	40	70	60	2	1,0	0,57	200
10 mm	10 R	60	35	60	50	1	1,0	0,58	125
5 mm	5 R	50	30	50	40	1	1,0	0,60	75
2,8 mm	2,8 R	40	25	40	30	0,5	1,0	0,62	40
1 mm	1 R	30	15	30	25	0,5	1,0	0,50	15
250 μm	0,25 R	15	10	15	12	0,3	1,0	0,67	2

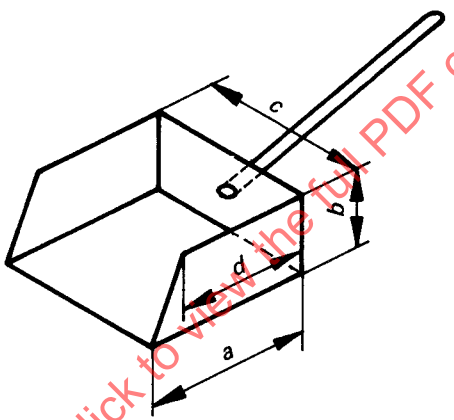
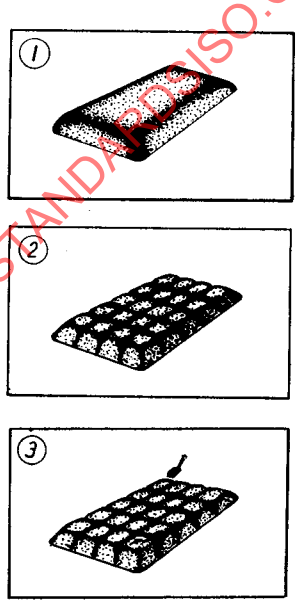


FIGURE 2 — **Shovel for increment division**



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

2) Arrange in 20 equal parts; for example into 5 equal parts lengthwise and 4 equal parts breadthwise.

3) Take a shovelful of sample from each of the 20 parts by inserting the shovel to the bottom of the sample layer and combine the 20 shovelfuls of sample into a divided sample.

FIGURE 3 — **Manual increment division method**

6.3 Division by riffle divider

6.3.1 *Selection of riffle divider*

1) The riffle divider shall be of the type shown in the Annex.

2) According to the whole-through sieve size of the sample, the appropriate riffle divider specified in Table 4 shall be selected. However, riffle dividers having opening widths greater than those specified in Table 4 may be used provided that the width of each opening does not exceed three times the whole-through sieve size.

TABLE 4 — **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		
16	22,4	50	50 ± 1
10	16	30	30 ± 1
5	10	20	20 ± 1
2,8	5	10	10 ± 0,5
	2,8	6	6 ± 0,5

6.3.2 *Division of individual increment or sub-sample*

When an individual increment or sub-sample is divided, the division shall be carried out in accordance with Table 5. The sample shall not be divided further than the mass corresponding to the whole-through sieve size which is specified in Table 5.

TABLE 5 — **Rule for division of individual increment or sub-sample (in case of method other than increment division method)**

Whole-through sieve size up to and including	**Minimum mass of divided sample** kg
22,4 mm	100
10 mm	10
5 mm	5
500 μm	1

6.3.3 *Division of gross sample*

When a gross sample is divided, the division shall be carried out in accordance with Table 6. The gross sample shall not be divided further than the mass corresponding to the whole-through sieve size which is specified in Table 6.

TABLE 6 — **Rule for division of gross sample (in case of method other than increment division method)**

Whole-through sieve size up to and including	**Minimum mass of divided sample** kg
10 mm	50
5 mm	25
2,8 mm	15
1,4 mm	10
500 μm	5

6.3.4 *Procedure*

1) Place the sample 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). In order to avoid any systematic error, select at random one of the two divided samples.

2) Care must be taken not to leave any material retained in the slots of the riffle, and shaking of the container shall be carried out in such a manner as to prevent any bias in the division.

