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# International Standard



# 6375

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## Carbonaceous materials for the production of aluminium — Cokes for electrodes — Sampling

*Produits carbonés utilisés pour la production de l'aluminium — Cokes pour électrodes — Échantillonnage*

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**Descriptors** : extractive metallurgy, aluminium, coke, pitch (materials), petroleum products, electrodes, sampling.

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

International Standard ISO 6375 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in December 1978.

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

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No member body expressed disapproval of the document.

# Carbonaceous materials for the production of aluminium — Cokes for electrodes — Sampling

## 1 Scope and field of application

This International Standard specifies methods for sampling and preparation of samples from lots of calcined coke so as to obtain, with sufficient accuracy, a sample that is representative of the average qualities of each entire lot.

It is applicable to cokes manufactured from pitch or petroleum intended specifically for use in the manufacture of electrodes used for the electrolytic production of aluminium.

Because of the special requirements of these cokes, this International Standard includes provisions which do not appear in the methods for sampling similar carbonaceous products, such as metallurgical cokes. These provisions are mainly concerned with the composition of samples for physical measurements and for particle size analysis.

The theoretical and practical aspects of sampling on which this International Standard is based are described more fully in ISO 1988 (coal) and ISO 2309 (metallurgical coke) and users are referred to these documents for greater detail.

Because of processing considerations, it is essential that the product shall be of uniform chemical composition. Two samples should therefore be taken :

- the first at the time of loading : in order to check the quality of the product as supplied;
- the second at the time of unloading at the customer's premises or at the nearest port : in order to check for possible contamination in transit.

## 2 References

ISO 1988, *Hard coal — Sampling*.

ISO 2309, *Coke — Sampling*.

ISO 6206, *Chemical products for industrial use — Sampling — Vocabulary*.

## 3 Practical considerations

### 3.1 Sampling apparatus (see ISO 2309, annex)

Use automatic sampling apparatus whenever possible.

Hand-operated sampling tools (scoops, frames placed on conveyor belts, etc.) shall have a width for taking samples which is at least 2.5 times the maximum dimension of the largest pieces and shall be capable of taking a sample of material of 1 to 2 kg at a time.

The sample containers shall not be filled to more than 9/10 of their volume.

### 3.2 Special cases of sampling

Refer to ISO 2309, clause 6, for sampling procedures in specific circumstances (conveyor belt, various types of wagon, ships, etc.).

Whenever possible, avoid sampling from a stationary bulk and sample the consignment to be checked during loading or unloading.

Do not sample material corresponding to the top 0,20 m of the initial load so that bias associated with surface contamination is avoided. Sample the remainder uniformly throughout its entire bulk.

### 3.3 Processing of samples

Homogenization should preferably be carried out mechanically. Manual methods using a suitable working surface area, are however, acceptable.

The parts of crushing machines which come into contact with the product to be crushed shall be made of hard material which is unlikely to yield impurities when in operation.

Carry out sample mass reduction, preferably using closed sample dividers.

In all such operations, avoid the loss of fine particles.

#### 4 Definitions of samples

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

**4.1 increment** : A quantity of material taken, at one time, from a sampling unit by a sampling device (see ISO 6206).

**4.2 partial sample** : A sample taken from each 1 000 t fraction of a load or consignment. One part is used for an initial check of uniformity, the second part for particle size analysis and the third part to prepare the representative sample (4.3).

**4.3 representative sample** : A sample assumed to have the same composition as the material sampled when the latter is considered a homogeneous whole (see ISO 6206).

For the purposes of this International Standard, the representative sample is the aggregate of the third part of the partial samples (for loads or consignments greater than 1 000 t), or the aggregate of the increments taken from the consignments of less than 1 000 t.

#### 5 Number and size of increments

##### 5.1 General

ISO 2309, clause 4, specifies the number of increments required to determine the moisture content of metallurgical coke to an accuracy of  $\pm 1\%$ . The number of such samples per 1 000 t lot is large (50 for a moving stream of coke, 75 for a wagon, 100 for a ship, 150 from a stockpile) because of the very wide range of moisture contents. In the case of coke for electrodes, the quality of which is much more uniform, experience has shown that this number may be reduced. On average, at least 30 increments per 1 000 t lot is sufficient.

These increments shall have masses of 1 to 2 kg, but each shall be at least 20 times the mass of the coarsest lump.

For lots of less than 1 000 t, the number of increments is defined in figure 1. The number of increments taken per tonne of product increases as the tonnage decreases because of the increasing risk of heterogeneity.

