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Iron ores — Method of sampling and sample preparation for physical testing

Minerais de fer — Méthode d'échantillonnage et préparation des échantillons pour les essais physiques

Reference number
ISO 10836:1994(E)

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 10836 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Sub-Committee SC 1, *Sampling*.

Annexes A and B of this International Standard are for information only.

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Iron ores — Method of sampling and sample preparation for physical testing

1 Scope

This International Standard specifies the procedure for sampling and sample preparation of iron ores for physical testing. The testing covered by this International Standard refers to the physical test methods specified in the relevant International Standards in clause 2. With respect to the bulk density test, only Method 1 in ISO 3852, using a small container for iron ores having a nominal top size of 40 mm or smaller, falls within the scope of this International Standard.

NOTE 1 The test methods specified in ISO 4696 and ISO 7992 are referred to throughout the text simply as LTD (low-temperature disintegration) test and RUL (reduction properties under load) test, respectively.

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 3081:1986, *Iron ores — Increment sampling — Manual method.*

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method.*

ISO 3085:1986, *Iron ores — Experimental methods for checking the precision of sampling.*

ISO 3271:1985, *Iron ores — Determination of tumbler strength.*

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

ISO 3310-2:1990, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate.*

ISO 3852:1988, *Iron ores — Determination of bulk density.*

ISO 4695:1984, *Iron ores — Determination of reducibility.*

ISO 4696:1984, *Iron ores — Low-temperature disintegration test — Method using cold tumbling after static reduction.*

ISO 4698:—[1], *Iron ore pellets — Determination of relative free-swelling index.*

ISO 4700:1983, *Iron ore pellets — Determination of crushing strength.*

ISO 7215:1985, *Iron ores — Determination of relative reducibility.*

ISO 7992:1992, *Iron ores — Determination of reduction properties under load.*

3 Definitions

For the purposes of this International Standard, the following definitions apply. The definitions not specified here are in accordance with the International Standards referred to in clause 2.

3.1 sample for physical testing: A sample for the determination of physical properties, e.g. tumble

1) To be published.

strength, reducibility, free-swelling index, crushing strength, reduction-disintegration index and bulk density.

3.2 split use of sample: The separate use of parts of a sample as test samples for the determination of two or more quality characteristics.

3.3 multiple use of sample: The use of the sample in its entirety for the determination of one quality characteristic, followed by the use of the same sample in its entirety for the determination of one or more other quality characteristics.

3.4 reserve sample: A sample, for testing physical properties, reserved for testing by an independent laboratory.

4 Fundamentals

4.1 Sample derivation

The sample for physical testing shall be a split-use or a multiple-use sample derived from the sample for size analysis or the sample for the determination of moisture content and/or chemical analysis of a lot.

However, the increments for a physical test sample may be taken independently of the increments for size analysis, moisture determination and chemical analysis, provided the required overall precision for the respective physical properties is demonstrated to be within the limits specified in table 1.

4.2 General procedure

The general procedure for sampling and sample preparation for physical testing shall be in accordance with ISO 3081, ISO 3082 and ISO 3083. The sampling and preparation of the test sample shall be in accordance with the following procedure:

a) establish a flow sheet for sampling and preparation of samples for physical testing, using split-use samples, multiple-use samples or independently obtained samples;

b) take increments and prepare the gross sample for physical testing;

c) prepare the test sample with the size and mass specified in the relevant International Standards.

CAUTION — Due consideration shall be given to the safety of operators when taking increments manually. The applicable regulations shall be respected.

4.3 Precision

In this International Standard, the overall precision (β_{SPM}), when determining the physical properties of the lot measured using the relevant International Standards, shall be within the limits specified in table 1 at a probability level of 95 %.

NOTE 2 The attainable precision in terms of standard deviation is given in annex A.

