
**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*

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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.

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.

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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.

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2 Normative references (standards.iteh.ai)

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 μ g 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 μ g of chloride.

4.8 Chloride standard solution B, 50 μ g 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 μ g of chloride.

4.9 Chloride standard solution C, 20 μ g 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 μ g 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 μ g, 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 μ g and 3,0 μ g 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)

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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.

5.4 Filtration apparatus, glass or polycarbonate plastic, with 25 mm to 50 mm diameter cellulose membrane microfilters of less than 1 µm pore size¹⁾.

The microfilters should at all times be handled only with clean tweezers.

5.5 Ion-selective electrode meter, or high-sensitivity pH meter, or high-impedance millivoltmeter, capable of reading with a sensitivity of 0,1 mV.

5.6 Chloride ion-selective electrode and separate double-junction, free-flowing, reference electrode.

Both of the electrodes shall be maintained and used in accordance with the manufacturers' specifications, and the outer chamber solution in the reference electrode should be changed as specified and replenished as necessary. The flow rate across the nitrate/test solution junction should be such that the level of the outer chamber solution falls at a rate of approximately 4 mm to 5 mm per day.

As some chloride ion-selective electrodes are light sensitive, they should not be used in direct sunlight or in very bright daylight.

NOTE "Combined" electrodes, which normally are not provided with a reference electrode incorporating a double junction, are not suitable.

6 Sampling and samples

6.1 Laboratory sample

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

NOTE A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

6.2 Preparation of predried test samples

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 °C ± 2 °C, as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with Annex A, independently, on one predried test sample.

The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure shall be carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

1) Millipore (XX10 apparatus with HAWP filters), Sartorius, Gelman are examples of suitable apparatus available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these types of apparatus.

7.2 Blank test and check test

NOTE For technical reasons, a blank test, as usually understood, cannot be conducted for methods using ion-selective electrodes. For this method, the procedure in 7.4.2 is substituted.

In each run, one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analyses of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.2.

The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar, to ensure that, in either case, no significant changes in the analytical procedure would become necessary.

When the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.3 Temperature setting

7.3.1 Magnetic stirrer/hotplate

Determine the temperature setting required to maintain a temperature of 90 °C to 95 °C in a 35 ml volume of water.

7.3.2 Laboratory hotplate

Preheat the hotplate and determine the temperature setting required to produce, in a 50 ml volume of water, a temperature of at least 90 °C (but without boiling) after heating for 25 min.

7.4 Preliminary tests

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7.4.1 Electrode-system check test

Before use of the electrodes for each series of tests, carry out the following check test of the electrode function.

Add to a 150 ml or 250 ml beaker, 100 ml of water, a stirring bar (5.3) and 2 ml of sodium nitrate solution (4.4). Place the electrodes in the solution, stir for 5 min and record the electrode potential in millivolts (E_1).

Stir at a rate that will displace any air bubbles from the electrode surface, but without causing a vortex.

Add 1 ml of chloride standard solution A (4.7), stir for 5 min to allow a stable reading to be obtained and record the electrode potential (E_2). Add 10 ml of chloride standard solution A (4.7), stir for 5 min and again record the electrode potential (E_3). The electrodes can be regarded as operating satisfactorily when the difference between E_2 and E_3 is $57 \text{ mV} \pm 2 \text{ mV}$, for a temperature of the test solutions within the range 20 °C to 25 °C.

7.4.2 Contamination check test

To ensure that the apparatus and reagents are free from chloride contamination, carry out the complete procedure specified in 7.6 without the addition of a test portion. The electrode potential reading obtained should be within 20 mV of the reading E_1 obtained in 7.4.1. If not, the cleaning procedures should be repeated. If the potential reading is again not within 20 mV of E_1 , use a different source of potassium sulfate and then, if necessary, potassium persulfate or buffer reagents.

7.5 Test portion

Weigh, to the nearest 0,001 g, approximately 2 g of the predried test sample (6.2).