INTERNATIONAL STANDARD

ISO
9055

First edition
1988-12-15

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

Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of sulfur content by the bomb method

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

Reference number
ISO 9055 : 1988 (E)

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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9055 was prepared by Technical Committee ISO/TC 47, *Chemistry.*

Annex A of this International Standard is for information only.

Printed in Switzerland

Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of sulfur content by the bomb method

WARNING — Strict adherence to all of the provisions prescribed in this International Standard should ensure against explosive rupture of the bomb, or a blow-out, provided that the bomb is of proper design and construction and in good mechanical condition. It is desirable that the bomb be enclosed in a shield of steel plate at least 13 mm thick, or that equivalent protection be provided against unforseeable contingencies.

1 Scope

This International Standard specifies 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 at least 0,1 % (*m*/*m*) sulfur.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6257 : 1980, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling.*

3 Principle

Oxidation of the sample by combustion in a bomb containing oxygen under pressure. Gravimetric determination of the sulfur, in the form of sulfate in the bomb washings, as barium sulfate.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, ϱ approximately 1,19 g/ml, approximately 37 % (*m*/*m*) solution.

4.2 Bromine water, saturated.

4.3 Barium chloride, 85 g/l solution.

Dissolve 100 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in water and dilute to 1 000 ml.

4.4 Sodium carbonate, 50 g/l solution.

Dissolve 135 g of sodium carbonate decahydrate ($Na_2CO_3.10H_2O$) or its equivalent mass in water and dilute to 1 000 ml.

4.5 Oxygen, free of combustible material and sulfur compounds, available at a pressure of 4,053 MPa*).

4.6 White oil: liquid paraffin B.P. or equivalent.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Bomb, of capacity of not less than 300 ml, and so constructed that it will not leak during the test and that quantitative recovery of the liquids from the bomb may be readily achieved.

The inner surface of the bomb may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the bomb assembly, such as the head gasket and the lead-wire insulation, shall be

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*) 1 MPa = 1 MN/m^2

resistant to heat and chemical action, and shall not undergo any reaction that will affect the sulfur content of the liquid in the bomb[3].

5.2 Platinum sample cup, of outside diameter 24 mm at the bottom and 27 mm at the top, height 12 mm and mass 10 g to 11 g.

5.3 Platinum firing wire, 0,4 mm diameter approximately.

5.4 Ignition circuit, capable of supplying sufficient current to ignite the cotton wicking or nylon thread without melting the wire.

The current shall not be drawn directly from the mains and the voltage shall not exceed 25 V.

WARNING — The switch in the ignition circuit shall be of a type which remains open, except when held in a closed position by the operator.

5.5 White cotton wicking, or white nylon sewing thread.

5.6 Emery polishing paper, No. 00 or equivalent.

5.7 Chromium(III) oxide paste, grit free.

5.8 Cold water bath.

5.9 Porcelain filter crucible, P 10 series (porosity 4 µm to 10 µm).

6 Sampling and test sample preparation

6.1 Sampling

Sample the pitch for electrodes in accordance with ISO 6257.

6.2 Preparation of test sample

Prepare the test sample immediately before the determination.

If it is sufficiently hard, grind the whole of the test sample with a pestle and mortar to a particle size of less than 200 µm.

If the pitch is too soft for grinding, melt and mix the sample, ensuring that its temperature does not exceed 150 °C and that the melting period does not exceed 10 min. The material for the test portion may be taken from the molten sample.

7 Procedure

7.1 Preparation of bomb and test portion

Cut a piece of firing wire (5.3) 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals.

Arrange the coil so that it will be above and to one side of the sample cup (5.2). Insert between two loops of the coil a wisp of the cotton or nylon thread (5.5) of such length that one end will extend into the sample cup. Place about 5 ml of the sodium carbonate solution ((4.4) in the bomb (5.1) and rotate the bomb in such a manner that the interior surface is moistened by the solution.

Introduce into the sample cup the quantities of sample and, if required, white oil (4.6) specified in table 1, weighing the sample to the nearest 0,1 mg. (When white oil is used, stir the mixture with a short length of silica rod and allow the rod to remain in the sample cup during the combustion.)

WARNING — Do not use more than 0,8 g total of sample and white oil or other low-sulfur combustible material.

Table 1

Sulfur content % (m/m)	Mass of test portion g	Mass of white oil g
⩽ 5	0,6 to 0,8	0
> 5	0,3 to 0,4	0,3 to 0,4

After repeated use of the bomb for sulfur determinations, a film may be noticed on the inner surface. This dullness shall be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 r/min and polish the inside surface with emery polishing paper (5.6), coated with a light machine oil to prevent deep cutting, and then with the chromium(III) oxide paste (5.7) and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before using the bomb, it shall be washed with soap and water to remove oil or paste left from the polishing operation.

Use of sample masses containing over 20 mg of chlorine may cause corrosion of the bomb. To avoid this, it is recommended that, for samples containing over 2 % (m/m) chlorine, the sample mass be based on the chlorine content as given in the following table :

Table 2

Chlorine content % (m/m)	Mass of sample g	Mass of white oil g
2 to 5	0,4	0,4
> 5 to 10	0,2	0,6
> 10 to 20	0,1	0,7
> 20 to 50	0,05	0,7

If the sample is not readily miscible with white oil, some other low-sulfur combustible diluent may be substituted. However, the combined mass of sample and non-volatile diluent shall not exceed 0,8 g.