Table 1 — Overall precision (tentative)

Type of iron ore	**Overall precision, β_{SPM}**					
	Tumble strength (Abrasion index) %	**Reducibility** % min.	**Relative reducibility** %	**Reduction-disintegration index** (RDI) RDI + 3,15 %	**Free-swelling index** %	**Crushing strength** daN
Pellets	0,4	0,24	2,8	3,8	2,6	32
Sinters	0,6	0,30	3,2	4,6	—	—
Sized ore	0,6	0,24	7,0	3,6	—	—

5 Apparatus

5.1 Riffles, scoops for increment division and other apparatus, as specified in ISO 3081, ISO 3082 and ISO 3083.

5.2 Test sieves, having square openings of the following nominal aperture sizes and conforming to ISO 3310-1 and ISO 3310-2:

40 mm, 25 mm, 22,4 mm, 20 mm, 16 mm, 10 mm and 6,3 mm

5.3 Rectangular frames, with 15 equal parts (Frame A) and 25 equal parts (Frame B).

6 Method of sampling

Before starting the sampling and sample preparation for physical testing, special attention shall be given to the number and mass of increments to be taken from the lot.

a) In the case of split use or multiple use of a sample, when the mass of the sample is expected to be less than that required for preparing the sample for physical testing, the number and/or mass of increments to be taken shall be increased to give the required mass. However, it is preferable to increase the number of increments taken, rather than take fewer increments of larger mass.

b) When taking increments independently, the number of increments (n_1) to be taken shall be calculated using the following formula:

$$n_1 = \left(\frac{2\sigma_W}{\beta_S} \right)^2$$

where

σ_W is the measured quality variation within strata;

β_S is the sampling precision which is equal to $2\sigma_S$ (σ_S is the sampling precision in terms of the standard deviation).

NOTE 3 When the value of σ_W is unknown, the number of increments should be in accordance with those for "large" quality variation in table 4 of ISO 3081:1986 or ISO 3082:1987.

Sampling should be carried out at the nearest possible point to the loading or discharging facilities, preferably immediately before or immediately after the point of weighing. In addition, free fall drops in the materials handling system should be kept to a minimum to reduce size degradation of the ore.

7 Method of sample preparation

7.1 Preparation of sample for physical testing

7.1.1 Selection of the sample preparation procedure

Selection of the sample preparation procedure for physical testing should take into consideration the sample derivation and the sampling apparatus. Examples of sample preparation for physical testing are shown in figure 3. The scheme for taking increments independently is shown in figure 4.

7.1.1.1 Split use of sample

Split each partial sample into four parts and use one part for physical testing and the other three parts for size analysis, determination of moisture content and chemical analysis [see figure 3 a)].

7.1.1.2 Combination of split use and multiple use of sample

Split each partial sample into two parts and use one part for size analysis and subsequent physical testing and the other part for preparation of moisture and chemical analysis samples [see figure 3 b)]. The moisture sample could also be used subsequently for physical testing [see figure 3 c)].

7.1.1.3 Independent sample

Take the increments for physical testing from the lot, independent of those taken for size analysis, determination of moisture content and chemical analysis (see figure 4). The preparation procedure shown in figure 5, 6 or 7 should be applied directly.

7.1.2 Preparation of gross sample

When the sample for physical testing is prepared from each increment or each partial sample, the samples thus prepared shall be combined to prepare the gross sample for physical testing.

The minimum mass of the gross sample for physical testing shall be determined from the test requirements and the number of required physical properties. In general, except for the sample for the bulk density test, a gross sample for physical testing should weigh at least 500 kg. When the bulk density test is carried out according to Method 1 of ISO 3852, the gross sample for physical testing should weigh at least 1 200 kg.

The gross sample for physical testing shall be divided to prepare test samples for determination of physical properties, irrespective of the division rules specified in ISO 3082 and ISO 3083. However, whatever division method is used, it should be demonstrated that

the precision detailed in table 1 and annex A is obtained using the procedures outlined in ISO 3085.

7.2 Preparation of test samples

7.2.1 General

The gross sample for physical testing should be divided into two parts; one for the preparation of test samples for the specified physical properties (Sample A) and the other for retention as a reserve sample (see figures 5 and 6).

NOTE 4 When the bulk density test is carried out, the gross sample for physical testing (approx. 1 200 kg) should be divided into two parts; one for preparation of the bulk density test sample (approx. 600 kg) and the other for further division into two parts, i.e., one for preparation of the physical test samples other than for bulk density determination (approx. 300 kg, Sample A) and the other for retention as the reserve sample (see also figure 7).

