

Methods of

Sampling and test for carbonaceous materials used in aluminium manufacture —

Part 1: Electrode pitch —

Section 1.6 Determination of coking
value

ICS 71.100.10

National foreword

This British Standard is the UK implementation of ISO 6998:1997, incorporating Technical Corrigendum No. 1. It supersedes BS 6043-1.6:1984 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/24, Raw materials for the aluminium industry, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find facility of the BSI Standards Electronic Catalogue.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the ISO title page, page ii, pages 1 to 3 and a back cover.

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Amendments issued since publication

Amd. No.	Date	Comments
10644 Corrigendum No. 1	April 2000	Amends clause 3

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INTERNATIONAL
STANDARD

ISO
6998

Second edition
1997-12-15

**Carbonaceous materials for the production
of aluminium — Pitch for electrodes —
Determination of coking value**

*Produits carbonés utilisés pour la production de l'aluminium — Brai pour
électrodes — Détermination du résidu de cokéfaction*



Reference number
ISO 6998:1997(E)

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.

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.

International Standard ISO 6998 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*.

This second edition cancels and replaces the first edition (ISO 6998:1984), of which it constitutes a minor revision. The main change concerns clause 8 (Expression of results) which has been amended to make it clearer.

Descriptors: Extractive metallurgy, aluminium, carbon electrodes, carbonaceous materials, pitch (materials), tests, determination, coking.

1 Scope

This International Standard specifies a method for determination of the coking value of pitch used for the production of aluminium.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards. ISO 565:1990, *Test sieves — Metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings*.

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*.

ISO 6257:1980, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling*.

3 Principle

A test portion of pitch is heated at $550\text{ °C} \pm 10\text{ °C}$ and the residue weighed.

4 Material

4.1 Calcined petroleum coke, sieved.

Use the fraction which passes a 1 mm mesh sieve and is retained by a $212\text{ }\mu\text{m}$ mesh sieve (see ISO 565).

5 Apparatus

Ordinary laboratory apparatus, plus the following:

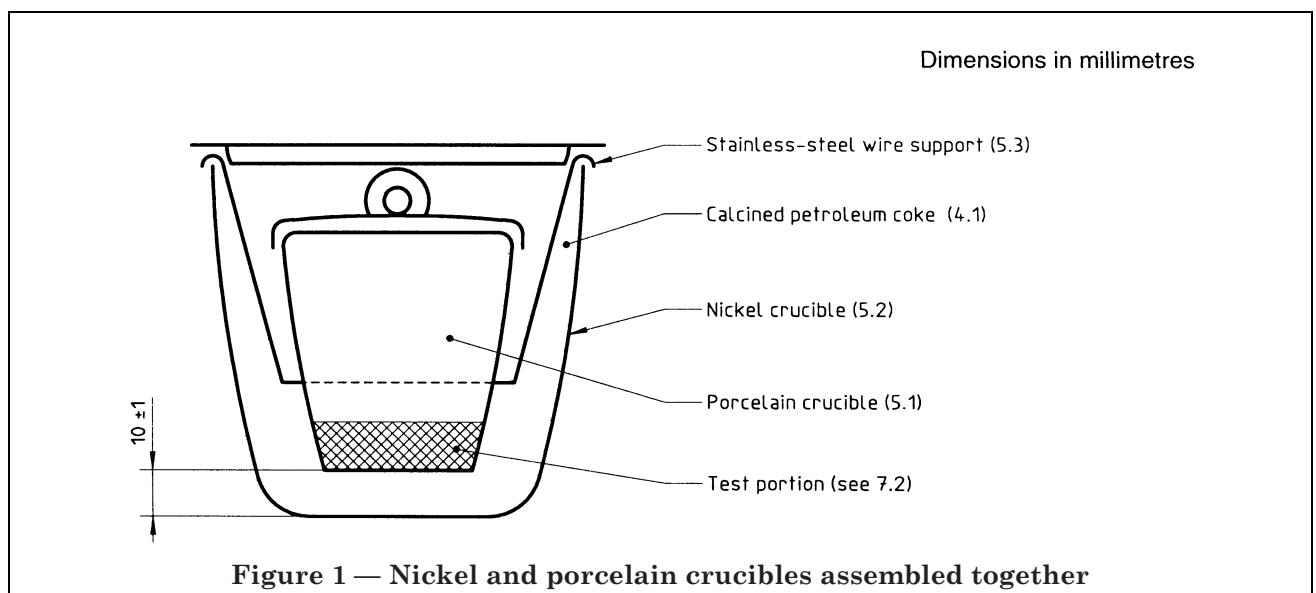
5.1 Porcelain crucibles, of 25 ml to 50 ml capacity, having a height to external top diameter ratio of 0,7 to 0,9, fitted with overlapping lids.

