International Standard

ISO 25319

Determination of metallic Fe in reduced iron (sponge Iron) and briquette — Iron (III) chloride titrimetric method

Dosage du Fe métallique dans le fer préréduit (fer spongieux) et dans la briquette — Méthode titrimétrique au chlorure de fer (III)

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First edition
2026-11

Reference number
ISO 25319:2026(en)

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Published in Switzerland

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

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Determination of metallic Fe in reduced iron (sponge Iron) and briquette — Iron (III) chloride titrimetric method

1 Scope

This method is applicable to a concentration range of 52,80 % mass fraction to 86,40 % mass fraction of the metallic Fe.

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 10835, *Direct reduced iron and hot briquetted iron — Sampling and sample preparation*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

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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

Iron present in the oxidation state Fe^0 in the sample is oxidized to Fe^{+2} by the action of $FeCl_3$. This reaction is catalysed by ammonium chloride according to the following reaction:

$$2FeCl_3 + Fe \rightleftarrows 3FeCl_2$$

Fe^{2+} is oxidized and titrated with potassium dichromate solution using the sodium diphenylamine sulfonate indicator (or barium diphenylamine sulfonate) according to the following reaction:

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightleftarrows 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

5 Reagents

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

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5.1 Iron (III) chloride solution 7 %

Add 117 g of iron (III) chloride hexahydrate to a 2 000 ml container holding 600 ml of water and agitate until it is completely dissolved. Dilute with water to a volume of 1 000 ml.

NOTE For better dissolution of the iron (III) chloride, use hot water (60 °C to 80 °C). The ideal condition is a complete dissolution of the salt resulting in a translucent solution.

5.2 Standard potassium dichromate solution, 0,016 67 mol/L

Add 4,903 1 g of dried potassium dichromate to a 2 000 ml container holding 600 ml of water and agitate until it is completely dissolved. Dilute with water to a volume of 1 000 ml.

Prepare a standard potassium dichromate solution following the directions in Annex B

5.3 Mixed acid

Add 700 ml of water to a 2 000 ml beaker. Place the beaker in a cool place and add slowly with stirring 150 ml of phosphoric acid, 1,7 b/ml. Allow cooling. Add slowly with stirring 150 ml of sulfuric acid, 1,84 g/ml and allow cooling (it is optional: placing the beaker in an ice-water bath). Dilute this mixture to 1 000 ml with stirring.

5.4 Sodium diphenylamine sulfonate 2 g/l

Dissolve 0,2 g of powdered sodium diphenylamine sulfonate ($C_6H_5NHC_6H_4SO_3Na$) in a small volume of water and dilute to 100 ml. Store this solution in a brown glass bottle.

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5.5 Barium diphenylamine sulfonate 2 g/l

Dissolve 0,2 g of powdered barium diphenylamine sulfonate ($(C_6H_5NHC_6H_4SO_3)_2Ba$) in 15 ml of hydrochloric acid 1 mol/L and put the container on the heater and heat (heat under 100 °C to prevent HCl volatilization or reagent decomposition due to excessive temperature) until the salt is completely solved. (Approximate time of dissolution is 15-30 minutes). Dilute this mixture to 100 ml and store this solution in a brown glass bottle.

NOTE As both sodium and barium diphenylamine sulfonate are equal as an indicator, there is no difference in the use of either of them.

5.6 Ammonium chloride solution 100 g/l.

Dissolve 100 g ammonium chloride (NH_4Cl) to a small volume of water and dilute to 1 000 ml.

5.7 Ammonium iron(II) sulfate hexahydrate

Ammonium iron(II) sulfate hexahydrate1,86g/cm^3(20 °C), Molar mass 392,14 g/mol.

6 Apparatus

6.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

6.2 Erlenmeyer flask, wide mouth (500 ml).

6.3 Magnet stirrer, with magnetic stirring bar.

6.4 Measuring cylinder, of appropriate volume.

6.5 A grade burette, mark A, shall conform to the specifications of ISO 385.

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6.6 Non-magnetic spatula.

6.7 Beaker

7 Sampling and sample preparation

7.1 Sample preparation

After receiving the sample, the sample shall be dried at or below a temperature of 105 °C until a constant weight is reached. Then let the sample cool to room temperature in a desiccator.

7.2 Laboratory sample

For analysis, use a laboratory sample of particle size of less than 160 µm which has been taken and prepared in accordance with ISO 10835.

8 Procedure

8.1 Number of determinations

All determinations should be carried out in duplicate. If necessary, make further determinations in accordance with the flowsheet presented in Annex A.

8.2 Preparation of test portions

Accurately weigh 0,100 0 g ± 0,005 0 g of the test sample (7.2 laboratory sample). Record the sample mass (m). Add the test sample to an Erlenmeyer flask or beaker with appropriate volume.

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8.3 Determination

8.3.1 Decomposition of the test portion

Add 100 ml of 7 % Iron (III) chloride solution and 50 ml of Ammonium chloride solution to the Erlenmeyer flask containing sample, put a magnet bar into the container and then cover the Erlenmeyer flask with a watch glass and stir the solution with a magnetic agitator for at least 30 min with high speed (600 to 700 rpm).

$$2Fe^{3+} + Fe \rightarrow 3Fe^{2+} + Fe^{3+} \text{ residual}$$

8.3.2 Titration

After 30 min, add 30 ml of sulfuric acid-phosphoric acid solution, 5 to 10 drops of sodium or barium diphenylamine sulfonate indicator and 200 ml to 300 ml distilled water.

NOTE The consumption amount of the indicator can be different depending on the type of brand used.

Titrate the solution with potassium dichromate solution 0,016 67 mol/l. The end point is reached when a final drop of the titrant imparts a permanent purple colour. In each run, one blank test shall be carried out in parallel with the analysis of the sample(s) under the same conditions. Perform a blank test by following the procedure without the sample. Record the blank titration volume as V_0. For every new batch of iron (III) chloride solution, a blank should be performed.

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