Sample A should then be divided into two parts; one for preparation of the sample for the tumble test (Sample A1) and the other for preparation of other test samples for determination of physical properties (Sample A2).

Samples A1 and A2 shall be dried at a temperature of 105 °C ± 5 °C before preparing the respective test samples. Examples of preparation procedures for test samples are shown in figures 5 and 6.

7.2.2 Test sample for tumble test

The test sample for the tumble test specified in ISO 3271 should be prepared as follows.

7.2.2.1 Pellets

Sieve Sample A1 (approx. 125 kg) using 40 mm and 6,3 mm sieves and discard the +40 mm and −6,3 mm fractions. Divide the −40 mm and +6,3 mm fraction using the increment division method to obtain four test portions, each having a mass of 15 kg ± 0,15 kg.

7.2.2.2 Sinters and sized iron ores

Sieve Sample A1 (approx. 125 kg) using 40 mm, 25 mm, 16 mm and 10 mm sieves and discard the +40 mm and −10 mm fractions. Weigh the three other fractions, i.e. the −40 mm +25 mm, −25 mm +16 mm and −16 mm +10 mm fractions, and calculate and record the percentage of each size fraction. From the three size fractions, reconstitute four test portions of 15 kg ± 0,15 kg by taking a proportionate mass of material from each of the three size fractions.

Alternatively, sieve Sample A1 (approx. 125 kg) using 40 mm and 10 mm sieves and discard the +40 mm and −10 mm fractions. Divide the −40 mm and +10 mm fraction using the increment division method to obtain four test portions, each having a mass of 15 kg ± 0,15 kg.

7.2.3 Test samples for physical tests other than tumble test and bulk density test

7.2.3.1 Pellets

The total mass of test samples required by the respective International Standards is about 15 kg as shown in table 2.

Table 2 — Total mass of test sample

Type of test	International Standard	Approx. mass of test sample
LTD test	ISO 4696	2 kg
Free-swelling test	ISO 4698	1 kg
Compression test	ISO 4700	1 kg
Reduction test:		
— Reducibility	ISO 4695	2,5 kg
— Relative reducibility	ISO 7215	2,5 kg
RUL test	ISO 7992	6 kg
		———
		Total 15 kg

Sample A2 (approx. 125 kg) is divided twice, and one 30 kg sample is set aside in reserve wile the other 30 kg sample is dried at 105 °C ± 5 °C for at least 2 h and sieved using 12,5 mm and 10 mm sieves. The +12,5 mm and −10 mm fractions are discarded and the −12,5 mm +10 mm fraction (approx. 15 kg) is further prepared as follows:

Divide the fraction into 15 equal parts, five lengthwise and three breadthwise, using a rectangular frame (Frame A), and spread it evenly over the frame.

7.2.3.1.1 Test sample for LTD test, crushing strength test, and free-swelling test

Extract four parts at random, mix and divide using a riffle to obtain three test samples as shown in figure 1 (see also figure 5).

From the LTD test sample, four test portions, each having a mass of approx. 500 g, are prepared.

From the crushing strength test sample, 60 (or more) pellets are selected at random as the test portion.

From the free-swelling test sample, 18 pellets are selected at random as the test portion.

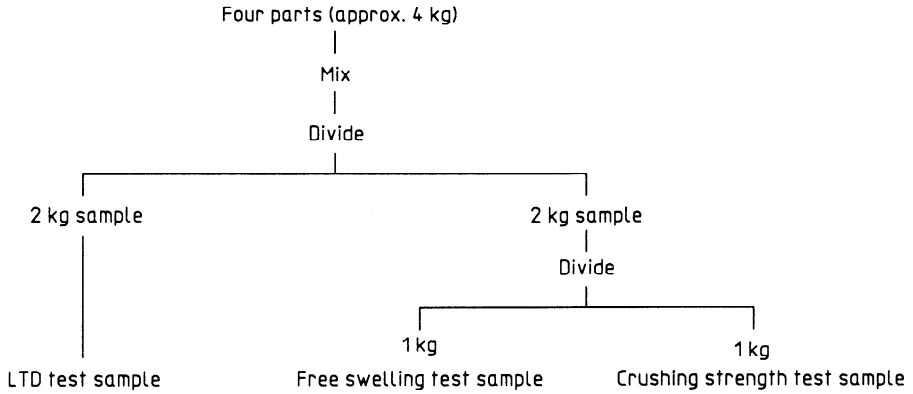


Figure 1 — Preparation of test samples for LTD test, crushing strength test and free-swelling test

