International Standard 5939

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

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)

First edition — 1980-07-15

UDC 665.775 : 669.713.7 : 543.812 **Ref. No. ISO 5939-1980 (E)**

Descriptors : pitch (materials), electrodes, chemical analysis, determination of content, water, Dean and Stark apparatus.

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

Printed in Switzerland

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, *Carbonaceaous 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 ± 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 ± 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.

6 Procedure

WARNING — Carry out the test in a well ventilated fume cupboard, to prevent the exposure of the operators to toxic xylene vapour.

6.1 Test portion

Weigh, to the nearest 0,1 g, 100 ± 0,5 g of the roughly crushed or melted sample (see ISO 6257) into the flask (5.1.1) of the Dean and Stark apparatus.

It is important to prevent loss of water when preparing the test portion. If the expected softening point of the sample is higher than 50 °C (see ISO 5940), make it brittle by cooling, crush it, and weigh it in such a way as to exclude extraneous moisture (see ISO 6257).

Melt softer samples using the minimum quantity of heat.

6.2 Determination

Add 100 ml of the xylene (4.1) to the distillation flask containing the test portion (6.1) and add a few glass beads to regulate boiling. Attach the flask to the Dean and Stark apparatus and circulate cold water through the jacket of the condenser. Heat the flask gently until the test portion has dissolved and then adjust the rate of heating so that the condensate falls from the end of the reflux condenser at a rate of 2 to 5 drops per second.

Continue the distillation until condensed water is no longer visible in any part of the apparatus except in the bottom of the graduated receiver and until the volume of water collected remains constant for 5 min. If a persistent ring of condensed water forms in the condenser tube, remove it by increasing the rate of distillation by a few drops per second for a few minutes.

When the carry-over of water is complete, allow the receiver and contents to cool to room temperature and read the volume of water to the nearest scale division, correcting for any certified error of the receiver.

7 Expression of results

7.1 Method of calculation

The water content, expressed as a percentage by mass, is given by the formula

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

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in grams, of the water collected in the receiver.

NOTE — The assumption that 1 ml of water weighs 1 g is sufficiently accurate for the purpose of this determination.

7.2 Precision

Repeatability : ± 0,1 g (absolute).

Reproducibility : ± 0,2 g (absolute).

8 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 Standards to which reference is made, or regarded as optional.