7.2 Addition of oxygen

Place the sample cup in position and arrange the cotton wisp or nylon thread so that the end dips into the sample. Assemble the bomb and tighten the cover securely.

WARNING — Do not add oxygen or ignite the sample if the bomb has been jarred, dropped or tilted.

Admit oxygen slowly (to avoid blowing the oil from the cup) until the pressure indicated in table 3 is reached.

Table 3

Capacity of bomb ml	Minimum gauge pressure[1] MPa	Maximum gauge pressure[1] MPa
300 to 350	3,95	4,15
350 to 400	3,65	3,85
400 to 450	3,14	3,34
450 to 500	2,84	3,04

[1] The minimum pressures are specified to provide sufficient oxygen for complete combustion and the maximum pressures represent a safety requirement.

7.3 Combustion

Immerse the bomb in the cold water bath (5.8). Connect the terminals to the open electrical circuit (5.4). Close the circuit to ignite the sample.

WARNING — Do not go near the bomb till at least 20 s after firing.

Remove the bomb from the bath after a further immersion period of at least 10 min.

Release the pressure at a slow, uniform rate such that the operation requires not less than 1 min. Open the bomb and examine the contents.

If traces of unburned pitch or sooty deposits are found, discard the determination and thoroughly clean the bomb before again putting it in use.

7.4 Collection of sulfur solution

Rinse the interior of the bomb, the oil cup, and the inner surface of the bomb cover with a fine jet of water, and collect the washings in a 600 ml beaker having a mark to indicate 75 ml.

Remove any precipitate in the bomb by means of a rubber policeman. Wash the base of the terminals until the washings are neutral to a suitable indicator. (The volume of the washings is normally in excess of 300 ml.) Add 10 ml of the saturated bromine water (4.2) to the washings in the beaker. Place the sample cup in a 50 ml beaker. Add 5 ml of the saturated bromine water, 2 ml of the hydrochloric acid (4.1) and enough water just to cover the cup. Heat the contents of the beaker to just below its boiling point for 3 min to 4 min and add to the beaker containing the bomb washings. Wash the sample cup and the 50 ml beaker thoroughly with water. Remove any precipitate in the cup by means of a rubber policeman.

Add the washings from the cup and the 50 ml beaker, and the precipitate, if any, to the bomb washings in the 600 ml beaker. Do not filter any of the washings, since filtering would remove any sulfur present as insoluble material.

7.5 Determination

Evaporate the combined washings to 200 ml on a hotplate or other source of heat. Adjust the heat to maintain slow boiling of the solution and add 10 ml of the barium chloride solution (4.3), either in a fine stream or drop by drop. Stir the solution during the addition and for 2 min thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume of approximately 75 ml as indicated by the mark on the beaker.

Remove the beaker from the hotplate (or other source of heat) and allow it to cool for 1 h before filtering.

Filter the supernatant liquid through an ashless, quantitative filter paper (see note 1).

Wash the precipitate with water, first by decantation and then on the filter, until free from chloride. Transfer the paper and precipitate to a tared crucible weighed to the nearest 0,1 mg and dry (see note 2) at low heat until the moisture has evaporated.

Char the paper completely without igniting it, and finally heat at a bright red heat until the residue is white in colour. After combustion is complete, allow the crucible to cool to room temperature, and weigh to the nearest 0,1 mg.

NOTES

1 The tared porcelain filter crucible (5.9) may be used in place of the filter paper. In this case the precipitate is washed free of chloride and then dried to constant mass at 500 °C ± 25 °C.

2 A satisfactory means of drying, charring and combusting the paper and precipitate is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and combustion usually will occur at the desired rate.

7.6 Blank

Make a blank determination whenever new reagents, white oil or other low-sulfur combustible material are used. When running a blank on white oil, use 0,3 g to 0,4 g and follow the normal procedure.

8 Expression of results

8.1 Method of calculation

The sulfur content of the sample, expressed as a percentage by mass, is given by the formula

$$\frac{(m_1 - m_2) \times 0{,}137\,3}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of barium sulfate obtained from the test portion;

m_2 is the mass, in grams, of barium sulfate obtained during the blank test;

0,137 3 is the conversion factor from $BaSO_4$ to S.

8.2 Precision

The permissible difference between two results shall not exceed the appropriate value given in table 4.

Table 4

Values in percentage by mass

Sulfur content	Repeatability	Reproducibility
0,1 to 0,5	0,04	0,05
> 0,5 to 1,0	0,05	0,09
> 1,0 to 1,5	0,08	0,15
> 1,5 to 2,0	0,12	0,25
> 2,0 to 5,0	0,18	0,27

9 Test report

The test report shall include the following particulars:

a) an identification of the sample;

b) a reference to this International Standard;

c) the results and the units in which they are expressed;

d) any unusual features noted during the determination;

e) any operations not included in this International Standard or regarded as optional.

Annex A
(informative)

Bibliography

[1] ASTM D 129-64, *Standard test method for sulfur in petroleum products (General bomb method).*

[2] BS 2000 : Part 61 : 1982, *Petroleum and its products — Part 61 : Sulphur in petroleum products (bomb method).*

[3] BS 4791 : 1985, *Specification for calorimeter bombs.*

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UDC 665.777 : 542.5 : 546.22

Descriptors : industrial products, electrodes, pitch (materials), chemical analysis, determination of content, sulphur, aluminium, production.

Price based on 4 pages