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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 24230-2009

**Chromium ores and concentrates -- Determination of chromium
content -- Titrimetric method
(English Translation)**

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1 Scope

This Standard specifies a titration method for the determination of chromium content in chromium ores and concentrates.

This Standard is applicable to the determination of chromium content in chromium ores and concentrates. The determination range (expressed by Cr, mass fraction) is >7.0%. The relevant provisions of GB/T 24228 shall be observed.

2 Normative References

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 12805 Laboratory glassware - Burettes (GB/T 12805-1991, neq ISO 385:1984)

GB/T 12806 Laboratory glassware - One-mark volumetric flasks (GB/T 12806-1991, eqv ISO 1042:1983)

GB/T 12808 Laboratory glassware - One-mark pipettes (GB/T 12808-1991, eqv ISO 648.1977)

GB/T 24228 Chromium ores and concentrates - Methods of chemical analysis - General instruction (GB/T 24228-2009, ISO 6629.1981, MOD)

GB/T 24220 Chromium ores - Determination of hygroscopic moisture content in analytical samples - Gravimetric method (GB/T 24220-2009, ISO 6129.1981, MOD)

GB/T 24243 Chromium ores - Increment sampling (GB/T 24243-2009, ISO 6153.1989, IDT)

ISO 6154 Chromium ores - Preparation of samples

3 Principle

The test material is fused with sodium peroxide; and the melt is leached with water; the solution is acidified with sulfuric acid and boiled to remove hydrogen peroxide. Chromium (III) ions are oxidized to chromate using ammonium persulfate with silver nitrate as a catalyst. Chromium (VI) is titrated with ferrous ammonium sulfate, followed by back-titration to the endpoint using a potassium permanganate standard solution, or directly titrate chromium (VI) by potentiometric titration.

4 Reagents

Unless otherwise specified, only recognized analytically pure reagents and distilled water, or water of equivalent purity complying with GB/T 6682, shall be used for the analysis.

4.1

Sodium peroxide (Na_2O_2), dry powder.

NOTE: Sodium peroxide shall be as dry as possible; it must not be used if caking has occurred.

4.2

Urea.

4.3

Sulfuric acid, ρ 1.84 g/mL.

4.4

Sulfuric acid, 1 + 1.

4.5

Sulfuric acid, 1 + 4.

4.6

Phosphoric acid, ρ 1.70 g/mL.

4.7

Manganese sulfate solution, 100 g/L.

Weigh 100 g of manganese (II) sulfate heptahydrate ($\text{MnSO}_4 \cdot 7\text{H}_2\text{O}$) and dissolve in 1 L of water.

4.8

Manganese sulfate solution, 1 g/L.

Pipette 10 mL of manganese sulfate solution (4.7) into 1 L of water.

4.9

Silver nitrate solution, 1 g/L; add 0.5 mL of nitric acid (ρ 1.42 g/mL) per liter of solution; permanganate standard solution (4.13.1);

V2 - volume of potassium permanganate standard solution (4.13.1) consumed by the titration, in mL;

V3 - volume of the added ammonium ferrous sulfate standard solution (4.14.1), in mL.

5 Apparatus

The common laboratory equipment and instruments. Burettes, one-mark volumetric flasks, and one-mark pipettes shall comply with the provisions of GB/T 12805, GB/T 12806, and GB/T

12808, respectively.

Potentiometric titration assembly.

a) Electrode pair. Platinum indicator electrode; calomel or tungsten electrode as reference electrode.

b) Magnetic stirrer.

c) Millivoltmeter. High-impedance millivoltmeter compatible with the electrode pair and suitable for pH measurement.

6.1 Laboratory sample

The laboratory sample for analysis shall be sampled and prepared in accordance with GB/T 24243 and ISO 6154; the particle size shall be less than 100 μm .

6.2 Preparation of pre-dried specimen

Thoroughly mix the laboratory sample and take a subsample using the incremental reduction method. Dry the specimen at a temperature of 105 °C to 110 °C in accordance with the provisions of GB/T 24220.

7.1 Number of determinations

Perform at least two independent determinations on the same pre-dried specimen.

NOTE: The term "independent" means that the results of the second and subsequent determination are not influenced by the result of the previous determination. In this analytical method, this condition implies that replicate determinations are performed by the same operator at different times or by different operators, including the use of appropriate recalibration.

7.2 Test material quantity

Weigh 0.25 g of the test material, accurate to 0.0002 g.

7.3 Blank test and verification test

Carry out a blank test alongside the test material following all analytical procedures; simultaneously analyze a standard sample of the same type for verification test.

7.4 Decomposition of the test material

WARNING. Safety goggles must be worn when handling sodium peroxide (from the start of heating until complete fusion).

Place the test material (7.2) into a corundum crucible; and add 3 g ~ 4 g of sodium peroxide (4.1). Mix the contents thoroughly with a glass rod; cover with 1 g ~ 2 g of sodium peroxide (4.1); and heat slowly at 400°C ~ 500°C. Then, fuse the mixture at 800°C ~ 850°C for 5 min ~

7 min, swirling the crucible frequently until the melt is homogeneous.

After the crucible has cooled, place it in a 600 mL beaker; and add 100 mL ~ 150 mL of hot water to dissolve the melt. Rinse the crucible with hot water. If particles of the melt adhere to the crucible walls, wash them with 7 drops ~ 8 drops of sulfuric acid solution (4.4) and 2 mL ~

3 mL of water; once the particles have dissolved, combine this solution with the solution in the beaker; and rinse the crucible again with water.

Add sulfuric acid solution (4.4) until the hydroxide precipitate dissolves; dilute with water to 300 mL ~ 350 mL. Add 10 mL of sulfuric acid (4.3) and 5 mL of phosphoric acid (4.6); and