INTERNATIONAL STANDARD

ISO 10237

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Carbonaceous materials for use in the production of aluminium — Calcined coke — Determination of residual-hydrogen content

Produits carbonés utilisés pour la production de l'aluminium — Coke calciné — Détermination de la teneur en hydrogène résiduel

Reference number
ISO 10237:1997(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 10237 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.*

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet: central@iso.ch
X.400: c=ch; a=400net; p=iso; o=isocs; s=central

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Carbonaceous materials for use in the production of aluminium — Calcined coke — Determination of residual-hydrogen content

1 Scope

Green coke is calcined prior to being used for the fabrication of anodes for the electrolytic production of aluminium. A criterion for the degree of calcination is the hydrogen residue.

This International Standard specifies a method for determining the residual-hydrogen content of calcined coke used in the production of aluminium. The method is only applicable to products having a residual-hydrogen content of less than 1 % (*m*/*m*).

NOTE — At concentrations of less than 1 % (*m*/*m*), hydrogen is mainly bound to condensed aromatic rings and less to side chains.

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 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

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

3 Principle

A crushed and dried test portion is completely combusted at 750 °C in a stream of oxygen. The gases evolved are passed over copper(II) oxide and lead(II) chromate. The water that has formed, in which the hydrogen of the sample is bound, is subsequently absorbed in a drying agent. The hydrogen content of the test portion is calculated from the increase in mass of the drying agent.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Copper(II) oxide (CuO), in wire form.

4.2 Lead(II) chromate ($PbCrO_4$), particle size approximately 1 mm to 3 mm.

4.3 Sulfuric acid (H_2SO_4), with a concentration by mass between 94 % (*m/m*) and 96 % (*m/m*).

or

4.4 Magnesium perchlorate [$Mg(ClO_4)_2$].

4.5 Synthetic graphite, hydrogen-free, grain size ⩽ 1 mm, water content ⩽ 0,001 % (*m/m*).

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Heating cabinet, capable of being maintained at a temperature of 220 °C ± 10 °C.

5.2 Balance, accurate to 0,1 mg.

5.3 Desiccator, with a suitable drying agent.

5.4 Wire screen, mesh 1 mm.

5.5 Combustion apparatus, as shown in figure 1, consisting of the items specified in 5.5.1 to 5.5.15.

5.5.1 Oxygen cylinder (1).

5.5.2 Pressure reducer (2).

5.5.3 Flowmeter (3a), e.g. a variable-section flowmeter, capable of measuring a gas throughput of 40 l/h.

5.5.4 Three 500 ml gas washing bottles (4, 5, 6), the first empty to remove foreign bodies, the second filled to about one-third with sulfuric acid (4.3) to dry the oxygen and the third empty to retain entrained sulfuric acid.

5.5.5 Quartz tube (7), diameter 20 mm, length 900 mm, containing two rolls of copper wire gauze with a copper content of more than 95 % (*m/m*) and a mesh of 0,3 mm to 0,7 mm, the space between the rolls being filled with copper(II) oxide (4.1).

5.5.6 Gas-cleaning furnace (8), surrounding the tube (7) and capable of being maintained at a temperature of 425 °C ± 25 °C.

5.5.7 350 ml gas washing bottle (9), with connector, sintered disc (porosity grade P160 as defined in ISO 4793, maximum inner pore size 100 µm to 160 µm, diameter 60 mm) and ball (minimum diameter 60 mm) and filled with sulfuric acid (4.3) to a level 20 mm to 30 mm above the sintered disc.

5.5.8 250 ml gas washing bottle (10), with sintered disc (porosity grade P160 as defined in ISO 4793), to retain entrained sulfuric acid.

5.5.9 Pipeline filter (11), porosity grade P160 as defined in ISO 4793, diameter 60 mm.

5.5.10 Mercury safety valve (12), with drilled stopper and a volume of 250 ml.

5.5.11 Stopcock (13) **and flowmeter** (3b), e.g. a variable-section flowmeter capable of measuring a gas throughput of 14 l/h.

