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Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of sulfur content by an instrumental method

Produits carbonés utilisés pour la production de l'aluminium — Brai pour électrodes — Dosage du soufre par une méthode instrumentale

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Foreword

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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 10238 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*.

Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of sulfur content by an instrumental method

1 Scope

This International Standard describes a method for determining the sulfur content of pitch used in the production of aluminium. The method is applicable to pitches of both coal tar and petroleum origin, provided that they contain between 0,1 % and 4,0 % by mass of sulfur.

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 3696, *Water for analytical laboratory use — Specification and test methods.*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

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

3 Principle

A known mass of the sample is burned in a stream of oxygen at a temperature of 1 350 °C. The oxides of sulfur formed, together with any chlorine present, are absorbed in neutral hydrogen peroxide and determined volumetrically. A correction is made to take account of the chlorine content of the sample. Aluminium oxide is added to the sample to prevent the retention of sulfur in the ash.

4 Reagents

Unless otherwise specified, use reagents of analytical reagent grade and water conforming to grade 3 of ISO 3696.

4.1 Disodium tetraborate solution, $c(\frac{1}{2}Na_2B_4O_7 \cdot 10H_2O)$ = 0,025 mol/l.

4.2 Sulfuric acid solution, $c(\frac{1}{2}H_2SO_4)$ = 0,025 mol/l.

4.3 Hydrogen peroxide solution, consisting of 3 % by volume of H_20_2 and 97 % by volume of water, neutralized with the disodium tetraborate solution (4.1) to the screened indicator (4.4).

4.4 Screened indicator, consisting of equal volumes of a) and b) prepared as follows and mixed immediately before use:

a) 0,125 g of methyl red dissolved in 100 ml of 95 % by volume ethanol solution;

b) 0,083 g of methylene blue dissolved in 100 ml of 95 % by volume ethanol solution.

Store in a dark glass bottle.

4.5 Mercury(II) oxycyanide solution, consisting of approximately 100 ml of water saturated with mercury(II) oxycyanide ($3Hg(CN)_2 \cdot HgO$).

Agitate thoroughly, filter and neutralize the filtrate with the sulfuric acid solution (4.2) to the screened indicator. Store the solution in a dark glass bottle. Do not keep longer than 4 days.

WARNING — This compound and its solution are toxic and should be handled with appropriate care.

4.6 Aluminium oxide, finely divided, particle size approximately 0,1 mm.

4.7 Oxygen.

4.8 Sodium hydroxide on an inert base, preferably of coarse grading, for example 1,7 mm to 1,2 mm, and preferably of the self-indicating type.

5 Apparatus

Ordinary laboratory apparatus and the following.

5.1 Combustion furnace and absorption train, as shown in Figure 1, and consisting of the following.

5.1.1 Furnace, electrically heated, designed to carry a tube of 28,5 mm outside diameter and to heat it over a length of about 125 mm to give a temperature of 1 350 °C at the centre of the hot zone and a temperature distribution curve similar to that shown in Figure 2.

5.1.2 Combustion tube, of 22 mm internal diameter and 28,5 mm outside diameter and 0,65 m long, made of refractory aluminous porcelain that is not permeable to gases at 1 400 °C.

5.1.3 Combustion boat, of iron-free unglazed porcelain, 70 mm long, 12,5 mm wide and 10 mm deep, that does not blister, discolour or change in mass on heating at 1 350 °C in oxygen for 3 h.

5.1.4 Silica pusher, comprising a sealed tube or rod of silica, 6 mm in diameter and approximately 450 mm long, flattened at one end to form a disc of 12 mm diameter for pushing the combustion boat into the furnace.

The pusher passes loosely through a glass or metal T-piece, one end of which fits into the rubber stopper that closes the inlet end of the combustion tube, the other being sealed with a rubber sleeve through which the pusher slides (see Figure 1). Oxygen is admitted through the limb of the T-piece.

The pusher is marked in millimetres from the disc end for convenience in measuring how far the combustion boat is pushed into the combustion tube.

5.1.5 Hook, comprising a length of stiff nickel chromium wire with a hooked end, to extract the combustion boat from the furnace on to a piece of refractory tile.

5.1.6 Flowmeter, capable of measuring rates of flow of oxygen of up to 300 ml/min.

5.1.7 Pressure gauge, to measure the back pressure on the system, normally 0,5 kPa to 0,7 kPa.