

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)

ICS 73.060.10
november 2015

Als Nederlandse norm is aanvaard:

- ISO 2597-2:2015, IDT

Normcommissie 342093 "Chemische analyse van metalen"



THIS PUBLICATION IS COPYRIGHT/PROTECTED

DEZE PUBLICATIE IS AUTEURSRECHTELIJK BESCHERMD

Apart from exceptions provided by the law, nothing from this publication may be duplicated and/or published by means of photocopy, microfilm, storage in computer files or otherwise, which also applies to full or partial processing, without the written consent of the Netherlands Standardization Institute.

The Netherlands Standardization Institute shall, with the exclusion of any other beneficiary, collect payments owed by third parties for duplication and/or act in and out of law, where this authority is not transferred or falls by right to the Reproduction Rights Foundation.

Auteursrecht voorbehouden. Behoudens uitzondering door de wet gesteld mag zonder schriftelijke toestemming van het Nederlands Normalisatie-instituut niets uit deze uitgave worden verveelvoudigd en/of openbaar gemaakt door middel van fotokopie, microfilm, opslag in computerbestanden of anderszins, hetgeen ook van toepassing is op gehele of gedeeltelijke bewerking.

Het Nederlands Normalisatie-instituut is met uitsluiting van ieder ander gerechtigd de door derden verschuldigde vergoedingen voor verveelvoudiging te innen en/of daartoe in en buiten rechte op te treden, voor zover deze bevoegdheid niet is overgedragen c.q. rechtens toekomt aan de Stichting Reprorecht.

Although the utmost care has been taken with this publication, errors and omissions cannot be entirely excluded. The Netherlands Standardization Institute and/or the members of the committees therefore accept no liability, not even for direct or indirect damage, occurring due to or in relation with the application of publications issued by the Netherlands Standardization Institute.

Hoewel bij deze uitgave de uiterste zorg is nagestreefd, kunnen fouten en onvolledigheden niet geheel worden uitgesloten. Het Nederlands Normalisatie-instituut en/of de leden van de commissies aanvaarden derhalve geen enkele aansprakelijkheid, ook niet voor directe of indirecte schade, ontstaan door of verband houdend met toepassing van door het Nederlands Normalisatie-instituut gepubliceerde uitgaven.

**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)*

Preview



Reference number
ISO 2597-2:2015(E)

© ISO 2015

Copyright
Preview



COPYRIGHT PROTECTED DOCUMENT

© ISO 2015, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Principle	1
3.1 Decomposition of the test portion.....	1
3.1.1 Acid decomposition.....	1
3.1.2 Fusion-filtration.....	2
3.2 Titration of iron.....	2
4 Reagents	2
5 Apparatus	3
6 Sampling and samples	4
6.1 Laboratory sample.....	4
6.2 Preparation of test samples.....	4
6.2.1 General.....	4
6.2.2 Ores having significant contents of combined water or oxidizable compounds.....	4
6.2.3 Ores outside the scope of 6.2.2	4
7 Procedure	5
7.1 Number of determinations.....	5
7.2 Blank test and check test.....	5
7.3 Determination of hygroscopic moisture content.....	5
7.4 Test portion.....	5
7.5 Determination.....	5
7.5.1 Decomposition of the test portion.....	5
7.5.2 Reduction.....	7
8 Expression of results	8
8.1 Calculation of total iron content.....	8
8.2 General treatment of results.....	8
8.2.1 Repeatability and permissible tolerance.....	8
8.2.2 Determination of analytical result.....	9
8.2.3 Between-laboratories precision.....	9
8.2.4 Check for trueness.....	9
8.2.5 Calculation of final result.....	10
8.3 Oxide factors.....	10
9 Test report	10
Annex A (normative) Flowsheet of the procedure for the acceptance of analytical values for test samples	12
Annex B (normative) Procedure of Japanese weighing method	13
Bibliography	14

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*

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.

ISO 2597-2:2015(E)

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 (H₂O₂)**, 30 % by volume solution. (Methods 1 and 2).
- 4.13 **Hydrogen peroxide (H₂O₂)**, 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 (SnCl₂·2H₂O) 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 (KMnO₄) solution, 25 g/l. (Methods 1 and 2).

4.16 Potassium dichromate (K₂Cr₂O₇) solution, 1 g/l. (Method 1).

4.17 Titanium(III) chloride (TiCl₃) solution, 20 g/l, dilute one volume of titanium(III) chloride solution (about 20 % TiCl₃) 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 (Na₂CO₃) and two portions of sodium peroxide (Na₂O₂). (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 (C₁₆H₈O₈N₂S₂Na₂)] 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 (C₆H₅NHC₆H₄SO₃Na) 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.

Bestelformulier

NEN

Stuur naar:

NEN Standards Products & Services
t.a.v. afdeling Klantenservice
Antwoordnummer 10214
2600 WB Delft

NEN Standards Products & Services

Postbus 5059
2600 GB Delft

Vlinderweg 6
2623 AX Delft

T (015) 2 690 390
F (015) 2 690 271

www.nen.nl/normshop

Ja, ik bestel

__ ex. NEN-ISO 2597-2:2015 en IJzererts - Bepaling van het ijzergehalte - € 79.70
Deel 2: Titrimetrische methode na titanium (III) chloride reductie

Wilt u deze norm in PDF-formaat? Deze bestelt u eenvoudig via www.nen.nl/normshop

Gratis e-mailnieuwsbrieven

Wilt u op de hoogte blijven van de laatste ontwikkelingen op het gebied van normen, normalisatie en regelgeving? Neem dan een gratis abonnement op een van onze e-mailnieuwsbrieven. www.nen.nl/nieuwsbrieven

Retourneren

Fax: (015) 2 690 271
E-mail: klantenservice@nen.nl
Post: NEN Standards Products & Services,
t.a.v. afdeling Klantenservice
Antwoordnummer 10214,
2600 WB Delft
(geen postzegel nodig).

Gegevens

Bedrijf / Instelling _____

T.a.v. _____ O M O V

E-mail _____

Klantnummer NEN _____

Uw ordernummer _____ BTW nummer _____

Postbus / Adres _____

Postcode _____ Plaats _____

Telefoon _____ Fax _____

Factuuradres (indien dit afwijkt van bovenstaand adres)

Postbus / Adres _____

Postcode _____ Plaats _____

Datum _____ Handtekening _____

Voorwaarden

- De prijzen zijn geldig tot 31 december 2016, tenzij anders aangegeven.
- Alle prijzen zijn excl. btw, verzend- en handelingskosten en onder voorbehoud bij o.m. ISO- en IEC-normen.
- Bestelt u via de normshop een pdf, dan betaalt u geen handeling en verzendkosten.
- Meer informatie: telefoon (015) 2 690 391, dagelijks van 8.30 tot 17.00 uur.
- Wijzigingen en typfouten in teksten en prijsinformatie voorbehouden.
- U kunt onze algemene voorwaarden terugvinden op: www.nen.nl/leveringsvoorwaarden.