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NATIONAL STANDARD OF THE
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**Lithium carbonate - Determination of calcium,
magnesium, copper, zinc, nickel, manganese and
cadmium contents - Ion exchange-flame atomic
absorption spectrometric method**

碳酸锂、单水氢氧化锂、氯化锂化学分析方法

离子交换火焰原子吸收光谱法 测定钙、镁、铜、锌、镍、锰、镉量

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Lithium carbonate - Determination of calcium, magnesium, copper, zinc, nickel, manganese and cadmium contents - Ion exchange-flame atomic absorption spectrometric method

1 Scope

This standard specifies the determination method of calcium, magnesium, copper, zinc, nickel, manganese and cadmium in lithium carbonate.

This standard applies to the determination of calcium, magnesium, copper, zinc, nickel, manganese and cadmium content in phosphor-grade lithium carbonate, as well as the individual determination of the content of one element. Measurement range: calcium 0.00010% ~ 0.010%; magnesium, cadmium, copper, zinc, nickel, manganese 0.00010% ~ 0.0060%.

2 Normative references

GB 1.4 Directives for the work of standardization - Rules for drafting chemical analysis standards

GB 1467 General rules and regulations of the chemical analysis methods for products of metallurgical industries

GB 7728 Chemical analysis of metallurgical products - General rule for flame atomic absorption spectrometric methods

3 Method summary

The sample is decomposed by hydrochloric acid and adjusted to pH 8 ~ 9. Use the potassium chelating resin to separate and enrich the trace precious impurity elements calcium, magnesium, copper, nickel, manganese, zinc and cadmium. The adsorbed impurity elements are respectively eluted by hydrochloric acid (0.2 mol/L and 1.0 mol/L). At the wavelengths listed in atomic absorption spectrometer Table 1, use an air-acetylene flame to measure each element separately.

The standard solution shall have the same acidity as the elution solution.

4.12 Calcium standard solution:

4.12.1 Pipette 25.00 mL of calcium standard stock solution (4.11). Place it in a 250 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 100 µg of calcium.

4.12.2 Pipette 10.00 mL of calcium standard stock solution (4.12.1). Place it in a 100 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 10 µg of calcium.

4.13 Magnesium standard stock solution: Weigh 1.000 g pure metallic magnesium (above 99.9%). Place it in a 300 mL beaker. Slowly add 34 mL of hydrochloric acid (4.1) to dissolve it. Heat to boil. Cool to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 1 mg of magnesium.

4.14 Magnesium standard solution:

4.14.1 Pipette 25.00 mL of magnesium standard stock solution (4.13). Put it in a 250 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 100 µg of magnesium.

4.14.2 Pipette 10.00 mL of magnesium standard stock solution (4.14.1). Put it in a 100 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 10 µg of magnesium.

4.15 Copper standard stock solution: Weigh 1.000 g of pure metallic copper (above 99.9%) in a 300 mL beaker. Add 20 mL of nitric acid (1 + 1, superior grade pure) to dissolve. Heat to evaporate it dry. Add 10.0 mL of sulfuric acid. Heat carefully to evaporate to sulfuric acid fumes. Cool it. Add water and boil until the salt is completely dissolved. Cool to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 1 mg of copper.

4.16 Zinc standard stock solution: Weigh 1.000 g of pure metallic zinc (above 99.9%) in a 300 mL beaker. Add 25.0 mL of hydrochloric acid (1 + 1, superior grade pure) to dissolve it. Heat to boil it. Cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 1 mg of zinc.

4.17 Nickel standard stock solution: Weigh 1.000 g of pure metallic nickel (above 99.9%) in a 300 mL beaker. Add 15 mL of nitric acid (ρ_{1.42} g/mL, superior grade pure). Heat at low temperature to completely dissolve it. Boil it. Cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 1 mg of nickel.

