BS ISO 21826-1:2022

BSI Standards Publication

Iron ores — Determination of total iron content using the EDTA photometric titration method

Part 1: Microwave digestion method

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Iron ores — Determination of total iron content using the EDTA photometric titration method —

Part 1: Microwave digestion method

Minerais de fer — Dosage du fer total par la méthode titrimétrique photométrique à l'EDTA —

Partie 1: Méthode de digestion par micro-ondes

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Contents

Page

Foreword ... **iv**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **1**

4 Principle ... **2**

5 Reagents ... **2**

6 Apparatus ... **5**

7 Sampling and samples ... **6**
- 7.1 Laboratory sample ... 6
- 7.2 Preparation of test samples ... 6
  - 7.2.1 General ... 6
  - 7.2.2 Ores having significant contents of combined water or oxidizable compounds ... 6
  - 7.2.3 Ores outside the scope of 7.2.2 ... 6

8 Procedures ... **6**
- 8.1 Number of determinations ... 6
- 8.2 Test portion ... 6
- 8.3 Blank test ... 7
- 8.4 Determination ... 7
  - 8.4.1 Decomposition of the test portion ... 7
  - 8.4.2 Titration ... 7
- 8.5 Procedure of blank test ... 7

9 Expression of results ... **8**
- 9.1 Calculation of total iron content ... 8
- 9.2 General treatment of results ... 8
  - 9.2.1 Repeatability and permissible tolerance ... 8
  - 9.2.2 Methods for checking the acceptability of the test results and determining the final quoted result ... 9
  - 9.2.3 Between-laboratories precision ... 9
  - 9.2.4 Check for trueness ... 9
  - 9.2.5 Calculation of final result ... 10
- 9.3 Oxide factors ... 10

10 Test report ... **11**

Annex A (informative) **Flowsheet of the procedure for the acceptance of analytical values for test samples** ... **12**

Annex B (informative) **Typical titration curves** ... **13**

Annex C (informative) **Titration parameters for reference** ... **15**

Bibliography ... **26**

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Full document details: BS ISO 21826-1:2022

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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This first edition of ISO 21826-1 cancels and replaces ISO/TS 21826:2020, which has been technically revised.

The main changes are as follows:

— “This method does not apply as a referee method” has been added to the Scope.

A list of all parts in the ISO 21826 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Iron ores — Determination of total iron content using the EDTA photometric titration method —

Part 1:
Microwave digestion method

WARNING — This document can involve hazardous materials, operations and equipment. It does not address all of the potential safety concerns associated with its use. It is the responsibility of the user of this method to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies a photometric titration method using EDTA for the determination of the total iron content of iron ores.

This method is applicable to a concentration range of a mass fraction of 37,00 % to 72,00 % of total iron in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

This method does not apply as a referee method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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*

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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4 Principle

The samples are digested in closed vessels in a microwave oven in a mixture of nitric acid, hydrochloric acid and hydrofluoric acid. The resulting solution is diluted with water and adjusted to pH 1 with sodium hydroxide. Sulphuric acid is added to the solution to eliminate titanium hydroxide precipitation. Boric acid is added to the solution to reduce the interference of fluoride. Iron is determined by automatic photometric titration with EDTA.

5 Reagents

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

5.1 Iron powder (purity > 99,98 %).

5.2 Hydrochloric acid, ρ 1,16 g/ml.

5.3 Nitric acid, ρ 1,42 g/ml.

5.4 Hydrofluoric acid solution, 1+9.

5.5 Sulphuric acid solution, 1+1.

5.6 Boric acid solution, 10 %.

Weigh 100 g of boric acid into a 1 000 ml beaker. Add 25 g of sodium hydroxide and dilute to 1 000 ml with water. The solution should be made at the time of use.

5.7 Sodium hydroxide solution, 200 g/l.

5.8 Iron standard solution, 0,100 0 mol/l.

Weigh 0,558 5 g of iron powder (5.1) into a 250 ml beaker. Add 5 ml of hydrochloric acid (5.2) and 5 ml of nitric acid (5.3). Cover the beaker with a watch glass and heat at 80 °C (hotplate) until completely dissolved. Cool and transfer to a 100 ml volumetric flask and dilute to the volume mark with water.

