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Carbonaceous materials for the production of aluminium — Anodes, cathodes blocks, sidewall blocks and baked ramming pastes — Determination of the thermal conductivity using a comparative method

Produits carbonés utilisés pour la production de l'aluminium — Anodes, cathodes blocs, blocs de façade et pâtes de brasquage cuites — Détermination de la conductivité thermique à température ambiante par une méthode comparative

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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.

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The main task of technical committees is to prepare International Standards. 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.

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ISO 12987 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*.

Introduction

Knowledge of the thermal conductivity of carbonaceous materials allows calculations of the heat transfer and losses in electrolysis cells.

Carbonaceous materials for the production of aluminium — Anodes, cathodes blocks, sidewall blocks and baked ramming pastes — Determination of the thermal conductivity using a comparative method

1 Scope

This International Standard specifies a method for the determination of thermal conductivity of carbonaceous material in the temperature range of 20 °C to 60 °C. The typical range of thermal conductivity for these materials is 2 W/(K·m) to 100 W/(K·m).

This method can be used for other carbon materials, such as graphitized electrodes for other applications.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1

thermal conductivity

λ

material property, expressed in W/(K·m), obtained by determining the heat flow $\frac{\mathrm{d}Q}{\mathrm{d}t}$ at a given temperature difference $\Delta\vartheta$ through a body with height h and cross-sectional area A as follows:

$$\lambda = \frac{\mathrm{d}Q}{\mathrm{d}t} \times \frac{1}{\Delta\vartheta} \times \frac{h}{A} \qquad (1)$$

where

$\frac{\mathrm{d}Q}{\mathrm{d}t}$ is the heat flow, expressed in watts;

$\Delta\vartheta$ is the temperature difference, expressed in kelvins;

h is the height, expressed in metres, of the body;

A is the cross-sectional area, expressed in square metres

NOTE 1 For cylindrical test specimens:

$$A = \pi \times \frac{d^2}{4} \qquad (2)$$

where d is the diameter of the test specimens, expressed in metres

NOTE 2 This temperature difference is compared to those obtained with reference specimens thereby allowing the determination of the thermal conductivity of the test specimen.

3 Reagents and materials

3.1 Contact agent, for testing, consisting of Vaseline, glycerol or contact paste capable of improving the contact between the heads and the specimen.

3.2 Calibration specimens, which have been calibrated by a recognized calibration authority such as the producer of the equipment or a national standards body.

Table 1 shows examples of reference materials for specimens to be calibrated by the producer of equipment or by national standards bodies. The thermal conductivity of these materials varies almost linearly with temperature over the range of 20 °C to 100 °C.

Table 1 — Examples of reference materials

Material	Thermal conductivity W/(K·m) at 20 °C
Flint glass	1,4
High alloy steel	14
Bronze	65
Brass	110
Aluminium 99,9 % pure	235

4 Apparatus

4.1 Typical measuring device, schematically shown in Figure 1 and consisting of the following:

4.1.1 Lower head, as heat sink cooled to 20 °C ± 0,1 °C.

4.1.2 Thermostatically controlled device, capable of maintaining the water temperature at 20 °C ± 0,02 °C and a circulating rate of approximately 10 l/min.

4.1.3 Differential thermocouple.

4.1.4 Suitable voltmeter.

4.1.5 Upper head, as heat source, heated to at least 40 °C, preferentially 60 °C, and accurate to 0,1 °C.

4.1.6 Liquid thermostat, capable of maintaining the water temperature at 40 °C ± 0,02 °C, and a circulating rate of approximately 10 l/min, or electrical heating device maintaining the temperature of the upper head at 60 °C ± 0,1 °C.

4.1.7 Clamping device, applying a minimum pressure of about 5 MPa and a cardanic suspension.

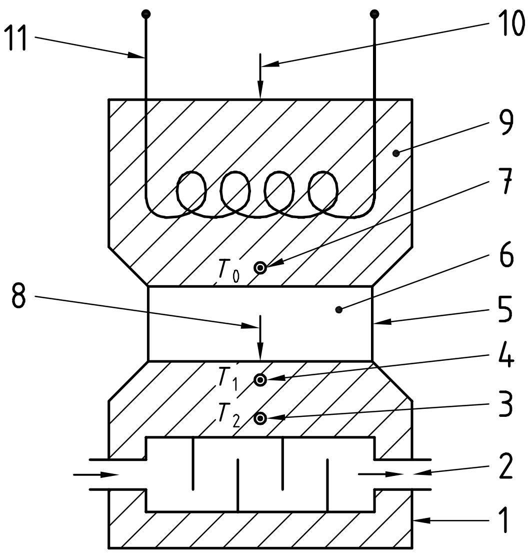
4.1.8 Insulating cylinder, to avoid heat loss.