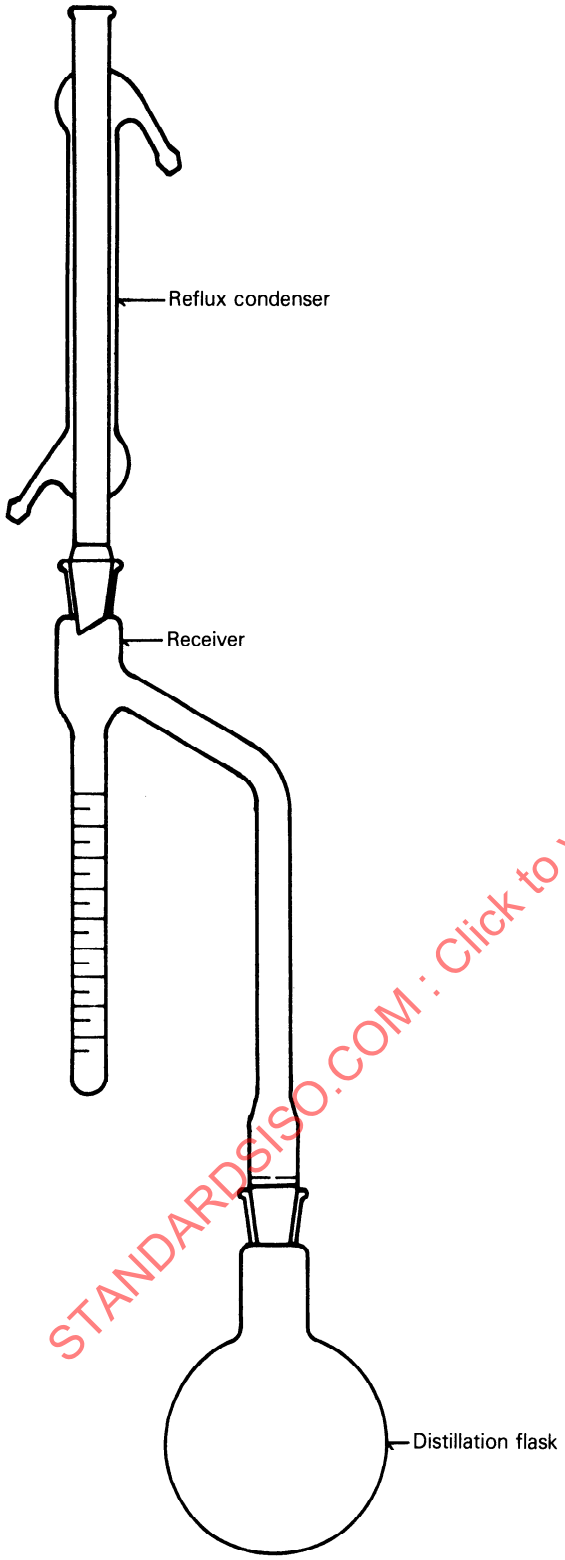

Figure 1 — Typical assembly of Dean and Stark apparatus (5.1)

Dimensions in millimetres

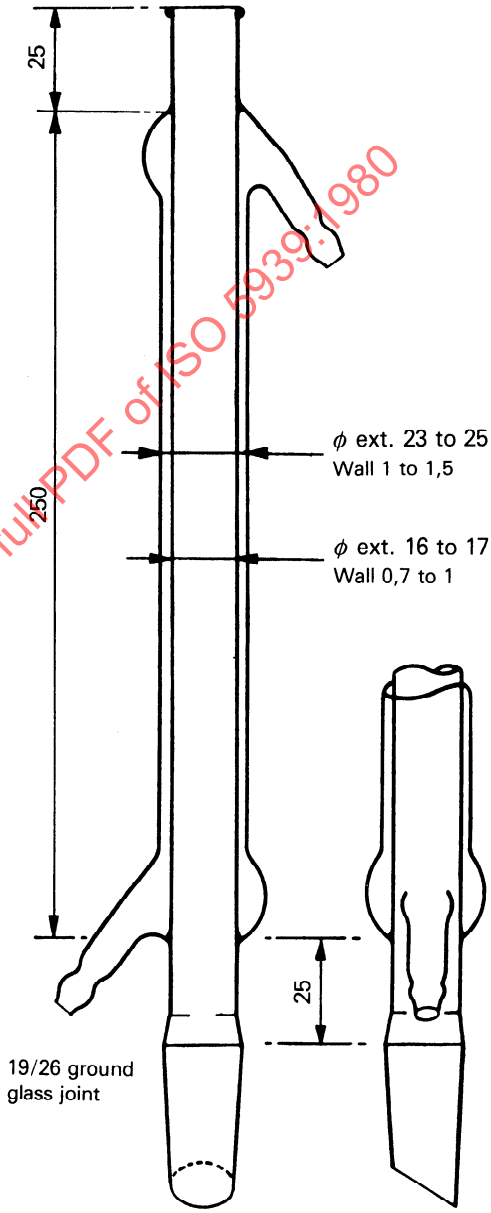

Figure 2 — Suitable form of reflux condenser (5.1.3)

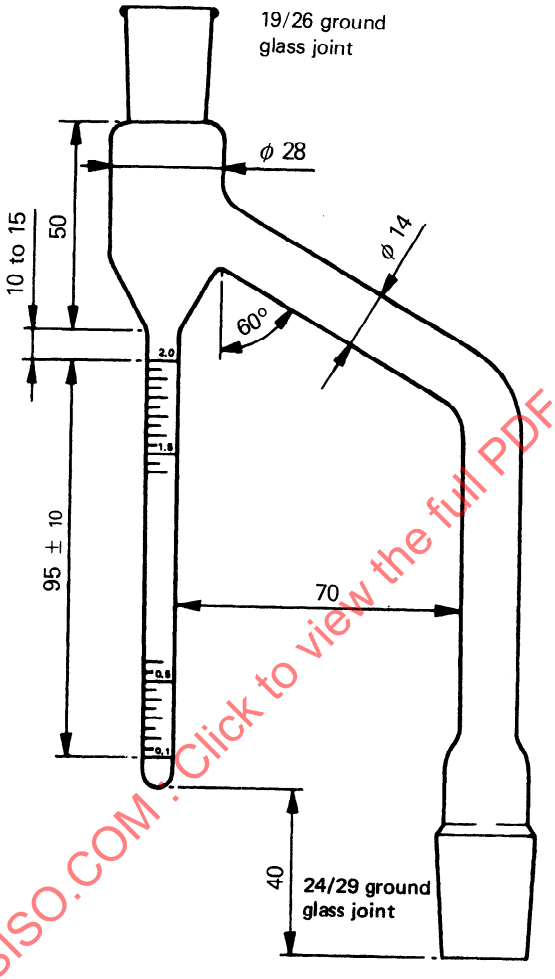

Figure 3 — 2 ml receiver (5.1.2)