International Standard 6609

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Aluminium ores — Determination of iron content — Titrimetric method

Minerais alumineux — Dosage du fer — Méthode titrimétrique

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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.

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.

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Aluminium ores — Determination of iron content — Titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of iron in aluminium ores. The method has been chosen to avoid interference from platinum which may occur with the use of platinum crucibles in the acid decomposition procedure.

The method is suitable for use with ores having iron oxide contents between 2 and 50 % (*m/m*).

2 Reference

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

3 Principle

Decomposition of the test portion by sintering with sodium peroxide followed by a brief fusion. Dissolution of the fusion mixture with water and hydrochloric acid. Determination of iron by titration with potassium dichromate after reduction with tin(II) chloride, and oxidation of excess reductant with mercury(II) chloride.

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.

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

4.2 Hydrochloric acid, ϱ_{20} = 1,17 g/ml.

4.3 Sulfuric acid, ϱ_{20} = 1,84 g/ml, diluted 1 + 19.

4.4 Sulfuric acid-phosphoric acid mixture

Cautiously pour 150 ml of sulfuric acid (ϱ_{20} = 1,84 g/ml) into about 300 ml of water while stirring. Cool in a water bath, add 150 ml of phosphoric acid (ϱ_{20} = 1,70 g/ml) and dilute to 1 litre.

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

Place 20 ml of the hydrochloric acid (4.2) into a 100 ml beaker and add 10 g of crystalline tin(II) chloride ($SnCl_2 \cdot 2H_2O$). Heat gently while stirring until the solution is clear. Cool the solution rapidly and dilute to 100 ml.

Store this solution in a brown glass bottle with the addition of a small quantity of granular tin metal.

4.6 Mercury(II) chloride, 40 g/l solution.

Dissolve 40 g mercury(II) chloride in water and dilute to 1 litre.

4.7 Ammonium iron(III) sulfate, standard volumetric solution.

4.7.1 Preparation of the solution

Dissolve 47,5 g of ammonium iron(III) sulfate — 24 water [$Fe_2(NH_4)_2(SO_4)_4 \cdot 24H_2O$] in the sulfuric acid solution (4.3). Transfer to a 1 000 ml one-mark volumetric flask and dilute to the mark with the same acid.

4.7.2 Standardization of the solution

Pipette 10 ml of the ammonium iron(III) sulfate solution (4.7.1) into a 400 ml beaker and dilute to about 80 ml. Add 10 ml of the hydrochloric acid (4.2). Boil gently and add the tin(II) chloride solution (4.5), drop by drop, whilst stirring the solution, until the yellow colour disappears. Add 1 to 3 drops of excess. Cool rapidly in a water bath. Add, all at once, 10 ml of the mercury(II) chloride solution (4.6), mix gently and allow to stand for 5 min.

Dilute to approximately 150 ml. Add 30 ml of the sulfuric acid-phosphoric acid mixture (4.4) and 5 drops of the indicator (4.9), then titrate with the standard potassium dichromate solution (4.8) to a permanent violet end-point (30 s). Record the volume of potassium dichromate solution used (V_1).

NOTE — In the absence of iron the diphenylaminesulphonate indicator does not react with the dichromate solution. The addition of ammonium iron(III) sulfate solution is therefore necessary to promote indicator response in the blank solution, and thus allow a suitable correction for the blank.

4.8 Potassium dichromate, standard solution, $c(K_2Cr_2O_7) = 0{,}016\,67$ mol/l.

Dry about 6 g of finely powdered potassium dichromate in an air oven at 140 to 150 °C for 30 to 60 min. Cool in a closed vessel. Weigh 4,904 g, dissolve in water, and dilute to the mark in a 1 000 ml one-mark volumetric flask.

1 ml of 0,016 67 mol/l $K_2Cr_2O_7 \mathrel{\widehat{=}} 0{,}005\,585$ g of Fe

1 ml of 0,016 67 mol/l $K_2Cr_2O_7 \mathrel{\widehat{=}} 0{,}007\,985$ g of Fe_2O_3

4.9 Barium diphenylaminesulfonate solution.

Dissolve 0,2 g of barium diphenylaminesulfonate $[(C_6H_5NHC_6H_4{\cdot}SO_3)_2Ba]$ in a small volume of hot water and dilute to 100 ml. Store in a cool, dark place. Discard when discoloured.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Microburette, 10 ml, class A.

5.2 Zirconium crucibles, 40 to 50 ml capacity.

5.3 Balance, capable of being read to 0,000 1 g.

5.4 Desiccator, containing phosphorus(V) oxide as desiccant.

5.5 Burette, 50 ml, class A.

5.6 Muffle furnace, capable of being maintained at 480 to 500 °C.

6 Sampling and preparation of samples

Laboratory samples shall be taken and ground to pass a 150 µm test sieve, in accordance with the methods specified in the relevant standards.[1)]

7 Procedure

7.1 Number of determinations

Carry out the analysis in duplicate and independently on each ore sample.

NOTE — The expression "independently" implies a change of the person carrying out the analysis. If the same person has to carry out the analysis, the procedure shall be carried out at different times.

7.2 Test portion

Weigh, to the nearest 0,000 1 g, approximately 0,40 g of test sample into a zirconium crucible and record the mass (m_1).

7.3 Blank test

In parallel with the decomposition of the test portion, prepare a blank test solution according to the method of decomposition, but omitting the test portion. When the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents used are from the same bottles.

