International Standard 6791

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Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of content of quinoline-insoluble material

Produits carbonés utilisés pour la production de l'aluminium — Brais pour électrodes — Détermination du taux de matières insolubles dans la quinoléine

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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 6791 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in December 1979.

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

Australia	Egypt, Arab Rep. of	Romania
Austria	France	South Africa, Rep. of
Belgium	Germany, F. R.	Sweden
Brazil	Hungary	Switzerland
Bulgaria	Italy	Thailand
Canada	Korea, Rep. of	United Kingdom
China	Poland	USSR
Czechoslovakia	Portugal	

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of content of quinoline-insoluble material

1 Scope and field of application

This International Standard specifies a conventional gravimetric method for the determination of the content of quinoline-insoluble material in pitch used in the production of aluminium.

2 Reference

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

3 Principle

Extraction of a test portion with quinoline, at a specified temperature and for a specified period. Filtration and weighing of the residue.

4 Reagents and materials

During the analysis, use only reagents of recognized analytical grade.

4.1 Quinoline, freshly distilled, boiling between 235 and 237 °C at 1 013 mbar[1].

WARNING — Risk of poisoning by inhalation or swallowing. Irritating to skin and eyes. Prevent inhalation of vapour and contact with skin, eyes and clothing.

4.2 Toluene.

WARNING — Highly flammable. Harmful by inhalation. Keep away from sources of ignition. No smoking. Do not empty into drains. Take precautionary measures against static discharges.

4.3 Filter aid, diatomaceous silica type, free from organic compounds, and chemically neutral.

The particle sizes shall be within the range 1 to 40 µm and the average particle size shall be within the range 6 to 10 µm.

Dry the material in the oven (5.4), controlled at 105 to 110 °C, for 1 h. Allow to cool in a desiccator and weigh. Repeat the process of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,005 g. Store the dried material in a desiccator.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Water bath, capable of being controlled at 70 to 80 °C.

5.2 Hartley funnel, or equivalent, having a disc diameter of 70 mm (a suitable type is shown in the figure).

5.3 Glass fibre filter pads, of diameter 70 mm, with retention equal to 98 % for particles of size 1,2 µm.

NOTE — A larger funnel (5.2) and pad (5.3), for example of diameter 120 mm, may be used, if available, as this largely obviates the need to transfer any residue from the walls of the funnel to the pad during the determination (7.3).

5.4 Electric oven, capable of being controlled at 105 to 110 °C.

6 Sample preparation

Sample in accordance with ISO 6257. Prepare the test sample immediately before the determination.

If it is sufficiently hard, grind the whole of the test sample, using a mortar and pestle, to a particle size not exceeding 200 µm.

If the pitch is too soft for grinding, melt and mix the sample, ensuring that its temperature does not exceed 150 °C and that the melting period does not exceed 10 min.

NOTE — For soft pitches, the material for the test portion may be taken from the molten sample.

1) 1 mbar = 100 Pa

7 Procedure

WARNING — Attention is drawn to the dangers involved in the use of quinoline and toluene (see WARNINGS to clauses 4.1 and 4.2). Carry out all operations involving the use of these products in an efficiently ventilated fume cupboard.

7.1 Test portion

Weigh, to the nearest 0,005 g, approximately 1 g of the test sample.

7.2 Filter preparation

Heat one of the filter pads (5.3) in the oven (5.4), controlled at 105 to 110 °C, for 1 h. Transfer the pad to a desiccator, allow to cool to ambient temperature and weigh to the nearest 0,005 g. Repeat the process of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,005 g. Fit the pad into the clean and dry Hartley funnel (5.2).

7.3 Determination

Transfer the test portion (7.1) quantitatively to a 100 ml beaker. Add approximately 1 g of the dried filter aid (4.3), weighed to the nearest 0,005 g, and 25 ml of the quinoline (4.1). Stir the contents of the beaker to break up any lumps and cover the beaker with a watch glass.

Partially immerse the beaker in the water bath (5.1), controlled at 70 to 80 °C, together with another suitable beaker containing about 100 ml of the quinoline. Bring the temperature of the contents of each beaker to 70 to 80 °C and continue heating for 15 to 20 min, occasionally stirring the contents of the beaker containing the test portion.

Fit the assembled Hartley funnel and filter pad (see 7.2) into the neck of a filter flask. Moisten the pad with a little of the hot quinoline and apply suction. As soon as filtration is complete, start filtering the dissolved test portion and any residue without unduly disturbing the solution, transferring about 2 ml of this suspension at a time to the filter pad and allowing each portion to filter completely before making the next addition. Between each addition, maintain the temperature of the solution remaining in the beaker at 70 to 80 °C by means of the heated water bath. When filtration is complete, rinse the beaker five times with approximately 5 ml portions of the hot quinoline from the other beaker, so as to transfer all remaining insoluble matter to the pad. Wash the residue on the filter pad with further 5 ml portions of the hot quinoline until the washings are clear. About ten washings are usually necessary.

Wash the beaker which had contained the test portion with ten approximately 5 ml portions of the toluene (4.2), allowing each washing to pass completely through the filter pad before making the next addition. During the entire filtering operation, ensure that as little as possible of the solution and residue is allowed to come into contact with the walls of the funnel.

