GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 73.060.10

CCS D 31

GB/T 6730.64-2022

Replacing GB/T 6730.64-2007

Iron Ores - Determination of Water Soluble Chloride Content - Ion-selective Electrode Method

铁矿石 水溶性氯化物含量的测定

离子选择电极法

(ISO 9517:2007, Iron Ores - Determination of Water-soluble Chloride - Ion-selective Electrode Method, MOD)

Issued on: October 12, 2022 **Implemented on: October 12, 2022**

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

Table of Contents

Iron Ores - Determination of Water Soluble Chloride Content - Ion-selective Electrode Method

WARNING---the personnel adopting this document shall have practical experience in regular laboratory work. This document does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated in relevant national regulations.

1 Scope

This document specifies the method for the determination of water soluble chloride content in iron ores using ion-selective electrodes.

This document is applicable to the determination of water soluble chloride content in natural iron ores, iron ore concentrates, sinters and pellets. The determination range (mass fraction) is: 0.007% ~ 0.1%.

NOTE: the water soluble chloride content in iron ores refers to the chlorine obtained by leaching iron ores with aqueous solution under near-neutral conditions.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 6730.1 Iron Ores - Preparation of Predried Test Samples for Chemical Analysis (GB/T 6730.1-2016, ISO 7764:2006, MOD)

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB/T 10322.1 Iron Ores - Sampling and Sample Preparation Procedures (GB/T 10322.1-2014, ISO 3082:2009, IDT)

GB/T 12805 Laboratory Glassware - Burettes (GB/T 12805-2011, ISO 385:2005, NEQ)

GB/T 12806 Laboratory Glassware - One-mark Volumetric Flasks (GB/T 12806-2011, ISO 1042:1998, NEQ)

GB/T 12808 Laboratory Glassware - One-mark Pipettes

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Principle

The specimen is leached with potassium sulfate aqueous solution, and the obtained suspension is transferred to a volumetric flask, diluted to the scale, and dry-filtered. Take part of the filtrate, add potassium persulfate solution, neutral buffer solution and ionic strength adjustment solution; use a chloride ion-selective electrode and a double-joint reference electrode to determine the concentration of chloride ions in the test solution.

5 Reagents and Materials

Unless otherwise stated, only approved analytically pure reagents and distilled water of Grade-2 or above that complies with the stipulations of GB/T 6682 or water of equivalent purity are used in the analysis.

The preparation of reagents and calibration solutions, as well as all operations specified in Chapter 6 ~ Chapter 8, shall be effectively isolated from the place where hydrochloric acid is used.

5.1 Potassium sulfate (K_2SO_4) solution, 2 g/L.

5.2 Potassium persulfate ($K_2S_2O_8$) solution, 15 g/L. Prepare it right before use.

5.3 Sodium nitrate ($NaNO_3$) solution, 5 mol/L. Dissolve 42.5 g of sodium nitrate in about 60 mL of water, transfer it to a 100 mL volumetric flask, use water to dilute to the scale, and mix it well.

5.4 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 about 40 mL of water, transfer it to a 100 mL volumetric flask. Use water to dilute to the scale and mix it well.

5.5 Cleaning fluid. Under constant stirring, carefully and slowly add 150 mL of phosphoric acid (ρ 1.84 g/mL) and 150 mL of phosphoric acid (ρ 1.7 g/mL) to 700 mL of water, and mix it well.

5.6 Chlorine standard solution A, 1.00 mg/mL. Weigh-take 0.8240 g of sodium chloride reference material, dissolve it in about 50 mL of water, then, transfer it to a 500 mL volumetric flask; use water to dilute to the scale and mix it well. Sodium chloride is pre-burned at 500 °C ~ 600 °C to a constant weight, then, cooled to room temperature.

5.7 Chlorine standard solution B, 100 μg/mL. Take 50.00 mL of chlorine standard solution A

Chlorine calibration solutions containing 1.0 μg/mL ~ 10.0 μg/mL shall be prepared on the same day.

6 Instruments

6.1 Burettes, one-mark volumetric flasks and one-mark pipettes: shall respectively comply with the stipulations of GB/T 12805, GB/T 12806 and GB/T 12808.

6.2 Magnetic stirrer.

6.3 Electric heating magnetic stirrer: or water bath with ultrasonic wave. Set the heating power to maintain 35 mL of water at 90 °C ~ 95 °C.

6.4 Electric hot plate: set the heating power to rise 50 mL of water to at least 90 °C (under non-boiling conditions) in 25 min.

6.5 Plastic stirring rod: coated with PTFE or polyethylene, length: 25 mm ~ 30 mm. Before use, the stirring rod shall be cleaned in the cleaning liquid (see 5.5) for 30 min, then, washed with water for 30 min, to avoid possible chlorine contamination caused by iron ore powder adhesion or other reasons. The cleaned stirring rod shall be picked up with clean tweezers. Or plastic magnetic stirrers coated with PTFE or polyethylene can also be used, and the cleaning method is the same.

6.6 Suction filtration device: diameter 25 mm ~ 50 mm, equipped with glass or polycarbonate plastic microporous filter membrane with a pore size less than 1 μm. The microporous filter membrane shall only be handled with clean tweezers.

