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Manganese Ores – Determination of Total Iron Content – Potassium Dichromate Titrimetric Method and 1,10-Phenanthroline Spectrophotometric Method

锰矿石 全铁含量的测定 重铬酸钾滴定法和邻菲啰啉分光光度法

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Manganese Ores – Determination of Total Iron Content – Potassium Dichromate Titrimetric Method and 1,10-Phenanthroline Spectrophotometric Method

Warning: This Part does not point out all possible safety issues. It is the responsibility of the user to take appropriate safety and health measures; and ensure compliance with the conditions stipulated by relevant national regulations.

1 Scope

This Part specifies the principle, reagents and materials, instruments, sampling and sample preparation, analytical procedures, result calculation, etc. of using potassium dichromate titrimetric method and 1,10-phenanthroline spectrophotometric method to determine the total iron content.

This Part is applicable to the determination of total iron in the manganese ores and the manganese concentrates with vanadium content (mass fraction) < 0.02%; the test range (mass fraction): Method-I potassium dichromate titrimetric method is 2.00%~25.00%; while Method-II 1,10-phenanthroline spectrophotometric method is 0.10%~3.00%.

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 2011 Method of Sampling and Sample Preparation of Manganese Ores in Bulk

GB/T 14949.8-1994 Manganese Ores - Determination of Hygroscopic Moisture Content in Analytical Samples

3.2.14 Ammonia water (1+50).

3.2.15 Hydrogen peroxide (volume fraction of 30%).

3.2.16 Tin dichloride solution (100g/L): take 10g of tin dichloride ($SnCl•2H_2O$); add 20mL of hydrochloric acid (3.2.4); heat and dissolve; after cooling off, use water to dilute to 100mL; mix evenly.

3.2.17 Titanium trichloride solution (1+3): pipette 10mL of titanium trichloride solution (15%~20%); add 30mL of hydrochloric acid (3.2.8); mix evenly.

3.2.18 Potassium dichromate standard titration solution.

3.2.18.1 Solution-A [$c(1/6K_2Cr_2O_7) = 0.05mol/L$]: take 2.4517g of reference potassium dichromate, which has been pre-dried for 2h at 150°C, then place into a desiccator, and cool off to the room temperature, into 300mL beaker; add 100mL of water to dissolve; transfer into 1000mL volumetric flask; use water to dilute to the scale; mix evenly. 1mL of such solution is equivalent to 0.002792g of iron.

3.2.18.2 Solution-B [$c(1/6K_2Cr_2O_7) = 0.02mol/L$]: take 0.980.7g of reference potassium dichromate, which has been pre-dried for 2h at 150°C, then place into a desiccator, and cool off to the room temperature, into 300mL beaker; add 100mL of water to dissolve; transfer into 1000mL volumetric flask; use water to dilute to the scale; mix evenly. 1mL of such solution is equivalent to 0.001117g of iron.

3.2.19 Iron standard solution: take 1.0000g of high-purity metallic iron (99.99%) or 1.4300g of high-purity ferric oxide (99.99%), which has been pre-dried for 2h at 105°C~110°C, then place into a desiccator, and cool off to the room temperature, into 200mL beaker; add 50mL of hydrochloric acid (3.2.7); heat and dissolve at low temperature; after cooling off, transfer into 1000mL volumetric flask. Use water to dilute to the scale, mix evenly. 1mL of such solution is equivalent to 1mg of iron.

3.2.20 Neutral red indicator solution (1g/L).

3.2.21 Sodium diphenylamine sulfonate indicator solution (2g/L).

3.3 Instrument

The general laboratory instruments are used for analysis.

3.4 Sampling and sample preparation

The sampling and sample preparation shall be carried out as per the provisions of GB/T 2011; the specimen shall pass through a 0.080mm sieve.

3.5 Analytical procedures

b) The degree of compliance with the provisions of this Standard;

c) Analysis results and their representations;

d) Anomalies observed during the initial determination;

e) There may be effects on the analysis results that are not covered by this Standard, or optional operations.

4 Method II -- 1,10-Phenanthroline Spectrophotometric Method

4.1 Principle

The sample is decomposed by hydrochloric acid; the remaining slag is melted by potassium pyrosulfate. Use hydroxylamine hydrochloride to reduce the ferric iron into ferrous iron. At the acidity of pH 4~5, the ferrous iron and 1,10-phenanthroline forms a red complex; then measure the absorbance at the wavelength of 510nm; calculate the total iron content.

4.2 Reagents and materials

Unless otherwise specified, the reagents determined to be analytically pure and distilled water or water with equivalent purity shall be used in the analysis.

4.2.1 Potassium pyrosulfate.

4.2.2 Hydrofluoric acid (ρ1.14g/mL).

4.2.3 Hydrochloric acid (ρ1.19g/mL).

4.2.4 Hydrochloric acid (1+50).

4.2.5 Sulfuric acid (1+1).

4.2.6 Hydroxylamine hydrochloride solution (100g/L).

4.2.7 1,10-phenanthroline solution (5g/L): take 5g of 1,10-phenanthroline; add 100ml of ethanol to dissolve; use water to dilute to 1000mL, mix evenly.

4.2.8 Acetic acid-sodium acetate buffer solution: take 450g of anhydrous sodium acetate into 500mL of water; add 240mL of glacial acetic acid; dilute with water to 1000mL, mix evenly.

4.2.9 Iron standard solution:

4.5.3.2 Transfer the filter paper and remaining slag into platinum crucible; carbonize and ash at low temperature; burn at 500°C~600°C; cool off, wet with water; add 3~4 drops of sulfuric acid (4.2.5), 5~6mL of hydrofluoric acid (4.2.2); slowly evaporate till the white fumes of sulfur trioxide are completely emitted; cool off. Add 2g of potassium pyrosulfate (4.2.1); cover the stopper, melt at 650°C~700°C till transparent. After cooling off, place the crucible in the main solution and heat it for leaching; wash the crucible by water; after cooling off, transfer into 250mL volumetric flask; dilute with water to the scale, mix evenly.

4.5.3.3 Separately-take test solution into 100mL volumetric flask according to Table 3; add 50mL of water; add 5mL of hydroxylamine hydrochloride solution (4.2.6), mix evenly. Stand for 5min; add 10mL of acetic acid-sodium acetate buffer solution (4.2.8), 10mL of 1,10-phenanthroline solution (4.2.7); stand for 1h at the room temperature or in the 30°C water bath for 15min; cool off; dilute with water to the scale, mix evenly. Measure the absorbance at the wavelength of 510nm by 1cm absorber with water as reference on the spectrophotometer. After deducting the absorbance measured in the blank test (4.5.2); find out the iron content form the calibration curve.

4.5.3.4 Drawing the calculation curve: separately pipette 0mL, 1.00mL, 2.00mL, 4.00mL, 6.00mL, 8.00mL of iron standard solution (4.2.9.2) into a group of 100mL volumetric flaks; add 50mL of water. Then develop color as per 4.5.3.3; measure the absorbance and draw the calibration curve.

4.6 Result calculation

Calculate the total iron content (mass fraction) in the specimen as per Formula (2):

$$w(\mathrm{Fe})(\%) = \frac{m_2 \times 10^{-6}}{m_1 \cdot \gamma} \times 100 \times \frac{100}{100 - A} \qquad \cdots\cdots(2)$$

Where:

m_2 – iron content found out from the calibration curve, in μg;

m_1 – sample mass, in g;

γ – separately-taking rate of test solution;

A – mass fraction of wet storage water in the specimen.

4.7 Tolerance

The difference in analysis results between laboratories shall be no more than the tolerance in Table 4.

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