INTERNATIONAL STANDARD

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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

Reference number
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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).

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 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.

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/

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.

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}.

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}.

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.

7 Sampling and samples

7.1 Laboratory sample

For analysis, use a laboratory sample of 100 µm nominal top size, which has been taken and prepared in accordance with ISO 3082. For ores with significant contents of combined water or oxidizable compounds, use a particle size of less than 160 µm nominal top size.

NOTE See ISO 7764 for guidance on the significant contents of combined water and oxidizable compounds.

7.2 Preparation of test samples

7.2.1 General

Depending on the ore type, proceed in accordance with either 7.2.2 or 7.2.3.

7.2.2 Ores having significant contents of combined water or oxidizable compounds

Prepare an air-equilibrated test sample in accordance with ISO 2596 with the following types of ore:

a) processed ores containing metallic iron;

b) natural or processed ores in which the sulfur content is higher than 0,2 % mass fraction;

c) natural or processed ores in which the content of combined water is higher than 2,5 % mass fraction.

7.2.3 Ores outside the scope of 7.2.2

Prepare a predried test sample as follows.

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test sample at (105 ± 2) °C as specified in ISO 7764.

8 Procedures

8.1 Number of determinations

Carry out the analysis at least in duplicate and independently on one prepared test sample (see 7.2).

NOTE The expression “independently” means that the second, and any subsequent, result is not affected by the previous result(s).

8.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 0,25 g of the predried air equilibrated test sample obtained in accordance with 7.2.2. Concurrently, determine the hydroscopic moisture content in accordance with ISO 2596 and calculate the dried mass of the test portion (m).

Where the test sample is obtained in accordance with 7.2.3, transfer 0,25 g of the test portion to a weighing bottle (6.8). Dry the open weighing bottle with the test portion and the lid for 2 h at (105 ± 2) °C. Close the weighing bottle with the lid, transfer to a desiccator and cool to room temperature (about 20 min).

Weigh the bottle (with the lid on) to the nearest 0,000 2 g (m_1). Transfer the test portion to the vessel and then weigh the weighing bottle with the lid on (m_2). The dried mass of the test portion (m) is the difference between the two weighings, m_1 and m_2.

8.3 Blank test

8.3.1 In each run, at least one blank test shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions.

8.3.2 Where the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents used are from the same reagent bottles.

8.4 Determination

8.4.1 Decomposition of the test portion

Transfer the test portion (see 8.2) to the 100 ml PTFE-TFM vessel (6.9). Add 5 ml of hydrochloric acid (5.2), 5 ml of nitric acid (5.3) and 0,5 ml of hydrofluoric acid solution (5.4).

For TFe < 40 %, add 1 ml of hydrofluoric acid solution (5.4).

If part of the sample stays on the inner wall of the vessel, wet it by adding acids drop by drop, then gently swirl the solution to homogenize the sample with the acids. Visually look for a reaction. If a reaction occurs, allow the reaction to subside completely before capping the vessel.

Close the vessel and introduce it into the rotor segment, then tighten by using a torque wrench. Insert the segment into the microwave cavity and connect the temperature sensor. Set the digest temperature to 210 °C and hold this temperature for 30 min. Run the microwave programme to completion.

Cool the rotor by air or by water until the temperature of the solution is below 50 °C. Open the vessels carefully. Wash the caps and collect the solution to the 250 ml beakers. Add 2 ml of sulphuric acid solution (5.5) to the vessels and transfer the solution of the vessels to the 250 ml beakers. Add 30 ml of boric acid solution (5.6) and dilute to 100 ml with water.

The microwave oven should be operated according strictly to the supplier's instructions.

To allow for a feedback process control, a reference vessel shall be prepared containing a chemistry identical to any other sample vessel being processed during the same run.

For TFe < 40 %, if white precipitate (SiO_2) in the solution is found, filtration should be applied to remove the precipitate before adding 30 ml of boric acid solution (5.6). Transfer the solution and the white precipitate on to the fast filter paper. Wash the beaker and the white precipitate with warm hydrochloric acid (2 %) until the yellow colour of iron(III) chloride is no longer observed, and then wash in warm water six to eight times. Collect the filtrate and washings in a 250 ml beaker.

