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Chromium ores and concentrates — Determination of aluminium content — Complexometric method

Minerais et concentrés de chrome — Dosage de l'aluminium — Méthode complexométrique

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

International Standard ISO 8889 was prepared by Technical Committee ISO/TC 65, *Manganese and chromium 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.

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Chromium ores and concentrates — Determination of aluminium content — Complexometric method

1 Scope and field of application

This International Standard specifies a complexometric method for the determination of the aluminium content of chromium ores and concentrates. The method is applicable to a concentration range of 1,5 to 20,0 % (m/m) of aluminium content in chromium ores.

It should be read in conjunction with ISO 6629.

2 Reference

ISO 6629, *Chromium ores and concentrates — Methods of chemical analysis — General instructions.*

3 Principle

Decomposition of a test portion by treatment with perchloric, nitric and hydrochloric acids with removal of the chromium by distillation as chromyl chloride. Separation of the insoluble residue, the filtrate being reserved as the main solution.

Ignition of the residue and treatment with sulfuric and hydrofluoric acids. Fusion of the ignited residue with sodium disulfate. Dissolution of the melt and combination of the solution obtained with the main solution.

Precipitation of the hydroxides with the ammonia solution, filtration and redissolution of the hydroxides in hydrochloric acid. Reduction of chromate ions to ions of Cr(III) with hydrogen peroxide. Separation of aluminium from iron and other elements with sodium hydroxide.

Addition of EDTA Na₂ solution to an aliquot portion of the solution. Titration of an excess of EDTA Na₂ with zinc acetate solution, using xylenol orange as an indicator. Decomposition of the aluminium complex with sodium fluoride. Titration of the EDTA Na₂ released with zinc acetate solution, using xylenol orange as an indicator.

4 Reagents

- 4.1 Hydrochloric acid**, ρ 1,19 g/ml.
- 4.2 Hydrochloric acid**, diluted 1 + 1.
- 4.3 Hydrochloric acid**, diluted 1 + 100.

- 4.4 Nitric acid**, ρ 1,40 g/ml.
- 4.5 Hydrofluoric acid**, 40 % (m/m) solution.
- 4.6 Perchloric acid**, ρ 1,50 g/ml.
- 4.7 Sulfuric acid**, ρ 1,84 g/ml, diluted 1 + 1.
- 4.8 Ammonia solution**, ρ 0,91 g/ml, diluted 1 + 1.
- 4.9 Sodium hydroxide**, 20 % (m/m) solution.
- 4.10 Sodium hydroxide**, 1 % (m/m) solution.
- 4.11 Sodium disulfate** (pyrosulfate, Na₂S₂O₇).
- 4.12 Ammonium chloride**, 2 % (m/m) solution with addition of ammonia solution to adjust the pH to 7,0 to 8,0.
- 4.13 Phenolphthalein indicator**, 10 g/l alcoholic solution.
- Dissolve 1 g of indicator in 100 ml of ethanol, 60 % (m/m) solution.
- 4.14 Sodium fluoride**, 4 % (m/m) solution.
- Store in a polyethylene bottle.
- 4.15 Buffer solution**, pH 5,5 to 5,9.
- Dissolve 500 g of the ammonium acetate in 1 000 ml of water, add 30 ml of acetic acid (ρ 1,054 9 g/ml) and mix.
- Check the pH with a pH-meter and adjust by adding sodium hydroxide (4.10) or acetic acid.
- 4.16 Xylenol orange indicator**, 1 g/l solution.
- Dissolve 0,1 g of indicator in 100 ml of water.
- 4.17 Disodium salt of ethylenedinitrilotetraacetic acid**, standard volumetric solution, $c(\text{EDTA Na}_2) = 0,05 \text{ mol/l}$.
- Dissolve 18,6 g of EDTA Na₂ in 200 to 250 ml of water, transfer to a 1 000 ml one-mark volumetric flask, dilute with water to the mark and mix.

4.18 Zinc acetate, standard volumetric solution, $c(\text{Zn}) = 0,05 \text{ mol/l}$.

Dissolve 3,269 0 g of zinc (purity 99,99 %) in 50 ml of hydrochloric acid (4.2), add several drops of nitric acid (4.4) and evaporate the solution until almost dry. Add 200 ml of water, mix the solution, heat until the salts dissolve and cool. Add 25 ml of the buffer solution (4.15) to the solution, transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution corresponds to 0,001 349 g of aluminium.

Store in a polyethylene bottle.

5 Apparatus

Ordinary laboratory apparatus and

5.1 pH-meter.

6 Sample

Use a test sample which has been crushed to a size not exceeding 160 μm (checked on a sieve of appropriate aperture size) and air dried under laboratory conditions.

7 Procedure

7.1 Decomposition of test portion

Weigh 0,25 g of the test sample, place it in a 400 ml beaker, add 30 ml of perchloric acid (4.6) and 5 ml of nitric acid (4.4). Cover the beaker with a watch-glass, heat until dense white fumes of perchloric acid appear and continue heating to oxidize the chromium. Remove the watch-glass and carefully add hydrochloric acid (4.1), drop by drop, along the walls of the beaker until brown fumes of chromyl chloride cease to evolve, the chromium being reduced to the trivalent state. Replace the watch-glass on the beaker and heat the solution to oxidize the chromium completely.

Repeat the distillation of chromyl chloride to decompose the test portion completely.

Cool the solution, add 50 ml of hot water, heat gently to dissolve the salts and then heat almost to boiling. Filter the solution through a medium-texture filter paper containing a small amount of ashless paper pulp and wash the precipitate 12 to 15 times with hot hydrochloric acid (4.3) and twice with hot water. Collect the filtrate and washings in a 400 ml beaker and reserve as the main solution.