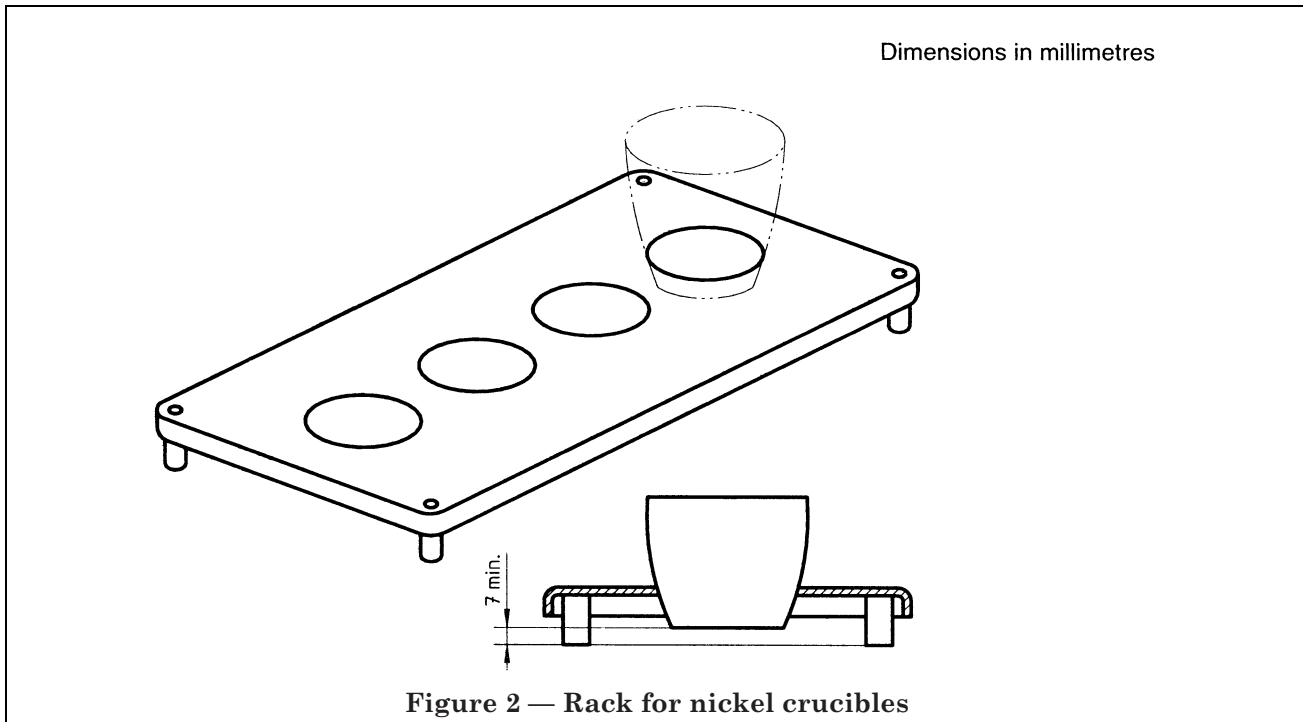
5.2 Nickel crucibles, of capacity approximately 130 ml, height 60 mm and diameter 60 mm, fitted with lids.

5.3 Stainless-steel wire supports (see Figure 1), to locate the porcelain crucibles (5.1) correctly in the nickel crucibles (5.2) and allowing a separation of $10\text{ mm} \pm 1\text{ mm}$ between the base of each crucible.

5.4 Rack for the nickel crucibles (see Figure 2), made from 1 mm thick stainless-steel plate and containing four holes. The rack is bent over at the edges, welded at the corners and fitted with four welded legs which ensure, when nickel crucibles are placed in the holes, that the bottoms of the crucibles are kept at least 7 mm above the floor of the furnace.

The rack is intended for four determinations (two duplicate determinations), but for reference testing only two of the holes are used.





5.5 Sieves, of 1 mm, 300 μm and 212 μm nominal mesh size (see ISO 565).

5.6 Electric furnace, capable of being maintained at $550\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$.

6 Sampling and preparation of test sample

6.1 Sampling

Carry out sampling by the procedure specified in ISO 6257.

