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**Carbonaceous materials used in the  
production of aluminium — Calcined  
coke — Determination of the reactivity to  
air —**

Part 1:

**Ignition temperature method**

*(standards.iteh.ai)*

*Produits carbonés utilisés pour la production de l'aluminium — Coke  
calciné — Détermination de la réactivité à l'air —*

*ISO 12982-1:2000*

*Partie 1: Méthode de la température d'inflammabilité*

*fd7c7cecf07f8/iso-12982-1-2000*



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## 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 12982 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12982-1 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*.

ISO 12982 consists of the following parts, under the general title *Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of the reactivity to air*:

— Part 1: *Ignition temperature method*

A thermogravimetric method will be the subject of a future part 2 to ISO 12982.

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# Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of the reactivity to air —

## Part 1: Ignition temperature method

### 1 Scope

This part of ISO 12982 describes an ignition temperature method for the determination of the reactivity to air of calcined petroleum coke used in the manufacture of anodes for the production of aluminium. A heating rate of 5 °C/min is used for petroleum coke specifications, whereas 10 °C/min is used for statistical process control of calcination kilns and for anode butt quality control.

NOTE ISO 12982-2 (in preparation) will give a thermobalance method for the determination of the reactivity to air of calcined coke.

### 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 12982. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 12982 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 8723, *Carbonaceous materials for the production of aluminium — Calcined coke — Determination of oil content — Method by solvent extraction.*

ISO 12984, *Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of particle size distribution.*

### 3 Principle

The reactivity to air of calcined coke is measured by calculation after determining the ignition temperature of a sample exposed to air, and thus oxygen.

A coke sample of 5 g having a grain size of 1 mm to 1,4 mm is exposed to an air stream of 50 l/h in a furnace heated at 10 °C/min or 0,5 °C/min, depending on the sample history and the intended application. Ignition is assumed to occur at the point where a sudden rise in the sample temperature occurs.

The reactivity to air is calculated using a correlation derived from thermogravimetric measurements made on coke samples<sup>[1][2]</sup>.

### 4 Reagents

**4.1 Air**, bottled or compressed, containing less than 100 mg/kg free water.

**4.2 Certified calibration standard**, having an ignition temperature of about 620 °C at a heating rate of 10 °C/min.

## 5 Apparatus

Ordinary laboratory apparatus and the following.

**5.1 Furnace**, capable of heating from 20 °C to 1 000 °C in less than 1 h. A vertical, single-zone tube furnace that ensures a good vertical temperature distribution shall be used. A furnace having suitable dimensions is shown in Figure 1.

**5.2 Tube reactor**, consisting of two quartz tubes and a cap, with ground-glass joints, assembled as described in 5.2.1 to 5.2.4.

NOTE A tube reactor having suitable dimensions for the furnace is shown in Figure 2 and a diagram of a complete apparatus is given in Figure 3.

**5.2.1 External tube**, consisting of the following:

- **gas inlet**, positioned near the top of the external tube, allowing gas to flow down to the bottom of the tube and to be preheated before flowing up through the coke bed;
- **protection tube**, for the thermocouple (5.3), positioned so that the tip of the thermocouple lies 5 mm underneath the fritted disc (5.2.3).

The gas inlet tube and the thermocouple protection tube shall extend outside the furnace.

**5.2.2 Reaction tube**, fitted inside the external tube (5.2.1).

**5.2.3 Fritted disc**, having a pore size of 250 µm to 500 µm, fitted inside the reaction tube and positioned so that the base of the coke bed lies in the middle of the furnace.

**5.2.4 Cap**, containing a gas outlet, clamped to the top of the reaction tube.

**5.3 Thermocouple**, chromel alumel, K-type, having an accuracy of better than  $\pm 0,375$  %, a diameter of 2 mm and a minimum length of 200 mm.

**5.4 Programmable temperature control unit**, consisting of a two-point temperature DPID controller with adjustable heating rate and temperature display.

**5.5 Chart recorder**, to record the temperature of the test sample versus time. Alternatively, a microprocessor which automatically detects the ignition temperature can be used.

**5.6 Flow meter**, with a calibrated scale for air ( $p = 0,1$  MPa), having a full-scale reading of 60 l/h and an accuracy of better than  $\pm 2$  %.

**5.7 Pressure control**, comprising a valve to regulate the pressure and a manometer having a scale reading from 0 MPa to 1,0 MPa.

## 6 Sampling

Take a sample of the coke in accordance with the procedure specified in ISO 6375.