4.18 Manganese standard stock solution: Weigh 1.000 g of pure metallic manganese (above 99.9%, use diluted sulfuric acid to wash off the oxide from the surface of the manganese before weighing; then use water to wash off the acid; bake it dry). Place it in a 300 mL beaker. Add 20 mL of hydrochloric acid (1 + 1, superior grade pure). Heat 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 it uniformly. Each 1 mL of this solution contains 1 mg of manganese.

4.19 Cadmium standard stock solution: Weigh 1.000 g of pure metallic cadmium (above 99.9%). Place it a 300 mL beaker. Add 25.0 mL of hydrochloric acid (4.1) to dissolve it. Heat to boil it. Cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 1 mg of cadmium.

4.20 Mixed standard solution:

4.20.1 Respectively pipette 25.00 mL of copper standard stock solution (4.15), zinc standard stock solution (4.16), nickel standard stock solution (4.17), manganese standard stock solution (4.18), cadmium standard stock solution (4.19) in a 250 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 100 µg of copper, zinc, nickel, manganese, cadmium.

4.20.2 Pipette 10.00 mL of mixed standard solution (4.21.1) in a 100 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. Each 1 mL of this solution contains 10 µg of copper, zinc, nickel, manganese, cadmium.

4.21 Ion exchange column: Made of polyethylene tube, as shown in the Figure below.

7.3.1.1.

7.3 Determination

7.3.1 Separation and enrichment:

7.3.1.1 Calcium and magnesium: Put the sample in a beaker. Use a small amount of water to moisten it. Add hydrochloric acid (4.1) according to Table 2 to decompose it. Heat to boil to drive off carbon dioxide. Remove the beaker. Respectively add water to about 40 mL. Cool to room temperature. Use ammonia (4.8) and hydrochloric acid (4.9) to adjust to pH 8 ~ 9 [use pH test paper (4.10) to test]. Transfer the test solution to the processed exchange column (4.22.1). Make it exchange at the flow rate of 1 ~ 2 mL/min. When the test solution flows to the resin surface, use water to rinse it twice, 10 mL each time. Add 40 mL acetic acid (4.5) to wash off and discard lithium. Then use water to rinse the resin twice, 10 mL each time. Use 15.00 mL of hydrochloric acid (4.2) to wash off calcium and magnesium. Collect them in a 25 mL dry colorimetric tube. Mix it uniformly.

7.3.1.2 Copper, nickel, manganese, zinc, cadmium: The sample is carried out according to 7.3.1.1, but finally eluted by 15.00 mL hydrochloric acid (4.3).

7.3.2 At the wavelength listed in atomic absorption spectrometer Table 1, use air-acetylene flame, parallel to the standard solution series; respectively use hydrochloric acid (4.2) to make zero adjustment to measure the absorbance of calcium and magnesium; use with hydrochloric acid (4.3) to make zero adjustment to measure the absorbance of copper, zinc, nickel, manganese, cadmium; find the concentration of the corresponding elements in the test solution and the blank solution from the working curve.

7.4 Drawing of working curve

7.4.1 Calcium working solution

7.4.1.1 Pipette 0, 2.00, 4.00, 6.00, 8.00, 10.00 mL of calcium standard solution (4.12.2) into a set of 100 mL volumetric flasks containing 2.85 mL hydrochloric acid (4.1), respectively. Use water to dilute it to the mark. Mix it uniformly. Transfer the solution into a plastic bottle for storage.

7.4.1.2 Pipette 0, 2.00, 4.00, 6.00, 8.00, 10.00 mL of calcium standard solution (4.12.1) into a set of 100 mL volumetric flasks containing 2.85 mL hydrochloric acid (4.1), respectively. Use water to dilute it to the mark. Mix it uniformly. Transfer the solution into a plastic bottle for storage.

7.4.2 Magnesium working solution:

Pipette 0, 2.00, 4.00, 6.00, 8.00, 10.00 mL of magnesium standard solution

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