
International Standard



6994

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Aluminium ores — Determination of aluminium content — EDTA titrimetric method

Minerais alumineux — Dosage de l'aluminium — Méthode titrimétrique à l'EDTA

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Foreword

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International Standard ISO 6994 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Aluminium ores — Determination of aluminium content — EDTA titrimetric method

1 Scope and field of application

This International Standard specifies an EDTA titrimetric method for the determination of the aluminium content of aluminium ores.

The method is applicable to products having aluminium contents, expressed as aluminium oxide (Al_2O_3), in the range 30 to 65 % (*m/m*).

2 Reference

ISO 8557, *Aluminium ores — Determination of hygroscopic moisture in analytical samples — Gravimetric method.*

3 Principle

Decomposition of the test portion by either

- a) treatment with hydrochloric, nitric and sulfuric acids;

NOTE — This method is recommended for ores containing gibbsite and/or boehmite and when the residue from the dissolution of the test portion after silica volatilization is less than 1 % of the mass of the test portion.

or

- b) sintering with sodium peroxide followed by brief fusion and dissolution of the melt with sulfuric acid;

NOTE — This method is recommended for ores containing diaspore and when the residue from the dissolution of the test portion after silica volatilization is greater than 1 % of the test portion.

or

- c) fusion with sodium tetraborate/sodium carbonate flux followed by dissolution of the melt with sulfuric acid.

NOTE — This method is suitable for all types of ore.

Removal of silica by filtration to give a solution containing aluminium and other elements. Measurement of an aliquot portion and precipitation of aluminium and other elements except chromium, manganese and vanadium, by ammonium benzoate in acid solution. Filtration and dissolution of the precipitated benzoates in acid followed by precipitation of elements except aluminium by sodium hydroxide. Filtration of the precipitate followed by acid dissolution, reprecipitation with sodium hydroxide and filtration to recover coprecipitated aluminium. Addition of an excess of EDTA to the combined filtrates and determination of aluminium content by back titration of the excess EDTA with zinc sulfate solution using xylenol orange as indicator.

4 Reagents

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

4.1 Sodium peroxide (Na_2O_2), powdered.

NOTE — Sodium peroxide should be protected against humidity and should not be used once it has begun to agglomerate.

4.2 Sodium carbonate/sodium tetraborate flux.

Mix intimately 3 parts by mass of anhydrous sodium carbonate (Na_2CO_3) and 1 part by mass of anhydrous sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$).

4.3 Ammonium acetate, 250 g/l solution.

Dissolve 250 g of ammonium acetate in 900 ml of water and adjust the pH to 6 by the addition of glacial acetic acid (ρ 1,050 g/ml). Dilute to 1 litre.

4.4 Ammonium benzoate, 100 g/l solution.

NOTE — To obtain a clear solution after warming, it may be necessary to add up to 5 ml of ammonia solution (4.6). Filter if necessary to remove any particulate residue.

4.5 Hexamethylenetetramine (hexamine), 250 g/l solution.

Dissolve 25 g of hexamethylenetetramine in 70 ml of water and dilute to 100 ml.

4.6 Ammonia solution, (ρ 0,9 g/ml), diluted 1 + 1.

4.7 Glacial acetic acid, (ρ 1,050 g/ml).

4.8 Hydrochloric acid (ρ 1,17 g/ml), diluted 2 + 3.

4.9 Sodium hydroxide, 160 g/l solution.

This solution shall be stored in a plastic bottle.

4.10 Sulfuric acid (ρ 1,84 g/ml), diluted 1 + 1.

4.11 Sulfuric acid (ρ 1,84 g/ml), diluted 1 + 3.

4.12 Sulfuric acid (ρ 1,84 g/ml), diluted 1 + 9.

4.13 Hydrofluoric acid, 40 % (m/m), (ρ 1,13 g/ml).

4.14 Sodium sulfate, ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$), 100 g/l solution.

4.15 Potassium permanganate, 1 g/l solution.

4.16 Acid mixture.

Measure into a 1 litre beaker 225 ml of water and add cautiously, with stirring, 175 ml of sulfuric acid (ρ 1,84 g/ml). Cool to room temperature and add 150 ml of hydrochloric acid (ρ 1,17 g/ml) and 50 ml of nitric acid (ρ 1,42 g/ml).

This solution shall be prepared for each series of determinations and any unused solution discarded.

4.17 Standard aluminium solution.

Dissolve approximately 1,00 g, weighed to the nearest 0,000 1 g, of aluminium metal (not less than 99,9 %) in 60 ml of the hydrochloric acid (4.8), heating if necessary. Cool and dilute to the mark in a 1 000 ml one-mark volumetric flask.

Calculate the equivalent aluminium oxide concentration a of the standard solution, in grams per litre, using the equation

$$a = m_1 \times 1,889 5 \times \frac{P}{100}$$

where

m_1 is the mass of aluminium metal, in grams;

P is the purity of aluminium used, in per cent.

4.18 Ethylenediaminetetraacetic acid disodium salt dihydrate (EDTA), approximately 0,05 mol/l (18,6 g/l) standard volumetric solution.

4.19 Zinc sulfate, standard volumetric solution, $c(\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}) = 0,05$ mol/l.

Dissolve 14,5 g of zinc sulfate heptahydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in water, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume and mix.

4.19.1 Standardization procedure

4.19.1.1 Titration of EDTA solution with standard zinc sulfate solution

Using a pipette, add 50 ml of the EDTA solution (4.18) to a 500 ml beaker and add 250 ml of water. Add 0,3 ml of the methyl orange indicator (4.20) and, if necessary, add the ammonia solution (4.6) drop by drop until the colour just changes to yellow.

Add 20 ml of the ammonium acetate solution (4.3) and adjust to $\text{pH } 5,7 \pm 0,1$, using either the ammonia solution (4.6) or the glacial acetic acid (4.7). Add 0,5 ml of the xylenol orange indicator (4.21) and titrate to the end-point (colour change yellow to purple) with zinc sulfate solution (4.19). Record the titre (V_1).

4.19.1.2 Determination of the equivalence factor for zinc sulfate solution

Pipette 50 ml (V_3) of the standard aluminium solution (4.17) into a 500 ml beaker, and add 200 ml of water.

With a pipette, add 50 ml of the EDTA solution (4.18), and 0,3 ml of the methyl orange indicator (4.20). Add the ammonia solution (4.6) drop by drop until the colour just changes.

Add 20 ml of the ammonium acetate solution (4.3) and a boiling aid. Boil for 10 min and cool. Adjust to $\text{pH } 5,7 \pm 0,1$, as in 4.19.1.1. Add 0,5 ml of the xylenol orange indicator (4.21) and titrate to the end-point with the zinc sulfate solution (4.19). Record the titre (V_2).

Calculate the equivalence factor A of the zinc sulfate solution, using the formula

$$1 \text{ ml zinc sulfate solution } (A) \equiv \frac{V_3 a}{V_1 - V_2} \text{ mg of Al}_2\text{O}_3$$

where a is the mass, in grams, of aluminium oxide per litre of standard aluminium solution (4.17).

4.20 Methyl orange indicator, 0,1 g/l solution.

4.21 Xylenol orange indicator, 1 g/l solution.

Prepare each month and store in a dark bottle.



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