International Standard 6376

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

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

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Foreword

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

Australia	Hungary	Sweden
Austria	Italy	Switzerland
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Canada	Libyan Arab Jamahiriya	United Kingdom
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The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

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

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

1 Scope and field of application

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

NOTE — The content of toluene-insoluble material is commonly called "sum of resins α + β".

2 Reference

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

3 Principle

Determination by weighing of the fraction of a test portion of pitch which is insoluble in toluene after boiling for a specified period.

4 Reagents and materials

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

4.1 Toluene.

4.2 Acetone.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Conical flask, of capacity 500 ml, of borosilicate glass, fitted with a ground glass socket (see ISO 4797).

5.2 Reflux condenser, effective length 300 mm, having a ground glass cone (see ISO 383) at its lower end, by means of which it can be fitted to the socket of the conical flask (5.1).

5.3 Filter crucible, of glass, of capacity about 30 ml, fitted with a sintered glass disc (see ISO 4793) of porosity grade P16 (pore size index 10 to 16 µm) and having the following approximate principal dimensions :

disc diameter : 30 mm

height (disc to upper rim) : 35 mm.

5.4 Electric oven, capable of being controlled at a temperature between 105 and 110 °C.

6 Sampling

See ISO 6257.

In the case of hard pitches, grind the sample so that it passes through a sieve of nominal aperture size 200 µm (see ISO 565).

In the case of soft pitches, use the sample as received.

7 Procedure

WARNING — Toluene is toxic and highly flammable. Carry out all operations involving its use in an efficiently ventilated fume cupboard.

7.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the sample (see clause 6).

7.2 Determination

Prepare the filter crucible (5.3) for the determination by heating it for about 1 h in the oven (5.4) controlled at a temperature between 105 and 110 °C, allowing it to cool to ambient temperature in a desiccator and weighing it to the nearest 0,001 g.

Place the test portion (7.1) in the conical flask (5.1). Add about 100 ml of hot (approximately 80 °C) toluene (4.1) and swirl to dissolve the test portion.

NOTE — Dissolution before boiling of soft pitches that are liquid on transfer to the flask is particularly necessary to prevent the formation of droplets of pitch which are subsequently not readily soluble in the boiling toluene.

Fit the condenser (5.2) to the flask and start the water circulation. Bring the contents of the flask to a steady boil and continue boiling under reflux for about 30 min.

Stop the heating and remove the condenser. Using gentle suction, immediately filter the contents of the flask through the dried and weighed filter crucible (5.3). Rinse the flask (5.1) with about 10 ml of the hot (approximately 80 °C) toluene (4.1) and filter the washings through the filter crucible (5.3). When filtration is complete, repeat the rinsing and filtering operations with further 10 ml portions of the hot toluene until any residue in the flask is transferred to the filter crucible and the filtrate is colourless. Wash the filter crucible and its contents with approximately 10 ml of the acetone (4.2) and, when filtration is complete, repeat the operation with a further 10 ml portion of the acetone (4.2).

Treat the filter crucible and its contents as follows :

Heat for approximately 1 h in the oven (5.4), maintained at a temperature between 105 and 110 °C, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,001 g.

8 Expression of results

8.1 Method of calculation

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

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

where

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

m_1 is the mass, in grams, of the crucible containing the insoluble material;

m_2 is the mass, in grams, of the empty crucible.

8.2 Precision

Repeatability : 0,5 %

Reproducibility : 1 %

9 Test report

The test report shall contain the following information :

a) 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.