4.2 Device for measuring dimensions, with error limit of ± 1 %, for example a vernier calliper as specified in ISO 6906 or a micrometer calliper as specified in ISO 3611.

4.3 Straight edge.

5 Sampling

Select test specimens in accordance with a suitable sampling scheme, for example using ISO 8007-1 or ISO 8007-2.



Key

1 lower head
2 water flowing through the lower head at 20 °C ± 0,1 °C and 10 l/min
3 differential thermocouple element T_2
4 differential thermocouple element T_1
5 insulating cylinder
6 test specimen
7 differential thermocouple element T_0
8 direction of heat flow
9 upper head
10 clamping pressure $(p) = 200$ N
11 heating element

Figure 1 — A typical device to perform the test shown schematically

The diameter of the cylindrical test specimens shall be between 20 mm and 50 mm, the height shall be from 5 mm to 50 mm and the end surfaces shall be plane.

The accuracy of the method is affected by the geometry of the test specimens. For high thermal conductivity materials, thinner and/or longer test specimens yield better results.

For test specimens of 20 mm height, the diameters in Table 2 have been found appropriate.

The flatness of the end planes shall be better than ± 0,05 mm, which can be checked by means of the straight edge (4.3). The test specimens shall be dried to constant mass.

Table 2 — Dimensions of test specimens

Typical range of thermal conductivity W/(K·m)	**Test specimen material**	**Diameter** [a] mm
Up to 10	Anodes Baked ramming paste	50
5 to 120	Cathodes	50 or 30
80 to 120	Graphite electrodes	20 to 30

[a] Height of test specimens $= 20$ mm. The larger diameters are chosen for anodes, baked ramming paste and cathodes due to their coarser grain structure.

6 Procedure

6.1 Measurement of test specimen dimensions

Measure the height (h) and diameter (d) of the test specimens.

6.2 Calibration

Close the upper (4.1.5) and lower (4.1.1) heads. Heat the apparatus to the operating temperature.

When the temperatures of both heads are stable within $\pm$ 0,1 °C, choose one or several calibration specimens with the same cross-section as the test specimens. A thin layer of contact agent (3.1) may be applied to both sample end faces. Separate the heads and centre the specimens between the heads, close the heads on the specimen and apply and maintain a suitable pressure.

When the reading of the differential thermocouple (4.1.3) is constant, record the thermoelectric voltage (4.1.4).

The calibration curve (thermoelectric voltage U as a function of $\lambda A/h$) can be determined from measurements of different calibration specimens with known thermal conductivity and dimensions (see DIN 51908). The thermoelectric voltage U yields the quantity dQ/d$t \times 1/\Delta\vartheta$ for any specimen.

If an insulating cylinder is used for the measurement of the test specimens, this cylinder should be used for calibration, as well.

6.3 Measurement of test specimens

Test specimens with the same cross-sections as the calibration specimens are measured according to the measuring procedure given for calibration specimens in 6.2.

7 Calculation

The thermal conductivity λ, expressed in W/(K·m), is calculated according to Equation (3) as follows:

$$\lambda = \frac{\mathrm{d}Q}{\mathrm{d}t} \times \frac{1}{\Delta\vartheta} \times \frac{h}{A} \qquad (3)$$

where

λ	is thermal conductivity, expressed in W/(K·m);
$\frac{\mathrm{d}Q}{\mathrm{d}t} \times \frac{1}{\Delta\vartheta}$	is taken from the calibration curve;
h	is the height, expressed in metres, of the test specimen;
A	is cross-sectional area, expressed in square metres, of the test specimen.

8 Precision

As specified in ASTM E691, the precision of this method is as follows.

a) The repeatability r in W/(m·K) is calculated by the following equation:

$$r = 0{,}083\,\lambda - 0{,}72$$

b) The reproducibility R in W/(m·K) is calculated by the following equation:

$$R = 0{,}124\,\lambda + 1{,}097$$

9 Test report

The test report shall include the following information:

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

b) a reference to this International Standard (ISO 12987:2004);

c) the measuring temperatures;

d) the results and the method used;

e) details of any unusual features noted during the determination;

f) 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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[5] ISO 8007-2, *Carbonaceous materials used in the production of aluminium — Sampling plans and sampling from individual units — Part 2: Prebaked anodes*

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[7] DIN 51908, *Testing of carbonaceous materials — Determination of thermal conductivity at room temperature by means of a comparative method — Solids*

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