7.2.3.1.2 Test sample for reduction tests (reducibility and relative reducibility)

Extract five parts at random, mix and divide using a riffle and obtain two test samples for the reducibility and the relative reducibility tests as shown in figure 2.

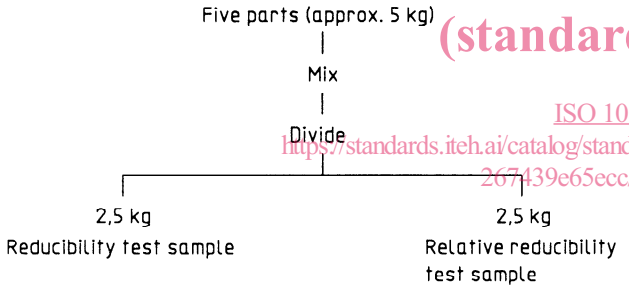


Figure 2 — Preparation of test samples for reduction tests

From each test sample, prepare five test portions, each having a mass of approx. 500 g. Of these test portions, one is for chemical analysis.

7.2.3.1.3 Test sample for RUL test

The remaining six parts (approx. 6 kg) are used as the RUL test sample. Prepare four test portions and one chemical analysis sample, each having a mass of approx. 1 200 g.

7.2.3.2 Sinters and sized iron ores

The total mass of test samples required by the respective International Standards is about 10,5 kg as shown in table 3.

Table 3 — Total mass of test sample

Type of test	International Standard	Approx. mass of test sample
LTD test	ISO 4696	2 kg
Reducibility test	ISO 4695	2,5 kg
RUL test	ISO 7992	6 kg
		Total 10,5 kg

Sample A2 (approx. 125 kg) is divided, and one 62,5 kg sample is set aside in reserve while the other 62,5 kg sample is dried at 105 °C ± 5 °C for at least 2 h and further divided into two portions (see figure 6).

One sample (approx. 30 kg) is used as the relative reducibility test sample. This sample is sieved using 22,4 mm and 20 mm sieves. The +22,4 mm and −20 mm fractions are discarded. The −22,4 mm +20 mm fraction is further divided, using the increment division method, to obtain four test portions and one chemical analysis sample, each having a mass of approx. 500 g.

The remaining 30 kg sample is sieved using a 12,5 mm sieve. The +12,5 mm fraction is crushed carefully and sieved using a 16,0 mm sieve. The +16,0 mm fraction is crushed to −16,0 mm. The −12,5 mm and −16,0 mm fractions are mixed and sieved using 12,5 mm and 10,0 mm sieves. The +12,5 mm and −10,0 mm fractions are discarded and the −12,5 mm +10,0 mm fraction (approx. 25 kg) is then prepared as follows.

Divide the fraction into 25 equal parts, five lengthwise and five breadthwise, using a rectangular frame (Frame B) and spread the sample evenly over the frame.

Extract two parts at random, mix and divide using a riffle to obtain four test portions for the LTD test, each having a mass of approx. 500 g.

Extract five parts at random, mix and divide using a riffle to obtain two samples. One is used as the reducibility test sample while the other is discarded. From the test sample, five test portions, each having a mass of approx. 500 g, are prepared. Of these test portions, one is for chemical analysis.

Extract six parts at random, mix and prepare four test portions and one chemical analysis sample for the RUL test, each having a mass of approx. 1 200 g.

