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Aluminium ores — Preparation of samples

Minerais alumineux — Préparation des échantillons

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Foreword

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

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

International Standard ISO 6140 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*, Sub-Committee SC 1, *Sampling*.

Annex A forms an integral part of this International Standard. Annexes B, C, D, E and F are for information only.

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Aluminium ores — Preparation of samples

1 Scope

This International Standard specifies methods of treatment of gross samples and subsamples of aluminium ores with a view to the preparation of samples for the determination of moisture, chemical analysis and physical testing.

The methods specified are applicable to all aluminium ores.

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 6138:1991, *Aluminium ores — Experimental determination of the heterogeneity of constitution.*

ISO 8685:—¹⁾, *Aluminium ores — Sampling procedures.*

ISO 9033:1989, *Aluminium ores — Determination of the moisture content of bulk material.*

3 Definitions

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

1) To be published.

3.1 chemical analysis sample: The sample crushed to pass a sieve aperture of 150 μm used for the determination of chemical characteristics of the ore.

3.2 division: The process of decreasing the sample mass (without modification of the particle size of the constituent parts) where a representative part of the sample is retained while rejecting the remainder.

3.3 gross sample: The quantity of an ore consisting of all of the increments or divided increments or subsamples or divided subsamples taken from a lot.

3.4 moisture sample: A sample used for the determination of moisture content of the lot or sampling unit.

3.5 nominal top size: The size of aperture of the finest sieve (complying with ISO 565) through which a minimum of 95 % of the mass of the material passes.

3.6 pass: The passage of the retained material once through a sample divider.

3.7 reduction: The process whereby a sample is reduced in particle size by crushing or grinding without a change in its mass or composition.

3.8 physical testing sample: A sample taken for the measurement of physical characteristics.

3.9 stage: A sequence of operations comprising reduction in particle size, mixing and culminating in sample division. The number of stages in sample preparation is equal to the number of divisions made.

4 Fundamentals of sample preparation

4.1 Introduction

Sample preparation involves several distinct operations, which are sometimes preceded by drying. These are as follows:

- a) reduction;
- b) mixing — to achieve homogeneity;
- c) division.

These three operations are generally considered to constitute only one stage of sample preparation.

As a general rule, reduction should always precede division. An exception occurs when high capacity streams are being sampled mechanically and it is permissible to divide large primary increments as specified in ISO 8685.

The procedures of sample preparation should be chosen to minimize errors of sample preparation without having to retain too large a mass. Examples of sample preparation schemes are given in annex A.

4.2 Minimum mass of sample to be retained after division

The amount of sample to be retained depends upon the nominal top size of the ore at that stage.

The recommended procedure for sample preparation should involve two or more stages, and the minimum mass of ore retained after each stage shall not be less than m_G , where

$$m_G = \left(\frac{C_v}{\sigma_s} \right)^2 \rho g D^3 \times 10^{-6}$$

where

- m_G is the minimum gross sample mass, in kilograms;
- C_v is the coefficient of variation between particles of the quality characteristic under investigation, determined in accordance with ISO 6138;
- σ_s is the required sampling error (standard deviation);
- ρ is the density, in tonnes per cubic metre, of the ore particles (not bulk density);
- g is the size range factor indicated in table 1;

D is the nominal top size, in millimetres, of the ore in the lot.

Table 1 — Size range factors

Size range	Value for g
Large size range ($D/D' > 4$)	0,25
Medium size range ($4 \geq D/D' \geq 2$)	0,50
Small size range ($D/D' < 2$)	0,75
Uniform size ($D/D' = 1$)	1,00
where D' is the sieve size retaining 95 % of the ore.	

4.3 Drying

For materials other than those used for moisture determination, the material may be air dried or oven dried to ensure that it can be handled freely without loss or contamination. No sample shall be exposed to a temperature exceeding 105 °C. The method for treatment of moisture samples is specified in clause 7.

4.4 Particle size reduction

4.4.1 General

The purpose for which the sample is to be used will determine whether it is permissible to reduce the particle size in the course of sample preparation. Several cases are considered in 4.4.2 to 4.4.4.

4.4.2 Chemical analysis sample

The ore shall invariably require size reduction to meet the specifications of 8.2.2. The sample may be reduced as is appropriate to facilitate the operations of division described in 4.6.

