INTERNATIONAL STANDARD

ISO 12982-1

First edition
2000-12-15

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

Part 1: Ignition temperature method

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

Partie 1: Méthode de la température d'inflammabilité

Reference number
ISO 12982-1:2000(E)

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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.

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[1][2].

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 ± 0,375 %, a diameter of 2 mm and a minimum length of 200 mm.

5.4 Progammable 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 ± 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

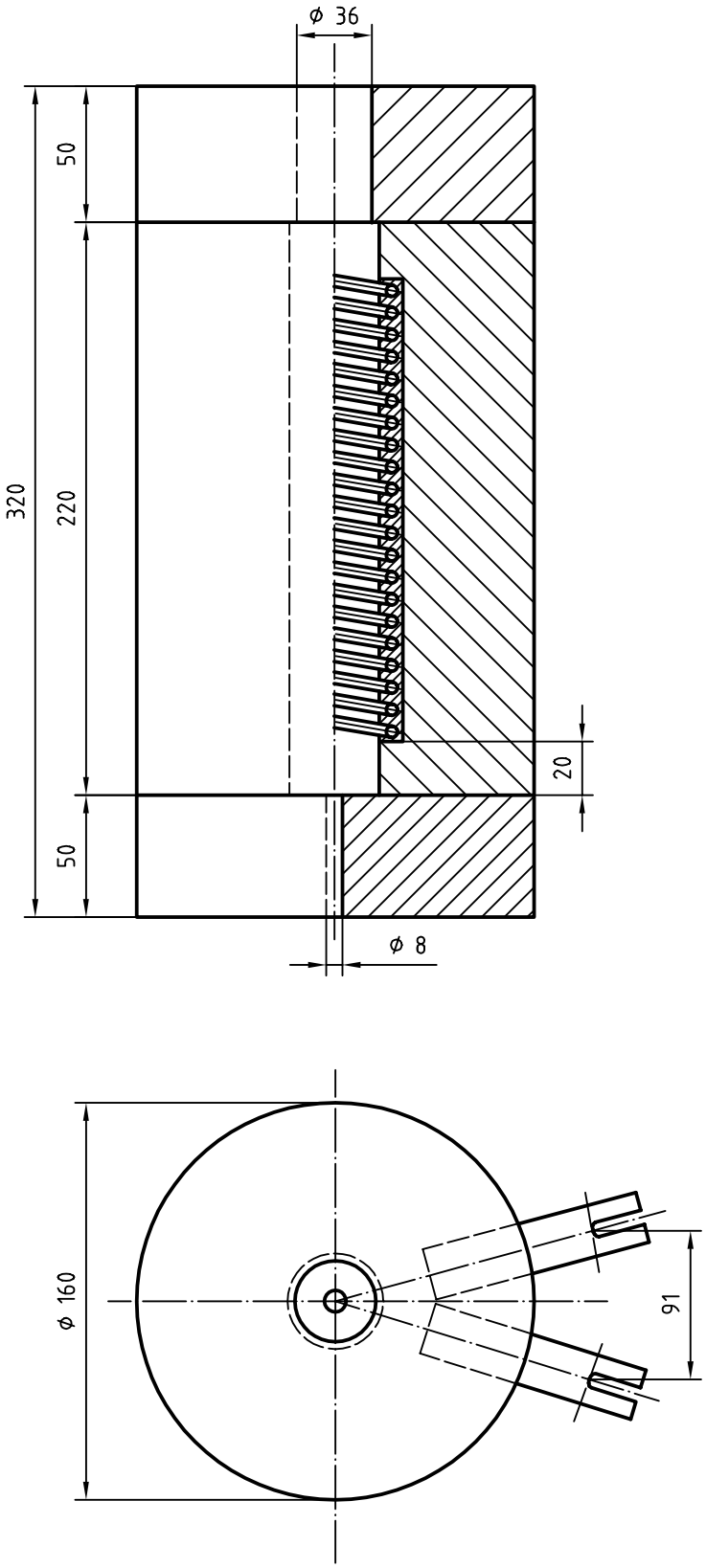
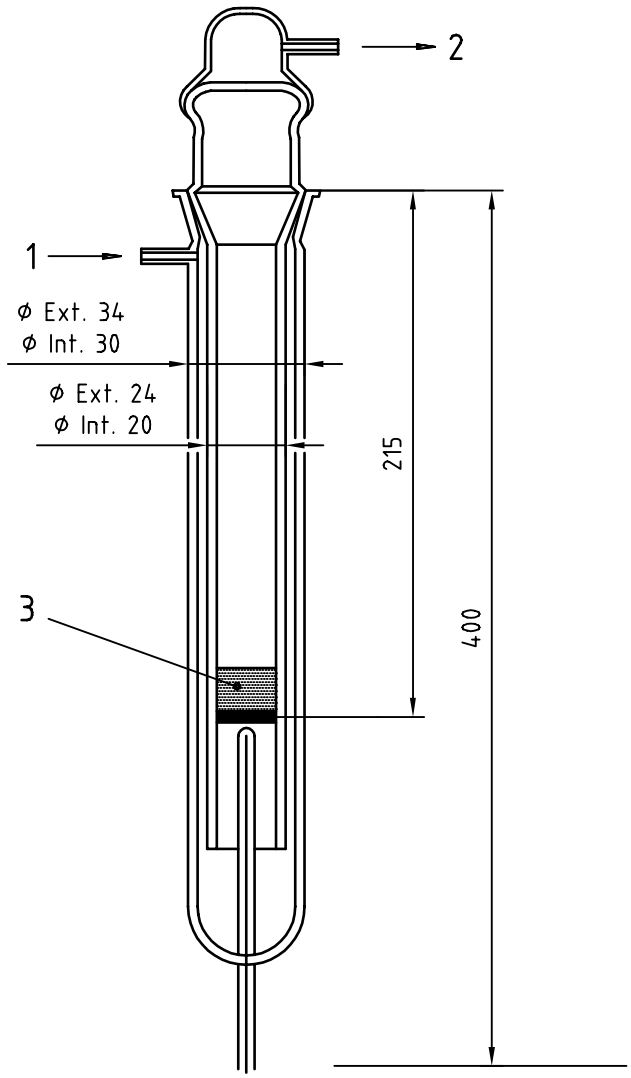


