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International Standard



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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

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6998 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in August 1982.

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It has been approved by the member bodies of the following countries :

Australia	India	Romania
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No member body expressed disapproval of the document.

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

1 Scope and field of application

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

2 References

ISO 565, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

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

3 Principle

Heating of a test portion under defined conditions at 550 ± 10 °C and weighing the coke residue.

4 Material

Calcined petroleum coke, sieved.

Use the fraction which passes a 1 mm mesh sieve and is retained by a 212 μm mesh sieve (see ISO 565).

5 Apparatus

Ordinary laboratory apparatus and

5.1 Porcelain crucibles, tall form, of capacity approximately 50 ml, 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 ± 1 mm between the bases of each crucible.

5.4 Support for the nickel crucibles (see figure 2), made from 1 mm thick stainless steel plate, which contains four holes. The support is bent over the edges, welded at the corners and fitted with four welded legs which allow, when holes are used to nest the nickel crucibles, to hold its bottom at least 7 mm above the base of the furnace.

The support is intended for four determinations (two duplicate determinations), but for reference testing, only two of the holes should be 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 controlled at 550 ± 10 °C.

6 Sampling and sample

6.1 Sampling

Sampling should be carried out according to the procedure specified in ISO 6257.

6.2 Preparation of test sample

If the sample is sufficiently hard, crush 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 °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 the porcelain crucible, without preliminary treatment.

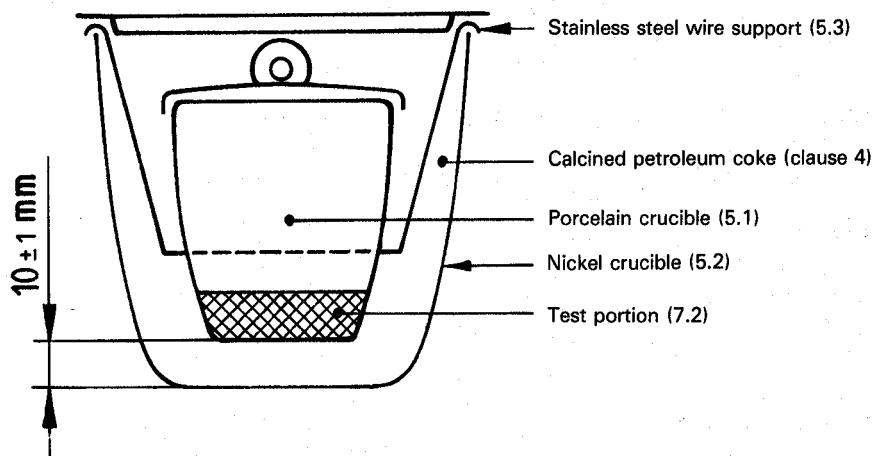


Figure 1 – Assembly of nickel and porcelain crucibles

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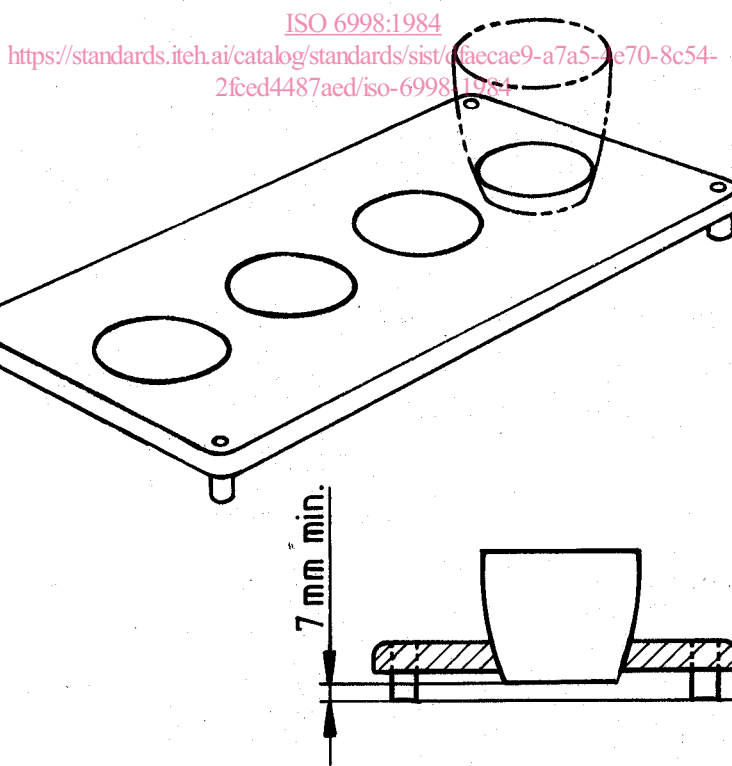


Figure 2 – Support for nickel crucibles

7 Procedure

7.1 Number of determinations

Carry out the determination in duplicate.

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), controlled at 550 ± 10 °C. Allow them to cool to ambient temperature in a desiccator and weigh each of them to the nearest 0,001 g (m_1). Weigh into each crucible, to the nearest 0,001 g, a test portion of $1 \pm 0,05$ g of the test sample (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 (clause 4), 10 ± 1 mm thick, on the base of this crucible. Place one of the porcelain crucibles with its test portion (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 by the coke. Close the nickel crucible with its lid.

Repeat the above operations, using the second porcelain crucible and test portion (7.2).

Place the prepared crucibles on the metallic support (5.4) and place the whole in the electric furnace (5.6), controlled at 550 ± 10 °C, as quickly as possible, in order to avoid heat losses.

NOTE — It is essential that the temperature of the furnace space used to contain the crucibles and their supports should be uniform and comply with the specified condition, i.e. 550 ± 10 °C. An initial temperature check should be made with a pyrometer and only that part of the furnace complying with this temperature should be used for the crucibles and their supports.

There should 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 the back wall of the furnace should 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 0,001 g (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 to 1 000 °C.

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 (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.

Report the mean value of the duplicate determinations.

8.2 Precision (see ISO 5725, sub-clause 3.1).

8.2.1 Repeatability

The results of duplicate tests (each determination with two crucibles) are to be considered doubtful if they differ by more than 1,0 % of the lower value.

8.2.2 Reproducibility

The results of one determination (mean of two determinations) obtained from two laboratories are to be considered doubtful if they differ by more than 2,0 % of the lower value.

9 Test report

The test report shall include the following particulars:

- a) an identification of the sample;
- b) the reference of the method used;
- c) the results and the method of expression used;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standards to which reference is made, or regarded as optional.