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Aluminium ores — Determination of total iron content — Titanium trichloride reduction method

*Minerais alumineux — Dosage du fer — Méthode de réduction au
trichlorure de titane*



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Foreword

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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.

International Standard ISO 10213 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*, Sub-Committee SC 2, *Methods of analysis*.

Annex A forms an integral part of this International Standard. Annex B is for information only.

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Aluminium ores — Determination of total iron content — Titanium trichloride reduction method

1 Scope

This International Standard specifies a titrimetric method, free from mercury pollution, for the determination of the total iron content in aluminium ores, using potassium dichromate as titrant after reduction of the trivalent iron by tin(II) chloride and titanium(III) chloride. The excess reductant is then oxidized by perchloric acid.

The method is applicable to ores containing between 2 % (m/m) and 50 % (m/m) of iron oxide (Fe_2O_3).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

ISO 6140:1991, *Aluminium ores — Preparation of samples*.

ISO 8558:1985, *Aluminium ores — Preparation of pre-dried test samples*.

ISO 8685:—¹⁾, *Aluminium ores — Sampling procedures*.

1) To be published.

3 Principle

Decomposition of the test portion by sintering with sodium peroxide followed by a brief fusion. Dissolution of the melt in water and hydrochloric acid. Reduction of the major portion of the iron(III) by tin(II) chloride and reduction of the remainder of the iron by titanium(III) chloride. Oxidation of the excess reductant with dilute perchloric acid. Titration of the reduced iron with potassium dichromate solution using sodium diphenylaminesulfonate indicator.

4 Reagents

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

4.1 Sodium peroxide, dry powder.

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

4.2 Hydrochloric acid, ρ_{20} between 1,16 g/ml and 1,19 g/ml.

4.3 Hydrochloric acid, ρ_{20} between 1,16 g/ml and 1,19 g/ml, diluted 1 + 10.

4.4 Perchloric acid, 72 % (m/m) (ρ_{20} = 1,67 g/ml), diluted 1 + 1.

4.5 Sulfuric acid-phosphoric acid mixture.

Pour 150 ml of phosphoric acid (ρ_{20} = 1,70 g/ml) into about 400 ml of water while stirring, add 150 ml of sulfuric acid (ρ_{20} = 1,84 g/ml), cool in a water bath and dilute with water to 1 litre.

4.6 Potassium permanganate, 25 g/l solution.

4.7 Tin(II) chloride, 100 g/l solution.

Dissolve 100 g of crystalline tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 200 ml of hydrochloric acid (4.2) by heating the solution on a water bath. Cool the solution and dilute with water to 1 litre.

This solution should be stored in a brown glass bottle with a small quantity of granular tin metal.

4.8 Titanium(III) chloride, 15 g/l solution.

Dilute 1 volume of titanium(III) chloride solution (about 15 % TiCl_3) with 9 volumes of hydrochloric acid diluted 1 + 1. Alternatively, dissolve 1 g of titanium sponge in about 30 ml of hydrochloric acid (4.2) in a covered beaker, by heating on a water bath. Cool the solution and dilute with water to 250 ml.

Prepare fresh solutions as needed.

4.9 Iron, 0,1 mol/l standard solution.

Transfer 5,585 g of pure iron to a 500 ml Erlenmeyer flask and place a small filter funnel in the neck. Add 75 ml of hydrochloric acid diluted 1 + 1 in small increments and heat until dissolved. Cool and oxidize with 5 ml of a 3 % (m/m) hydrogen peroxide solution added in small portions. Heat to boiling and boil to decompose the excess hydrogen peroxide and to remove chlorine. Transfer to a 1000 ml volumetric flask (5.3) and dilute to the mark with water.

1,00 ml of this standard solution is equivalent to 1,00 ml of the standard volumetric potassium dichromate solution (4.10).

4.10 Potassium dichromate, standard volumetric solution, $c(\text{K}_2\text{Cr}_2\text{O}_7) = 1/60 \text{ mol/l}$.

Pulverize about 6 g of potassium dichromate in an agate mortar, dry at 140 °C to 150 °C for 2 h, and cool to room temperature in a desiccator. Dissolve 4,9035 g of this dried powder in water, cool to 20 °C and transfer to a 1000 ml volumetric flask (5.3). Dilute to the mark with water and mix.

4.11 Sodium diphenylaminesulfonate, indicator solution.

Dissolve 0,2 g of the reagent ($\text{C}_6\text{H}_5\text{NHC}_6\text{H}_4\text{SO}_3\text{Na}$) in water and dilute to 100 ml.

Store the solution in a brown glass bottle.

5 Apparatus

Use ordinary laboratory apparatus and the following.

5.1 Crucible, zirconium metal or vitreous carbon, of capacity 25 ml to 30 ml.

5.2 Burette, class A.

5.3 One-mark volumetric flasks, of capacity 1000 ml.

5.4 Muffle furnace, suitable for operation in the range 500 °C to 800 °C.

6 Sampling and samples

6.1 Sampling

Laboratory samples shall be taken and crushed to pass a 150 μm test sieve in accordance with the procedures specified in ISO 6140 and ISO 8685.

6.2 Preparation of the test sample

Prepare the test sample by drying it in an oven in accordance with the procedure described in ISO 8558. (This is the pre-dried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate, independently, on one test sample (6.2).

NOTE 1 The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure shall be carried out either by the same operator at a different time, or by a different operator, with appropriate recalibration in both cases.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,2 mg, approximately 0,4 g of the test sample (6.2).

NOTE 2 When pre-dried test samples are used, the test portion should be taken on the day of pre-drying and weighed immediately to avoid reabsorption of moisture.

7.3 Blank test

Carry out a blank test using the same quantities of each reagent and the same procedure as in the actual determination, but omit the test portion.

When the analysis is carried out on several samples at the same time, the blank test value may be represented by one test, provided that the procedure is the same and the reagents used are from the same bottles.