8.4.2 Titration

Follow the procedure given in 5.9.2.2 to 5.9.2.5 and record the volume of the end point as V.

8.5 Procedure of blank test

8.5.1 Using 1,00 ml of the iron standard solution (5.8) instead of the test portion, add the same amounts of all reagents in 8.4.1 and run the remaining steps specified in 8.4.1 and 8.4.2. Carry out the analysis in duplicate and record the results as V_{S1} and V_{S2}.

8.5.2 If $|V_{S1}-V_{S2}|$ is equal to or less than 0,02 ml, the arithmetic mean of V_{S1} and V_{S2} shall be reported as the final quoted result, V_{S0}.

If $|V_{S1}-V_{S2}|$ is greater than 0,02 ml, the blank test shall be repeated and the median of the four test results shall be reported as the final quoted result, V_{S0}.

8.5.3 The blank test value of the titration (V_S) is calculated to two decimal places using Formula (5):

$$V_s = V_{S0} - 1{,}00 \times \frac{0{,}100\,0}{C_{EDTA}} \quad (5)$$

where

V_{S0} is the volume at end point, in millilitres, of EDTA;

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

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

C_{EDTA} is the concentration, in mole per litre, of EDTA solution.

9 Expression of results

9.1 Calculation of total iron content

The total iron content, w_{Fe}, as a percentage by mass, is calculated to two decimal places using Formula (6):

$$w_{Fe} = \frac{C_{EDTA} \times (V - V_S) \times 0{,}055\,847}{m} \times 100 \quad (6)$$

where

C_{EDTA} is the concentration, in mole per litre, of EDTA solution;

V is the end point volume for the test portion, in millilitres, of EDTA solution;

V_S is the blank test value as defined in 8.5.3, in millilitres, of EDTA solution;

m is the dried mass, in grams, of the test portion;

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

9.2 General treatment of results

9.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by Formulae (7) to (10):

$$\sigma_d = 0{,}084 \quad (7)$$

$$\sigma_L = 0{,}067 \quad (8)$$

$$R_d = 0{,}23 \quad (9)$$

$$P = 0{,}27 \quad (10)$$

where

σ_d is the independent duplicate standard deviation;

σ_L is the between-laboratories standard deviation;

R_d is the independent duplicate limit;

P is the permissible tolerance between laboratories.

9.2.2 Methods for checking the acceptability of the test results and determining the final quoted result

The two test results should be obtained under repeatability conditions. The absolute difference between the two test results should then be compared with the independent duplicate limit. This procedure is summarized in the flowchart given in Annex A.

9.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final laboratory results reported by two laboratories.

Compute the following quantity using Formula (11):

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \tag{11}$$

where

μ_{12} is the mean of the final results;

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2.

If $|\mu_1 - \mu_2| \leq P$, the final results are in agreement, where P is the permissible tolerance between laboratories.

9.2.4 Check for trueness

The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM) or a reference material (RM). Calculate the analytical result, μ, for the CRM/RM using the procedures given in 9.1 and 9.2, and compare it with the certified or reference value, A_c.

There are two possibilities:

a) $|\mu_c - A_c| \leq C$, in which case the difference between the reported result and the certified/reference value is statistically insignificant;

b) $|\mu_c - A_c| > C$, in which case the difference between the reported result and the certified/reference value is statistically significant

where

μ_c is the final result for the CRM;

A_c is the certified/reference value for the CRM/RM;

C is a value dependent on the type of CRM/RM used, see Formula (12).

CRMs used for this purpose should be prepared and certified in accordance with ISO Guide 35.

C shall be calculated using Formula (12):

$$C = 2\sqrt{\frac{s_{\mathrm{Lc}}^2 + \frac{s_{\mathrm{Wc}}^2}{n_{\mathrm{Wc}}}}{N_{\mathrm{c}}} + \sigma_L^2 + \frac{\sigma_d^2}{n}} \tag{12}$$

where

s_{Lc} is the between-laboratories standard deviation of the certifying laboratories;

s_{Wc} is the within-laboratory standard deviation of the certifying laboratories;

n_{Wc} is the average number of replicate determinations in the certifying laboratories;

N_{c} is the number of certifying laboratories;

n is the number of replicate determinations carried out on the CRM/RM;

σL and σd are as defined in 9.2.1.

