NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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

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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.

5.3 Sodium nitrate (NaNO3) 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 (KH2PO4)

and 2.84 g of disodium hydrogen phosphate (Na2HPO4) 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

(rho 1.84 g/mL) and 150 mL of phosphoric acid (rho 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 ?g/mL. Take 50.00 mL of chlorine standard solution A

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

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.

6.3 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 ?m. 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.

7.1 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 ?m. If the content of combined water or readily oxidizable substances in the specimens is high, the particle size shall be less than 160 ?m.

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.