Figure 1 — Characteristics and dimensions of a typical furnace

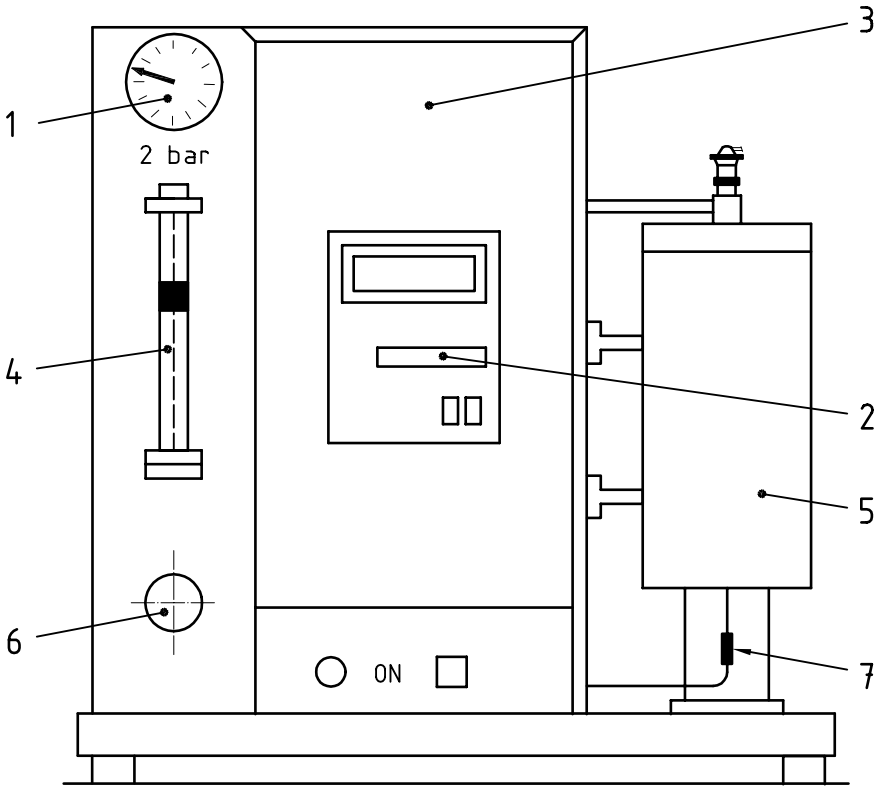
Dimensions in millimetres



Key

1	Air inlet
2	Air outlet
3	Test sample

Figure 2 — Tube reactor with test sample



Key

1	Manometer
2	Chart recorder
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.

Many granular materials are coated with oil. In such cases, remove the oil from the mixture of fractions IIa and IIb with dichloromethane, using the procedure specified in ISO 8723.

