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(en)

Iron ores - Determination of water-soluble chloride - Ion-selective electrode method (ISO 9517:2007,IDT)

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INTERNATIONAL STANDARD

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Iron ores — Determination of water-soluble chloride — Ion-selective electrode method

Minerais de fer — Dosage des chlorures solubles dans l'eau — Méthode par électrode sélective des ions

Reference number
ISO 9517:2007(E)

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Contents

Page

Foreword ... **iv**

1 Scope ... **1**

2 Normative references ... **1**

3 Principle ... **1**

4 Reagents ... **1**

5 Apparatus ... **3**

6 Sampling and samples ... **4**
6.1 Laboratory sample ... **4**
6.2 Preparation of predried test samples ... **4**

7 Procedure ... **4**
7.1 Number of determinations ... **4**
7.2 Blank test and check test ... **5**
7.3 Temperature setting ... **5**
7.3.1 Magnetic stirrer/hotplate ... **5**
7.3.2 Laboratory hotplate ... **5**
7.4 Preliminary tests ... **5**
7.4.1 Electrode-system check test ... **5**
7.4.2 Contamination check test ... **5**
7.5 Test portion ... **5**
7.6 Determination ... **6**
7.6.1 Leaching of water-soluble chloride ... **6**
7.6.2 Filtration ... **6**
7.6.3 Treatment of the test solution ... **6**
7.6.4 Measurement of electrode potential ... **6**
7.6.5 Preparation of calibration graph ... **6**
7.6.6 Measurement of chloride concentration ... **7**

8 Expression of results ... **7**
8.1 Calculation of mass fraction of water-soluble chloride ... **7**
8.2 General treatment of results ... **8**
8.2.1 Repeatability and permissible tolerance ... **8**
8.2.2 Determination of analytical result ... **8**
8.2.3 Between-laboratories precision ... **8**
8.2.4 Check for trueness ... **9**
8.2.5 Calculation of final result ... **9**

9 Test report ... **10**

Annex A (normative) **Flowsheet of the procedure for the acceptance of analytical values for test samples** ... **11**

Annex B (informative) **Derivation of repeatability and permissible tolerance equations** ... **12**

Annex C (informative) **Graphical presentation of precision data obtained by international analytical trials** ... **13**

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 9517 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 9517:1989), which has been technically revised. It has been updated to alter the manner in which precision data are presented.

INTERNATIONAL STANDARD **ISO 9517:2007(E)**

Iron ores — Determination of water-soluble chloride — Ion-selective electrode method

WARNING — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies an ion-selective electrode method for the determination of the mass fraction of water-soluble chloride in iron ores. This method is applicable to a mass-fraction range of 0,007 % to 0,1 % of water-soluble chloride in natural iron ores, concentrates and agglomerates, including sinter products.

NOTE Water-soluble chloride is the part of the mass fraction of chloride in an iron ore that is extractable by leaching with aqueous solution under substantially neutral conditions.

2 Normative references

The following referenced documents are indispensable for the application 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 648, *Laboratory glassware — One-mark pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

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

3 Principle

The test portion is digested in water containing potassium sulfate, the suspension is transferred to a volumetric flask and diluted to volume. The solution is dry filtered, an aliquot is treated with potassium persulfate solution, and neutral buffer is added. Ionic-strength adjuster solution is added and the chloride concentration is determined potentiometrically using a chloride-ion electrode and a double-junction reference electrode.

4 Reagents

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

The preparation of reagent and calibration solutions, and all operations specified in Clauses 5, 6 and 7, shall be conducted in an area adequately isolated from any areas in which hydrochloric acid is used.

4.1 Potassium sulfate (K_2SO_4) solution, 2 g/l.

4.2 Potassium sulfate (K_2SO_4) solution, 4 g/l.

4.3 Potassium persulfate ($K_2S_2O_8$) solution, 1,5 g/100 ml.

Prepare freshly for each series of tests.

4.4 Sodium nitrate solution, $c(NaNO_3)$ = 5 mol/l.

Dissolve 42,5 g of sodium nitrate in about 60 ml of water, transfer to a 100 ml volumetric flask, dilute to volume and mix.

