
**Carbonaceous materials for the
production of aluminium — Calcined
coke for electrodes — Determination
of the electrical resistivity of granules**

*Produits carbonés utilisés pour la production de l'aluminium — Coke
calciné — Détermination de la résistivité électrique granulaire*

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 226, *Materials for the production of primary aluminium*.

This second edition cancels and replaces the first edition (ISO 10143:1995), which has been technically revised.

Carbonaceous materials for the production of aluminium — Calcined coke for electrodes — Determination of the electrical resistivity of granules

1 Scope

This International Standard specifies a method for the determination of the electrical resistivity of granular carbon (calcined or graphitized) used in the manufacture of carbon electrodes for the production of aluminium.

The measurement of electrical resistivity assists in assessing the extent of coke calcination. The electrical resistivity of the coke aggregate will influence that of the coke electrodes made from it.

In general, a more highly calcined coke will have a lower electrical resistivity if other factors, such as grain size, are similar.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 6997, *Carbonaceous materials for the production of aluminium — Calcined coke — Determination of apparent oil content — Heating method*

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

ISO 11412, *Carbonaceous materials for the production of aluminium — Calcined coke — Determination of water content*

3 Principle

A test portion of the granular carbon is placed in a cylindrical holder which has electrical contacts at the top and bottom. A fixed pressure is applied to the test portion to ensure good electrical contact and a fixed, constant direct current is applied. The voltage drop and the height of the column of granules are measured and the electrical resistivity is calculated.

4 Apparatus

Ordinary laboratory apparatus, plus the following:

4.1 Sample holder and plunger, with removable base for cleaning as shown in [Figure 1](#).

4.2 Length-measuring device, capable of measuring the movement of the compression plunger to $\pm 0,02$ mm.

4.3 Brass reference cylinder, having a height of $20 \text{ mm} \pm 0,01 \text{ mm}$ and a diameter of 29 mm, used for calibrating the length-measuring device ([4.2](#)).

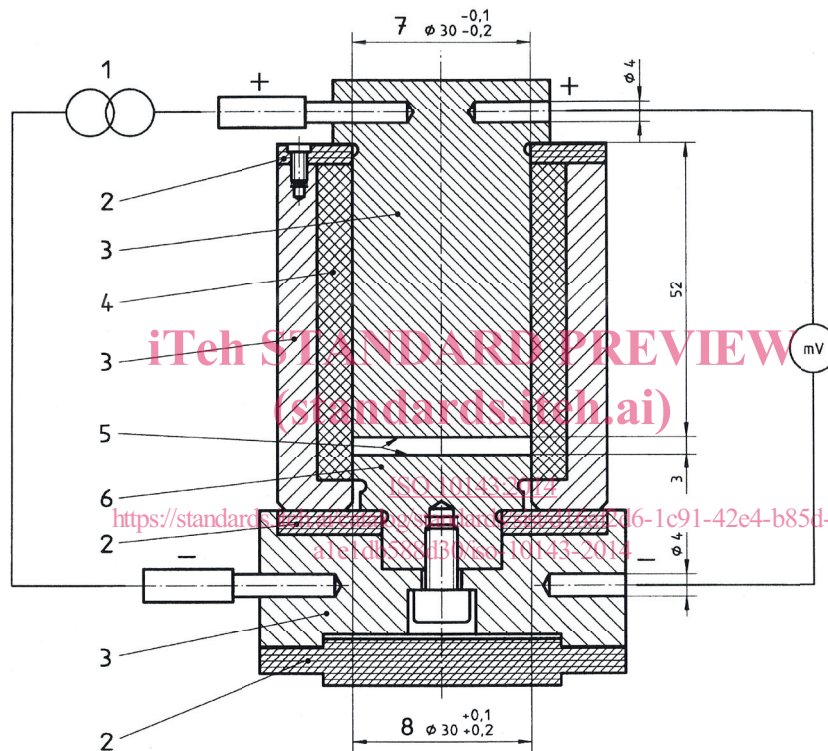
4.4 **Test machine**, capable of applying a constant pressure of 3 MPa \pm 1 % to the plunger (4.1). Giving a velocity to the plunger of not more than 50 mm/s and having a centering device for the sample holder (4.1).

4.5 **Power supply**, capable of providing a constant direct current of 1 A \pm 0,002 A.

4.6 **Voltmeter**, capable of measuring to the nearest \pm 0,01 mV.