5.9 Disodium dihydrogen ethylenedinitrilotetraacetate dihydrate (EDTA) solution, $C(C_{10}H_{14}N_2O_8Na_2{\cdot}2H_2O)$ = 0,1 mol/l.

5.9.1 Preparation of the EDTA solution

Dissolve 40 g of disodium EDTA dihydrate in water and dilute to 1 l with water. Store this solution in a polyethylene bottle.

5.9.2 Standardization of the EDTA solution

5.9.2.1 Weigh, to the nearest 0,000 2 g, approximately 0,20 g (m_1) of iron powder (5.1) to a 250 ml beaker. Add 5 ml of hydrochloric acid (5.2) and 5 ml of nitric acid (5.3). Cover the beaker with a watch glass and heat at 80 °C (hotplate) until completely dissolved. Add 2 ml of sulphuric acid solution (5.5) and dilute to 100 ml with water.

5.9.2.2 While stirring the solution with a glass rod, add 15 ml of sodium hydroxide (5.7), 1 ml of sulfosalicylic acid (5.10) and dilute to 150 ml with water. Wash the glass rod. Cover the beaker with a watch glass and heat the solution at 210 °C (hotplate) to near boiling. Wash the watch glass and the inner wall of the beaker.

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5.9.2.3 Place the beaker on the titration stand (6.3). (The temperature of the solution is above 80 °C at this point.) Place the combined glass pH electrode (6.4), stir the solution with a magnetic bar (6.6) or plastic propeller stirrer/rod stirrer. Adjust the acidity of the solution to pH (1 ± 0,05) with sodium hydroxide (5.7) by manual operation through a dropper.

There should be no temperature compensation at the time of pH adjustment.

5.9.2.4 Place the photometric sensor (6.5) and the dispensing tube tip into the beaker. The detection window of the photometric sensor and the dispensing tube tip shall be immersed. Ensure there are no bubbles between the light path in the detection window of the photometric sensor.

5.9.2.5 Titrate with the EDTA solution (5.9) using the potentiometer titration apparatus (6.2). The colour of the solution changes to light yellow at the end point. The end point is detected automatically. Record the volume of the end point as V_1.

The temperature of the solution shall be above 50 °C at the end of the titration with EDTA.

NOTE The photometric sensor is immersed into the solution after the solution is adjusted to pH 1 in order to avoid the sensor being corroded by acids.

Typical titration curves are given in Annex B.

Titration parameters for reference are given in Annex C.

5.9.2.6 Carry out the analysis in quadruplicate and record the volume of the end point as V_1, V_2, V_3 and V_4.

5.9.2.7 The un-blank calibration concentration of the EDTA solution, $C_{n(\mathrm{u})}$ (n = 1, 2, 3, 4), expressed in mole per litre, is calculated to four decimal places using Formula (1):

$$C_{n(\mathrm{u})} = \frac{m_n}{0{,}055\,847 \times V_n} \tag{1}$$

where

n	is 1, 2, 3, 4;
m_n	is the mass, in grams, of the iron powder;
V_n	is the volume at end point, in millilitres, of EDTA;
0,055 847	is the multiple of the atomic mass of iron.

If the range ($C_{\max} - C_{\min}$) of the four test results ($C_{1(\mathrm{u})}$, $C_{2(\mathrm{u})}$, $C_{3(\mathrm{u})}$, $C_{4(\mathrm{u})}$) is equal to or less than 0,000 2 mol/l, the arithmetic mean of the four test results shall be reported as the final quoted result, $C_{\mathrm{EDTA(u)}}$.

If the range of the four test results is greater than 0,000 2 mol/l, the median of the four test results shall be reported as the final quoted result, $C_{\mathrm{EDTA(u)}}$.

5.9.3 Blank test for the standardization of EDTA

5.9.3.1 In a 250 ml beaker, add 1,00 ml of iron standard solution (5.8), 5 ml of hydrochloric acid (5.2), 5 ml of nitric acid (5.3), 2 ml of sulphuric acid solution (5.5) and 100 ml of water. Follow the procedure given in 5.9.2.2 to 5.9.2.5. Record the volume of the end point as V_{01}, V_{02}, V_{03} and V_{04}.