Dimensions in millimetres

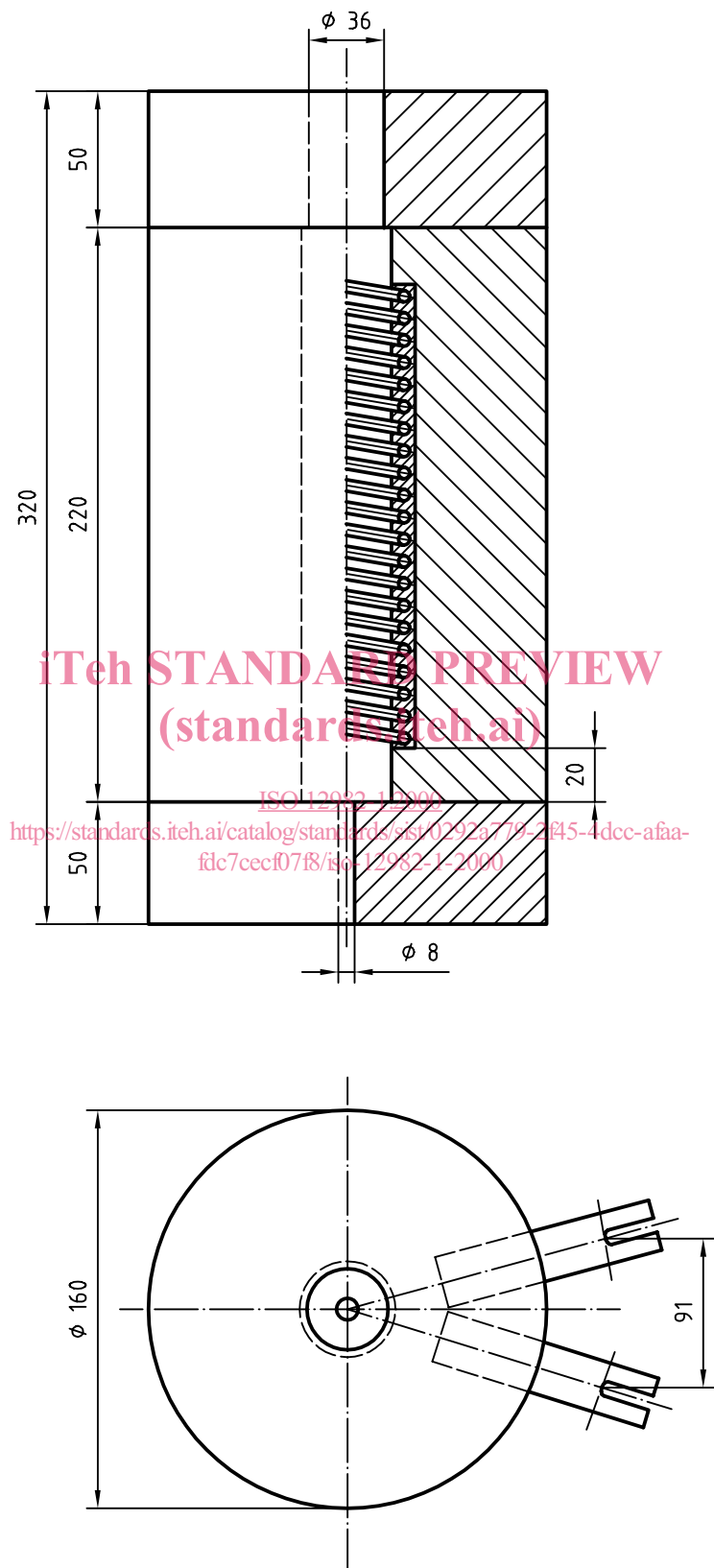
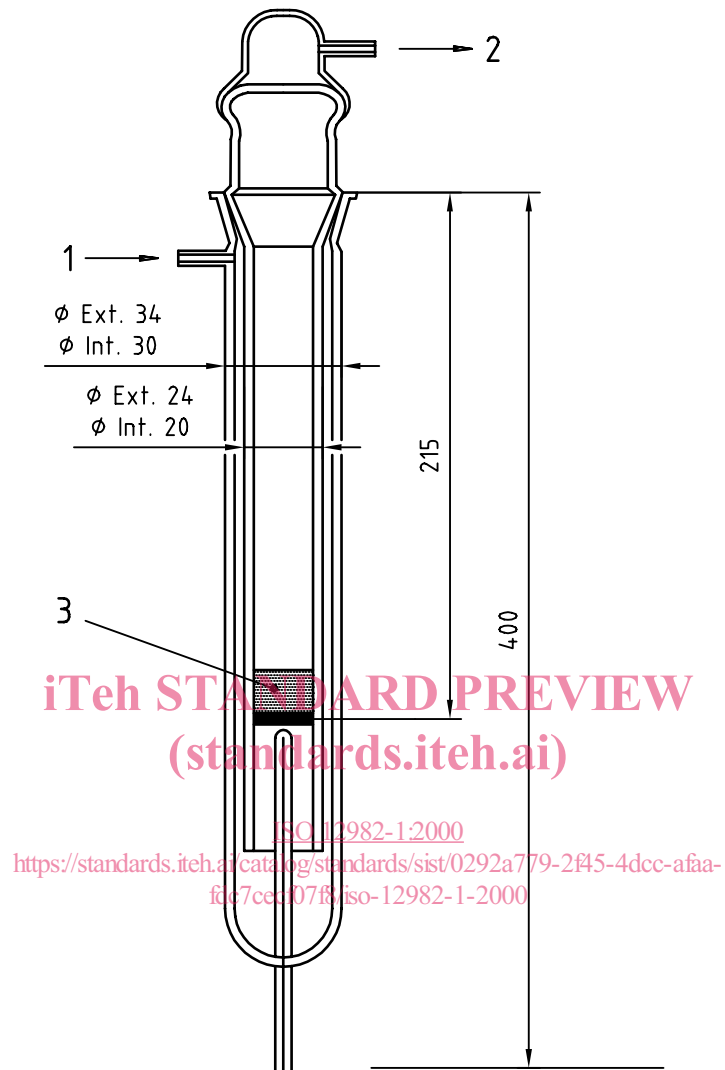


