International Standard 6997

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Carbonaceous materials for the production of aluminium — Calcined coke — Determination of apparent oil content — Heating method

Produits carbonés utilisés pour la production de l'aluminium — Coke calciné — Détermination de la teneur apparente en huile — Méthode par chauffage

First edition — 1985-06-15

UDC 665.777 : 669.713 : 543.8 **Ref. No. ISO 6997-1985 (E)**

Descriptors : industrial products, coke, tests, heating tests, determination of content, oils, gravimetric analysis, aluminium, production.

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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6997 was prepared by Technical Committee ISO/TC 47, *Chemistry.*

Carbonaceous materials for the production of aluminium — Calcined coke — Determination of apparent oil content — Heating method

0 Introduction

Calcined coke may be treated with different types of oil in order to avoid the formation of dust during loading and transportation. The method specified in this International Standard allows the determination of loss in mass of calcined coke resulting from the elimination by heating of oil adhering to the particles. However, loss of mass determined in this way does not give the true oil content of the coke, but rather a useful indication of the loss of mass which may occur during use.

1 Scope and field of application

This International Standard specifies a conventional heating gravimetric method for the determination of the apparent oil content of the calcined coke used in the production of aluminium.

The method provides two ways of determination :

A : normal and reference determination (6.2.1);

B : simplified determination (6.2.2);

2 References

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

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

ISO 6375, *Carbonaceous materials for the production of aluminium — Cokes for electrodes — Sampling.*

3 Principle

Heating of a test portion in a non-oxidizing atmosphere at a fixed temperature and determination of the loss in mass.

Calculation of the apparent oil content in terms of loss of mass.

To ensure a non-oxidizing atmosphere, add some drops of toluene to the crucibles.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Porcelain crucibles, capacity approximately 150 ml, fitted with lids.

4.2 Electric oven, capable of being controlled between 105 and 110 °C.

4.3 Electric furnace, capable of being controlled up to 800 °C.

4.4 Steel box, resistant at 550 °C, with lid, of dimensions 200 mm × 100 mm × 100 mm.

5 Sampling and sample

5.1 Sampling

Sampling shall be carried out according to the procedure specified in ISO 6375.

5.2 Preparation of test sample

Using a mortar and pestle, crush approximately 200 g of the laboratory sample (see ISO 6375) sufficiently to pass through a sieve of nominal mesh apertures 4 mm (see ISO 565). Dry the crushed and sieved product in the electric oven (4.2), controlled at a temperature between 105 and 110 °C, for 1 h, or a longer period, until two successive weighings do not differ by more than 0,01 g.

6 Procedure

6.1 Test portion

Heat two of the crucibles, with their lids (4.1), for about 1 h in the electric furnace (4.3) at a temperature of about 800 °C. Allow them to cool to ambient temperature in a desiccator and weigh each of them, without their lids, to the nearest 0,001 g. Weigh into each crucible, to the nearest 0,001 g, a test portion of approximately 75 g from the test sample (5.2). Add a few drops of toluene to the crucibles to ensure a non-oxidizing atmosphere.

6.2 Determination

6.2.1 A : Normal and reference determination

Put the lids on each of the porcelain crucibles containing the test portion (6.1) and place them in the steel box (4.4). Embed the crucibles into screened petroleum coke of a particle size of 0,25 to 2 mm, as indicated in the figure. Cover the steel box and place it in the cold electric furnace (4.3). Heat up the furnace to a temperature of 550 ± 10 °C and maintain this temperature for 2 h. After this period, shut down the furnace and let it cool.

Remove the steel box from the furnace and take out the porcelain crucibles. Take care to avoid contamination of the samples with petroleum coke particles of the packing material. Brush and wipe away adhering dust from the still covered crucibles and place them in a desiccator to cool to room temperature. Remove the lids and weigh each crucible with its contents, to the nearest 0,001 g.

NOTE — The determination may be also carried out introducing directly the steel box containing the crucibles in the furnace heated to 550 ± 10 °C. In this case, the box should be left in the furnace for 2,5 h. After this period remove the box from the hot furnace and let it cool covered on a refractory plate. Then proceed as indicated above.

6.2.2 B : Simplified determination

Put the lids on each of the porcelain crucibles containing the test portion (6.1) and place them in the electric furnace, previously heated to 500 ± 10 °C. Wait until the temperature of the furnace, which falls, returns to the value indicated, and maintain the crucibles at this temperature for 20 min. After this period, remove the crucibles from the furnace and allow them to cool slightly on a refractory plate. Transfer them to a desiccator and allow to cool to ambient temperature. Remove the lids, and weigh each crucible with its contents, to the nearest 0,001 g.