Dry the mixture of fractions IIa and IIb at (110 ± 5) °C to constant mass, i.e. until consecutive weighings at 5 min intervals differ by less than 0,1 %.

Take a test sample of $(5 \pm 0{,}01)$ g from the mixture of fractions IIa and IIb and weigh it to the nearest 0,001 g.

8 Procedure

8.1 Calibration

Carry out two measurements on a calibration standard having an ignition temperature T_R, and calculate the mean of the results, T_M, in order to calibrate the thermocouple which measures the sample temperature. Perform the calibration procedure once a week and after any maintenance of the apparatus (replacement of reactor tube or thermocouple, etc.). Calibrate the chart recorder periodically.

Calculate the difference between the calibration standard reference temperature and the actual temperature determined, ($T_M - T_R$), which is subtracted from the values determined for test samples.

8.2 Determination

Programme the temperature controller in accordance with Table 1 for the desired application.

Table 1 — Temperature controller programmes

Application	Fast method	Slow method
Standby temperature	300 °C	450 °C
Temperature gradient	10 °C/min	0,5 °C/min

Switch on the furnace, insert the empty inner quartz tube and fix the cover with the clamp. Heat the furnace to the standby temperature. Open the air valve and regulate the pressure to 0,2 MPa and the flow rate to 50 l/h. When the furnace temperature has stabilized at the standby temperature (± 3 °C), insert the weighed test sample into the reaction tube and start to increase the furnace temperature, plotting the temperature of the sample on the chart recorder. When a sharp increase in the sample temperature occurs, stop the furnace and the gas flow.

9 Calculation and expression of results

Determine the ignition temperature, T_I, of the coke from the time-temperature plot by extending the two linear sections of the curve (before and after ignition) towards the point at which the sharp increase in temperature occurs. Read off the ignition temperature as the point of intersection of the lines.

Express the ignition temperature, T_I, to the nearest 0,1 °C.

If a heating rate of 0,5 °C/min was used, calculate the reactivity to air at 525 °C, $\dot{\omega}_{525}$, expressed as a percentage loss in mass per minute, using the equation

$$\log\dot{\omega}_{525} = -9{,}519 - \left(\frac{4{,}159 \times 10^2}{T_I}\right) + \left(\frac{6{,}158 \times 10^6}{T_I^2}\right)$$

or, if a heating rate of 10 °C/min was used, calculate the reactivity to air at 600 °C, $\dot{\omega}_{600}$, expressed as a percentage loss in mass per minute, using the equation

$$\dot{\omega}_{600} = -50{,}064 + \left(\frac{75\,364}{T_I}\right) - \left(\frac{2{,}798\,3 \times 10^7}{T_I^2}\right)$$

where T_i is the measured ignition temperature, in kelvins, for the heating rate used (for the derivation of these equations, see references [1] and [2]).

Express the reactivity to air to the nearest 0,01 %/min.

NOTE Alternatively, the above calculations may be carried out automatically by a microprocessor.

10 Precision

10.1 Repeatability

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus but at different times on representative test samples taken from the same laboratory sample, should not differ by more than the figures given in Table 2 for the "Fast method" (10 °C/min) and the figures given in Table 3 for the "Slow method" (0,5 °C/min).

10.2 Reproducibility

The means of the results of duplicate determinations, carried out in each of two laboratories on representative test samples taken from the same sample after the last stage of sample preparation, should not differ by more than the figures given in Table 2 for the "Fast method" (10 °C/min) and the figures given in Table 3 for the "Slow method" (0,5 °C/min).