7.2.4 Test sample for bulk density test

When the bulk density test is required, divide the gross sample for physical testing (approx. 1 200 kg) into two parts (see note 4 to 7.2.1). Divide one 600 kg part twice to obtain four test portions, each of approx. 150 kg mass. Three test portions are for the bulk density test using Method 1 of ISO 3852 and one is used as a check sample for the determination of moisture content and size analysis (see figure 7).

7.2.5 Test samples for other tests

When the preparation of test samples for the determination of physical properties other than those given in 7.2.2 to 7.2.4 is required, such samples should be prepared from the physical properties test sample in parallel with the preparation of various test samples according to 7.2.2 and 7.2.3, to meet the requirements given in the specific relevant International Standard.

7.2.6 Preparation of test sample from the reserve sample

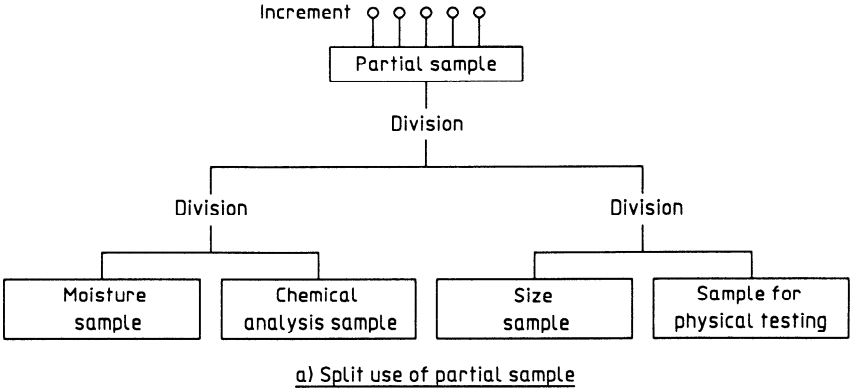
When testing for certain physical properties is requested by an independent laboratory, the test sample shall be prepared from the reserve sample in accordance with the relevant procedure in 7.2.2 to 7.2.5, taking into consideration the specific property.

8 Reserve samples

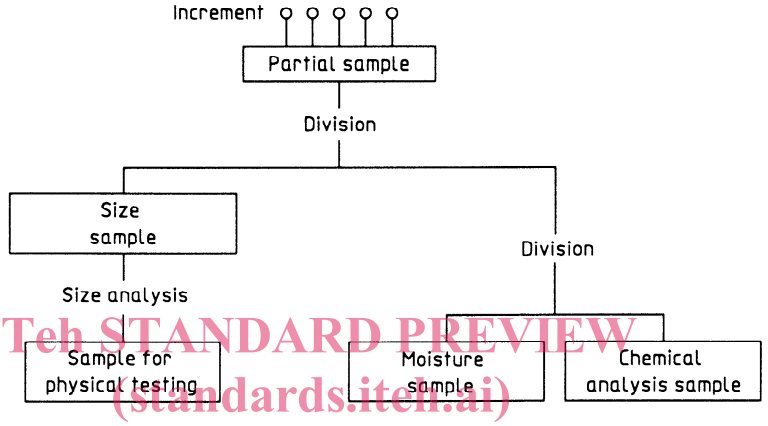
The reserve sample shall be prepared from the gross sample for physical testing at a convenient stage of the preparation of test samples for determination of physical properties, as specified in 7.2.

The reserve samples shall be put into containers, and the containers shall be sealed and marked with the required items of identification.

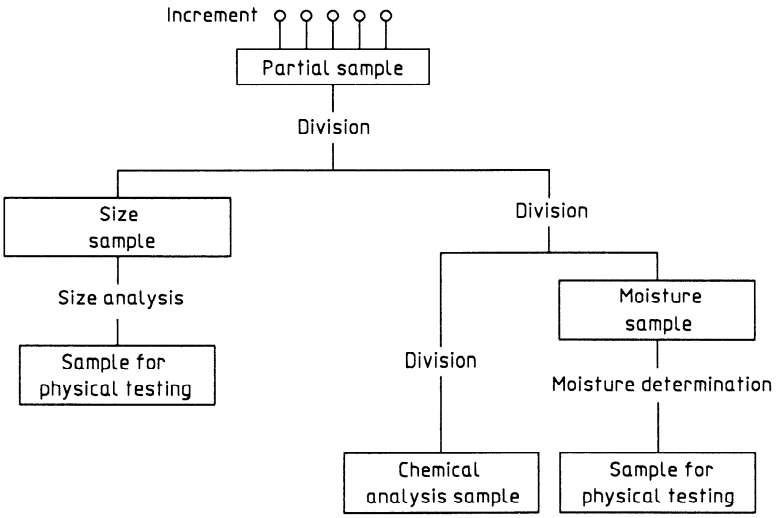
The reserve samples should be kept for a period of six months. If the test report is accepted by the supplier and purchaser within six months, the reserve samples may be discarded.



