
International Standard



6995

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Aluminium ores — Determination of titanium content — 4,4'-Diantipyrylmethane spectrophotometric method

Minerais alumineux — Dosage du titane — Méthode spectrophotométrique au diantipyrylméthane-4,4'

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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 6995 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*.

Aluminium ores — Determination of titanium content — 4,4'-Diantipyrilmethane spectrophotometric method

1 Scope and field of application

This International Standard specifies a 4,4'-diantipyrilmethane spectrophotometric method for the determination of the titanium content of aluminium ores.

The method is applicable to ores containing between 0,5 and 8 % (*m/m*) of titanium dioxide.

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:

- a) Treatment with a mixture of hydrochloric acid, nitric acid and sulfuric acid.

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 a brief fusion. Dissolution of the melt with sulfuric acid.

NOTE — This method is recommended for ores containing diaspor and when the residue after silica volatilization for acid decomposition is greater than 1 % of the mass of the test portion;

or

- c) Fusion with sodium tetraborate/sodium carbonate flux followed by treatment with sulfuric acid.

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

Dehydration of silica, dissolution of salts, and filtration and ignition of the residue. Removal of the silica by evaporation with hydrofluoric and sulfuric acids. Fusion of the residue with sodium carbonate/sodium tetraborate flux, dissolution of the melt with sulfuric acid and addition to the main solution.

Reduction of the iron with ascorbic acid; colour development with 4,4'-diantipyrilmethane and measurement of the absorbance at about 390 nm.

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.

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 3 parts by mass of anhydrous sodium carbonate and 1 part by mass of anhydrous sodium tetraborate.

4.3 Hydrofluoric acid, 40 % (*m/m*), ρ_{20} 1,13 g/ml.

4.4 Sulfuric acid, ρ_{20} 1,84 g/ml, diluted 1 + 1.

4.5 Sulfuric acid, ρ_{20} 1,84 g/ml, diluted 1 + 9.

4.6 Hydrochloric acid, ρ_{20} 1,17 g/ml, diluted 1 + 1.

4.7 Acid mixture.

Measure into a one-litre beaker 225 ml of water and add cautiously, with mixing, 175 ml of sulfuric acid (ρ_{20} 1,84 g/ml). Cool to room temperature and add 150 ml of hydrochloric acid (ρ_{20} 1,17 g/ml) and 50 ml of nitric acid (ρ_{20} 1,42 g/ml).

Prepare this solution fresh for each series of determinations and discard any unused solution.

4.8 Ascorbic acid, 100 g/l solution.

Prepare this solution as required.

4.9 4,4'-Diantipyrilmethane, 15 g/l solution.

Dissolve 15 g of 4,4'-diantipyrilmethane ($C_{23}H_{24}N_4O_2$) in 1 l of sulfuric acid solution (ρ_{20} 1,84 g/ml, diluted 1 + 49).

Prepare fresh as required.

4.10 Ammonium iron(III) sulfate solution.

Dissolve 6 g of ammonium iron(III) sulfate — 24 water [$Fe_2(NH_4)_2(SO_4)_4 \cdot 24H_2O$] in 1 litre of water containing 10 ml of the sulfuric acid solution (4.5).