The following procedure should be used when the information on the RM certificate is incomplete:

— if there are sufficient data to enable the between-laboratories standard deviation to be estimated, delete the expression $\frac{s_{\mathrm{Wc}}^2}{n_{\mathrm{Wc}}}$ and regard s_{LC} as the standard deviation of the laboratory means;

— if the certification has been made by only one laboratory or if the interlaboratory results are missing, use Formula (13):

$$C = 2\sqrt{2\sigma_L^2 + \frac{\sigma_d^2}{n}} \tag{13}$$

A CRM certified by only one laboratory should be avoided unless it is known to have an unbiased certified value.

9.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations specified in Annex A, calculated to four decimal places and rounded off to the second decimal place as follows:

a) where the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place in accordance with ISO 80000-1:2009, B.3, Rule A, is kept unchanged;

b) where the figure in the third decimal place is 5 and there is a figure other than 0 in the fourth decimal place, or where the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;

c) where the figure in the third decimal place is 5 and there is a figure 0 in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

9.3 Oxide factors

$$w_{\mathrm{Fe_2O_3}} = 1{,}430\, w_{\mathrm{Fe}}$$

$$w_{\mathrm{FeO}} = 1{,}286\, w_{\mathrm{Fe}}$$

$$w_{Fe_3O_4} = 1{,}382\, w_{Fe}$$

10 Test report

The test report shall include the following information:

a) the name and address of the testing laboratory;

b) the date of issue of the test report;

c) a reference to this document, i.e. ISO 21826-1;

d) the details necessary for the identification of the sample;

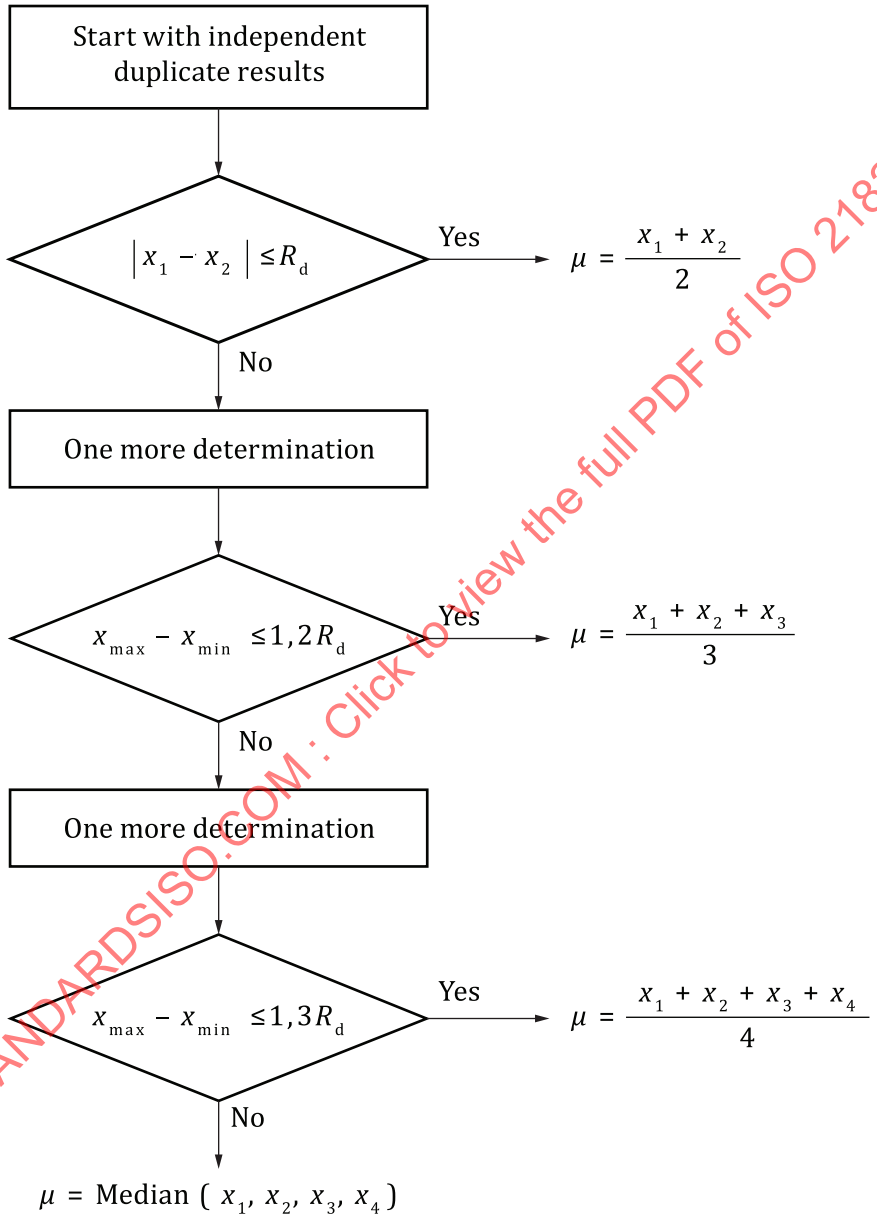
e) the result of the analysis;

