

Iron ores — Determination of total iron content

Part 2: Titrimetric methods after titanium(III) chloride reduction

ICS 73.060.10

National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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*Partie 2: Méthodes titrimétriques après réduction au chlorure de
titane(III)*



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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2597-2 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

ISO 2597 consists of the following parts, under the general title *Iron ores — Determination of total iron content*:

- *Part 1: Titrimetric method after tin(II) chloride reduction*
- *Part 2: Titrimetric methods after titanium(III) chloride reduction*

Iron ores — Determination of total iron content —

Part 2:

Titrimetric methods after titanium(III) chloride reduction

1 Scope

This part of ISO 2597 specifies two titrimetric methods, free from mercury pollution, for the determination of total iron content in iron ores, using potassium dichromate as titrant after reduction of the iron(III) by tin(II) chloride and titanium(III) chloride. The excess reductant is then oxidized by either dilute potassium dichromate (method 1) or perchloric acid (method 2).

Both methods are applicable to a concentration range of 30 % mass fraction to 72 % mass fraction of iron in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

WARNING — This part of ISO 2597 may involve hazardous materials, operations and equipment. This part of ISO 2597 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 2597 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 31-0:1992, *Quantities and units — Part 0: General principles*

ISO Guide 35, *Reference materials — General and statistical principles for certification*

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

3 Principle

3.1 Decomposition of the test portion

3.1.1 Acid decomposition

For samples containing not more than 0,05 % mass fraction of vanadium, the test portion is treated with hydrochloric acid in the presence of tin chloride and the residue is filtered, ignited and treated with hydrofluoric and sulfuric acids. The mixture is fused with potassium disulfate and the cold melt is dissolved in water and hydrochloric acid then neutralized with ammonia solution. The precipitate is filtered, washed in water, dissolved in hydrochloric acid and combined with the main iron solution, which is treated with potassium permanganate and evaporated.

3.1.2 Fusion-filtration

For samples containing more than 0,05 % mass fraction of vanadium, the test portion is fused with a mixture of fluxes, the cold melt is leached with water and the precipitate is filtered, washed in sodium hydroxide solution, dissolved in hydrochloric acid and evaporated.

3.2 Titration of iron

The major portion of the iron(III) is reduced by tin(II) chloride and the remainder of the iron(III) is reduced by titanium(III) chloride. The excess reductant is oxidized with either dilute potassium dichromate solution (method 1) or dilute perchloric acid (method 2). The reduced iron is titrated with potassium dichromate solution using the 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.

	Method
4.1 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml.	1 and 2
4.2 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.	1 and 2
4.3 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 12.	1 and 2
4.4 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 100.	1 and 2
4.5 Hydrofluoric acid, 40 % mass fraction (ρ 1,13 g/ml) or 48 % mass fraction (ρ 1,19 g/ml).	1 and 2
4.6 Sulfuric acid, ρ 1,84 g/ml.	1 and 2
4.7 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1, carefully pour 1 volume of reagent 4.6 into 1 volume of cold water.	1 and 2
4.8 Orthophosphoric acid, ρ 1,7 g/ml.	1 and 2
4.9 Perchloric acid, 72 % mass fraction (ρ 1,7 g/ml), diluted 1 + 1.	2