5.5.12 Combustion furnace (15), capable of being maintained at a temperature of 750 °C ± 10 °C.

5.5.13 Quartz combustion tube (14), diameter 32 mm, length 1 500 mm, containing, at one end, copper(II) oxide (4.1) and lead(II) chromate (4.2) held in place by rolls of copper wire gauze with a copper content of more than 95 % (*m/m*) and a mesh of 0,3 mm to 0,7 mm.

When filling the combustion tube, a space of 20 mm shall be left between the lead(II) chromate and the copper wire gauze to allow for expansion of the lead(II) chromate.

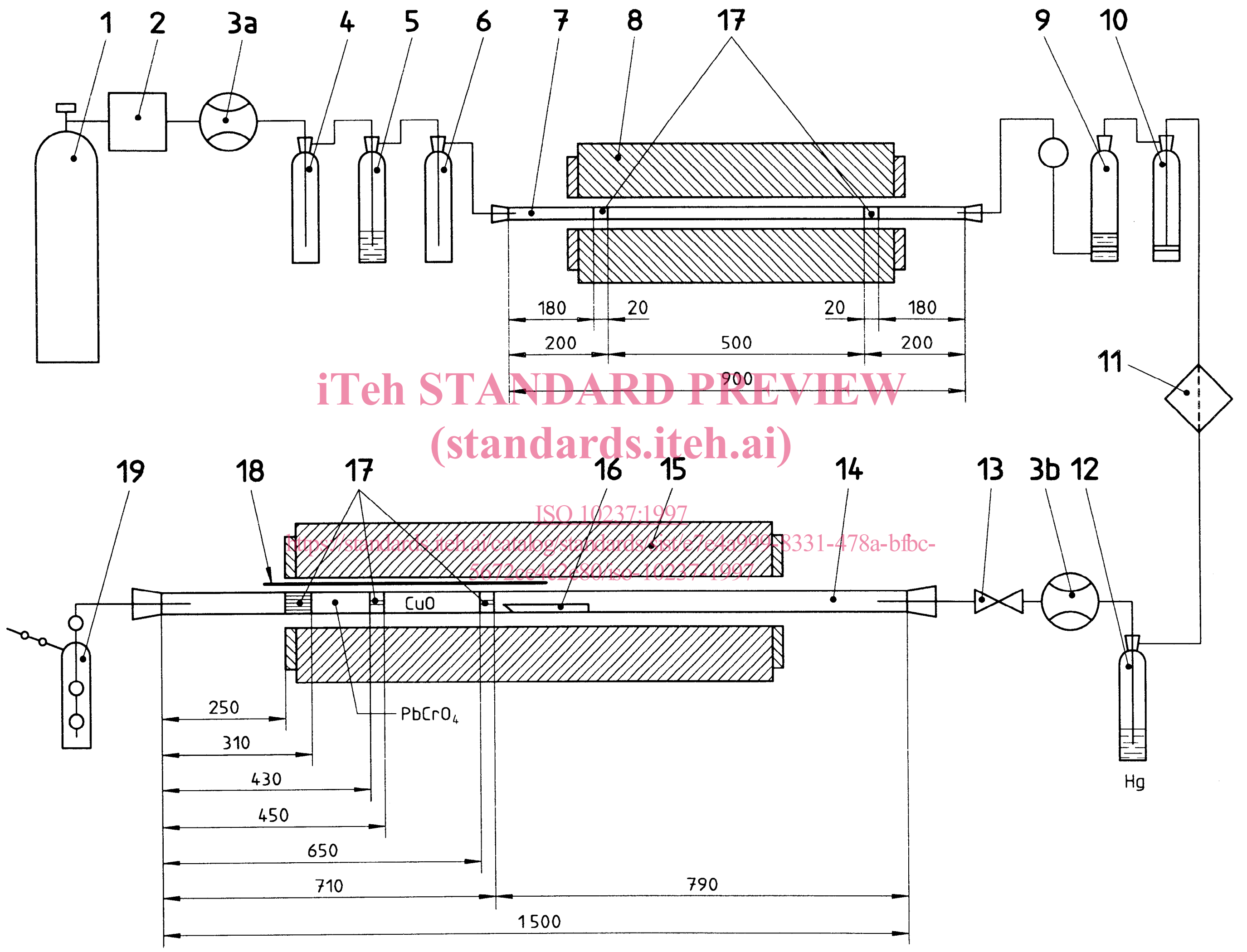
NOTE — Several combustion furnaces may be connected to the same oxygen supply by inserting a stopcock manifold in place of the single stopcock at 13.