a) Split use of partial sample



b) Multiple use of one part of the partial sample (for size analysis and physical testing) and split use of the other part of the partial sample (for moisture determination and chemical analysis)



c) Preparation of two samples for physical testing

Figure 3 — Examples of preparation of samples for physical testing

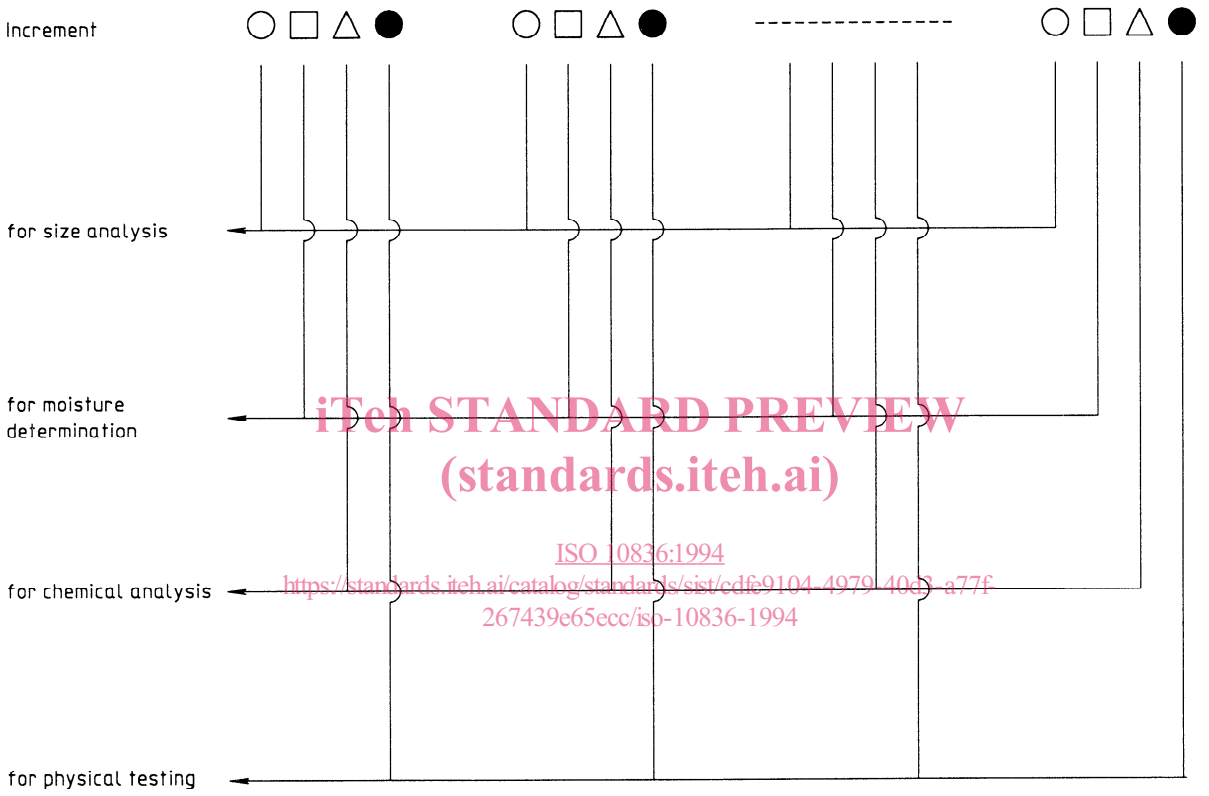


Figure 4 — Independent sampling scheme

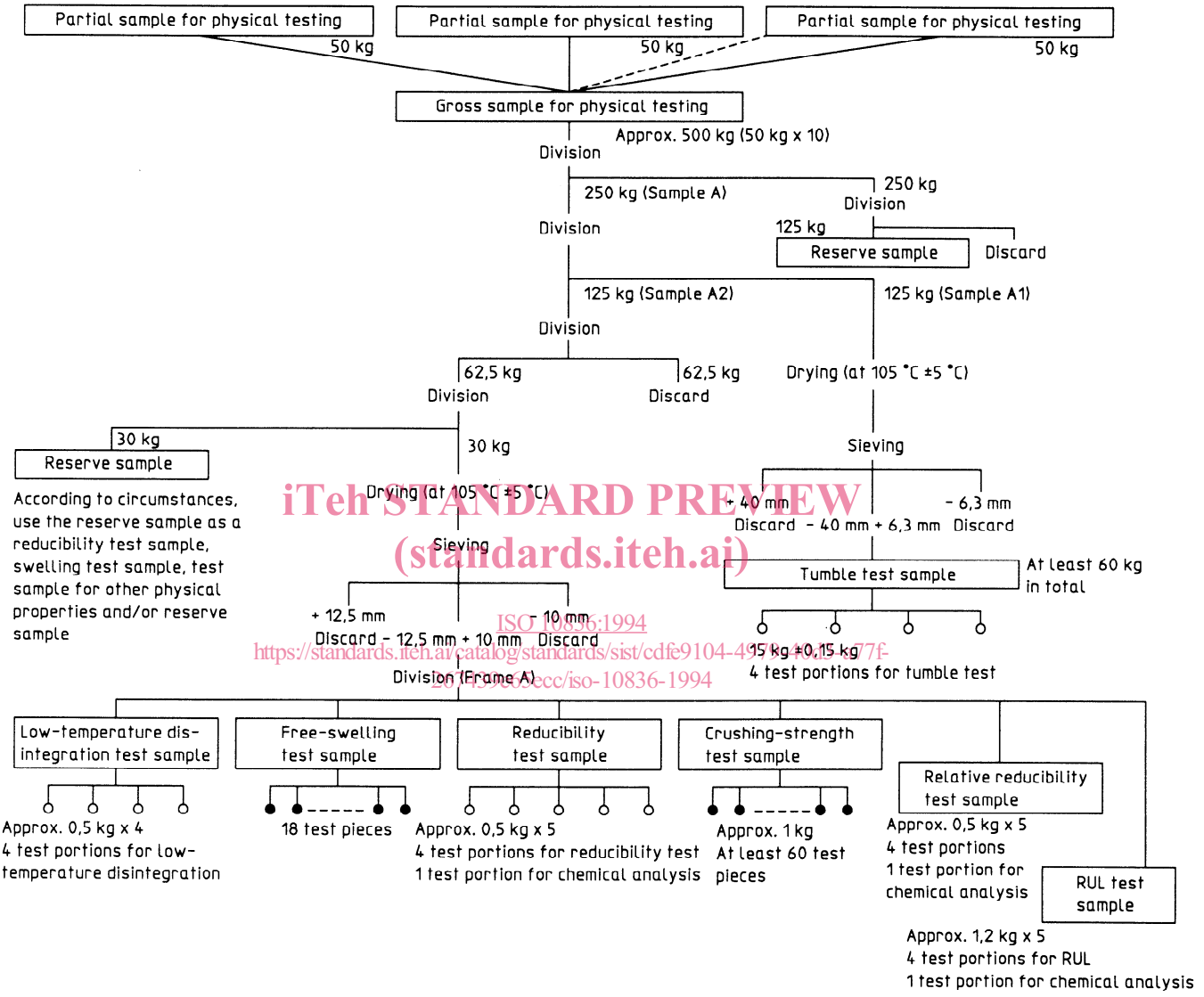


Figure 5 — Example of preparation of test samples for determination of physical properties of iron ore pellets