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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

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*.
<https://standards.iteh.ai/catalog/standards/iso/e9879909-6714-47ab-9d4d-253ee11687fd/iso-12979-1999>

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.

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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