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Methods for chemical analysis of zinc concentrate roasting - Part 5: Determination of iron content - Na_2EDTA titrimetric method

锌精矿焙砂化学分析方法 第 5 部分：铁量的测定 Na_2EDTA 滴定法

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Methods for chemical analysis of zinc concentrate roasting - Part 5: Determination of iron content - Na_2EDTA titrimetric method

1 Scope

This Part of YS/T 1149 specifies the method for the determination of iron content, in zinc concentrate roasting.

This Part applies to the determination of iron content in zinc concentrate roasting, which has a tin content < 0.40%. The measurement range is 2.00% ~ 20.00%.

2 Method summary

The sample is dissolved in hydrochloric acid, nitric acid, sulfuric acid. It is evaporated to near dryness. The salts are dissolved by hydrochloric acid; ammonia water is added to precipitate iron into ferric hydroxide; it is filtered, to separate coexisting elements. The precipitate is dissolved by diluted hydrochloric acid; the acidity is adjusted. The temperature is controlled at 60 °C ~ 80 °C; the sulfosalicylic acid is used as an indicator; the Na_2EDTA standard titration solution is used for titration, until the solution changes from brown to bright yellow, which is taken as the end point.

3 Reagents

Unless otherwise specified, the reagents used in this Part are analytical reagents and distilled water or deionized water or water of equivalent purity, that meet national standards or industry standards.

3.1 Ammonium chloride.

3.2 Hydrochloric acid (ρ1.19 g/mL).

3.3 Nitric acid (ρ1.42 g/mL).

3.4 Sulfuric acid (ρ1.84 g/mL).

3.5 Ammonia (ρ0.90 g/mL).

3.6 Hydrochloric acid (1 + 1).

3.7 Hydrochloric acid (1 + 11).

3.8 Ammonia (1+1).

3.9 Washing solution: Weigh 25 g of ammonium chloride (3.1). Dissolve it in 500 mL of water. Add 20 mL of ammonia water (3.5). Mix well.

3.10 Sulfosalicylic acid solution (100 g/L).

3.11 Iron standard solution (0.01 g/mL): Weigh 1.4297 g of ferric oxide reference material (pre-dried at 100 °C ~110 °C for 1 h ~ 2 h and cooled to room temperature). Put it in a 300 mL beaker. Add 10 mL of hydrochloric acid (3.6). Heat it at low temperature, until it is completely dissolved. Cool it. Add 10 mL of hydrochloric acid (3.6). Transfer it into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 0.01 g of iron.

3.12 Disodium ethylenediaminetetraacetate (Na_2EDTA) standard titration solution (about 0.025 mol/L).

3.12.1 Preparation: Weigh 10 g of Na_2EDTA ($C_{10}H_4N_2O_8Na_2 \cdot 2H_2O$) in a beaker. Add about 400 mL of hot water to dissolve it. Cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well. Let it stand for 3 days, before calibration.

3.12.2 Calibration: Pipette three parts of 5.00 mL of iron standard solution (3.11) into 300 mL beakers, respectively. Add water to about 120 mL. Use ammonia water (3.8) to neutralize it, until ferric hydroxide precipitate appears. Add 10 mL of hydrochloric acid (3.7). Heat to 60 °C ~ 80 °C. Add 1 mL of sulfosalicylic acid solution (3.10). Use Na_2EDTA standard titration solution to titrate it, until the solution changes from brown to bright yellow, which is taken as the end point.

A blank test is performed along with the calibration.

The actual concentration of Na_2EDTA standard titration solution is calculated, according to formula (1):

$$c = \frac{\rho_{Fe} \cdot V_{Fe} \times 1\,000}{55.85 \times (V_2 - V_1)} \qquad \cdots\cdots (1)$$

Where:

c - The concentration of Na_2EDTA standard titration solution, in mole per liter (mol/L);

5.4.1 Put the sample (5.1) in a 300 mL beaker. Use a small amount of water to wet it (if the sample contains high silica, add 0.2 g of ammonium fluoride). Add 10 mL of hydrochloric acid (3.2). Dissolve it at low temperature for 5 min. Add 10 mL of nitric acid (3.3). Continue to heat to dissolve it completely. Add 5 mL of sulfuric acid (3.4). Heat it until thick white smoke appears. Evaporate it to 2 mL. Remove and cool it. Add 2 mL ~ 3 mL of hydrochloric acid (3.2). Use water to wash the watch glass and the wall of the beaker. Heat to dissolve the salts. Add 4 g ~ 5 g of ammonium chloride (3.1). Add water to a volume of 70 mL ~ 80 mL. Use ammonia water (3.5) to neutralize it, until ferric hydroxide is completely precipitated. Add another 3 mL. Heat it to slight boiling, for 3 min ~ 5 min. Remove it. Add 3 mL of ammonia water (3.5). Use fast filter paper to filter it. Use hot washing solution (3.9) to wash the beaker and filter paper 4 times each. Then use water to wash them once each.

5.4.2 Use hot hydrochloric acid (3.6) to dissolve the precipitate in the original beaker. Then use water and hydrochloric acid (3.6) to alternately wash it, until the filter paper is colorless. Put the solution at a low temperature, to evaporate it to 1 mL ~ 2 mL. Remove it. Add water to about 120 mL. Use ammonia water (3.8) to neutralize it, until ferric hydroxide precipitates. Add 10 mL of hydrochloric acid (3.7). Heat it to 60 °C ~ 80 °C. Add 1 mL of sulfosalicylic acid solution (3.10). Use Na_2EDTA standard titration solution (3.12) to titrate it, until the solution changes from brown to bright yellow, which is taken as the end point.

6 Calculation of analysis results

The amount of iron is expressed as the mass fraction of iron w_{Fe}; the value is expressed as %; it is calculated according to formula (2):

$$w_{Fe}(\%) = \frac{c \times 55.85 \times (V_3 - V_0)}{m} \times 100 \qquad \cdots\cdots (2)$$

Where:

c - The concentration of Na_2EDTA standard titration solution, in mole per liter (mol/L);

V_3 - The volume of Na_2EDTA standard titration solution, which is consumed by the titrating the sample solution, in milliliter (mL);

V_0 - The volume of Na_2EDTA standard titration solution, which is consumed by the titrating the blank test solution, in milliliters (mL);

55.85 - The molar mass of iron, in grams per mole (g/mol);

m - The mass of the sample, in grams (g).

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