

International Standard



5939

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Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of water content — Azeotropic distillation (Dean and Stark) method

Produits carbonés utilisés pour la production de l'aluminium — Brai pour électrodes — Dosage de l'eau — Méthode par entraînement azéotropique (Dean et Stark)

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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 5939 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in November 1978.

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

Australia	India	Sweden
Belgium	Israel	Switzerland
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The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of water content — Azeotropic distillation (Dean and Stark) method

1 Scope and field of application

This International Standard specifies an azeotropic distillation method for the determination of the water content of pitch used in the production of aluminium.

The method is applicable to pitches having water contents in the range 0,1 to 2 % (m/m).

NOTE — Water contents in excess of 2 % (m/m) may be determined by means of this method by using a larger receiver.

2 References

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*.

ISO 5940, *Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of softening point by the ring and ball method*.

ISO 6257, *Carbonaceous materials for the production of aluminium — Pitch for electrodes — Sampling*.¹⁾

3 Principle

Heating the pitch under reflux with xylene. Collection in a receiver of the condensate of xylene and water and measurement of the volume of water which separates out in the graduated part of the receiver.

4 Reagent

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

4.1 Xylene, or any of its isomers, or a mixture of these isomers in any proportion, provided that the boiling range is between 130 and 140 °C. (See WARNING, clause 6.)

5 Apparatus

Ordinary laboratory apparatus and

5.1 Dean and Stark apparatus, comprising the following items :

5.1.1 Distillation flask, of glass, capacity 500 ml, having a ground-glass joint, complying with ISO 383 (see figure 1) to fit the receiver (5.1.2).

5.1.2 Graduated receiver, of capacity 2 ml. The receiver shall be graduated at every 0,05 ml with a maximum error of $\pm 0,02$ ml (see figure 3).

NOTE — For pitches having water contents in excess of 2 % (m/m), use a receiver of 10 ml capacity, graduated at every 0,2 ml and having a maximum error of $\pm 0,06$ ml.

5.1.3 Reflux condenser, connected to the receiver 5.1.2 (see figure 2).

NOTE — Before use, remove all traces of contamination from the graduated receiver and from the interior of the reflux condenser by washing them successively with, for example, chromic-sulphuric acid mixture, distilled water and acetone, and dry. Perfect cleanliness of the apparatus is essential to the success of the test.

1) At present at the stage of draft.