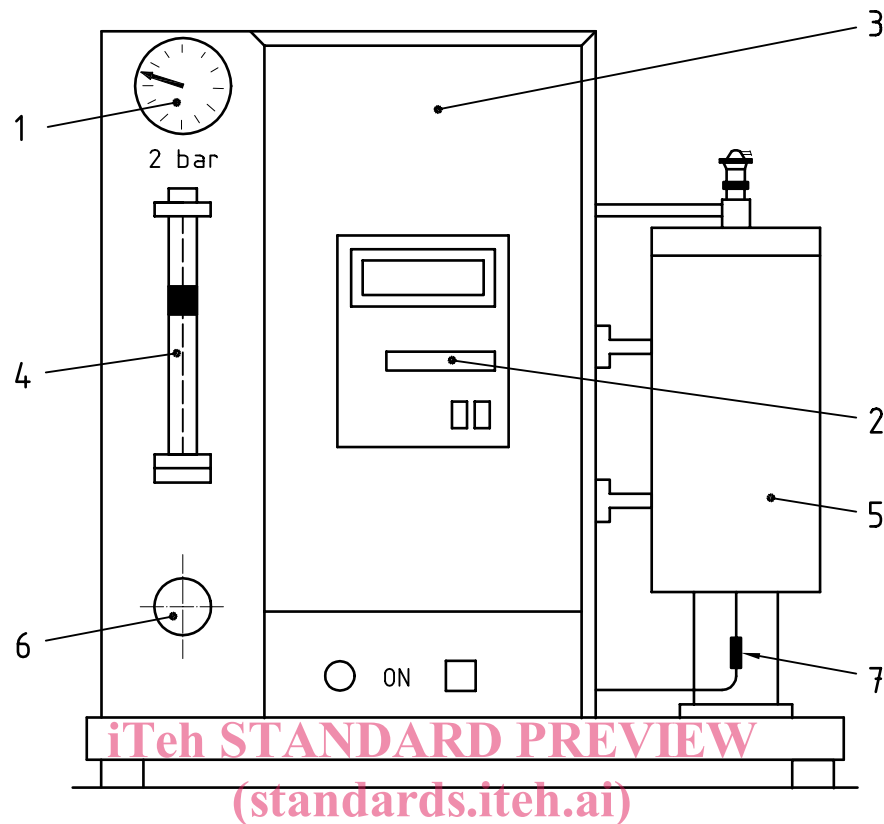
Figure 1 — Characteristics and dimensions of a typical furnace



- Key**
- 1 Air inlet
  - 2 Air outlet
  - 3 Test sample

**Figure 2 — Tube reactor with test sample**



**Key**

1	Manometer	<a href="https://standards.iteh.ai/catalog/standards/sist/0292a779-2f45-4dcc-afaa-fdc7cecf07f8/iso-12982-1-2000">ISO 12982-1:2000</a>
2	Chart recorder	<a href="https://standards.iteh.ai/catalog/standards/sist/0292a779-2f45-4dcc-afaa-fdc7cecf07f8/iso-12982-1-2000">https://standards.iteh.ai/catalog/standards/sist/0292a779-2f45-4dcc-afaa-fdc7cecf07f8/iso-12982-1-2000</a>
3	Reactivity-to-air apparatus	
4	Gas flow meter	
5	Furnace	
6	Pressure valve	
7	Thermocouple	

**Figure 3 — Reactivity-to-air apparatus****7 Preparation of test sample**

Divide the sample into three fractions by sieving in accordance with ISO 12984. The fractions shall have the following dimensions:

I > 1,4 mm

IIa 1 mm to 1,4 mm

III < 1 mm

Crush fraction I to produce fraction IIb so that most of fraction IIb has the following dimensions after sieving:

IIb 1 mm to 1,4 mm

Thoroughly mix fractions IIa and IIb.