
**Carbonaceous materials used in the
production of aluminium — Pitch for
electrodes — Determination of C/H ratio in
the quinoline-insoluble fraction**

*Produits carbonés utilisés pour la production de l'aluminium — Brais pour
électrodes — Détermination du rapport C/H dans la fraction insoluble dans
la quinoléine*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12979 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

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Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of C/H ratio in the quinoline-insoluble fraction

1 Scope

This International Standard describes a method for the determination of the atomic carbon/hydrogen ratio of quinoline-insoluble material isolated from coal-tar pitches. A procedure for the isolation of the quinoline-insoluble material in a form suitable for the analysis is described.

NOTE The isolation procedure is not an alternative to ISO 6971¹⁾, which is to be used in all cases where the amount of quinoline-insoluble matter present in a pitch is to be determined.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 4788, *Laboratory glassware — Graduated measuring cylinders.*

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

3 Principle

The quinoline-insoluble material is isolated from a finely divided sample of the coal-tar pitch by digestion with hot quinoline followed by pressure-filtration through a membrane filter. The isolated material is washed on the filter with hot quinoline then with hot toluene and dried.

The carbon and hydrogen contents of the isolated quinoline-insoluble material are determined by combustion of a weighed aliquot portion of that material in a stream of oxygen and trapping and weighing the carbon dioxide and water which are formed. The atomic carbon/hydrogen ratio of the quinoline-insoluble material is calculated from its carbon and hydrogen contents and the relative atomic masses of carbon and hydrogen.

1) ISO 6971, *Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of contents of quinoline-insoluble material.*

4 Isolation of quinoline-insoluble material for analysis

4.1 Reagents

WARNING — Refer to the reagent supplier's Health and Safety data sheets for the precautions which are to be taken for the safe use of quinoline and toluene.

4.1.1 Quinoline, of purity at least 97 %, freshly distilled, boiling between 235 °C and 237 °C at 0,101 3 MPa.

Store the distilled quinoline between 5 °C and ambient temperature in a stoppered bottle of dark glass. Use it within two weeks; after this time period either redistill or discard the quinoline.

4.1.2 Toluene, general purpose reagent grade.

4.1.3 Nitrogen or other inert gas, supplied via a two-stage pressure regulator with a maximum outlet pressure of 0,45 MPa.

4.2 Apparatus

WARNING — Before using pressure-filtration equipment ensure that it is provided with adequate safety devices and that the equipment has been tested to ensure compliance with relevant Health and Safety legislation.

Ordinary laboratory apparatus and the following.

4.2.1 Thermostatically controlled electric hotplate, capable of maintaining liquids in beakers at 70 °C to 80 °C.

4.2.2 Pressure-filtration apparatus, accepting filter discs of 47 mm diameter and designed for safe operation at up to 120 °C with applied gas pressures of up to 1 MPa. A typical pressure-filtration apparatus is shown in Figure 1.

4.2.3 Membrane filter discs, of polytetrafluoroethylene (PTFE), 47 mm in diameter and having a nominal pore size of 0,2 µm.

4.2.4 Hand-held hot-air blower, general-purpose laboratory type, typically of 500 W power rating and giving an air exit temperature of approximately 300 °C.

4.2.5 Test sieve, of 250 µm nominal aperture, conforming to the requirements of ISO 565, and having a lid and receiver.

4.2.6 Watch glass, approximately 70 mm in diameter, dried at 105 °C to 110 °C for 1 h and cooled in a desiccator.

4.2.7 Measuring cylinder, of borosilicate glass, of 50 ml capacity with pouring spout, conforming to the requirements of ISO 4788.

4.3 Sampling and preparation of the test sample

Prepare a representative sample of approximately 10 g of the pitch by the method described in ISO 6257. Grind the sample with a pestle and mortar until it all passes the test sieve (4.2.5).

4.4 Procedure

4.4.1 Weigh a 100 ml borosilicate glass beaker to the nearest 0,1 mg. Transfer approximately 1 g of the test sample (4.3) to the beaker and weigh it to the nearest 0,1 mg. Using the measuring cylinder (4.2.7), add 25 ml of the quinoline (4.1.1) and stir the mixture with a glass rod to break up any agglomerates. Cover the beaker with a watch glass and place it on the hotplate (4.2.1). Allow the mixture to digest at 70 °C to 80 °C for 20 min. During the

digestion stir the contents of the beaker at approximately 5 min intervals. While the digestion is proceeding, heat the liquid reservoir and filter-support assembly of the pressure-filtration apparatus (4.2.2; see Figure 1) to approximately 100 °C, either in an oven or by use of the hot-air blower (4.2.4).

When the digestion is approaching completion, weigh a membrane filter disc (4.2.3) to the nearest 0,1 mg and insert it into the heated filter-support assembly. Assemble the filtration unit ensuring that it is satisfactorily gas-tight.