It is permissible, when handling samples intended for chemical analysis, to dry the material as received so that handling characteristics are improved. When drying is used, the following precautions should be observed:

- a) contamination or physical loss must be avoided;
- b) the sample shall not be heated to a temperature at which combined water or any other volatile component is lost. The maximum temperature reached by any portion of the sample shall not exceed 105 °C.

4.4.3 Moisture sample

One with a nominal top size of less than 22,4 mm shall not be reduced in particle size prior to moisture determination.

When handling ore with a nominal top size of greater than 22,4 mm, and which is not adhesive or excessively wet, the ore may be reduced in particle size to less than 22,4 mm, care being taken to minimize any change in moisture content.

When handling ore with a nominal top size of greater than 22,4 mm, and which is not adhesive or excessively wet, the total sample may be weighed and air dried. When it has dried to a free-flowing state, the sample shall be re-weighed. The sample may then be reduced in particle size prior to division and determination of residual moisture in accordance with ISO 9033.

When this procedure is followed, the final moisture value shall take into account the moisture loss in the air-drying stage.

4.4.4 Physical testing sample

Size reduction is not permitted when samples are to be used for

- a) determination of particle size distribution;
- b) determination of bulk density.

4.4.5 Particle size reduction apparatus

Throughout this International Standard the machines used for particle size reduction are referred to as "mills".

Particle size reduction mills used for aluminium ore samples include types such as jaw crushers, roll crushers, plate mills, hammer mills (impact pulverizers), and ring mills, the latter being preferred equipment for the final stages of grinding to a nominal top size of 150 μm . Examples of equipment are given in annex F.

Those parts of the apparatus which come into contact with the ore shall be of wear-resistant material to minimize contamination. This is particularly important with samples in which trace elements are to be determined and every effort should be made to use equipment which does not contain any of the elements to be determined.

The sample shall be fed uniformly into mills in such a way that choking of the mill or changes in mill speed, which may result in variation in the size distribution of the product, are avoided.

Errors of sample division and analysis are increased by the presence of oversize material, and mill performance should be checked regularly to ensure that the mill product meets the required nominal top size.

Certain types of particle size reduction apparatus, such as high speed impact pulverizers, ring mills

and plate mills, tend to become heated and samples must not be allowed to remain in them long enough to become affected. If a mill is used for a series of samples, it shall be cooled between each operation.

High speed impact pulverizers may be seriously damaged by the presence of hard extraneous material in the sample and precautions must be taken to prevent such material from entering the mill. The use of a magnetic separator is not permissible due to the magnetic susceptibility of bauxite ores.

All mills should be easy to clean and shall be cleaned between each sample.

4.5 Sample mixing

4.5.1 General

Errors in sample division can be reduced by thorough mixing of the sample prior to division. The need for mixing is particularly important when samples from more than one source are combined. Some methods of mixing, for example forming and reforming into a conical pile or rotary methods of mixing, may have the opposite effect to that intended, leading to increased segregation. Consequently, the choice of the most appropriate method of mixing is critical, and depends on the nature of the ore.

NOTE 1 Except when a moisture sample is to be taken, samples that are not free-flowing should be dried at a temperature not exceeding 105 °C before mixing. Where samples are taken for moisture determination, mixing is to be carried out as quickly as possible to minimize changes in moisture content.

4.5.2 Methods of mixing

Mixing can be carried out using any of the methods described in 4.5.2.1 to 4.5.2.3.

4.5.2.1 Passing the sample through a riffle or rotary sample divider at least three times in succession, recombining the sample after each pass.

4.5.2.2 Strip mixing. In this case, the ore is formed into a strip by careful distribution of the ore from a shovel. The length/width ratio of the strip shall be not less than 10 : 1. A complete cross-section of the ore strip is taken randomly and spread out to form a new strip. Successive cross-sections are taken randomly and spread out on top of the preceding cross-section, layer upon layer, until the old strip has been converted into a new strip. The process of taking cross-sections and reforming a new strip is repeated twice.

4.5.2.3 The use of mechanical mixers. Examples of mechanical mixers are shown in annex B.

4.6 Division

4.6.1 General

Sample division may be carried out by a variety of mechanical or manual methods. The minimum number of increments shall be 20.