6.4 Coning and quartering method

6.4.1 *Division of sample*

The division shall be carried out in accordance with 6.3.2 for individual increment or sub-sample and 6.3.3 for gross sample.

6.4.2 *Procedure*

1) The sample shall be crushed to minus 10 mm and thoroughly mixed on a flat, clean steel plate, by heaping into a cone and turning over onto a different place three times. The conical heap shall be formed by depositing each shovelful on top of the preceding one, taking care to place it on the apex of the cone, so that the portions which slide down the sides will be distributed as evenly as possible and that the centre of the cone will not be displaced.

A new cone shall then be formed twice in a similar way, taking care to work steadily around the previous one until it is all transferred.

2) The third cone shall now be flattened by repeated vertical insertions of the edge of a shovel or board commencing about the centre and working radially around the cone, lifting the shovel or board clear of the sample after each insertion. The flattened heap shall be of uniform thickness and diameter, and its centre shall coincide with the centre of the original cone.

3) From the flattened heap, the moisture sample shall be taken by the increment division method. The flattened heap shall be cut into quarters by two lines

which intersect at right angles at the centre of the heap. If available, a quartering iron should be used. Two diagonally opposite sectors shall be completely removed and rejected and the remainder shall be further crushed if necessary (see Table 5 or 6), mixed and heaped into a cone after turning over three times. The first two opposite sectors that are rejected each time shall be selected at random to avoid any systematic error.

4) The procedures mentioned in 2) and 3) shall be repeated.

6.5 Division by mechanical dividing apparatus

The rule of division shall be as specified in Tables 5 and 6. It is necessary to check the apparatus beforehand by check experiments to find whether there is no bias in it or whether the planned precision is satisfied by the apparatus. Check experiments shall be conducted against riffle divider method for iron content and particle size distribution and against increment division method for moisture content with reference to ISO 3086 and ISO 3085.

7 METHOD OF CRUSHING AND GRINDING

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

7.2 The inside of the crusher and grinder shall be cleaned well before charging samples, and examined for contamination and wear.

7.3 After the completion of crushing, there shall be no remnant in the crusher.

8 METHOD OF MIXING

8.1 By mixing the sample thoroughly, it may be made homogeneous and consequently the error in sample division can be decreased.

8.2 The mixing may be conducted either by a mechanical mixer or by hand.

8.3 The mixer shall be selected to suit the sample and particle size.

9 METHOD OF PRE-DRYING

1) The pre-drying mentioned in 4.5 shall be conducted by air-drying below the temperature where there is likely to be a change in quality.

When pre-drying is applied to normal iron ores, it shall be conducted at 105 °C or less.

If necessary, the pre-drying moisture contents shall be determined according to ISO 3087.

2) The air-drying shall be conducted by spreading the sample on a plate in a thin layer and repeating mixing and scattering occasionally until the sample preparation can be carried out without trouble.

ANNEX

TYPE OF RIFFLE DIVIDER

TABLE 7 — **Dimension of riffle divider**

Riffle divider number		50	30	20	10	6
Number of riffles		12	12	16	16	16
Dimensions mm	*A*	50 ± 1	30 ± 1	20 ± 1	10 ± 0,5	6 ± 0,5
	B	630	380	346	171	112
	C	250	170	105	55	40
	D	500	340	210	110	80
	E	300	200	135	75	60
	F	50	30	30	20	20
	G	340	340	210	110	80
	H	200	140	85	45	30
	I	640	390	360	184	120
	J	220	220	140	65	55
	K	220	220	140	65	55
	L	340	340	210	110	80
	M	250	170	105	55	40
	N	75	55	35	20	15
	O	340	340	210	110	80
	P	630	380	346	171	112
	Q	400	300	200	120	80
	R	265	200	135	70	45
	S	200	150	105	50	35

NOTES

1 *A* is the specified dimension. The other dimensions are shown for example.

2 θ shall be 60° or less.

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

4 The sample receivers shall be fitted tightly to the opening of the divider to avoid scattering of fine powders.

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