Release the suction, remove the filter pad with the residue and, after transferring to it any solid remaining on the walls of the funnel, place in the oven (5.4), controlled at 105 to 110 °C, for about 1 h. Transfer from the oven to the desiccator and allow to cool to ambient temperature. Weigh to the nearest 0,005 g. Repeat the operations of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,005 g. A drying time of 2 to 3 h is usually sufficient.

8 Expression of results

8.1 Method of calculation

The content of quinoline-insoluble material, expressed as a percentage by mass, is given by the formula

$$\frac{m_3 - m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in grams, of the dried filter pad (5.3);

m_2 is the mass, in grams, of the filter aid (4.3) added;

m_3 is the mass, in grams, of the dried filter pad, the filter aid and the residue.

8.2 Precision

Repeatability : 1 %

Reproducibility : 1,50 %

9 Test report

The test report shall include the following particulars :

a) an identification of the sample;

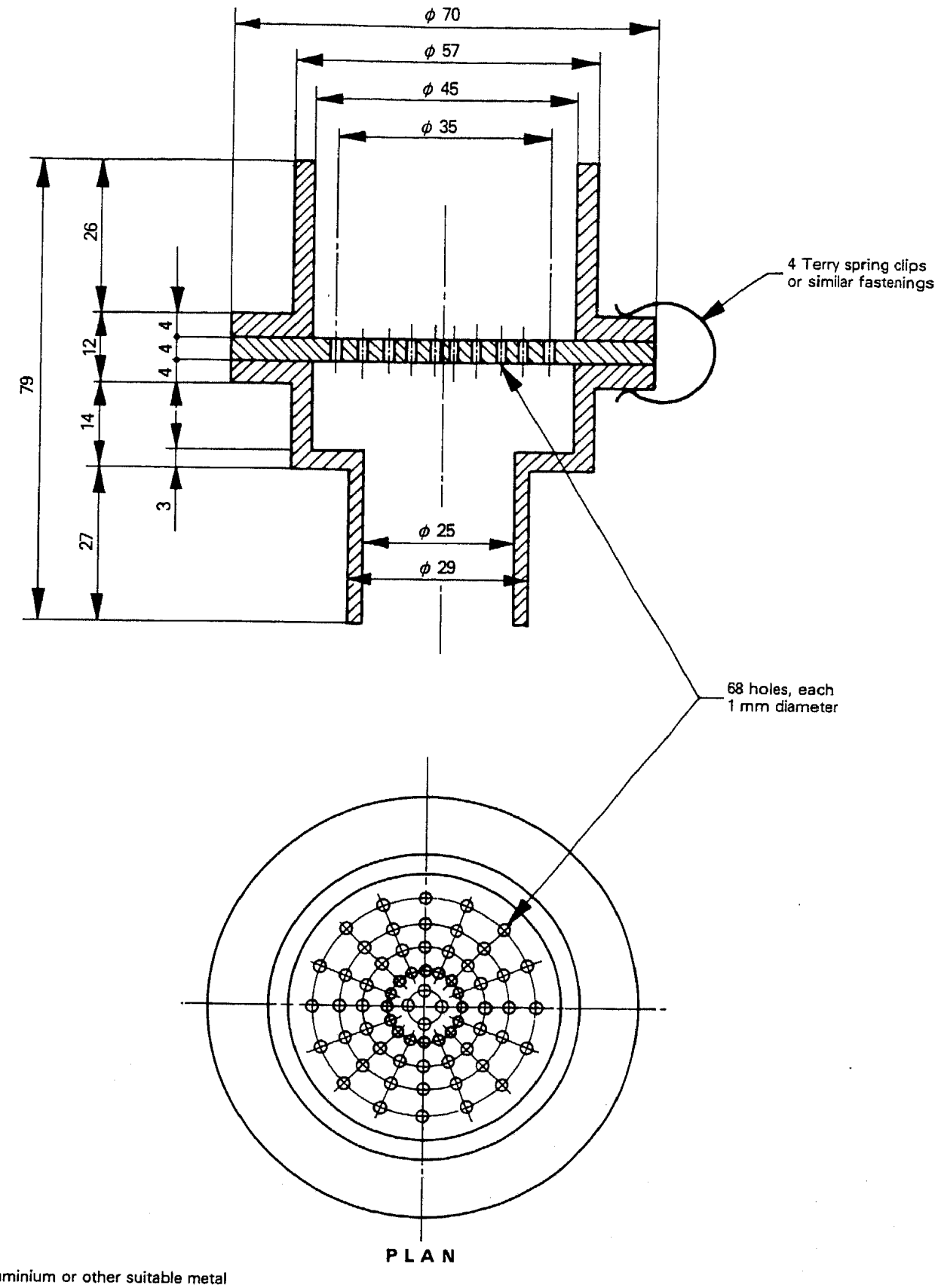
b) the reference of the method used;

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 Standard or in the International Standard to which reference is made, or regarded as optional.

Dimensions in millimetres



Material : Aluminium or other suitable metal

Finish : Fine machine all over polished bores and mating surfaces, i.e. flanges

Figure — Details of Hartley funnel