6.7 Ion-selective potentiometer: or high-sensitive pH meter, or high-impedance millivolt meter, with a reading accuracy of 0.1 mV.

6.8 Chloride ion-selective electrode and independent double salt bridge reference electrode: both electrodes shall be maintained and used in accordance with the instructions provided by the manufacturer. The solution in the outer chamber of the reference electrode shall be replaced as specified and filled when necessary. The flow rate along the nitrate / test solution interface shall be adjusted, so that the liquid level in the outer chamber maintains a falling speed of approximately 4 mm/d ~ 5 mm/d.

Chloride ion-selective electrodes are sensitive to light and shall not be used in direct sunlight or under strong light.

7 Sampling and Sample Preparation

7.1 Laboratory Specimens

In accordance with GB/T 10322.1, take and prepare the specimens. Generally, the particle size of the specimens shall be less than 100 μm. If the content of combined water or readily oxidizable substances in the specimens is high, the particle size shall be less than 160 μm.

The stipulations for high content of combined water and readily oxidizable substances shall comply with GB/T 6730.1.

7.2 Preparation of Pre-dried Specimens

Thoroughly mix the laboratory specimens and adopt the sample reduction method for sampling. In accordance with the stipulations of GB/T 6730.1, at 105 °C ± 2 °C, dry the specimens, and cool them to room temperature in a desiccator for later use.

8 Analytical Procedures

8.1 Number of Determinations

In accordance with Appendix B, the same dried specimen shall be independently determined at least twice.

NOTE: "independent" means that the results of the second and any subsequent determinations are not affected by the previous one. In this document, this condition means repeated determinations conducted by the same operator at different times or by different operators, including the application of appropriate re-calibration.

8.2 Blank Test and Verification Test

8.2.1 Blank test

Due to technical reasons, the conventional blank test does not apply to the ion-selective electrode method. This method uses the step of 8.3.2 to replace the blank test.

8.2.2 Verification test

Along with each batch of test specimens, under the same conditions, analyze the same type of reference materials for verification test. The pre-drying of the reference materials shall be carried out in accordance with the stipulations of 7.2.

If multiple specimens of the same type are simultaneously analyzed, the analysis value of reference material can be used.

8.3 Preparation before Test

8.3.1 Electrode inspection

Before using the electrode for each test, the electrode performance shall be inspected in accordance with the following steps.

a) Add 100 mL of water and 2 mL of sodium nitrate solution to a 150 mL or 250 mL beaker, and add a plastic stirring rod (see 6.5). Put the electrode into the solution, stir be left for enough time to make the temperature between the test solution and the calibration solution consistent.

In accordance with the potential value determined in the first round, and the sequence from low to high concentration, re-arrange the calibration solution and the test solution, then, determine another set of potential value. Take the second set of data as the determined value.

In accordance with the known chlorine mass concentration and corresponding determined potential value in the calibration, draw the lg [Cl]–*E* calibration curve. When necessary, respectively draw the calibration curves for concentration segments that are not of the same order of magnitude.

From the calibration curve, in accordance with the determined value of electrode potential of the test solution, calculate the corresponding chlorine mass concentration.

NOTE: when the chlorine mass concentration is lower than 3 μg/mL, the curve may bend to a certain extent. The extent of bending may vary due to differences in electrode performance.

9 Result Calculation and Expression

9.1 Calculation of Water Soluble Chloride Content

In accordance with Formula (1), calculate the water soluble chloride content (mass fraction) w_{Cl} in the specimen, expressed in (%).

$$w_{\mathrm{Cl}}=\frac{\rho_{\mathrm{Cl}}}{100m}\times 100 \quad \cdots\cdots\cdots\cdots (1)$$

Where,

ρ_{Cl}---the chlorine mass concentration in the test solution obtained from the calibration curve, expressed in (μg/mL);

m---the mass of specimen, expressed in (g).

9.2 General Processing of Analysis Results

9.2.1 Precision

The functional relation of precision in this document is shown in Table 4. The specimen used for precision test is shown in Appendix C.

Through the *C* value of the certified reference material (CRM) or reference material (RM) determined among multiple laboratories, calculate in accordance with Formula (3):

$$C=\frac{1}{\sqrt{2}}\sqrt{R^2-\frac{n-1}{n}r^2+8u^2} \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (3)$$

Where,

R---the inter-laboratory reproducibility limit;

r---the intra-laboratory repeatability limit;

n---the number of repeated determinations of the reference material;

u---the uncertainty of the standard value of CRM or RM.

9.2.5 Calculation of final result

The final result of the specimen is the arithmetic mean of the acceptable analysis values, or the value determined in accordance with the stipulations of Appendix B. The calculation is accurate to the fifth decimal place, and the report is accurate to the third decimal place. The values shall be rounded off in accordance with the stipulations of GB/T 8170.

10 Test Report

The test report shall include the following information:

a) Laboratory name and address;

b) Release date of test report;

c) Serial No. of this document;

d) Detailed description necessary for specimen identification;

e) Analysis results;

f) Serial No. corresponding to the analysis results;

g) Any abnormal characteristics during the determination and any operations not specified in this document that may affect the analysis results of the specimen or reference material.

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading…): https://www.chinesestandard.net/AboutUs.aspx

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: https://www.linkedin.com/in/waynezhengwenrui/

------ The End ------