International Standard 2072

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Aluminium oxide primarily used for the production of aluminium — Determination of zinc content — PAN photometric method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage du zinc — Méthode photométrique au PAN

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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 2072 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in May 1980.

It has been approved by the member bodies of the following countries :

Austria	Germany, F. R.	Poland
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Canada	Ireland	Sweden
China	Italy	Switzerland
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Egypt, Arab Rep. of	Mexico	USSR
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Australia
USA

This International Standard cancels and replaces ISO Recommendation R 2072-1971, of which it constitutes a technical revision.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

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INTERNATIONAL STANDARD ISO 2072-1981 (E)

Aluminium oxide primarily used for the production of aluminium — Determination of zinc content — PAN photometric method

WARNING — Attention is drawn to the dangers involved in the use of chloroform (see the note to 4.11).

1 Scope and field of application

This International Standard specifies a 1-(2-pyridylazo)-2-naphthol (PAN) photometric method for the determination of the zinc content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having zinc contents, expressed as ZnO, in the range 0,002 to 0,12 % (*m/m*).

2 Reference

ISO 804, *Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by alkaline fusion.*

3 Principle

Alkaline fusion of a test portion, and dissolution of the fused mass in nitric acid.

Separation of the zinc in the form of its thiocyanate by extraction with 4-methylpentan-2-one.

Re-extraction of the zinc with an aqueous hydrochloric acid solution.

Formation of the PAN-zinc complex, at pH 7,5, in the presence of tartaric acid, sodium thiosulphate and nitroso-R salt.

Extraction of the coloured PAN-zinc complex with chloroform, and photometric measurement at a wavelength of about 560 nm.

NOTE — The addition of tartaric acid eliminates interference by traces of aluminium and iron and the addition of thiosulphate and nitroso-R salt eliminates interference by copper and cobalt.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water which has been doubly distilled from a borosilicate glass apparatus with ground joints. Avoid the use of lead glass.

The reagents specified in ISO 804, clause 4, and

4.1 Hydrochloric acid, 220 g/l solution.

4.2 Ammonium thiocyanate, 500 g/l solution, purified.

4.2.1 Preparation of the solution

Dissolve 50 g of ammonium thiocyanate in water and dilute to 100 ml.

4.2.2 Purification of the solution

Adjust the pH of the solution (4.2.1) to 7,5, checking with the pH meter (5.2), by adding the sodium hydroxide solution (4.7), followed by the sodium hydroxide solution (4.8). Transfer the solution to a separating funnel of suitable capacity, add 10 ml of the PAN solution (4.10) and 30 ml of the chloroform (4.11), and shake vigorously for a few minutes. Allow to settle, draw off the organic phase and discard it. Again add to the aqueous phase 10 ml of the PAN solution and 30 ml of the chloroform, shake, allow to settle and draw off and discard the organic phase. Repeat the extractions until the organic phase is completely colourless. Remove the last traces of PAN by shaking with the chloroform and, after decantation, discard the organic phase.

Place the aqueous phase in a borosilicate glass or plastics flask.

4.3 4-methylpentan-2-one [Methyl isobutyl ketone].

4.4 Sodium acetate trihydrate, 250 g/l solution, purified.

4.4.1 Preparation of the solution

Dissolve 25 g of sodium acetate trihydrate in water and dilute to 100 ml.

4.4.2 Purification of the solution

Proceed as specified in 4.2.2, using 36,5 g/l hydrochloric acid solution to adjust the pH to 7,5.

4.5 Tartaric acid, 500 g/l solution, purified.

4.5.1 Preparation of the solution

Dissolve 50 g of tartaric acid in water and dilute to 100 ml.

4.5.2 Purification of the solution

Proceed as specified in 4.2.2, using a 400 g/l sodium hydroxide solution, followed by a 40 g/l sodium hydroxide solution, to adjust the pH to 7,5.

4.6 1-nitroso-2-hydroxynaphthalene-3,6-disulphonic acid, disodium salt [nitroso-R salt], 10 g/l solution.

4.7 Sodium hydroxide, 200 g/l solution.

4.8 Sodium hydroxide, 20 g/l solution.

4.9 Sodium thiosulphate pentahydrate, 250 g/l solution, purified.

4.9.1 Preparation of the solution

Dissolve 25 g of sodium thiosulphate pentahydrate in water and dilute to 100 ml.

4.9.2 Purification of the solution

Proceed as specified in 4.2.2.

4.10 1-(2-pyridylazo)-2-naphthol [PAN], 1 g/l methanolic solution.

