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

**Part 1:
Loss in mass method**

*Produits carbonés utilisés pour la production de l'aluminium —
Coke calciné — Détermination de la réactivité au dioxyde de carbone —*

Partie 1: Méthode par perte de masse



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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 12981 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

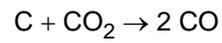
International Standard ISO 12981-1 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

ISO 12981 consists of the following parts, under the general title *Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of the reactivity to carbon dioxide*:

- *Part 1: Loss in mass method*
- *Part 2: Thermogravimetric method*

Introduction

The reactivity of a calcined coke to carbon dioxide is assessed by determining the loss in mass of a sample exposed in accordance with the following chemical reaction:



This determination allows an assessment of the later anode reactivity to carbon dioxide in the electrolysis cell.

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

Part 1: Loss in mass method

1 Scope

This part of ISO 12981 specifies a method for the determination, by a loss in mass method, of the reactivity of calcined petroleum coke, used in the manufacture of anodes for the production of aluminium, to carbon dioxide.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 12981. 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 12981 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 383, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 5725-2, *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, *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

A coke sample of 5 g having a grain size of 1 mm to 1,4 mm is exposed to a carbon dioxide (CO₂) stream of 50 l/h for approximately 100 min at 1 000 °C. The loss in mass is then measured.

4 Reagents

4.1 CO₂, having a minimum purity of 99,9 % by volume and maximum impurity levels of

- O₂: 100 µmol/mol,
- H₂O: 130 µmol/mol,
- N₂ + Ar: 800 µmol/mol.

4.2 Calibration standard, certified with a precisely known value of reactivity to carbon dioxide of about 10 % by mass.

NOTE Suitable materials are commercially available.

5 Apparatus

A diagram of a complete apparatus is shown in Figure 1.

5.1 Ordinary laboratory apparatus.

5.2 Furnace, with a vertical, single-zone tube ensuring good vertical temperature distribution and capable of heating from 20 °C to 1 000 °C in less than 1 h and maintaining the temperature at $(1\ 000 \pm 3)$ °C.

The characteristics and dimensions of a typical furnace are shown in Figure 2.

5.3 Tube reactor, consisting of two quartz tubes and a cap, with ground-glass joints (ISO 383, NS 29/32).

The characteristics and dimensions of a typical tube reactor assembly are shown in Figure 3 with the following components:

5.3.1 External tube, containing

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

The bottom of the thermocouple protection tube and the gas inlet shall be external to the furnace.

5.3.2 Inner reaction tube, fitted inside the external tube.

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

5.3.4 Cap, containing a gas outlet, clamped to the top of the inner reaction tube.

5.4 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.

5.5 Temperature control unit, consisting of a two-point temperature PID controller with a set-value adjuster (adjustment error $< 0,5$ %) and with a digital temperature display. The set-point value shall be selected so that the tube reactor thermocouple indicates 1 000 °C.