NOTE The membrane filter disc tends to curl up when placed on the heated filter-support assembly. It is helpful to place the membrane filter disc and the silicone rubber O-ring seal which lies above it on to the heated filter-support assembly in a single operation, thus holding the membrane filter disc flat during the subsequent assembly of the apparatus.

4.4.2 Place the measuring cylinder (4.2.7) under the outlet of the pressure filtration-assembly. Transfer the digested mixture of pitch and quinoline from the beaker to the liquid reservoir of the pressure-filtration apparatus, using a small glass funnel to prevent any of the liquid from entering the gas line. Close the filter unit and pressurize it with the inert gas (4.1.3), gradually increasing the applied pressure until the filtrate begins to flow into the graduated receiver.

NOTE 1 The filtrate typically begins to flow when the applied gas pressure reaches 0,12 MPa to 0,14 MPa.

Allow the filtration to proceed using the minimum applied gas pressure which is consistent with a satisfactory flow rate. Use the volume of the collected filtrate as a guide to the progress of filtration. If filtration is slow, maintain the temperature of the liquid-containing part of the pressure-filtration apparatus at approximately 70 °C to 80 °C by use of the hot-air blower.

NOTE 2 The filtration rate which is achieved depends very markedly on the characteristics of the pitch being tested. Electrode binder pitches which have had little or no thermal polymerization usually give solutions which filter readily in under 1 min with an applied gas pressure of 0,13 MPa. However, solutions of pitches which have had extensive thermal treatment usually filter more slowly, with filtration times up to 1 h being common. In these cases, it is essential to keep the filter unit hot during filtration and occasional depressurization and repressurization of the filter unit may prove helpful. The use of applied pressures above approximately 0,2 MPa does not normally produce any useful improvement in the rate of filtration.

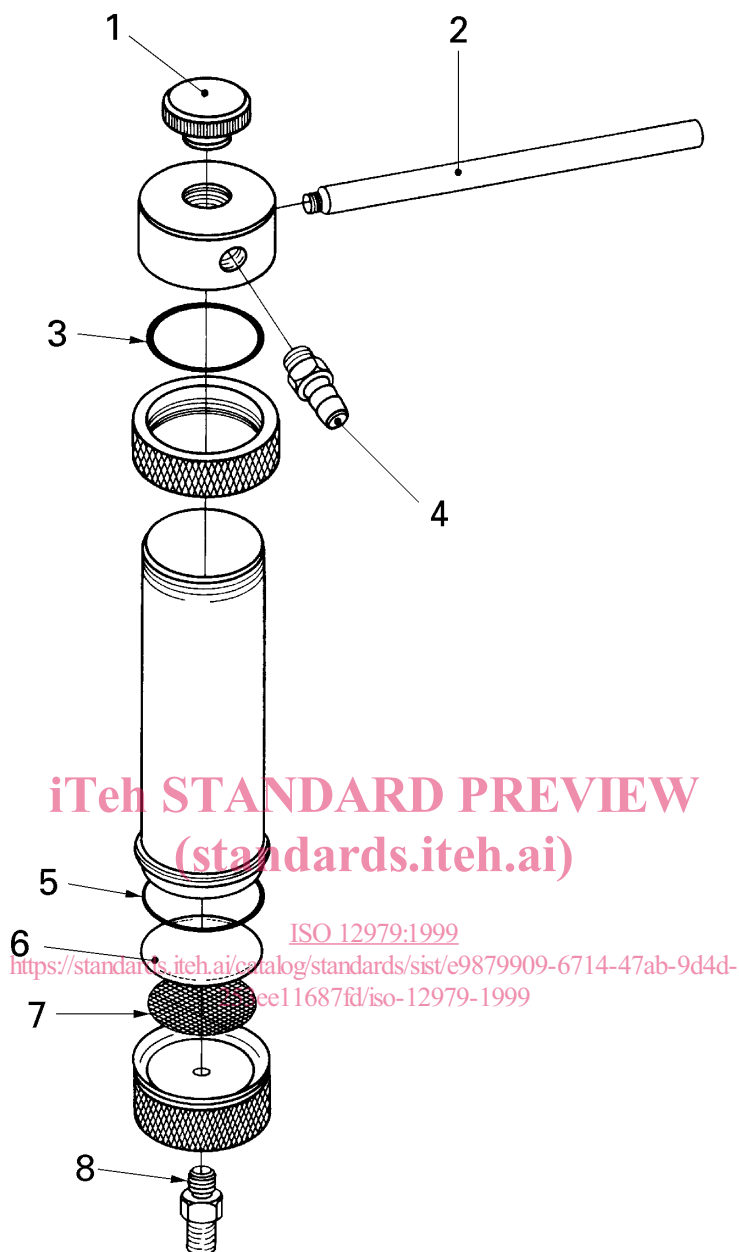
4.4.3 When the filtration of the quinoline solution is complete, depressurize the filtration apparatus, rinse the beaker with approximately 5 ml of quinoline at 70 °C to 80 °C, and transfer the washings via the glass funnel to the filtration apparatus. Close the apparatus and gradually repressurize it until filtration commences. This will typically occur at approximately 0,1 MPa. Adjust the applied pressure to achieve a satisfactory flow rate. Repeat the rinsing of the beaker and the washing of the insoluble matter on the filter a further nine times using approximately 5 ml of quinoline at 70 °C to 80 °C for each operation, by which stage the washings passing into the graduated receiver should be very little darker than the distilled quinoline (4.1.1). If the washings are still deeply coloured, continue washing with further portions of hot quinoline until they show no further lightening of colour. Wash the insoluble matter in the pressure-filtration apparatus with ten successive approximately 5 ml portions of the toluene (4.1.2) at 70 °C to 80 °C, using an applied gas pressure reduced to approximately 0,01 MPa to 0,02 MPa to achieve a controllable flow rate. Allow the final washings to drain for 1 min to 2 min.

