

International Standard



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Iron ores — Determination of aluminium content — Oxine gravimetric and titrimetric methods

Minerais de fer — Dosage de l'aluminium — Méthodes titrimétrique et gravimétrique à l'oxine

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

International Standard ISO 2771 was developed by Technical Committee ISO/TC 102, *Iron ores*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 2771-1973), which had been approved by the member bodies of the following countries :

Australia	Ireland	Spain
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France	Portugal	USA
India	South Africa, Rep. of	USSR

The member body of the following country had expressed disapproval of the document on technical grounds :

Netherlands

Iron ores — Determination of aluminium content — Oxine gravimetric and titrimetric methods

Second edition

1 Scope and field of application

This International Standard specifies gravimetric and titrimetric methods for the determination of the aluminium content in iron ores using 8-hydroxyquinoline.

These methods are applicable to natural iron ores and iron ore concentrates, and agglomerates including sinter products.

2 References

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples*.

ISO 3081, *Iron ores — Increment sampling — Manual method*.

ISO 3083, *Iron ores — Preparation of samples*.

3 Principle

Treatment of the sample with hydrochloric, nitric, and perchloric acids and filtration of the solution. Extraction of the filtrate with methyl isobutyl ketone to remove the bulk of the iron and retention of the aqueous solution containing most of the aluminium, which forms the principal solution.

Treatment of the insoluble residue with hydrofluoric and sulphuric acids to expel silicon tetrafluoride, then fusion with sodium pyrosulphate. Dissolution of the melt in hydrochloric acid and combination with the main solution. Treatment of this solution with ammonia in slight excess to precipitate aluminium, titanium, and residual iron. After filtration, dissolution of the precipitate in hydrochloric acid and re-precipitation with ammonia. After filtration, dissolution of the precipitate in hydrochloric acid.

Removal of titanium, vanadium and residual iron by precipitation with cupferron from an acid medium, followed by filtration. Evaporation of the filtrate with nitric and perchloric acids to decompose the excess cupferron and dilution of the solution with water and hydrochloric acid. Addition of tartaric acid to the hydrochloric solution, adjustment of the pH to 5,5 and precipitation of the aluminium as the oxinate by the addition of an 8-hydroxyquinoline solution. Collection of the aluminium ox-

inate by filtration and completion of the final determination of the aluminium by one of the three following procedures :

1) Aluminium content up to 2,5 % (m/m) particularly when less than 0,25 % (m/m) : Titrimetrically by solution of the oxinate precipitate in hydrochloric acid and the addition of a slight excess of a standard solution of potassium bromate. Addition of potassium iodide and titration of the iodine liberated by the reaction with the excess of bromate with a standard volumetric solution of sodium thiosulphate.

NOTE — The titrimetric method is to be preferred for samples containing less than 0,25 % of aluminium.

For samples containing more than 2,5 % of aluminium, it is extremely difficult to remove the excess of reagent by washing, and one of the two gravimetric procedures should be used.

Elimination of the excess reagent may be aided by heating the precipitate to 135 °C before dissolving it in hydrochloric acid.

2) Aluminium content up to 5 % (m/m) : Gravimetrically by drying the oxinate precipitate and weighing.

NOTE — Weighing of the oxinate is quite satisfactory for aluminium contents ranging from 0,25 to 5 %. For greater amounts of aluminium the precipitate becomes so voluminous that the expulsion of the excess reagent by drying at 135 °C may become uncertain.

3) Aluminium content greater than 2,5 % (m/m) and always if aluminium is greater than 5 % (m/m) : Gravimetrically as Al_2O_3 , after ignition of the oxinate precipitate.

NOTE — Al_2O_3 as the final weighing form offers several advantages : larger quantities of aluminium oxide can be handled and the ignition of the oxinate eliminates the possibility of high results due to retained reagent.

4 Reagents

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

4.1 Hydrochloric acid, ρ_{20} 1,19 g/ml.

4.2 Hydrochloric acid, diluted 1 + 10.