5.6 Flow meter, with a calibrated scale, for the CO₂ ($p = 0,1$ 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.

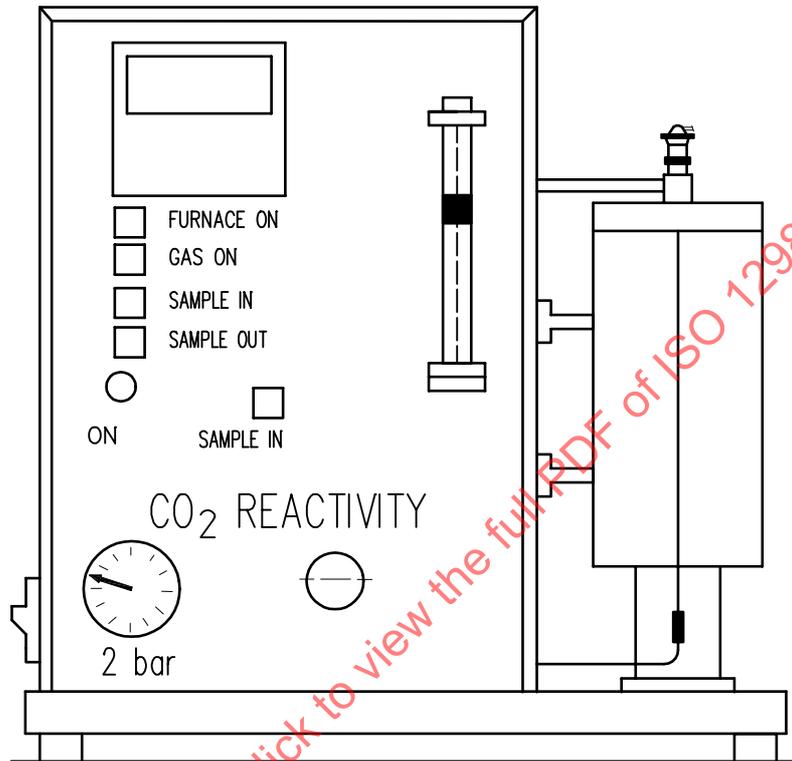


Figure 1 — CO₂ reactivity apparatus

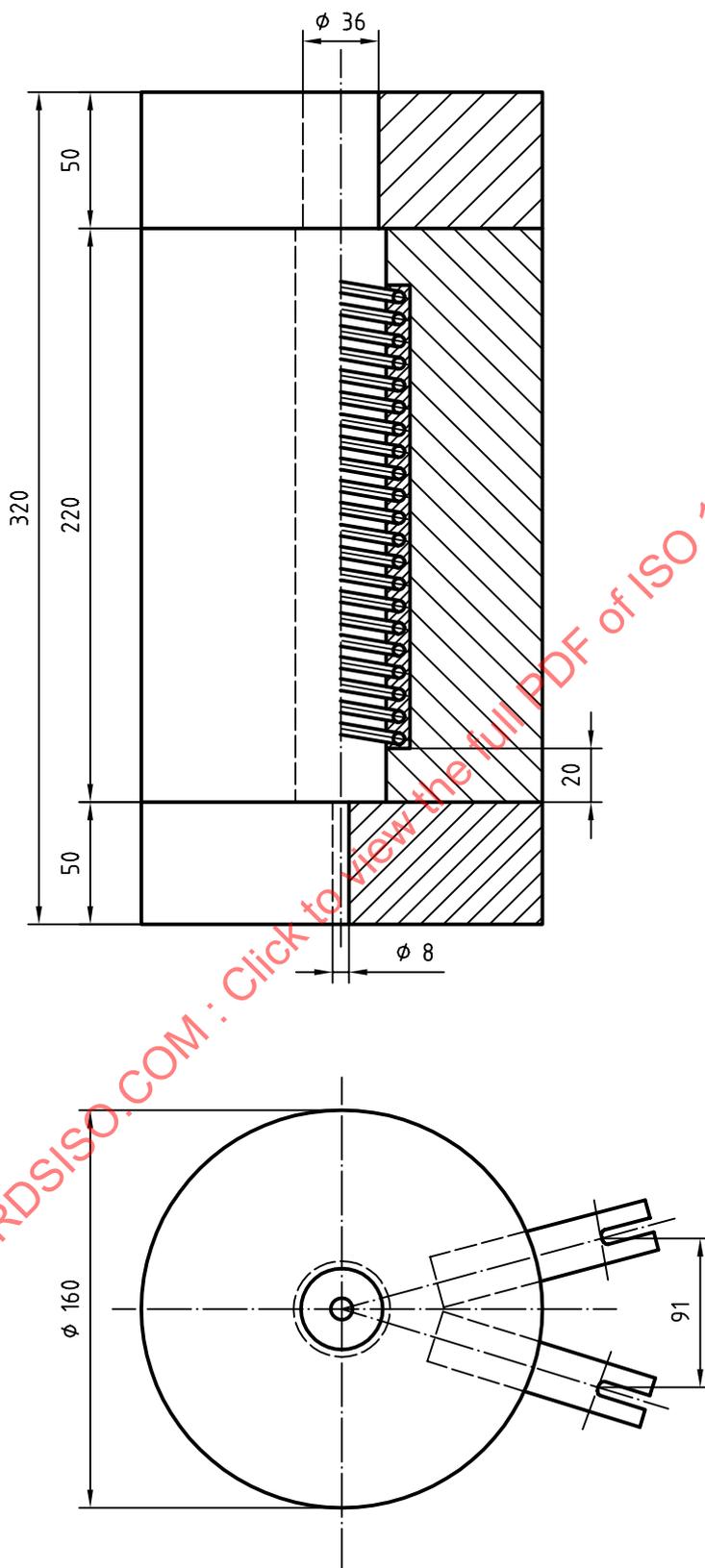
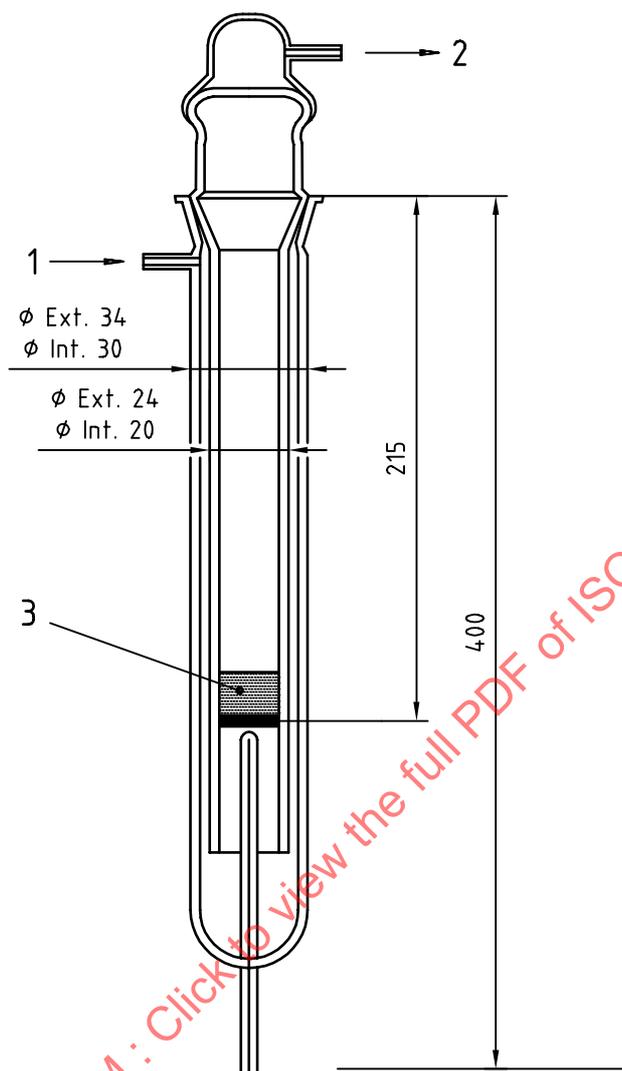


Figure 2 — Characteristics and dimensions of a typical furnace

Dimensions in millimetres

**Key**

- 1 CO₂ in
- 2 CO₂ out
- 3 Test sample (5 g)

Figure 3 — Tube reactor with test sample**6 Sampling**

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