4.4.4 Weigh the watch glass (4.2.6) to the nearest 0,1 mg. Transfer the membrane filter disc from the pressure-filtration apparatus to the watch glass. If any significant amount of the quinoline-insoluble matter adheres to the O-ring seal, place this on the watch glass also. Place the watch glass and contents in an oven maintained at 105 °C to 110 °C for 2 h, then transfer them to a desiccator to cool.

If quinoline-insoluble material has adhered to the O-ring seal, gently brush this on to the watch glass. Weigh the watch glass with the membrane filter disc and the quinoline-insoluble matter to the nearest 0,1 mg. Using forceps, hold the membrane filter disc, which will have rolled into a tube, over the watch glass and detach as much adhering quinoline-insoluble matter as possible by sharply tapping the forceps. Hold the membrane filter disc flat on a clean, smooth piece of paper and remove any residual quinoline-insoluble material by carefully brushing.

Transfer the recovered quinoline-insoluble material to a clean vial of suitable size to await analysis.

NOTE An artist's sable brush, size 5 has been found to be suitable for brushing off the quinoline-insoluble material.



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Key

- | | |
|---|--|
| <p>1 Cap
2 Clamping rod
3 Silicone O-ring
4 Pressure hose connector</p> | <p>5 Silicone O-ring
6 Perforated support plate
7 Coarse mesh support
8 Outlet connector</p> |
|---|--|

Figure 1 — Typical pressure-filtration apparatus

4.4.5 If the amount of recovered quinoline-insoluble material is insufficient for the ensuing analysis, repeat the extraction with amounts of pitch and quinoline increased proportionately up to a maximum of 5 g of pitch and 125 ml of quinoline for a single extraction. In the event that the increased amounts still yield an inadequate quantity of quinoline-insoluble matter, repeat the entire extraction procedure using further 5 g portions of the test sample until the required amount is obtained.

4.5 Calculation and expression of results

Calculate the yield of quinoline-insoluble material, w_{qi} , expressed as a percentage by mass, using the equation:

$$w_{qi} = \frac{100(m_1 - m_2 - m_3)}{m_4 - m_5} \quad (1)$$

where

- m_1 is the combined mass of the watch glass, the membrane filter disc and the isolated quinoline-insoluble material, in grams;
- m_2 is the mass of the watch glass, in grams;
- m_3 is the mass of the membrane filter disc, in grams;
- m_4 is the mass of the beaker containing the pitch sample, in grams;
- m_5 is the mass of the empty beaker, in grams.

Express the result of the calculation to two significant figures.

NOTE This result should not be quoted as the content of quinoline-insoluble matter present in the pitch because the quinoline-insoluble matter is empirical. ISO 6791 should be used for the determination of quinoline-insoluble matter.

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5 Analysis of quinoline-insoluble material for carbon and hydrogen content

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5.1 Reagents

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Warning — Refer to the reagent supplier's Health and Safety data sheets for the precautions which are to be taken for the safe use of magnesium perchlorate and sodium hydroxide supported on synthetic silicate.

5.1.1 Magnesium perchlorate, anhydrous, analytical reagent grade, of particle size range approximately 0,7 mm to 1,2 mm.

5.1.2 Sodium hydroxide on synthetic silicate support, analytical reagent grade, of particle size range approximately 1,5 mm to 3,0 mm.

5.1.3 Silver metal woven mesh, of wire diameter approximately 0,3 mm and mesh aperture approximately 0,8 mm.

5.1.4 Silver metal wool, microanalytical quality, typically of wire diameter approximately 0,05 mm.

5.1.5 Quartz wool.

5.1.6 Oxygen, of purity at least 99,5 %, supplied via a two-stage pressure regulator and a needle-valve capable of controlling the flow rate in the range 0 ml/min to 500 ml/min.

5.2 Apparatus

Ordinary laboratory apparatus and the following.

5.2.1 Carbon/hydrogen analyser, comprising the items in 5.2.2 to 5.2.8.