4.7 **Oven**, capable of being maintained at 120 °C \pm 2 °C.

Dimensions in millimetres



Key

- 1 power supply
- 2 PTFE
- 3 brass
- 4 agate
- 5 hard-chromed surfaces
- 6 stainless steel
- 7 plunger
- 8 mould

Figure 1 — Sample holder and plunger, showing electrical connections

5 Sampling and sample preparation

5.1 Sampling

Sample the coke in accordance with ISO 6375.

5.2 Sample preparation calcined petroleum coke

Heat the sample to $110\text{ °C} \pm 5\text{ °C}$ during 12 h, using the oven (4.7) in compliance with ISO 11412.

De-oil the sample by washing with dichloromethane in accordance with ISO 8723 or by heating in accordance with ISO 6997.

Crush all the sample material $>1,4\text{ mm}$ in a jaw crusher with a minimum aperture of $1,5\text{ mm}$ so that all the sample material passes the $1,4\text{ mm}$ sieve.

Sieve the fraction $1,4\text{ mm}$ to $1,0\text{ mm}$ for the analysis.

5.3 Sample preparation all samples

Divide the sample, by sieving, into three fractions (I, Ila, and III) as follows:

- fraction I $>1,0\text{ mm}$;
- fraction Ila $0,5\text{ mm}$ to $1,0\text{ mm}$;
- fraction III $<0,5\text{ mm}$.

Crush fraction I so that it is predominantly $0,5\text{ mm}$ to $1,0\text{ mm}$ and sieve to form fraction I Ib.

Mix fractions I la and I lb thoroughly.

Wash the sample with dichloromethane in accordance with ISO 8723.

Heat the sample to $120\text{ °C} \pm 2\text{ °C}$ for 30 min using the oven (4.7), allow to cool in a desiccator and weigh to the nearest 1 g or better. Repeat the operations of heating, cooling, and weighing until the difference between two successive weightings does not exceed 1 % of the original sample mass.

6 Procedure

6.1 Test portion

Weigh out $15\text{ g} \pm 0,1\text{ g}$ of the mixture of fraction $1,4\text{ mm}$ to $1,0\text{ mm}$ or of I la and I lb (5.3).

6.2 Setting up the test machine

Place the brass reference cylinder (4.3) in the sample holder (4.1).

Place the sample holder with plunger (4.1) in the test machine (4.4) and apply a pressure of 3 MPa. Set the length-measuring device (4.2) to the reference level of 20 mm. Connect up the electrical wires and switch on the power supply (4.5).

Place the sample holder with plunger (4.1) in the test machine (4.4) and apply a pressure of 3 MPa. Set the length-measuring device (4.2) to the reference level of 20 mm. Connect up the electrical wires and switch on the power supply (4.5).

6.3 Determination

Pour the test portion into the sample holder, insert the plunger and place the assembly in the test machine.

Apply a pressure of 3 MPa to the test portion and after 30 s record the voltage drop and the height of the column of granules.

6.4 Number of determinations

Carry out two determinations using a fresh test portion for each determination.

Clean the base and the plunger surfaces carefully after each determination.

7 Expression of results

Calculate the electrical resistivity, ρ , expressed in microhm metres, of the calcined coke using Formula (1):

$$\rho = \frac{S \times U}{I \times h} \quad (1)$$

where

S is the surface area, in square millimetres, of the sample holder (707 mm² for the sample holder shown in [Figure 1](#));

U is the voltage drop, in millivolts;

I is the current applied, in amps (1 A \pm 0,002 A);

h is the height, in millimetres, of the column of granules.

Express the result as the arithmetic mean of two determinations.

8 Precision¹⁾

8.1 Repeatability

The difference between the values of duplicate determinations, carried out in rapid succession by the same operator using the same apparatus on granules taken from the same test sample, shall not exceed the repeatability limit $r = 3 \%$.

8.2 Reproducibility

The difference between the values of the average of duplicate determinations obtained by two laboratories using this method for the analysis of the same laboratory sample is not expected to exceed the reproducibility limit $R = 5 \%$.

9 Test report

The test report shall include the following information:

- a) all details necessary for complete identification of the sample;

1) ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn), was used to obtain the precision data.

- b) a reference to this International Standard (i.e. ISO 10143);
- c) the size distribution of the grain fraction used;
- d) the results, expressed in accordance with [Clause 7](#);
- e) the details of any unusual features noted during the determination;
- f) the details of any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional.

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