7.2 Treatment of residue

Transfer the residue from 7.1 with the filter paper to a platinum crucible, dry, ash the paper and ignite at 800 to 900 °C. Cool the crucible, moisten the residue with 3 to 5 drops of sulfuric

acid (4.7), add 5 ml of hydrofluoric acid (4.5), heat the contents of the crucible until fumes of sulfuric acid cease to evolve, and ignite at 800 to 900 °C. Cool the crucible and fuse the residue with 2 g of sodium disulfate (4.11).

Leach the melt while heating with 30 ml of hot water. Add 5 to 10 ml of perchloric acid (4.6), and evaporate the solution until dense white fumes of perchloric acid appear. Remove the residual chromium by distillation as chromyl chloride (see 7.1).

Cool the solution, add 50 ml of hot water and heat to dissolve the salts. Add the solution thus obtained to the main solution.

7.3 Separation of aluminium

Dilute the solution obtained in 7.2 with 200 ml of water. Adjust the pH of the solution to between 3,0 and 4,0 with ammonia solution (4.8) (checking the pH using an indicator test paper) and heat the solution to boiling. Add ammonia solution (4.8) drop by drop to adjust the pH to between 6,5 and 7,5, using an indicator test paper, and boil the solution for 2 min. Add 2 or 3 drops of ammonia solution (4.8) and allow the precipitate to settle. Filter the precipitate on a medium-texture filter paper containing a small amount of paper pulp and wash 5 or 6 times with hot ammonium chloride solution (4.12).

Dissolve the precipitate on the filter paper with 20 ml of hot hydrochloric acid (4.2), collecting the solution in the beaker in which the precipitation took place. Wash the filter 7 or 8 times with hot hydrochloric acid (4.3).

Evaporate the solution to a volume of 50 to 100 ml. Add sodium hydroxide solution (4.9) to adjust the pH to between 5,0 and 6,0, using an indicator test paper, and then add an excess of 30 ml.¹⁾ Heat the solution to boiling and boil for 3 to 5 min.

Cool the solution and filter through a medium-texture filter paper containing a small quantity of paper pulp. Wash the beaker and the precipitate on the filter 7 or 8 times with sodium hydroxide solution (4.10). Collect the filtrate in a 600 ml beaker and reserve it.

Dissolve the precipitate on the filter paper with 20 ml of hot hydrochloric acid (4.2), collecting the solution in the beaker in which the precipitation took place. Wash the filter 7 or 8 times with hot hydrochloric acid (4.3).

Evaporate the solution to a volume of 50 to 100 ml. Add sodium hydroxide solution (4.9) to adjust the pH to between 5,0 and 6,0, using an indicator test paper, and then an excess of 10 ml. Heat the solution to boiling and boil for 3 to 5 min. Cool the solution and filter through a medium-texture filter paper containing a small amount of paper pulp. Wash the beaker and the precipitate on the filter 7 or 8 times with sodium hydroxide solution (4.10).

Add the filtrates thus obtained to the main solution and acidify the solution by addition of hydrochloric acid (4.3).

Evaporate to a volume of 50 to 100 ml and cool the solution. Transfer the solution into a 250 ml one-mark volumetric flask, dilute with water to the mark and mix.

1) It is preferable to use fluoroplastic beakers, etc., while carrying out analysis with alkaline solutions.

7.4 Titration

Take a 200 ml aliquot portion in the case of aluminium contents from 1,5 % (m/m) to 7,5 % (m/m), a 100 ml aliquot portion in the case of aluminium contents from 7,5 % (m/m) to 15 % (m/m), or a 50 ml aliquot portion in the case of aluminium contents from 15 % (m/m) to 20 % (m/m). Add 25 ml of EDTA Na₂ solution (4.17), 3 to 5 drops of phenolphthalein solution (4.13) and, while mixing, add sodium hydroxide solution (4.9) until the colour of the solution turns violet. Add hydrochloric acid (4.2) until the solution becomes colourless, then add 15 ml of buffer solution (4.15) and boil the solution for 3 to 4 min.

Cool the solution. Titrate an excess of the EDTA Na₂ with zinc acetate solution (4.18) in the presence of 10 drops of xylenol orange solution (4.16); the yellow colour changes to crimson-red at the end point.

Adjust the pH to between 5,2 and 5,9 using a pH-meter, add 40 ml of sodium fluoride solution (4.14) and boil again for 2 or 3 min. Cool the solution and titrate with zinc acetate solution (4.18); the yellow colour changes to crimson-red at the end point. Record the volume of zinc acetate solution used.

8 Expression of results

8.1 Calculation

The aluminium (Al) content, expressed as a percentage by mass, is given by the formula

$$\frac{(V_1 - V_2) \times 0,001\,349 \times 100}{m} \times K$$

where

V_1 is the volume, in millilitres, of zinc acetate solution (4.18) used in the titration of EDTA Na₂ solution (4.17) corresponding to the aluminium content (see 7.4);

V_2 is the volume, in millilitres, of the zinc acetate solution used in the titration of the blank test solution;

0,001 349 is the titre of the zinc acetate solution, in grams of aluminium per millilitre of solution;

m is the mass, in grams, of the test portion corresponding to the aliquot portion of the test solution;

K is the conversion factor for the expression of the aluminium content on the dry basis.

8.2 Permissible tolerances on results of parallel determinations

Aluminium content	Permissible tolerance
% (m/m)	% (m/m)
From 1,5 to 3,0	0,08
From 3,0 to 5,0	0,12
From 5,0 to 10,0	0,20
From 10,0 to 20,0	0,30

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