Dissolve 0,1 g of PAN in methanol, and dilute to 100 ml with the same methanol.

4.11 Chloroform.

NOTE — **Harmful vapour. Avoid breathing vapour and contact with eyes.**

4.12 Zinc, standard solution corresponding to 0,100 g of ZnO per litre.

Weigh, to the nearest 0,000 1 g, 0,100 g of zinc oxide, previously calcined at 1 000 °C for 1 h and cooled in a desiccator, into a 100 ml beaker and dissolve it in 10 ml of the hydrochloric acid solution (4.1).

Dilute the solution and transfer quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 100 µg of ZnO.

4.13 Zinc, standard solution corresponding to 0,010 g of ZnO per litre.

Take 50,0 ml of the standard zinc solution (4.12), place it in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 µg of ZnO.

5 Apparatus

Use glassware, including reagent flasks, made of borosilicate glass or a glass which does release zinc or lead; alternatively, a suitable plastics material may be used. Use only ground glass or plastics (not rubber) stoppers.

The apparatus specified in ISO 804, clause 5, and

5.1 Burette, graduated in 0,05 ml divisions, complying with the requirements of ISO/R 385.

5.2 pH meter.

5.3 Spectrophotometer, or

5.4 Photoelectric absorptiometer, fitted with filters ensuring maximum transmission at a wavelength of about 560 nm.

6 Procedure

6.1 Test portion

Take an aliquot portion of the principal solution P prepared in accordance with ISO 804, sub-clauses 6.1, 6.2 and 6.3, containing 10 to 50 µg of ZnO, and place it in a beaker of suitable capacity (for example 250 ml).

6.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure, on a solution prepared without the addition of extra-pure aluminium oxide, in accordance with ISO 804, sub-clause 6.4.2, using the same quantities of reagents as used for the determination.

6.3 Preparation of the calibration graph

6.3.1 Preparation of standard colorimetric solutions, for photometric measurements carried out in cells of optical path length 1 cm

By means of the burette (5.1), transfer into a series of seven beakers of suitable capacity (for example 100 ml), the volumes of the standard zinc solution (4.13) shown in the following table.

Standard zinc solution (4.13)	Corresponding mass of ZnO
ml	µg
0*	0
1,0	10
2,0	20
3,0	30
4,0	40
5,0	50
6,0	60

* Blank test of the reagents for calibration.

6.3.2 Colour development

Treat each of the solutions (6.3.1) as follows.

Add 10 ml of the sodium acetate solution (4.4), 5 ml of the tartaric acid solution (4.5), 1 ml of the nitroso-R salt solution (4.6) and, checking with the pH meter (5.2), adjust the pH of the solution to about 6 by adding the sodium hydroxide solution (4.7). Then add 10 ml of the sodium thiosulphate solution (4.9) and, checking with the pH meter, adjust the pH of the solution to 7,5 ± 0,1 by adding the sodium hydroxide solution (4.8).

The volume of the solution should be about 70 ml. Transfer the solution to a separating funnel of suitable capacity (for example 250 ml), washing the beaker with about 10 ml of water. Add 2,5 ml of the PAN solution (4.10) and 20 ml of the chloroform (4.11), and shake vigorously for 5 min. Allow to settle and draw off the organic phase into a 50 ml one-mark volumetric flask.

Repeat the extraction of the aqueous phase twice more using, each time, 2,5 ml of the PAN solution (4.10) and 10 ml of the chloroform (4.11). Finally, extract the organic phase dispersed in the aqueous phase using several small quantities of the chloroform. Collect all the organic phase in the same 50 ml one-mark volumetric flask. Dilute to the mark with the same chloroform and mix.

Use this solution for the photometric measurements.

6.3.3 Photometric measurements

Fill one of the optical cells with the chloroform solution (6.3.2) which has been filtered through a piece of dry cotton wool.

Carry out the photometric measurements using the spectrophotometer (5.3) set at the wavelength of maximum absorption (about 560 nm), or the photoelectric absorptiometer (5.4) fitted with suitable filters, after having, in each case, adjusted the apparatus to zero absorbance against the chloroform (4.11).

Deduct the absorbance of the blank test of the reagents for calibration from those of the standard colorimetric solutions (6.3.1).

6.3.4 Plotting the graph

Plot a graph having, for example, the masses, in micrograms, of zinc oxide (ZnO) contained in 50 ml of the standard colorimetric solutions (6.3.1) as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 Preparation of the test solution

Adjust the volume of the test portion (6.1) to about 30 ml, either by evaporation or by dilution.