Sample division may be carried out in as many passes as needed to obtain the required divided sample mass; for example, when using a riffle, three passes are required to obtain a one-eighth fraction.

Examples of sample division apparatus are illustrated in annex C. When division is performed for the purpose of extracting moisture samples, the preferred methods are mechanical division (see 4.6.2) or increment division (see 4.6.3.2).

4.6.2 Mechanical sample division

4.6.2.1 General

The main advantage of mechanical sample division is that the divided sample is obtained by taking a much larger number of increments than is generally obtainable manually. The minimum number of increments shall be 20.

The design criteria for mechanical sample dividers shall be as specified in ISO 8685.

4.6.2.2 Mechanical sample dividers

Examples of acceptable mechanical sample dividers are given in 4.6.2.2.1 to 4.6.2.2.6

4.6.2.2.1 Rotating cone (figure C.1)

This machine consists of a feed hopper, a low speed slotted rotating cone, a reject chute and a sample pipe. Ore is allowed to flow from the feed hopper onto the rotating cone and is diverted into the reject chute. The slot in the cone allows the ore to fall directly into the sample pipe for part of each revolution.

4.6.2.2.2 Rotary sample divider (figure C.2)

This machine comprises a number of sector-shaped canisters positioned on a platform, and a feeding device. The uniform ore stream flows to the hopper spout and by relative rotation of these two components the flow is intercepted by the top edge of the sector-shaped canisters, dividing the sample into representative parts.

4.6.2.2.3 Slotted belt (figure C.3)

An endless conveyor belt having slots spaced at equal pitch with lips which act as cutting edges. The ore is fed in a uniform stream through a feed chute onto the belt. As each slot passes through the ore stream, an increment falls to sample. The remainder of the stream is carried over the head pulley to reject.

4.6.2.2.4 Rotating chute (figure C.4)

A hollow shaft, to which is attached one or more cutters, rotates in an offset, cone-shaped housing. A feed pipe is positioned above the rotating cutter. As ore falls through the feed pipe, the cutter intersects the stream and diverts an increment through the hollow shaft.

4.6.2.2.5 Diverter chute (figure C.5)

A traversing chute is positioned at the head end of a belt feeder such that it will intercept the flow of ore as it is discharged over the head pulley. The chute diverts an increment to sample at each pass.

4.6.2.2.6 Bottom dump cutter (figure C.6)

A bucket type cutter with an opening bottom is arranged to traverse a falling stream of ore and travel to a position over a sample discharge chute where the bottom is opened to discharge the increment.

4.6.3 Manual sample division

4.6.3.1 Procedure

Sample division may be carried out using one of the following procedures:

- a) increment division;
- b) coning and quartering;
- c) riffling;
- d) fractional shovelling;
- e) strip mixing and splitting.

4.6.3.2 Increment division

Mix the ore thoroughly and spread it on a flat plate in the form of a rectangle of uniform thickness. The thickness shall be dependent on the nominal top size and is indicated in table 2.

Table 2 — Relationship between nominal top size, minimum increment mass and thickness of flattened sample

Minimal top size mm	Minimum mass of increment kg	Thickness of flattened sample mm
≤ 11,2	0,14	30 to 35
16,0	0,40	40 to 50
22,4	1,10	55 to 65
31,5	3,00	80 to 90
45,0	8,70	110 to 120

Mark a matrix, of minimum 5 × 4, on the spread sample (see figure 1) and collect one increment of the minimum mass given in table 2 from each part of the matrix with the appropriate flat-bottomed scoop chosen from the series shown in annex D.

Insert a flat bump plate vertically through the spread ore until it comes into contact with the mixing plate. Then insert the scoop to the bottom of the spread ore and take the increment by moving the scoop horizontally until its open end comes into contact with the bump plate, ensuring that all ore particles are collected off the top of the mixing plate. Lift the scoop and bump plate together so that ore is prevented by the bump plate from falling from the open end of the scoop.

If the mass of the combined increments is less than that determined in accordance with the indications of 4.2, further complete sets of increments shall be collected until the minimum mass is exceeded.

