Nederlandse norm

NEN-ISO 2597-2

(en)

IJzererts - Bepaling van het ijzergehalte - Deel 2: Titrimetrische methode na titanium (III) chloride reductie (ISO 2597-2:2015,IDT)

Iron ores - Determination of total iron content - Part 2: Titrimetric methods after titanium(III) chloride reduction (ISO 2597-2:2015,IDT)

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- ISO 2597-2:2015,IDT

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INTERNATIONAL STANDARD

ISO 2597-2

Second edition
2015-11-01

Iron ores — Determination of total iron content —

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

Minerais de fer — Dosage du fer total —

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 2597-2:2008), which has been technically revised.

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*

INTERNATIONAL STANDARD **ISO 2597-2:2015(E)**

Iron ores — Determination of total iron content —

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

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.

1 Scope

This part of ISO 2597 specifies a titrimetric method, 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.

This method is 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.

2 Normative references

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

ISO 80000-1:2009, *Quantities and unit — Part 1: General*

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.

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 more 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. 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.

4.1 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml. (Methods 1 and 2).

4.2 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1. (Methods 1 and 2).

4.3 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 12. (Methods 1 and 2).

4.4 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 100. (Methods 1 and 2).

4.5 Hydrofluoric acid, 40 % mass fraction (ρ 1,13 g/ml) or 48 % mass fraction (ρ 1,19 g/ml). (Methods 1 and 2).

4.6 Sulfuric acid, ρ 1,84 g/ml. (Methods 1 and 2).

4.7 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1, carefully pour 1 volume of reagent 4.6 into one volume of cold water. (Methods 1 and 2).

4.8 Orthophosphoric acid, ρ 1,7 g/ml. (Methods 1 and 2).

4.9 Perchloric acid, 72 % mass fraction (ρ 1,7 g/ml), diluted 1 + 1. (Method 2).

4.10 Sulfuric acid–orthophosphoric acid mixture, pour 150 ml of orthophosphoric acid (4.8) into about 400 ml of water while stirring, add 150 ml of sulfuric acid (4.6), cool in a water bath, dilute with water to 1 l and mix well. (Methods 1 and 2).

4.11 Sodium hydroxide (NaOH), solution, 20 g/l. (Methods 1 and 2).

4.12 Hydrogen peroxide (H_2O_2), 30 % by volume solution. (Methods 1 and 2).

4.13 Hydrogen peroxide (H_2O_2), 30 % by volume solution, diluted 1 + 9. (Method 1).

4.14 Tin(II) chloride solution, 100 g/l, dissolve 100 g of crystalline tin(II) chloride ($SnCl_2 \cdot 2H_2O$) in 200 ml of hydrochloric acid (4.1) by heating the solution in a water bath. Cool the solution and dilute with water to 1 l. This solution should be stored in a brown glass bottle with a small quantity of granular tin metal. (Methods 1 and 2).

4.15 Potassium permanganate ($KMnO_4$) solution, 25 g/l. (Methods 1 and 2).

4.16 Potassium dichromate ($K_2Cr_2O_7$) solution, 1 g/l. (Method 1).

4.17 Titanium(III) chloride ($TiCl_3$) solution, 20 g/l, dilute one volume of titanium(III) chloride solution (about 20 % $TiCl_3$) with nine volumes of hydrochloric acid (4.2). (Methods 1 and 2).

Alternatively, dissolve 1,3 g of titanium sponge in about 40 ml of hydrochloric acid (4.1) in a covered beaker by heating in a water bath. Cool the solution and dilute with water to 200 ml. Prepare fresh solution as needed.

4.18 Flux mixture, mix one portion of anhydrous sodium carbonate (Na_2CO_3) and two portions of sodium peroxide (Na_2O_2). (Methods 1 and 2).

4.19 Iron standard solution, 0,1 mol/l, transfer 5,58 g of iron(III) oxide (purity greater than 99,9 % mass fraction) to a 500 ml beaker flask and place a small filter funnel in the neck. Add 75 ml of hydrochloric acid (4.2) in small increments and heat until dissolved. (Methods 1 and 2).

Cool and oxidize with 5 ml of hydrogen peroxide (4.13) added in small portions. Heat to boiling and boil to decompose the excess hydrogen peroxide and to expel chlorine. Cool, transfer to a 1 000 ml volumetric flask and mix well.

1,00 ml of this solution is equivalent to 1,00 ml of the standard potassium dichromate solution (4.20).

4.20 Potassium dichromate (99,9 % minimum purity), standard solution, 0,016 67 mol/l, pulverize about 6 g of potassium dichromate reagent in an agate mortar, dry at 140 °C to 150 °C for 2 h, and cool to room temperature in a desiccator. (Methods 1 and 2).

Transfer 4,903 g of this material to a 300 ml beaker, dissolve in about 100 ml of water, transfer quantitatively to a 1 000 ml volumetric flask, make up to volume with water after cooling to 20 °C and mix well. Record the temperature at which this dilution was made (20 °C) on the stock bottle. Measure the temperature at each use to correct the volume of titrant used.

The volumetric flask should previously be calibrated by weighing the mass of water contained at 20 °C and converting to volume.

Water used for preparation should previously be equilibrated at room temperature.

A calibrated mercury thermometer, graduated in 0,1 °C divisions and having a marked dipping line, should be used. Take a sufficient volume of standard solution for dipping the thermometer and transfer to a suitable beaker. Measure the temperature of the solution to the nearest 0,1 °C, after dipping for more than 60 s.

4.21 Indigo carmine [5,5'-disulfonic acid disodium salt ($C_6H_8O_8N_2S_2Na_2$)] solution, 0,1 g/100 ml, dissolve 0,1 g of indigo carmine in a cold mixture of 50 ml sulfuric acid (4.7) and 50 ml of water. (Method 1).

4.22 Sodium diphenylaminesulfonate indicator solution, 0,2 g/100 ml, dissolve 0,2 g of sodium diphenylaminesulfonate ($C_6H_5NHC_6H_4SO_3Na$) in a small volume of water and dilute to 100 ml. (Method 1 and Method 2).

Store the solution in a brown glass bottle.

5 Apparatus

The pipette and volumetric flask specified are complying with ISO 648 and ISO 1042 respectively.

Ordinary laboratory apparatus, and the following.

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