f) the reference number of the result;

g) any characteristics noticed during the determination and any operations not specified in this document which may have had an influence on the results, either for the test sample or the CRM(s).

Annex A
(informative)

Flowsheet of the procedure for the acceptance of analytical values for test samples



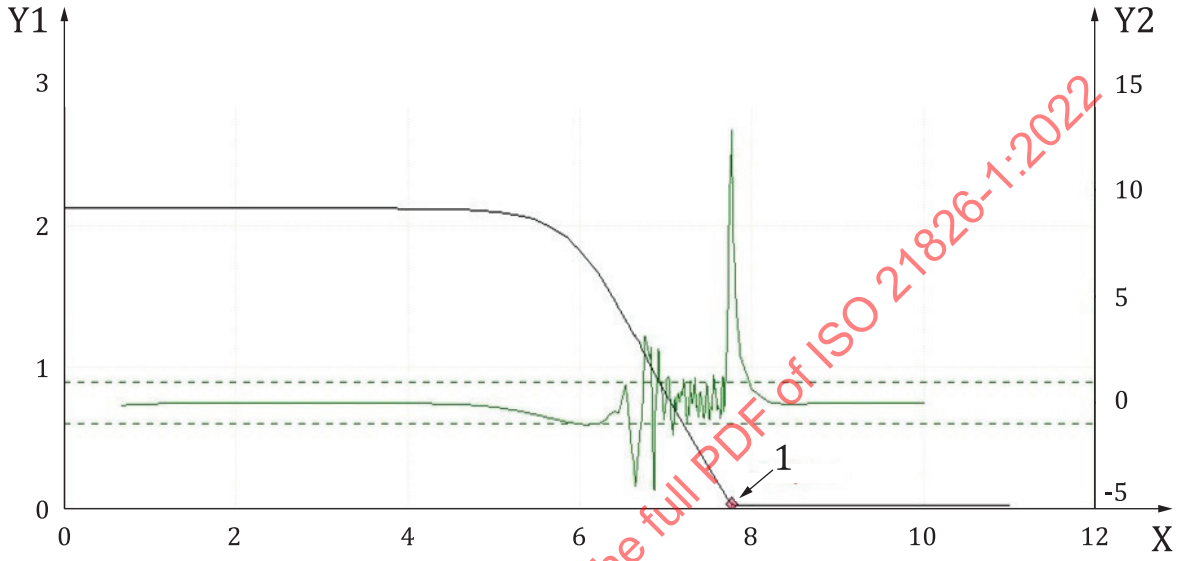
Key

R_d permissible tolerance within a laboratory (repeatability)

Figure A.1 — Flowsheet of the procedure for the acceptance of analytical values for test samples

Annex B
(informative)