NOTES

2 This method is regarded as a high precision method in spite of the large division ratio, i.e. the ratio of the total

sample mass to the retained sample mass. It is a recommended manual method for obtaining moisture samples.

3 This division process may be used for ore having a nominal top size up to 45 mm.

4 The matrix should be designed in conjunction with table 2 to give the total amount of sample required for subsequent analysis. The ratio of the length to the width should never be more than 5 : 4.

4.6.3.3 Coning and quartering (see figure 2)

Spread the ore on a smooth steel plate. Pile the ore into a conical heap using a shovel, each shovelful being taken in sequence from the periphery of the original heap and dropped directly onto the apex of the new heap. Repeat two or three times. Drag the material down by means of a shovel so as to form first a truncated cone, then a flat circular cake. Divide the flat circular cake into quarters using a steel cross.

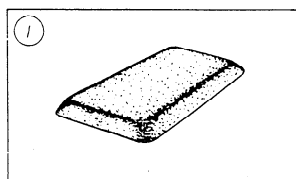
With the steel cross still in place, take two diagonally opposite quarters to form the sample and reject the remaining two quarters. The choice of the two quarters should be made at random.

If further reduction of the quantity of material is required, the process is repeated until the required mass is obtained.

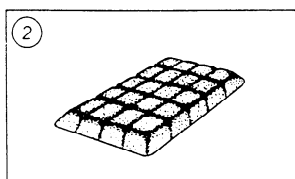
NOTES

5 This technique may be applied to the division of ore of nominal top size less than 45 mm and of sample mass of less than 1 t.

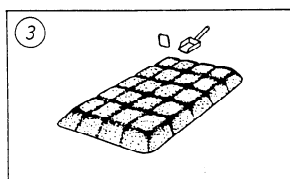
6 The method is not recommended for obtaining a moisture sample.



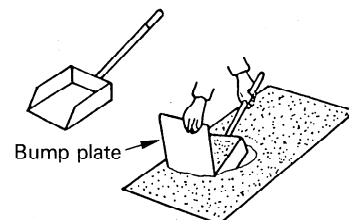
Spread the crushed gross sample into a rectangle with a thickness as specified in table 2.



Arrange in 20 equal parts, e.g. into 5 equal parts lengthwise and 4 equal parts breadthwise.



Take a scoopful of sample at random from each of the 20 parts by inserting the scoop to the bottom of the sample layer and combine the 20 scoopful of sample into a divided sample.



Outline of taking an increment by using a bump plate shown in (3)

Figure 1 — Manual increment division (Example for arranging in 20 parts)

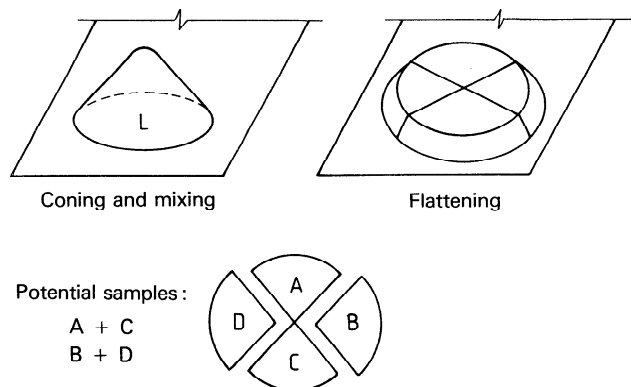


Figure 2 — Sampling by coning and quartering

4.6.3.4 Riffing

A riffle is a sample divider which is used to divide the ore fed onto it into halves, one being retained and the other rejected. It operates by allowing the ore to fall through a set of parallel slots of uniform width, adjacent slots feeding opposite containers.

A riffle shall be symmetrical (so that a part-sample may be taken from either side) and all surfaces on which ore might rest should be inclined at not more than 30° to the vertical. Receivers should fit closely against the body of the riffle, to minimize loss of dust. It is essential that the riffle used be appropriate for the nominal top size of the ore to be divided, as serious errors may be introduced if the slots are too small or there are too few.

The slot width shall be at least twice the nominal top size of the ore. There shall be at least eight slots for each half of the riffle.

Riffing shall be carried out as follows.