Table 2 — Fast method (10 °C/min)

Parameter	Repeatability	Reproducibility
Ignition temperature	3 °C	6 °C
Reactivity to air at 600 °C in %/min	10 % relative	20 % relative

Table 3 — Slow method (0,5 °C/min)

Parameter	Repeatability	Reproducibility
Ignition temperature	3 °C	6 °C
Reactivity to air at 525 °C in %/min	20 % relative	40 % relative

Figures 4 and 5 show the precision as a function of the reactivity to air for each of the methods.

11 Test report

The test report shall include the following information:

a) complete identification of the sample tested;

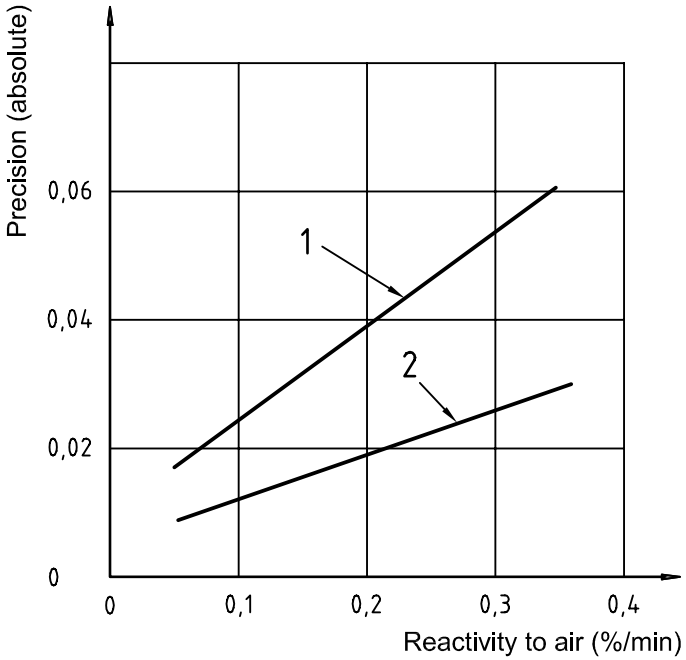
b) a reference to this part of ISO 12982, i.e. ISO 12982-1;

c) the date of the test and the results, expressed in accordance with clause 9;

d) any unusual features noted during the determination;

e) any operation not included in this part of ISO 12982 or in the International Standards to which reference is made, or regarded as optional;

f) the name and address of the certifying organization for the calibration standard.

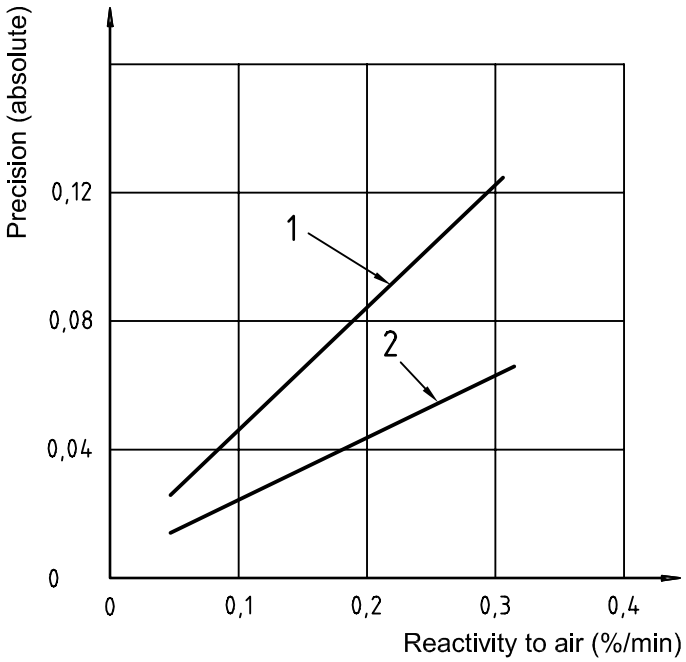


Key

1 Reproducibility, R

2 Repeatability, r

Figure 4 — Precision as function of the reactivity to air at 600 °C



Key

1 Reproducibility, R

2 Repeatability, r

Figure 5 — Precision as function of the reactivity to air at 525 °C

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[2] HUME, PERRUCHOUD, FISCHER and WELCH: Model for petroleum coke reactivity, *Light Metals*, Subodh K. Das, AIME Denver, 1993, pp. 525-531.

ICS 71.100.10

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