4.5 Phosphate buffer solution.

Dissolve 2,72 g of potassium dihydrogen phosphate (KH_2PO_4) and 2,84 g of disodium hydrogen phosphate (Na_2HPO_4) in 40 ml of water. Transfer to a 100 ml volumetric flask, dilute to volume and mix.

4.6 Stirring-bar cleaning solution.

Add carefully to about 700 ml of water, 150 ml of sulfuric acid (ρ 1,84 g/ml) and 150 ml of phosphoric acid (ρ 1,7 g/ml) and mix.

4.7 Chloride standard solution A, 1 000 µg of chloride per ml.

Dry about 2 g of sodium chloride at 105 °C for 1 h and cool in a desiccator. Weigh 0,824 g of the dried material, dissolve in about 50 ml of water and transfer to a 500 ml volumetric flask. Dilute to volume and mix.

1 ml of chloride standard solution A contains 1 000 µg of chloride.

4.8 Chloride standard solution B, 50 µg of chloride per ml.

Measure 25,0 ml of standard chloride solution A into a 500 ml volumetric flask, dilute to volume and mix.

1 ml of chloride standard solution B contains 50 µg of chloride.

4.9 Chloride standard solution C, 20 µg of chloride per ml.

Measure 10,0 ml of chloride standard solution A into a 500 ml volumetric flask, dilute to volume and mix.

1 ml of chloride standard solution C contains 20 µg of chloride.

Standard solutions B (4.8) and C (4.9) should be prepared freshly.

4.10 Calibration solutions.

Prepare the calibration solutions specified in Table 1 for the expected range of mass fractions of chloride.

If the mass fraction of chloride is unknown, prepare calibration solutions containing 5,0 µg, 10,0 g and 50,0 g of chloride per ml. If the mass fraction of chloride is then found to be less than 0,012 %, prepare additional calibration solutions containing 2,0 µg and 3,0 µg of chloride per ml. For higher mass fractions of chlorides, prepare any additional solutions required in accordance with Table 1.

Table 1 — Calibration solutions required for each range of mass fraction of chloride

Mass fraction of chloride in test sample %	Concentration in calibration solution µg/ml
0,005 to 0,025	2,0; 3,0; 5,0; 10,0
0,012 to 0,025	5,0; 10,0
0,025 to 0,10	10,0; 25,0; 50,0

For the preparation of the required calibration solutions, measure into a series of 100 ml volumetric flasks the aliquots of chloride standard solutions specified in Table 2.

Table 2 — Preparation of calibration solutions

Chloride concentration in calibration solution µg/ml	Standard-solution aliquot volume ml	Standard solution
2,0	10,0	C (4.9)
3,0	15,0	C (4.9)
5,0	10,0	B (4.8)
10,0	20,0	B (4.8)
25,0	50,0	B (4.8)
50,0	5,0	A (4.7)

Add to the aliquots of standard solution in the 100 ml volumetric flasks, 6 ml of potassium persulfate solution (4.3), 35 ml of potassium sulfate solution (4.2), 2 ml of phosphate buffer solution (4.5) and 2 ml of sodium nitrate solution (4.4) (ionic strength adjuster). Dilute to volume and mix.

Calibration solutions containing from 2,0 µg to 10,0 µg of chloride per ml should be prepared on the day of use.

5 Apparatus

Any one-mark pipettes and volumetric flasks required shall comply with the specifications of ISO 648 and ISO 1042, respectively.

Ordinary laboratory equipment and the following.

5.1 Magnetic stirrer (optional, see fourth paragraph of 7.6.4).

5.2 Magnetic stirrer/hotplate.

5.3 PTFE or polyethylene-covered stirring bars, 25 mm to 30 mm long.

Before use, stirring bars shall be cleaned to remove adhering iron ore and chloride contamination by leaching in the cleaning solution (4.6) for 30 min, and then in water for 30 min. Only clean tweezers should be used for handling the cleaned stirring bars.

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