5.5.14 Absorption unit (19) (see also figure 2), containing sulfuric acid (4.3) or magnesium perchlorate (4.4).

5.5.15 Stoppers and flexible tubing connections, made of silicone rubber.

5.6 Porcelain combustion boat (16), measuring 160 mm × 14 mm × 20 mm.

Dimensions in millimetres



Key

1 Oxygen cylinder
2 Pressure reducer
3a, 3b Flowmeters (e.g. variable-section flowmeters)
4, 5, 6 500 ml gas washing bottles
7 Quartz tube
8 Gas-cleaning furnace
9 350 ml gas washing bottle with sintered disc and ball
10 250 ml gas washing bottle with sintered disc
11 Pipeline filter
12 Over pressure safety bottle, containing mercury
13 Stopcock
14 Combustion tube
15 Combustion furnace
16 Porcelain boat
17 Copper wire gauze
18 Thermocouple
19 Absorption unit (see figure 2)

Figure 1 — General layout of combustion apparatus

Dimensions in millimetres

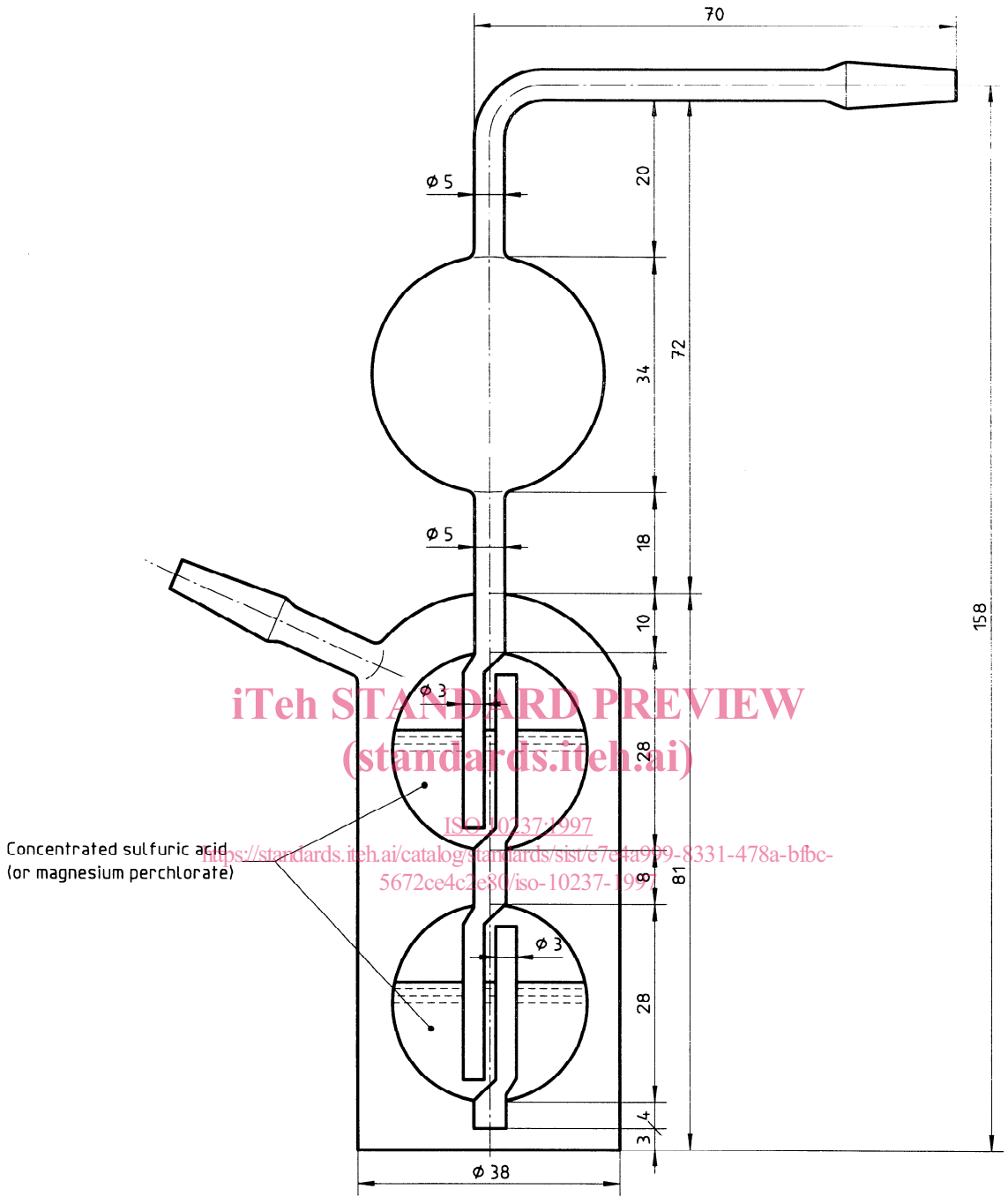


Figure 2 — Absorption unit

6 Sampling and preparation of test portion

6.1 Sampling

Sampling shall be carried out in accordance with ISO 6375.

6.2 Preparation of test portion

Crush the sample to a grain size ⩽ 1 mm and dry in the cabinet (5.1) for 2 h at 220 °C ± 10 °C. Keep the dried sample in the desiccator (5.3) until needed. For each analysis, a test portion of 10 g is normally required. If the

hydrogen concentration exceeds 0,3 % (*m*/*m*), however, reduce the test portion to 5 g and mix it with about the same quantity of hydrogen-free synthetic graphite (4.5) to dilute it.

In the case of oil-sprayed carbon materials, extract the spraying agent with dichloromethane (CH_2Cl_2).

7 Procedure

Set the oxygen flow through the combustion apparatus by means of the flowmeters (3a, 3b) at 8 l/h ± 1 l/h. Subsequently heat the gas-cleaning furnace (8) to 425 °C ± 25 °C and the combustion furnace (15) to 750 °C ± 10 °C.

Connect the absorption unit (19) to the apparatus for 20 min, then disconnect it, close it and after allowing it to reach room temperature (18 °C to 28 °C), weigh it to the nearest 0,1 mg.