7 Expression of results

7.1 Method of calculation

The apparent oil content, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the crucible containing the test portion before heating;

m_2 is the mass, in grams, of the crucible containing the test portion after heating.

Report the mean value to the nearest 0,01 % of two simultaneous determinations.

7.2 Precision (according to ISO 5725)

7.2.1 A : Normal determination (six laboratories)

m	(mean)	= 0,256 %
r	(repeatability)	= 0,050
R	(reproducibility)	= 0,078

7.2.2 B : Simplified determination (same sample; three laboratories)

m	(mean)	= 0,237 %
r	(repeatability)	= 0,036
R	(reproducibility)	= r (because of the negative value of the variance of reproducibility)

The results are rounded to the third decimal place.

8 Test report

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used [normal reference determination (6.2.1) or simplified determination (6.2.2)];

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

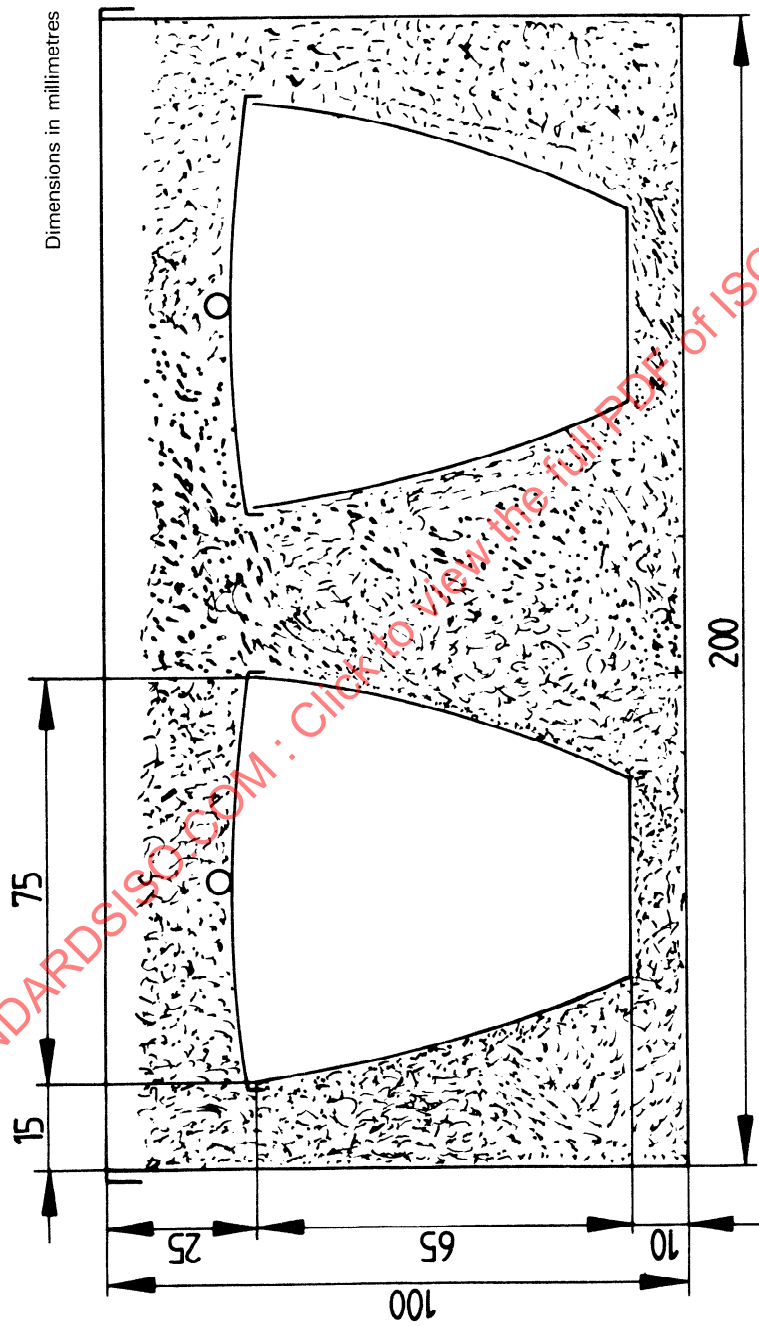

Figure — Arrangement of the crucibles in the heat-resistant steel box

This page intentionally left blank