Typical titration curves

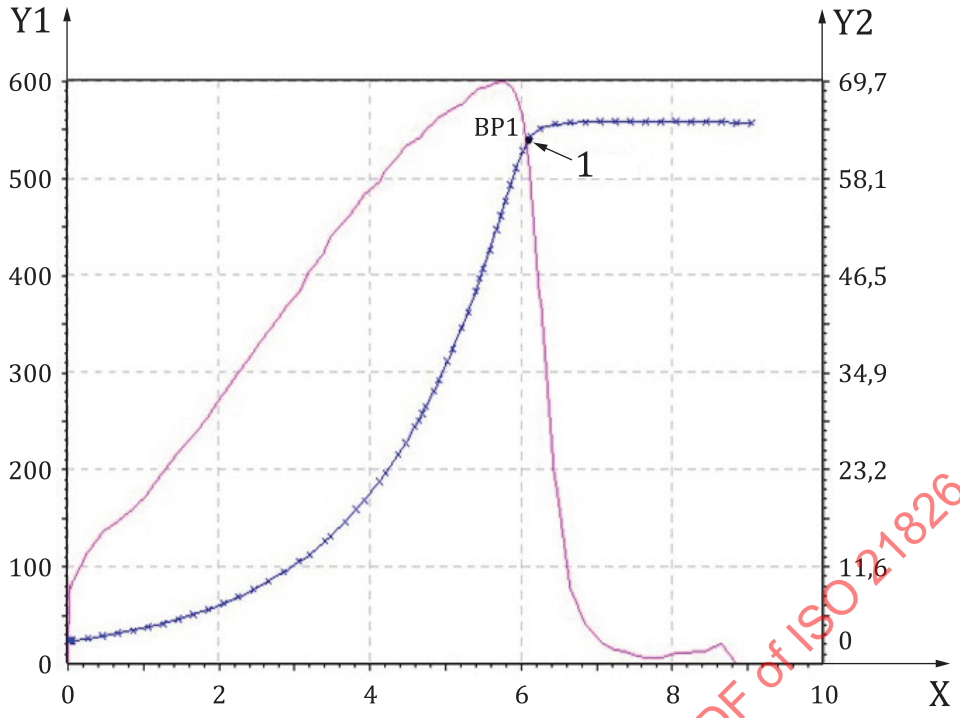


Key

X volume/ml
Y1 signal/A
Y2 $d^2E/dV^2/A/ml^2$
1 end point

Figure B.1 — Typical titration curve for Mettler Toledo[1)]

1) Mettler Toledo and Metrohm are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.



Key

X V (ml)
Y1 U (mV)
Y2 ERC
1 end point

Figure B.2 — Typical titration curve for Metrohm[1)]

Annex C
(informative)

Titration parameters for reference

C.1 Mettler Toledo[2] (for sample test)

Method

Method ID WG53

Sample

Sample type ..... Sample

Number of IDs 1

ID 1 ..... Fe

Entry type ..... Mass

Lower limit (g) 00

Upper limit (g) 025

Density (g/ml) 5,584 7

Correction factor 1,0

Temperature (°C) ..... 25(DO NOT CHANGE TO 80)

Entry ..... Arbitrary

Titrator reader ..... None

Number of sample factors 0

Titration stand (Titration stand 1)

Titration stand

Type ..... Manual stand

Titration stand ..... Manual stand 1

Stir (Stir 1)

Stir

Speed (%) 40

Duration (s) 5

2) Mettler Toledo and Metrohm are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Condition ..... No

Dispense (normal) (1) (Dispense normal 1)

Dispense

Titrant ..... EDTA

Concentration (mol/l) 0,1

Volume (ml) ..... 15(an appropriate value according to the iron content)

Dosing rate (ml/min) 60,0

Condition ..... No

Titration (EQP) (1) (Titration EQP 1)