Mix the ore and place it in a feed container. Spread the ore in the feed container so that it is spread uniformly along the full length of the container. Tip the container to feed the ore uniformly into the feed chute, so that the ore passes through the riffle, and collect it in two canisters. Retain the sample from one of the receiving canisters, chosen at random.

NOTES

7 If the ore is found to choke the riffle, it should be cleared before the operation is continued.

8 If further subdivision is required, the retained sample may be passed through the riffle again.

Care should be taken to ensure that loss of moisture from a moisture sample is minimized when division is being carried out using a riffle. Closed riffles shall

be used for dividing moisture samples, or for dry ores to prevent loss of dust. Examples of riffles are given in annex E.

4.6.3.5 Fractional shovelling (see figure 3)

Mix the ore and form a conical heap (L). Using an appropriate shovel as shown in figure D.2, take successive shovelsful from the base of the heap, working around the base. Place the shovelsful on separate successive heaps, the number of heaps being determined by the division ratio. For example, if a 1 in 5 division ratio is required, five heaps, N_1 to N_5 , are formed as shown in figure 3. Select at random the heap to be retained.

NOTES

9 This division process may be used for ore having a nominal top size up to 45 mm.

10 This method is not recommended for obtaining moisture samples.

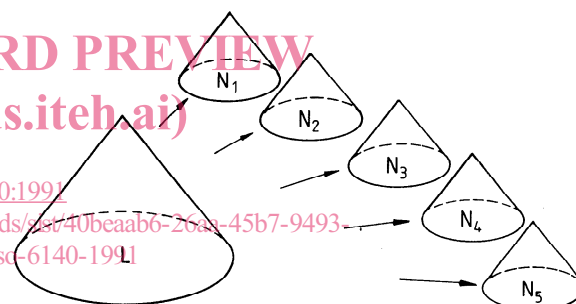


Figure 3 — Sampling by fractional shovelling

4.6.3.6 Strip mixing and splitting

Strip mixing and splitting simulates the conditions obtained in the sampling of ore from a conveyor belt. It shall be carried out as follows.

The ore is formed into a strip by careful distribution of the ore from a shovel along the length of strip as evenly as possible, working from end to end of the strip and from both sides of the strip. The end plates ensure that size segregation only occurs laterally. The general appearance of a completed strip is shown in figure 4. The subsample is then taken by inserting a suitable sampling frame across the strip and removing the ore from between the frame by the use of a scoop (figure D.1), ensuring that all fine particles are included in the increment. The number of required increments (minimum 20) shall be taken.

NOTE 11 This procedure is not recommended for obtaining a moisture sample.

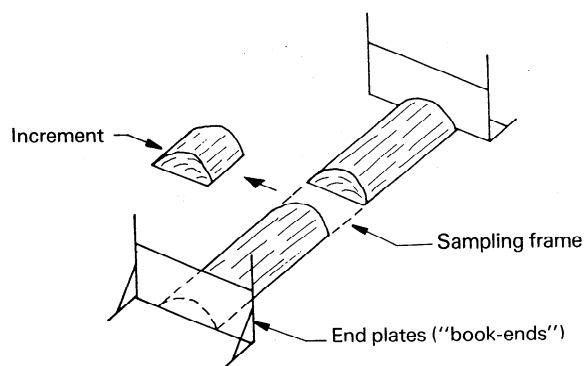


Figure 4 — Completed strip

5 Precision of sample preparation

The precision of sample preparation shall be checked periodically and whenever a new scheme is brought into operation. Details of test procedures for the determination of precision are given in the appropriate International Standard.

6 Bias in sample preparation

The bias in sample preparation shall be checked periodically and whenever a new scheme is brought into operation.

7 Preparation of samples for the determination of total moisture

7.1 Types of sample to be tested

The sample to be tested will be either

- a sample collected exclusively for the determination of moisture;
- a sample on which determinations of moisture and other quality characteristics are required.

7.2 Precautions against loss of moisture

One of the main difficulties in determining moisture is that of minimizing changes in the moisture content of the sample while preparing the final sample. Every precaution shall be taken to minimize changes in moisture content due to unsuitable containers and by evaporation during handling, particularly when the ore is extremely wet. All moisture samples shall be kept in sealed containers in a cool place before and after preparation as well as during any interval between stages of sample preparation.