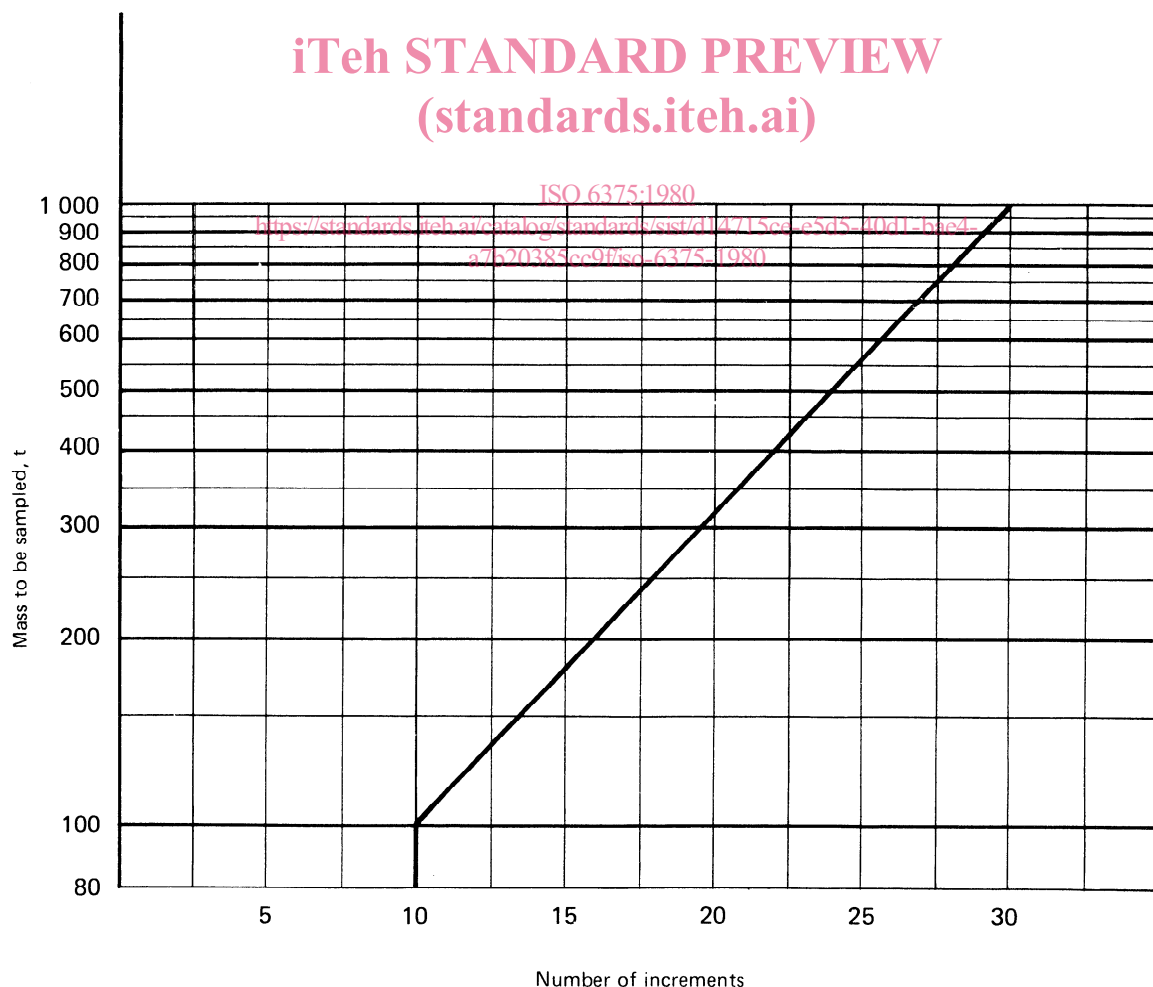


Figure 1 — Number of increments to be taken as a function of the mass to be sampled

5.2 Particular cases

5.2.1 Wagons

Take at least two increments from each wagon sampled, during loading (or unloading) :

- a) if the consignment (or load) consists of less than 15 wagons, sample all the wagons;

NOTE — In the case of only one wagon, three increments shall be taken for 10 kg at the beginning, midway and end of coke flow during loading or unloading.

- b) for a consignment of less than 1 000 t consisting of more than 15 wagons, sample 10 wagons selected at random;

- c) for a consignment of more than 1 000 t, take the 30 increments per 1 000 t fraction from 5 wagons selected at random from each 1 000 t fraction.

5.2.2 Ships

If the load is distributed throughout several holds, sample each hold separately, regardless of its contents, dividing it, if necessary, into approximately 1 000 t lots.

6 Sampling of consignments or loads

6.1 General

In view of the importance of certain characteristics of coke for electrodes, it is useful to obtain, in addition to the representative sample, a partial sample from each 1 000 t fraction if the size of the consignment so warrants, in order to carry out rapid checks to assess the uniformity of the product. The preparation of these samples is described in 6.2 and shown schematically in figure 2.