7.4 Check test

In each run, one analysis of a certified reference material of the same type of ore shall be carried out in parallel and under the same conditions with the analysis of one ore sample.

NOTE — The certified reference material should be of the same type as the sample to be analysed. Such certified reference materials cannot, however, be considered as being of the same type if the properties of the sample to be analysed differ from those of the certified reference material to such an extent that the analytical procedure must be changed substantially.

7.5 Determination

7.5.1 Decomposition of the test portion

To the test portion taken in (7.2), add 5 ± 0,1 g of the sodium peroxide (4.1). Mix using a nickel spatula. Place the crucible and contents in the muffle furnace (5.6), maintained at 480 to 500 °C for 45 min. Remove the crucible and contents from the furnace and heat over a burner to melt the sinter (about 30 s). Continue heating the melt while swirling for a total time of 2 min.

Allow the crucible to cool to room temperature (a metal block can be used to expedite this process), and place the crucible on its side in a 400 ml beaker. Carefully add 50 ml of water and cover the beaker immediately with a watch glass.

When reaction ceases add 30 ml of the hydrochloric acid (4.2) and cover with a watch glass until the reaction subsides. Rinse down the cover and wall of the beaker with water.

7.5.2 Determination of iron oxide

Remove the crucible and rinse well with water, keeping the total volume in the beaker to less than 100 ml. Ensure that all iron hydroxides have dissolved. Heat if necessary. Add 1 ml of the ammonium iron(III) sulfate solution (4.7) to the solution from the blank test.

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1) Where no International Standards exist, the relevant standards shall be the national standards. Two International Standards on this subject are currently in preparation: ISO 6137, *Aluminium ores — Method of sampling*, and ISO 6140, *Aluminium ores — Preparation of samples.*

Heat to about 80 °C and add the tin(II) chloride solution (4.5), drop by drop, while stirring the solution until the yellow colour disappears or ceases to fade. Then add 1 to 3 drops in excess. Cool rapidly in a water bath.

NOTE — High titaniferrous bauxites may give rise to a permanent yellow colouration.

Add, all at once, 10 ml of the mercury(II) chloride solution (4.6), mix gently and allow to stand for 5 min. Dilute to approximately 250 ml. Add 30 ml of the sulfuric acid-phosphoric acid mixture (4.4) and 5 drops of the indicator (4.9) and titrate with the potassium dichromate solution (4.8) to a permanent violet end-point (30 s). Record the volumes for the test solution (V_2) and for the blank test solution (V_3).

NOTE — For samples having an Fe_2O_3 content greater than 20 % (m/m), use a 50 ml burette.

8 Expression of results

8.1 Calculation of total iron (as iron oxide Fe_2O_3)

The total iron content (as iron oxide, Fe_2O_3), expressed as a percentage by mass, is given by the formula

$$\frac{V_2 - (V_3 - 0{,}1V_1)}{m} \times 0{,}007\,985 \times 100$$

where

V_1 is the volume, in millilitres, of standard potassium dichromate solution (4.8) used to titrate 10 ml of the ammonium iron(III) sulfate solution (4.7);

V_2 is the volume, in millilitres, of standard potassium dichromate solution (4.8) used for titration of the analytical sample;

V_3 is the volume, in millilitres, of standard potassium dichromate solution (4.8) used for titration in the blank test;

m is the mass, in grams, of the test portion.

8.2 General treatment of results

8.2.1 Precision

The precision of this method, as determined in an International test programme, is expressed in terms of repeatability, reproducibility and reproducibility index in the table.

Table — Precision data for iron determination

Sample	Mean Fe_2O_3 content	Components of standard deviation		Reproducibility index
	% (m/m)	r	R	$2s$
MT/12/3	5,63	0,06	0,08	0,20
MT/12/1	17,41	0,14	0,14	0,39
MT/12/5	19,50	0,09	0,12	0,30
MT/12/2	43,30	0,13	0,12	0,35

where

r is the repeatability;

R is the reproducibility.

8.2.2 Acceptance of analytical values (see ISO 5725)

The analytical value for the test sample shall be accepted when the analytical value obtained for the corresponding certified reference material agrees with the certified value within the limit of the reproducibility index contained in the table, and when the difference between the two values for the test sample does not exceed 2,77 r, as calculated from the appropriate value of r given in the table.

When the analytical value obtained for the certified reference material is outside the reproducibility index, an analysis shall be carried out simultaneously on one test sample and one certified reference material, with one blank test. The analytical value obtained for the certified reference material shall be examined for the acceptance of the value for the test sample, as above. If the value obtained for the certified reference material is again outside the limits, the procedure shall be repeated with a different reference material of the same type of ore until two acceptable values for the test sample are obtained.

8.2.3 Calculation of final result

The final result is the arithmetical mean of the acceptable analytical values calculated to four decimal places and rounded off to the second decimal place as follows:

a) when the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place is kept unchanged;

b) when the figure in the third decimal place is 5 and there are figures other than 0 in the fourth decimal place, or when the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;

c) when the figure in the third decimal place is 5 and there are no figures other than 0 in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8, and increased by one if it is 1, 3, 5, 7 or 9.

9 Test report

The test report shall include the following information:

a) details necessary for the identification of the sample;

b) reference to this International Standard;

c) results of the analysis;

d) reference number of the results;

e) any characteristics noticed during the determination and any operations not specified in this International Standard which may have had influence on the results.

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