INTERNATIONAL STANDARD

ISO 9406

Second edition
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Carbonaceous materials for the production of aluminium — Green coke — Determination of volatile matter content by gravimetric analysis

Produits carbonés utilisés pour la production de l'aluminium — Coke cru — Détermination de la teneur en matières volatiles par gravimétrie

Reference number
ISO 9406:1995(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 9406 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

This second edition cancels and replaces the first edition (ISO 9406:1988), of which it constitutes a minor revision (see figure 1).

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Introduction

Green coke is the end product of the distillation of mineral oil. To prepare it for the production of carbonaceous materials for electrolytic production of aluminium, its volatile components have to be removed by calcination.

The volatile matter content is an important factor technically and economically.

The method eliminates the volatile matter in green coke, thus permitting the volatile matter content to be determined. The method is based on that described in the standard DIN 51 720, *Prüfung fester Brennstoffe — Bestimmung des Gehaltes an Flüchtigen Bestandteilen.*

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Carbonaceous materials for the production of aluminium — Green coke — Determination of volatile matter content by gravimetric analysis

1 Scope

This International Standard specifies a method for the determination, by gravimetric analysis, of the volatile matter in green coke used in the production of aluminium.

The method is applicable to green cokes having volatile matter contents greater than 1 % (m/m).

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 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 6375:1980, *Carbonaceous materials for the production of aluminium — Coke for electrodes — Sampling.*

3 Principle

A test portion is heated in a crucible at a specified temperature and the loss in mass determined. The volatile matter content is then calculated as a percentage by mass.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Silica crucibles, ten, undamaged, of mass 10 g to 15 g, with ground lids and with the dimensions shown in figure 1.

Dimensions in millimetres

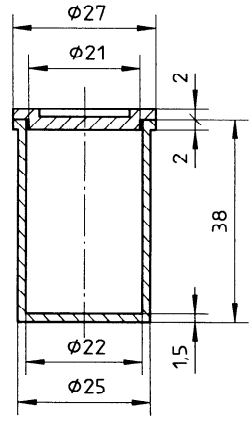

Figure 1

4.2 Electric muffle furnace, with horizontal heating elements and capable of being maintained at a temperature of 900 °C ± 20 °C, with useful heating chamber dimensions as follows:

height: 100 mm;
width: 230 mm;
length: 350 mm,

and with a central opening in the door to allow a thermocouple to be inserted.

4.3 Temperature-measuring device, with thermocouple, range 0 °C to 1 000 °C ± 10 °C.

4.4 Crucible stand, made of heat-resistant steel, for 6 crucibles, with the dimensions shown in figure 2.

4.5 Electric oven, capable of being maintained at 120 °C ± 5 °C.

5 Sampling and sample

5.1 Sampling

Carry out sampling of the green coke in accordance with the procedure specified in ISO 6375.

5.2 Preparation of test sample

Crush approximately 200 g of the laboratory sample in a mortar (see ISO 6375) and pass through a sieve of nominal mesh aperture 250 µm (see ISO 565). Dry the crushed and sieved product in the electric oven (4.5) to constant mass at a temperature of 120 °C ± 5 °C.

6 Procedure

6.1 Test portions

Heat six of the crucibles (4.1), with their lids, for about 1 h in the electric muffle furnace (4.2), maintained at a temperature of about 900 °C. Allow them to cool to ambient temperature in a desiccator and weigh each of them, without their lids, to the nearest 1 mg.

Weigh into each crucible, to the nearest 1 mg, a test portion of approximately 1 g from the dried test sample (5.2).

6.2 Checking the furnace temperature control

The furnace shall be checked at least once every 3 months, as follows:

Insert the crucible stand (4.4) with empty crucibles (4.1) into the furnace, pre-heated to 900 °C ± 20 °C. Close the door immediately.

Measure the temperature in the furnace by inserting the thermocouple (4.3) through the central opening of the door, with the measuring point near the centre of the furnace. Set the furnace temperature control device in such a way that the temperature comes back to 900 °C ± 20 °C 3 min to 4 min after introducing the crucible stand.

6.3 Determination

Tap the silica crucibles containing the test portions (6.1) gently on to a flat surface a few times to smooth the samples, close the crucibles with the lids and then insert them into the crucible stand. During coking, the crucible stand shall be full of crucibles in order that the heating rate, once set, is maintained. Fill any empty spaces in the crucible stand with empty crucibles. Put the crucible stand into the furnace, preheated to 900 °C ± 20 °C, as quickly as possible.

After 7 min ± 5 s, remove the crucible stand from the furnace. Place the crucibles on a cold base for 3 min. Then place them in a desiccator and leave for 30 min. Reweigh to the nearest 1 mg.

Take two test portions from each representative sample. Where a silica crucible is found to have become damaged, reject the result and repeat the determination for that test sample.

7 Expression of results

7.1 Method of calculation

Calculate the volatile matter content, expressed as a percentage by mass, from the formula

$$\frac{m_0 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in grams, of the residue.

Report, to the nearest 0,1 %, the mean value of two duplicate determinations.

7.2 Precision (in accordance with ISO 5725)

r (repeatability) = 0,2 % (m/m)

R (reproducibility) = 0,7 % (m/m)