Weigh, to the nearest 0,1 mg, about 10 g (or 5 g – see 6.2) of the sample into the porcelain boat (5.6). Immediately push the boat into the combustion tube (14) to the centre of the combustion furnace (15). Then reconnect the combustion tube to flowmeter 3b and check that the oxygen flow is still 8 l/h ± 1 l/h. After 5 min, connect the weighed absorption unit to the combustion tube. After 3,5 h, disconnect and close the absorption unit, allow it to reach a temperature between 18 °C and 28 °C, and weigh it to the nearest 0,1 mg.

Run blank determinations (without a test portion) to check the apparatus. The test should last at least 1 h and the increase in the mass of the absorption unit shall be ≤ 1 mg.

8 Expression of results

8.1 Calculation

The hydrogen content $w(H_2)$ is given, as a percentage by mass, by the equation

$$w(H_2) = \frac{(m_2 - m_1) \times f}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the absorption unit prior to the determination;

m_2 is the mass, in grams, of the absorption unit after the determination;

f is the ratio of the relative molecular mass of hydrogen to that of water (i.e. 2/18).

8.2 Precision

The precision data for this method were obtained in accordance with ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests* (now withdrawn).

8.2.1 Repeatability, *r*

Two successive results obtained by the same operator, working with the same apparatus under constant conditions and using the same test material, are considered acceptable if they do not differ by more than 0,005 % (absolute).

8.2.2 Reproducibility, *R*

Two independent results obtained by different operators, working in different laboratories under comparable conditions and using the same test material, are considered acceptable if they do not differ by more than 0,01 % (absolute).

9 Test report

The test report shall include at least the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the sample;

c) the number of test portions analysed;

d) the hydrogen content of each test portion, expressed as a percentage by mass to the nearest 0,001 % (m/m);

e) the mean value of the individual determinations;

f) details of any operation not included in this International Standard or in the International Standards to which reference is made, as well as details of any incident which may have affected the results;

g) the date of the test.

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