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Determination of copper content for brass - Iodimetry

黄铜中铜量的测定 碘量法

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Determination of copper content for brass - Iodimetry

1 Scope

This Standard specifies the method to determine the copper content in brass.

This Standard applies to the determination of copper content in brass. Determination range: 50.00%~97.00%.

This Standard is not intended as a method of arbitration.

2 Method summary

The specimen is dissolved in nitric acid. Trivalent arsenic and antimony are oxidized with bromine. The pH value of the control solution is 3~4. Use ammonium bifluoride to mask iron. Add potassium iodide to act with divalent copper. The precipitated iodine is indicated by starch. Use sodium thiosulfate standard titration solution to titrate.

3 Reagents

Unless otherwise specified, it shall only use the confirmed analytically-pure reagents AND distilled water or deionized water or equivalent-pure water in analysis.

3.1 Ammonia ($\rho=0.90\text{g/mL}$).

3.2 Potassium iodide solution (100g/L).

3.3 Nitric acid (1+2).

3.4 Glacial acetic acid ($\rho=1.05\text{ g/mL}$).

3.5 Hydrofluoric acid ($\rho=1.13\text{ g/mL}$).

3.6 Ammonium bifluoride saturated solution: store in polyethylene bottles.

3.7 Bromine saturated solution.

3.8 Potassium thiocyanate solution (200g/L).

3.9 Starch solution (5g/L).

Preparation: Weigh 5g of soluble starch. Slowly add to 1L of distilled water. Stir and mix well. Boil until the solution is transparent.

3.10 Standard titration solution of sodium thiosulfate [$c(\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}) \approx 0.1\text{mol/L}$].

3.10.1 Preparation

Weigh 250g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$). Dissolve in a 1000mL beaker. Add 2g of anhydrous sodium carbonate. Transfer to a 10L brown reagent bottle. Use freshly boiled cooled distilled water to dilute to about 10L. Shake well. Let stand for more than 10d.

3.10.2 Calibration

Weigh three portions of 0.30g (accurate to 0.0001g) of treated pure copper sheet (mass fraction is $\geq 99.95\%$). Determine simultaneously with the specimen according to 5.3.1~5.3.4. Carry out a blank test along with the calibration.

NOTE: Processing of pure copper sheets: Put the pure copper sheet into slightly boiling glacial acetic acid (1+4) and boil for 1min. Rinse twice with water and absolute ethanol after removal. Bake in a 100°C oven for 4min. Cool down. Store in a ground-mouth reagent bottle for later use.

Calculate the actual concentration of sodium thiosulfate standard titration solution according to formula (1):

$$c = \frac{w \times m \times 1\,000}{(V_1 - V_0) \times 63.55} \dots\dots\dots (1)$$

Where,

c - Actual concentration of the standard titration solution of sodium thiosulfate, in moles per liter (mol/L);

w - Mass fraction of copper in pure copper sheet, %;

m - Mass of pure copper sheet, in grams (g);

V_1 - Volume of sodium thiosulfate standard titration solution consumed by titration of pure copper solution during calibration, in milliliters (mL);

V_0 - Volume of sodium thiosulfate standard titration solution consumed by titrating copper blank solution during calibration, in milliliters (mL);

63.55 - Molar mass of copper, in grams per mole (g/mol).

3.11 Parallel calibration

Conduct parallel calibration in triplicate. When the relative error of the measured value is not more than 0.1%, take the average value; otherwise re-calibrate.

NOTE: The sodium thiosulfate standard titration solution must be re-calibrated every other week.

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