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Methods for chemical analysis of iron, steel and alloy - The sodium carbonate separation-diphenyl carbazine photometric method for the determination of chromium content

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Methods for chemical analysis of iron, steel and alloy

- The sodium carbonate separation-diphenyl carbazide photometric method for the determination of chromium content

1 Subject and scope

This Standard specifies the sodium carbonate separation-diphenyl carbazide photometric method for the determination of chromium content.

This Standard is applicable to the determination of chromium content in carbon steel, low alloy steel and precision alloy. The determination range: 0.005% ~ 0.500%.

2 Method summary

In the sulfuric acid solution, use potassium permanganate to oxidize chromium to hexavalent. High-price chromium and diphenylcarbazide generate purple complex. Measure its absorbance. Pre-use sodium carbonate precipitation to precipitate and separate iron and other elements coexist.

When 400 mg of iron, 60 mg of nickel, 40 mg of cobalt, 1 mg of copper, 2 mg of molybdenum, aluminum, 12 mg of tungsten coexist, it shall have no effect on determination of chromium after separation.

3 Reagents

- 3.1** Hydrochloric acid (ρ 1.19 g/mL)
- 3.2** Nitric acid (1+3)
- 3.3** Sulfuric acid (1+1)
- 3.4** Sulfuric acid (1+6)
- 3.5** Potassium permanganate solution (1%)
- 3.6** Sodium carbonate solution (20%)

3.7 Urea solution (20%)

3.8 Diphenyl carbazide solution (0.25%): weigh 0.25 g of diphenyl carbazide to dissolve in 94 mL of absolute ethanol and 6 mL of glacial acetic acid (ρ 1.05 g/mL). Store in a brown bottle.

3.9 Sodium nitrite solution (2%)

3.10 Chromium standard solution

3.10.1 Weigh 0.2829 g of potassium dichromate (reference) that have been dried to constant weight at 150°C. Dissolve in water. Remove to a 1000 mL volumetric flask. Use water to dilute to scale. Well mix. This solution shall contain 100 μ g of chromium per 1 mL.

3.10.2 Weigh 20.00 mL of chromium standard solution (3.10.1) into a 1000 mL volumetric flask. Use water to dilute to scale. Well mix. This solution shall contain 2 μ g of chromium per 1 mL.

4 Analysis steps

4.1 Sample quantity

Weigh 0.2000 ~ 0.3000 g of sample (when the chromium content in the sample is less than 0.01%, it shall weigh 0.3000 or 0.4000 g of sample).

4.2 Blank test

Perform the blank test with sample.

4.3 Determination

4.3.1 Sample dissolving

Place the sample (4.1) in to a 200 mL beaker. Add into 10 mL of nitric acid (3.2) to heat and dissolve [it shall add hydrochloric acid (3.1) to assist dissolving it is difficult to dissolve]. Add 5 mL of sulfuric acid (3.3). Heat to evaporate till sulfuric acid smokes. Cool for little while. Add 30 mL of water to heat and dissolve the salt.

4.3.2 Oxidation and separation

Add 2 mL of potassium permanganate solution (3.5). Boil till manganese dioxide is completely precipitated. Use water to dilute to 80 ~ 90 mL. While stirring, slowly add 30 mL of sodium carbonate solution (3.6) (30 mL of sodium carbonate solution is usually sufficient, but it shall have an excess of 5 mL when sodium carbonate solution precipitates; if 30 mL is not enough,

add a few milliliters). Slowly to heat till it is boiling 2 ~ 3 min. Cool to room temperature. Move to a 250 mL volumetric flask. Dilute with water to scale and mix well.

Use double-layer medium-density filter paper to carry out the dry filtration. Discard the original filtrate. Pipette the filtrate (pipette 50.00 mL when the chromium content is less than 0.01%, 25.00 mL when it is between 0.01% ~ 0.1%, 10.00 mL when it is between 0.1% ~ 0.2%, 5.00 mL when it is more than 0.2%) into a 100 mL volumetric flask. Add 4.0 mL of sulfuric acid (3.4). At this point, if the solution is pink, there may be some potassium permanganate is not decomposed, then add 5 mL of urea solution (3.7). Carefully drop sodium nitrite solution (3.9) until the test solution is colorless and then an excess of 1 drop.

4.3.3 Color

Use water to dilute to about 90 mL. Add 3.0 mL of diphenyl carbazide solution (3.8). Well mix. Use water to dilute to scale. Well mix.

4.3.4 Absorbance measurement

Move the partial solution into a 2 ~ 3 cm absorption dish. Take water as a reference. Measure its absorbance at a wavelength of 540 nm on spectrophotometer. Subtract the absorbance of the blank solution along with the sample, and check the corresponding chromium content in the color developing solution from the working curve.

4.4 Drawing of working curve

Pipette 0, 1.00, 2.00, 4.00, 7.00, 10.00 mL of chromium standard solution (3.10.2) into 6 100 mL flasks, respectively. Add 4.0 mL of sulfuric acid (3.4). The followings shall be carried out according to 4.3.3 and 4.3.4. Use reagent blank as reference to measure its absorbance. Take absorbance as the vertical axis, the corresponding chromium content as abscissa to draw the working curve.

5 Calculation of analysis results

Calculate the percentage of chromium by the following formula:

$$\text{Cr}(\%) = \frac{m_1 \cdot V}{m \cdot V_1} \times 100$$

where,

V_1 - volume of sub-test solution, mL;

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