Titrant

Titrant ..... EDTA

Concentration (mol/l) 0,1

Sensor

Type ..... Phototrode

Sensor DP5

Unit ..... A

Temperature acquisition

Temperature acquisition ..... No

Stir

Speed (%) 40

Predispense

Mode ..... Potential

Potential (A) 0,8

Wait time (s) 3

Control

Control ..... User

Titrant addition ..... Dynamic

dE (set value) (mV) 1,0

dV(min) (ml) 0,04

dV(max) (ml) 0,4

Mode ..... Equilibrium controlled

dE (mV) 1,0

dt (s) 1

t(min) (s) 2

t(max) (s) 5

Evaluation and recognition

Procedure ..... Segmented

Threshold (A/ml^2) 1

Tendency ..... None

Ranges

Add. EQP criteria ..... Steepest jump

Steepest jumps 1

Termination

At Vmax (ml) 40

At potential ..... Yes

Potential (A) 0,6

Termination tendency ..... None

At slope ..... No

After number of recognized EQPs ..... No

Combined termination criteria ..... No

Accompanying stating

Accompanying stating ..... No

Condition

Condition ..... No

Titration (EQP) (2) (Titration EQP 2)

Titrant

Titrant ..... EDTA

Concentration (mol/l) 0,1

Sensor

Type ..... Phototrode

Sensor DP5

Unit ..... A

Temperature acquisition

Temperature acquisition ..... No

Stir

Speed (%) 40

Predispense

Mode ..... None

Wait time (s) 0

Control

Control ..... User

Titrant addition ..... Dynamic

dE (set value) (mV) 1,0

dV(min) (ml) 0,02

dV(max) (ml) 0,4

Mode ..... Equilibrium controlled

dE (mV) 1,0

dt (s) 1

t(min) (s) 2

t(max) (s) 15

Evaluation and recognition

Procedure ..... Segmented

Threshold (A/ml) 0,5

Tendency ..... None

Ranges 1

Range type 1 ..... Potential

Lower limit – Potential 1 (A) 0

Upper limit – Potential 1 (A) 0,5

Add. EQP criteria ..... Steepest jump

Steepest jumps 1

Termination

At Vmax (ml) 5

At potential ..... No

At slope ..... No

After number of recognized EQPs ..... No

Combined termination criteria ..... No

Accompanying stating

Accompanying stating ..... No

Condition

Condition ..... No

EDTA (R1)

Calculation

Result ..... EDTA

Result unit ..... ml

Formula ..... R1 = VENDDi(1)+VEND(1)+V(2)

Constant C = ... 1

M ..... M(Fe)

z ..... z(Fe)

Decimal places 2

Result limits ..... No

Extra statistical functions ..... No

Send to buffer ..... No

Write to Smart Tag ..... None

C.2 Mettler Toledo (for blank test)

Method

Method ID WG53

Sample

Sample type ..... Sample

Number of IDs 1

ID 1 ..... Fe

Entry type ..... Mass

Lower limit (g) 0,0

Upper limit (g) 0,25

Density (g/ml) 5,584 7

Correction factor 1,0

Temperature (°C) ..... 25(DO NOT CHANGE TO 80)

Entry ..... Arbitrary

Titrator reader ..... None

Number of sample factors 0

Titration stand (Titration stand 1)

Titration stand

Type ..... Manual stand

Titration stand ..... Manual stand 1

Stir (Stir1)

Stir

Speed (%) 40

Duration (s) 5

Condition ..... No

Titration (EQP) (1) (Titration EQP 1)

Titrant

Titrant ..... EDTA

Concentration (mol/l) 0,1

Sensor

Type ..... Phototrode

Sensor DP5

Unit ..... A

Temperature acquisition

Temperature acquisition ..... No

Stir

Speed (%) 40

Predispense

Mode ..... None

Wait time (s) 0

Control

Control ..... User

Titrant addition ..... Dynamic

dE (set value) (mV) 1,0

dV(min) (ml) 0,02

dV(max) (ml) 0,04

Mode ..... Equilibrium controlled

dE (mV) 1,0

dt (s) 1

t(min) (s) 2

t(max) (s) 15

Evaluation and recognition

Procedure ..... Segmented

Threshold (A/ml) 0,5

Tendency ..... None

Ranges 1

Range type 1 ..... Potential

Lower limit – Potential 1 (A) 0

Upper limit – Potential 1 (A) 0,5

Add. EQP criteria ..... Steepest jump

Steepest jumps 1

Termination

At Vmax (ml) 3

At potential ..... No

At slope ..... No

After number of recognized EQPs ..... No

Combined termination criteria ..... No

Accompanying stating

Accompanying stating ..... No

Condition

Condition ..... No

EDTA (R1)