Care should be taken to minimize changes in humidity when carrying out sample division and such operations should be carried out as quickly as possible. In some circumstances, it may be necessary to carry out moisture determinations on each increment in order to minimize moisture changes (see annex A).

When conditions are such that a rapid change in moisture content may occur, the procedure given in A.2 shall be used.

7.3 Samples for the determination of moisture

Samples for the determination of moisture shall be as specified in table 3 and be taken and prepared in accordance with procedures specified in ISO 8685. Moisture determination shall be carried out in accordance with the procedure specified in ISO 9033. Samples of nominal top size greater than 22,4 mm may undergo a two-stage drying procedure as described in 4.4.3.

Table 3 — Minimum mass of sample for determination of moisture

Nominal top size of ore mm	Minimum mass kg
63,0	110
45,0	40
31,5	14
22,4	5
16,0	2
11,2	1

7.4 Reserve samples

It is recommended that two reserve samples be collected so that in the case of dispute, or if the results of the first moisture determinations are lost or invalid, the reserve samples may then be examined. Reserve samples shall be collected in the same way, and at the same time, as the final samples for the determination of moisture. Four sample containers shall be used and increments placed sequentially into each container.

8 Preparation of samples for chemical analysis

8.1 General

The procedure for preparation of chemical analysis samples is described in 8.2 and shown schematically in annex A. It is permissible to use the moisture sample after drying as the chemical analysis sample.

8.2 Procedure

8.2.1 First stage in the preparation

If necessary, the sample shall be dried in accordance with 4.3. The whole of the sample shall be passed through the mill (see 4.4), mixed and then divided (see 4.6) to decrease the mass to the value appropriate to the nominal top size of the crushed ore (see 4.2).

8.2.2 Further stages in the preparation

In the further stages of preparation, the retained portion from the first stage shall be reduced to a nominal top size of 150 μm (see 4.4) and then divided to obtain as many samples as contractually required, each of mass not less than 50 g.

The chemical analysis sample shall be placed in a sealed container and labelled with all necessary identification details.

9 Preparation of samples for physical testing

9.1 General

The procedure for preparation of samples for physical testing to measure particle size distribution or bulk density is described in 9.2. It is permissible to use the moisture sample after drying for the determination of particle size distribution or for determination of the bulk density of oven-dried material.

Where it is necessary to determine the bulk density of as-received material, a sample shall be collected exclusively for this purpose and the same precautions as detailed in 7.2 shall apply. If the determination of the bulk density of air-dried material is required, a sample shall also be collected exclusively for this purpose but no precautions to guard against loss of moisture by evaporation are necessary.

It is not permissible to reduce the particle size during any stage of preparation of samples to be used for the determination of particle size distribution or bulk density.

The provisions of 4.2 regarding minimum mass of samples shall be implemented for physical testing operations.

9.2 Procedure

9.2.1 Preparation of sample for the determination of particle size distribution

The appropriate procedure for preparation of samples to be used for the determination of particle size distribution depends on the characteristics of the ore. Two types shall be taken into account, namely

— Type A

An ore containing a fine fraction high in clay minerals which consolidate on drying. Consolidation of this type of fines may render it very difficult to obtain a true determination of particle size distribution of the fine portion of the ore even when the ore is rewetted in an effort to re-disperse the fines.

When dealing with type A ore, the samples taken in accordance with this International Standard shall be protected from moisture evaporation in a similar manner to that described in 7.2. Wet screening is used for this type of ore.

— Type B

An ore containing no fine fraction high in clay minerals. Type B ores may be dried by exposure to the atmosphere or by the use of an oven. When testing type B material for the determination of particle size distribution, the sample previously used for determination of moisture can be used.

9.2.2 Preparation of sample for bulk density determination

Three types of bulk density determination may be required:

- material as-received at natural moisture content;
- air-dried material;
- oven-dried material.

When the determination is to be made on as-received material, the sample shall be given the same consideration as the moisture sample described in 7.2. For air-dried and oven-dried material, such consideration is unnecessary and, where appropriate, the sample used for determination of moisture content may be used for the determination of bulk density. Multiple use of samples for physical tests is not recommended because of the potential for contamination and degradation.