6.4.2 Extraction of zinc

Transfer the test solution (6.4.1) to a separating funnel of suitable capacity (for example 250 ml), washing the beaker with about 5 ml of water.

Add 6 ml of the hydrochloric acid solution (4.1), 40 ml of the ammonium thiocyanate solution (4.2) and 10 ml of the methyl isobutyl ketone (4.3). Shake vigorously for 5 min. Allow to settle and draw off the aqueous phase into another separating funnel. Add 10 ml of the methyl isobutyl ketone to the aqueous phase, shake vigorously for 5 min and, after separation, draw off and discard the aqueous phase. Combine the organic phases quantitatively in a separating funnel and wash twice, using 10 ml of water for each washing, and shaking vigorously for several minutes. Discard the washings. Add, to the separating funnel containing the organic phase, 10 ml of the hydrochloric acid solution (4.1) and shake vigorously for 2 min. Allow to settle and draw off the aqueous phase, collecting it in a beaker of suitable capacity (for example 100 ml). Repeat the extraction twice more with, each time, 10 ml portions of the hydrochloric acid solution (4.1), collecting the aqueous phases in the same beaker.

Discard the organic phase. Place the beaker containing the combined aqueous phases on a boiling water bath and evaporate cautiously until the volume is reduced to about 3 ml. Remove the beaker from the water bath and allow to cool.

6.4.3 Colour development

Carry out the procedure as specified in 6.3.2.

6.4.4 Photometric measurements

Carry out photometric measurements on the chloroform extracts (6.4.3) from the test solution (6.4.1) and from the blank test solution (6.2), as specified in 6.3.3, after having adjusted the apparatus to zero absorbance against the chloroform (4.11).

7 Expression of results

By means of the calibration graph (6.3.4), determine the masses of ZnO corresponding to the values of the photometric measurements (6.4.4).

The zinc content, expressed as zinc oxide (ZnO) as a percentage by mass, is given by the formula

$$\frac{(m_1 - m_2) \times r_D \times 100}{10^6 \times m_0}$$

where

m_0 is the mass, in grams, of the test sample taken for the preparation of the principal solution P;

m_1 is the mass, in micrograms, of ZnO found in the aliquot portion of the test solution (6.4.1);

m_2 is the mass, in micrograms, of ZnO found in a corresponding aliquot portion of the blank test solution (6.2);

r_D is the ratio of the volume of the principal solution P to the volume of the aliquot portion (see 6.1) taken for the extraction of the zinc (6.4.2).

Express the result to three decimal places.

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 Standard to which reference is made, or regarded as optional.

Annex

ISO publications relating to aluminium oxide primarily used for the production of aluminium

ISO 802 — Preparation and storage of test samples.

ISO 803 — Determination of loss of mass at 300 °C (conventional moisture).

ISO 804 — Preparation of solution for analysis — Method by alkaline fusion.

ISO 805 — Determination of iron content — 1,10-Phenanthroline photometric method.

ISO 806 — Determination of loss of mass at 1 000 °C and 1 200 °C.

ISO 900 — Determination of titanium content — Diantipyrylmethane photometric method.

ISO 901 — Determination of absolute density — Pyknometer method.

ISO 902 — Measurement of the angle of repose.

ISO 903 — Determination of untamped density.

ISO 1232 — Determination of silica content — Reduced molybdosilicate spectrophotometric method.

ISO 1617 — Determination of sodium content — Flame emission spectrophotometric method.

ISO 1618 — Determination of vanadium content — *N*-Benzoyl-*N*-phenylhydroxylamine photometric method.

ISO 2069 — Determination of calcium content — Flame atomic absorption method.

ISO 2070 — Determination of calcium content — Naphthalhydroxamic acid spectrophotometric method.

ISO 2071 — Determination of zinc content — Flame atomic absorption method.

ISO 2072 — Determination of zinc content — PAN photometric method.

ISO 2073 — Preparation of solution for analysis — Method by hydrochloric acid attack under pressure.

ISO 2828 — Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method.

ISO 2829 — Determination of phosphorus content — Reduced phosphomolybdate spectrophotometric method.

ISO 2865 — Determination of boron content — Curcumin spectrophotometric method.

ISO 2926 — Particle size analysis — Sieving method.

ISO 2927 — Sampling.

ISO 2961 — Determination of an adsorption index.

ISO 3390 — Determination of manganese content — Flame atomic absorption method.