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5.9.3.2 If the range of the four test results (V_{01}, V_{02}, V_{03}, V_{04}) is equal to or less than 0,02 ml, the arithmetic mean of the four test results shall be reported as the final quoted result, V_B.

If the range of the four test results is greater than 0,02 ml, the median of the four test results shall be reported as the final quoted result, V_B.

5.9.3.3 The blank value of the titration, V_0, is calculated to two decimal places using Formula (2):

$$V_0 = V_B - A \tag{2}$$

where

V_B is the final quoted result;

A is the volume of EDTA (5.9), in millilitres, equivalent to 1,00 ml of the iron standard solution (5.8).

A is calculated to two decimal places using Formula (3):

$$A = 1{,}00 \times \frac{0{,}100\,0}{C_{\mathrm{EDTA(u)}}} \tag{3}$$

where

1,00 is the volume, in millilitres, of the iron standard solution;

0,100 0 is the concentration, in mole per litre, of the iron standard solution;

$C_{\mathrm{EDTA(u)}}$ is the un-blank calibration concentration (5.9.2.7), in mole per litre, of the EDTA solution.

NOTE In the absence of iron, there is no iron sulfosalicylate in the solution and the photometric titration cannot be carried out. The addition of the iron solution is therefore necessary to promote an indicator response in the blank solution and thus allow a suitable correction for the blank in terms of its equivalent in millilitres of the EDTA standard solution.

The 1 ml one-mark pipette shall be previously calibrated by weighing the mass of water delivered and converting to volume.

5.9.4 Calculation of the concentration of EDTA solution

The concentration of the EDTA solution, C_n (n = 1, 2, 3, 4), expressed in mole per litre, is calculated to four decimal places using Formula (4):

$$C_{0n} = \frac{m_n}{0{,}055\,847 \times (V_n - V_0)} \tag{4}$$

where

n is 1, 2, 3, 4;

m_n is the mass, in grams, of the iron powder;

V_n is the volume at end point, in millilitres, of EDTA;

V_0 is the blank value as defined in 5.9.3.3, in millilitres, of the EDTA solution;

0,055 847 is the multiple of the atomic mass of iron.

If the range ($C_{max} - C_{min}$) of the four test results (C_{01}, C_{02}, C_{03}, C_{04}) is equal to or less than 0,000 2 mol/l, the arithmetic mean of the four test results shall be reported as the final quoted result, C_{EDTA}.

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If the range of the four test results is greater than 0,000 2 mol/l, the median of the four test results shall be reported as the final quoted result, C_{EDTA}.

5.10 Sulfosalicylic acid, 100 g/l.

6 Apparatus

Use one-mark volumetric flasks that conform to the requirements of ISO 1042, Class A. Use single-volume pipettes that conform to the requirements of ISO 648, Class A.

6.1 Analytical balance, designed for weighing to the nearest 0,1 mg.

6.2 Potentiometric titration apparatus.

An automatic potentiometric titrimeter capable of titrating to a fixed volume using either variable or fixed titrant increments.

Use burettes that conform to the requirements of ISO 385, Class A.

6.3 Titration stand.

The accessories of the automatic potentiometric titrimeter should be used.

6.4 Combined glass pH electrode, with a measuring range of pH 0 to pH 14.

6.5 Photometric sensor, specifically designed for colour-indicated titrations in acidic solution. The wavelength 520 nm shall be used. The photometric sensor shall be connected to the potentiometer by means of a suitably screened cable. Before used, the photometric sensor output potential should be optimized to 1 000 mV.

6.6 Magnetic stir bar, which should have a chemically inert surface.

6.7 Weighing spatula, of a non-magnetic material or demagnetized stainless steel.

6.8 Weighing bottle, of approximate volume 10 ml.

6.9 Modified polytetrafluoroethylene (PTFE-TFM) pressure vessel, of 100 ml.

6.10 Microwave digestion oven, equipped with temperature sensors, enabling direct temperature control in a single reference vessel up to 250 °C. The feedback from the sensors and terminal controller can provide accurate control and reproducibility of all digestion parameters such as microwave power, time and temperature. Temperature sensors should be accurate to (210 ± 5) °C.

The temperature measurement system should be periodically calibrated at an elevated temperature.

Follow the microwave manufacturer's instructions for the specific temperature sensor calibration procedure.

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