6.2 Preparation of test sample

If the sample is sufficiently hard, crush it with a small jaw crusher and grind it in a mortar to pass at least a 300 μm mesh sieve and, if possible, a 212 μm mesh sieve. If the ambient temperature is high, the operation will be facilitated by chilling the sample beforehand.

If the pitch is too soft to crush, melt the sample and take sufficient of the molten mass for the test. The melting temperature shall not exceed $150\text{ }^{\circ}\text{C}$ and the melting period shall not exceed 10 min. It is also possible to transfer the required portion of a soft pitch directly to a porcelain crucible, without preliminary treatment.

7 Procedure

7.1 Number of determinations

Carry out the determination in duplicate, using two crucibles in each determination.

7.2 Test portion

Heat two of the porcelain crucibles (5.1), fitted with their lids, for about 2 h in the electric furnace (5.6), maintained at $550\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$. Allow them to cool to ambient temperature in a desiccator and weigh each of them to the nearest 1 mg (m_1). Weigh into each crucible, to the nearest 1 mg, a test portion of $1\text{ g} \pm 0,05\text{ g}$ of the test sample (see 6.2).

7.3 Determination

Place one of the wire supports (5.3) in one of the nickel crucibles and make a bed of petroleum coke (4.1), 10 mm \pm 1 mm thick, on the base of the crucible. Place one of the porcelain crucibles with its test portion (see 7.2) in the wire support so that it rests on the coke bed.

Close the porcelain crucible with its lid and fill the space between the two crucibles with more of the petroleum coke so that the porcelain crucible is completely embedded in the coke. Close the nickel crucible with its lid.

Repeat the above operations, using the second porcelain crucible and test portion.

Place the prepared crucibles in the rack (5.4) and place the whole in the electric furnace (5.6), maintained at $550\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$, as quickly as possible, in order to avoid heat losses.

It is essential that the temperature of that part of the furnace occupied by the crucibles in their rack is uniform and complies with the specified condition, i.e. $550\text{ °C} \pm 10\text{ °C}$. An initial temperature check shall be made with a pyrometer and only that part of the furnace complying with this temperature shall be used for the crucibles in their rack.

There shall be a space of not less than 7 mm between the nickel crucibles and the floor, walls and roof of the furnace. The space between any crucible and the front wall containing the door, and between any crucible and the back wall of the furnace, shall be not less than 50 mm.

After 2,5 h, remove the nickel crucibles from the furnace and allow to cool. Remove each porcelain crucible and clean carefully to remove any adhering coke powder, taking care to avoid contamination. Place the porcelain crucibles and contents, covered, in a desiccator, allow to cool to room temperature and weigh each of them to the nearest 1 mg (m_2).

NOTE To clean the porcelain crucibles and lids for further use, discard the coke residues and remove any carbonaceous material by heating the crucibles at 700 °C to $1\ 000\text{ °C}$.

Repeat the above operations for the second determination.

8 Expression of results

8.1 Calculation

The coking value, expressed as a percentage by mass, is given by the formula

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

- m_0 is the mass, in grams, of the test portion (see 7.2);
- m_1 is the mass, in grams, of the empty porcelain crucible;
- m_2 is the mass, in grams, of the porcelain crucible and residue (see 7.3).

Calculate the result for each of the four crucibles.

Discard doubtful results (see 8.2.1 and 8.2.2).

Repeat the determination in 7.3 as necessary to obtain four valid results.

Report the mean value of the four results as a percentage by mass to the nearest 0,1 % (m/m).

8.2 Precision (see ISO 5725-1:1994, clause 3)

8.2.1 Repeatability for pairs of crucibles heated together

The pair of results from the two crucibles heated together in the furnace shall be considered doubtful if the two results differ by more than $\sqrt{2}$ times the repeatability of the method (see 8.2.2).

8.2.2 Repeatability of the method

The results of duplicate determinations carried out by the same operator using the same apparatus (each result being the mean of the values obtained with two crucibles heated together) shall be considered doubtful if they differ by more than 1,0 % (m/m).

8.2.3 Reproducibility of the method

The results of determinations carried out on the same sample but in two different laboratories (each result being the mean of the values obtained with two crucibles heated together) shall be considered doubtful if they differ by more than 2,0 % (m/m).

9 Test report

The test report shall include the following particulars:

- a) all details necessary for identification of the sample;
- b) a reference to this International Standard;
- c) the results and the units in which they are expressed;
- d) details of any unusual incident noted during the determination;
- e) details of any operation not included in this International Standard or in the standards to which reference is made, as well as any operation regarded as optional;
- f) the date of the determination.

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