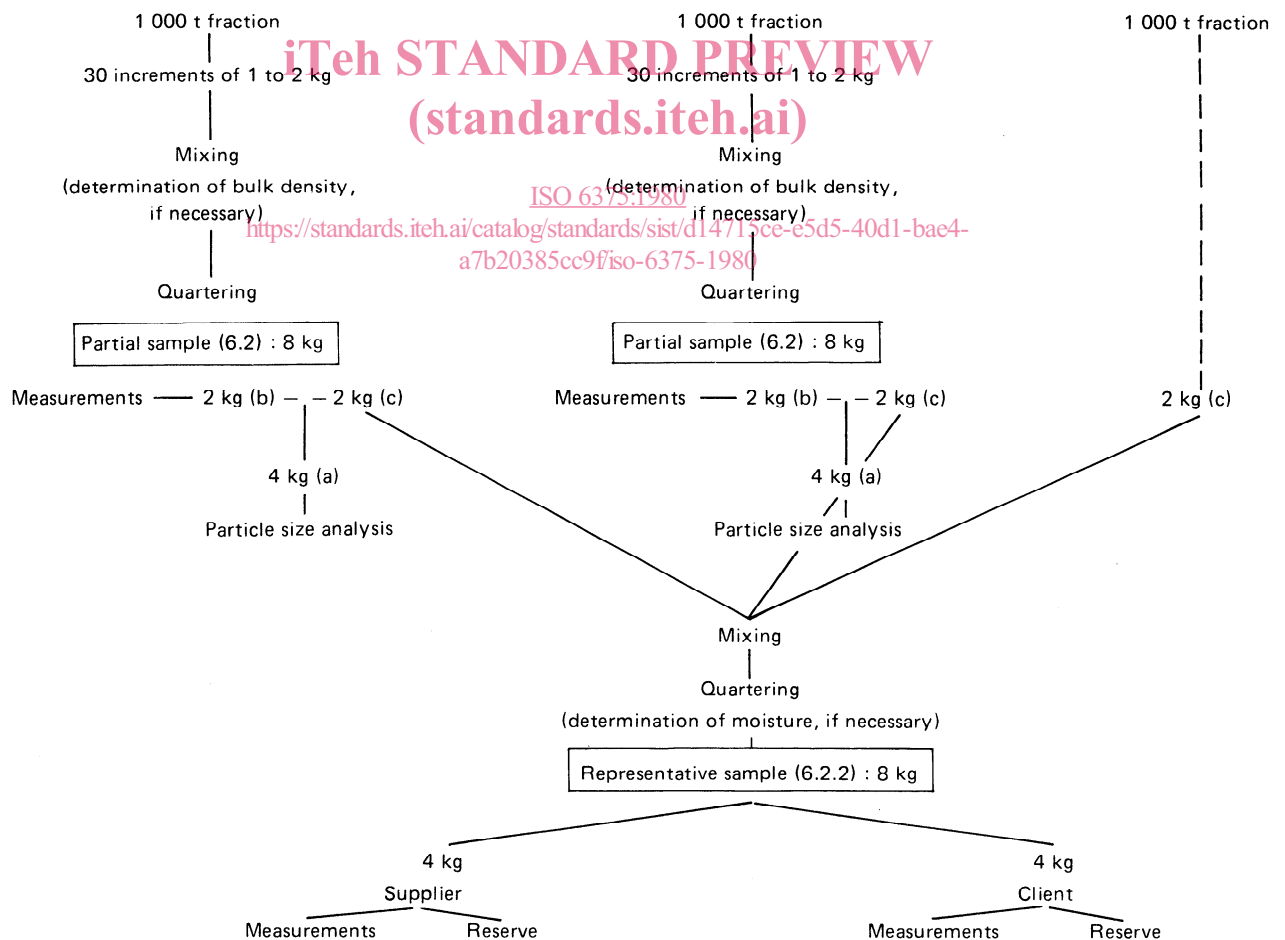


Figure 2 — Preparation of partial and representative samples

## 6.2 Sample preparation

Combine at least 30 increments taken from a 1 000 t lot,<sup>1)</sup> mix and then reduce by quartering to approximately 8 kg. From such samples obtain, by quartering, the samples specified in 6.2.1 and 6.2.2.

### 6.2.1 Partial samples representative of each 1 000 t fraction

Take

- a fraction of approximately 4 kg (a) for particle size analysis (immediately or subsequently) if this information is required;
- a fraction of 2 kg (b) which shall be checked immediately for the agreed characteristics.

### 6.2.2 Representative sample

Take a fraction of 2 kg (c) which, combined with the similar fractions taken from the other 1 000 t lots, comprises a sample representative of the whole consignment or load and on which determinations of all the characteristics agreed between the interested parties are carried out.

