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**Determination of Mercury in Inorganic Chemical
Product - Atomic Fluorescence Spectrometry**

无机化工产品中汞的测定

原子荧光光谱法

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Determination of Mercury in Inorganic Chemical Product - Atomic Fluorescence Spectrometry

Warning: personnel adopting this Standard shall have practical experience of working at regular laboratories. This Standard does not point out all the possible safety problems. Users shall undertake the responsibility of adopting appropriate safety and health measures; guarantee the compliance with the conditions stipulated by relevant national laws and regulations. Some reagents adopted in the test method are toxic or corrosive, operators shall be cautious! If they splash onto the skin, immediately use water to rinse them; medical treatment shall be necessary under severe circumstances. The test method adopts high-pressure argon cylinder, which shall be operated in accordance with the safety procedure of high-pressure steel cylinder.

1 Scope

This Standard specifies the principle, reagents or materials, instruments and equipment, test procedure, instrument reference conditions and test data processing of determining mercury content in inorganic chemical products through atomic fluorescence spectrometry.

This Standard is applicable to the determination of mercury content in inorganic chemical products through atomic fluorescence spectrometer. In the test solution for determination, mercury (Hg) mass concentration shall be within the range of 0.04 µg/L ~ 10 µg/L.

2 Normative References

The following documents are indispensable to the application of this Standard. In terms of references with a specified date, only versions with a specified date are applicable to this Standard. The latest version (including all the modifications) of references without a specified date is applicable to this Standard.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

HG/T 3696.2 Inorganic Chemicals for Industrial Use - Preparations of Standard and Reagent Solutions for Chemical Analysis - Part 2: Preparations of Standard Solutions for Impurity

HG/T 3696.3 Inorganic Chemicals for Industrial Use - Preparations of Standard and Reagent Solutions for Chemical Analysis - Part 3: Preparations of Reagent

keep away from light.

5.8 Mercury standard using solution: 1 mL of solution contains 0.05 µg of mercury (Hg); transfer 0.50 mL of mercury standard intermediate solution, place it in a 100 mL volumetric flask. Use potassium dichromate-nitric acid solution to dilute to the volume, then, mix it up. This solution shall be prepared right before usage.

6 Instruments and Equipment

Atomic fluorescence spectrometer: equipped with mercury hollow cathode lamp.

7 Test Procedures

7.1 Pre-cleaning of Glass Instruments

Before usage, glass vessels and PTFE digestion inner tank, which are about to be used in test, shall be soaked in nitric acid solution for 24 h. Then, use water to rinse them.

7.2 Preparation of Test Solutions

Test solutions shall be prepared in accordance with the stipulation in relevant product standards.

7.3 Sample Pre-treatment Method

Sample pre-treatment method shall refer to Appendix A.

7.4 Standard Curve Drawing

Respectively transfer 0.00 mL, 2.00 mL, 4.00 mL, 6.00 mL and 8.00 mL of mercury standard using solution; place them into 5 100 mL volumetric flasks. Respectively add 5 mL of hydrochloric acid; dilute to the constant volume, then, mix it up. In this series of solutions, mercury (Hg) mass concentration shall respectively be 0.00 µg/L, 1.00 µg/L, 2.00 µg/L, 3.00 µg/L and 4.00 µg/L. Standard solutions can also be prepared in accordance with the practically determined mercury content.

After the instrument is stabilized, use potassium borohydride (or sodium borohydride) solution as the reducing agent; take hydrochloric acid solution as the carrier solution. Under the optimal working conditions of the instrument, determine the fluorescence value of the standard solution. Take the measured fluorescence value as the y-coordinate; take the corresponding mercury mass concentration (µg/L) as the x-coordinate; draw a standard curve.

7.5 Test

Appendix A

(Informative)

Sample Pre-treatment Method

A.1 Open Container Digestion Method

Heat up and digest samples and reagents on a heating plate or electric furnace through an open container.

A.2 Sealed Container Digestion Method

In a sealed container (generally, PTFE material), use acid or other reagents to conduct wet digestion under raised temperature and pressure.

A.3 Microwave Digestion Method

Under the effect of microwave and the condition of sealed pressurization, mix up sample and acid, till they are completely digested.

A.4 Alkali Melting Method

Use alkali melting reagents, such as lithium metaborate, lithium tetraborate, sodium carbonate, sodium hydroxide, sodium peroxide, and corresponding fluoride of potassium salt and alkali metal (for example, potassium bifluoride); mix up with sample; melt them at high temperature.

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