Mill the 2 kg fraction (b) in a jaw crusher until it passes completely through a sieve of aperture 5 mm. After mixing, divide this fraction into two equal parts :

- retain one of these fractions (b<sub>1</sub>) until completion of testing of the representative sample (6.2.2), in case additional checks are required on each 1 000 t fraction;
- mill the other fraction (b<sub>2</sub>) until it passes completely through a sieve of aperture size appropriate to the required determinations. Take from this fraction, by quartering, the quantities required for the measurements.

## 7.2 Representative sample (6.2.2)

Treat as follows all the 2 kg fractions (c) taken from the partial samples in the case of loads greater than 1 000 t, or the whole of the increments in the case of loads less than 1 000 t.

**7.2.1** Intimately mix on a suitable working surface or in a device capable of accommodating all the partial samples.

**7.2.2** Quarter until 4 kg of product is obtained. (If the sample is to be divided between the supplier and the customer, approximately 8 kg of product should be obtained by quartering, increasing if necessary the quantity taken from the partial samples in the case of a load of 2 000 to 3 000 t, for example, which would normally give only 4 or 6 kg of representative sample.)

## 7 Treatment of samples

### 7.1 Partial samples taken per 1 000 t lot (see 6.2.1)

Carefully mix all the increments from a 1 000 t lot on a suitable working surface, or use a mechanical device capable of accommodating all the increments.

After mixing and, if necessary, determining the bulk density, reduce by quartering until 8 kg of product are obtained.

Then take

- approximately 4 kg (a) for particle size analysis;
- approximately 2 kg (b) for checking the uniformity of the consignment on the basis of certain agreed physico-chemical properties;
- approximately 2 kg (c) for the preparation of the representative sample (6.2.2).

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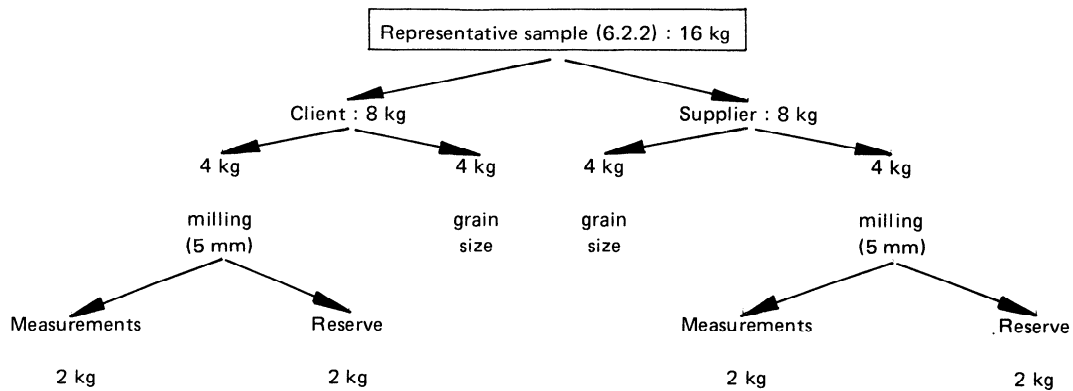
Mill the 4 kg sample thus obtained in a jaw crusher until it passes completely through a sieve of aperture 5 mm.

Mix it and then divide it by quartering into two 2 kg fractions, retaining one for possible repeat testing and milling the other until it passes completely through a sieve of aperture suitable for the required determinations. (The required particle size is specified in each of the methods of test for coke for electrodes.)

If the moisture content is to be determined, store the samples in air-tight tins. Determine the moisture content on the representative sample (6.2.2) after milling to 5 mm followed by homogenization.

NOTE — If the consignment is less than or equal to 1 000 t, the representative sample (6.2.2) consists of the sum of the increments. The final mass of this sample after quartering should be adjusted to suit the number of determinations required : for example, if the measurement of particle size is agreed between the customer and the supplier, 4 kg (analysis + reserves) + 4 kg (size analysis), i.e. 8 kg should be supplied to each party making a total of 16 kg. This sample should be divided by quartering in the following manner :

1) If the bulk density (mass contained in a given volume) at the time of delivery is to be checked, this determination shall be carried out immediately after mixing the increments but before quartering.



**8 Sampling report**

**8.1** The sampler shall attach to each sample a report containing at least the following information :

- a) a reference to this International Standard;
- b) the number of increments and mass of the sample taken;
- c) observations concerning any special or unusual phenomena, such as abnormal atmospheric conditions at the time of sampling, possible damage during transport;
- d) any operation not included in this International Standard or in those International Standards to which reference is made, or regarded as optional.

**8.2** The sample container shall be clearly labelled, allowing the sample to be unambiguously identified and giving, at least, the following information :

- a) the type of product;
- b) the name of the supplier;
- c) the name of the ship;
- d) the identification of the hold;
- e) the identification of the wagons;
- f) the total mass of the load;
- g) the number of partial samples;
- h) the nature of the samples : partial or representative;
- j) the mass of the sample.

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