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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 71.060.01

G 10

GB/T 30902-2014

**Inorganic chemicals for industrial use - Determination of
impurity element - Inductively coupled plasma optical
emission spectrometry (ICP-OES)**

无机化工产品 杂质元素的测定

电感耦合等离子体发射光谱法(ICP-OES)

Issued on: July 08, 2014

Implemented on: December 01, 2014

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine;
Standardization Administration of the People's Republic of China.**

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Inorganic chemicals for industrial use - Determination of impurity element - Inductively coupled plasma optical emission spectrometry (ICP-OES)

Warning: Some of the reagents used in this test method are toxic or corrosive, so be careful when operating! If it splashes on the skin, rinse it with water immediately, and in serious cases, treat it immediately; high-pressure argon gas cylinders are used in this test method and shall be operated according to the safety operation regulations of high-pressure cylinders; after igniting the plasma, the torch chamber door shall not be opened to prevent high-frequency radiation from harming the body; pay attention to safe use of electricity.

1 Scope

This Standard specifies the principle, reagents, instruments, apparatuses, analysis steps, precision, and recovery rate for the determination of metallic and non-metallic impurity elements in inorganic chemicals for industrial use using inductively coupled plasma optical emission spectrometry (ICP-OES).

This Standard applies to direct injection of liquid samples containing various impurities in inorganic chemicals for industrial use or test solutions after removing the matrix, using an inductively coupled plasma optical emission spectrometer for measurement.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 4470, Analytical spectroscopic methods - Flame emission, atomic absorption and atomic fluorescence - Vocabulary

GB/T 4842, Argon

GB/T 6379.2, Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

GB/T 6682-2008, Water for analytical laboratory use - Specification and test methods

The air flow between the outermost tube and the middle tube of the torch, which serves to cool the torch and maintain the plasma.

3.7

auxiliary gas

The gas flow between the middle tube and central tube of the torch, which serves to ignite the plasma, keep the bottom of the high-temperature ICP at a certain distance from the central tube and the middle tube, protect the tops of the central tube and the middle tube, especially the central tube mouth, from being melted or overheated, and reduce the excessive deposition of salt carried by aerosols on the central tube mouth. In addition, it also plays a role in raising the ICP and changing the plasma observation degree.

3.8

carrier gas

The gas flow in the central tube of the torch, which serves to atomize the liquid into an aerosol and carries the aerosol into the plasma.

3.9

washing time

The time to flush the sampling system with sample solution before exposure.

4 Principle

After the liquid sample is brought into the atomization system by the carrier gas for atomization, it enters the axial channel of the plasma in the form of an aerosol, and is fully evaporated, atomized, ionized and excited in the high temperature and inert gas. The characteristic spectral lines of the emitted elements enter the spectrum detector through the spectroscopic system, and the spectrum detector performs qualitative, semi-quantitative, and quantitative analysis methods based on the characteristic spectrum.

5 Reagents

5.1 Unless other requirements are specified, the reagents used refer to reagents of analytical grade or above. Inorganic acids, such as hydrochloric acid, nitric acid, perchloric acid, hydrofluoric acid, are commonly used in sample processing. They shall be checked before use to ensure that they do not contain the metal elements to be measured.

5.2 Laboratory water shall meet the specifications for water of grade 2 or above in GB/T 6682-2008.

5.3 Standard stock solution: The standard stock solution of each analytical element shall be prepared according to the regulations in HG/T 3696.2. Alternatively, mixed solutions and single standard solutions of certified series of national standard materials with corresponding concentrations can be used, and diluted to the required concentration. See Appendix A for the preparation of multi-element group standard solutions.

6 Instruments and apparatuses

Inductively coupled plasma atomic emission spectrometer: consisting of a sampling system, an excitation light source, an optical system, a detection system and a data processing system.

7 Procedure

7.1 Selection of measurement conditions

7.1.1 Analysis spectral line of elements to be measured

Refer to Appendix B for the wavelength of the analysis spectral line of elements to be measured.

7.1.2 Incident power

Select the optimal power according to the characteristics of the sample to be tested and the instrument conditions. The general range is 0.8 kW ~ 1.6 kW.

7.1.3 Observation height

The distance from the upper end of the induction coil to the measuring axis, which is generally 14 mm ~ 18 mm. When measuring a single element, the optical observation height of the element to be measured shall be selected; when measuring multiple elements, a moderate observation height shall be selected.

7.1.4 Solution lifting rate

The solution lifting rate is generally 0.6 mL/min ~ 2 mL/min.

7.1.5 Gas flow

Determine the optimal flow rate of each gas according to the torch and analysis requirements. The argon gas used shall comply with the requirements of GB/T 4842.

7.1.6 Analysis time

7.3.2.3 Microwave digestion method

Under the action of microwave (generally in the frequency of 2 450 MHz) and under closed pressure conditions, after the mixture of sample and acid absorbs microwave heat, the boiling point of the inorganic acid increases, and then the oxidation reaction activity increases, causing the surface layer of the sample to agitate and rupture, constantly producing new sample surfaces that are in contact with the acid solvent until the sample is completely digested and then undergo acid treatment.

7.3.2.4 Alkali metal fusion method

Use various alkali metal solvents, such as lithium metaborate, lithium tetraborate, sodium carbonate, sodium hydroxide, sodium peroxide, corresponding potassium salts and alkali metal fluorides (especially potassium bifluoride), to mix with the sample and melt at high temperature.

7.3.2.5 Separation and preconcentration methods

The separation process can remove possible matrix effects and interference, achieve preconcentration, and reduce the quantitation-limit. The main methods include solvent extraction, ion exchange, and co-precipitation/adsorption.

7.3.3 Liquid solution requirements

After the sample is processed, adjust the volume to an appropriate volume according to the content of the element to be measured, and make the sample solution.

The sampling volume is determined based on the mass concentration of the element to be measured in the sample and the detection limit of the method; the mass concentration of the element to be measured in the sample solution shall be at least three times the detection limit of the element. See Appendix C for the determination method of detection limit.

7.4 Determination

7.4.1 Qualitative analysis

Based on three or more sensitive lines of the element to be measured in the spectrum, qualitatively determine whether the element to be measured is present. Generally, there are two qualitative methods: comparative spectral line method and semi-automatic qualitative analysis.

7.4.2 Semi-quantitative analysis

The approximate content of the element to be measured in the sample can be measured, and semi-quantitative analysis results can be obtained using the software provided by the ICP-OES instrument. Generally, there are two semi-quantitative analysis methods: partial calibration method and persistence curve method.

7.4.3 Quantitative analysis

7.4.3.1 Standard curve method

According to the provisions of the product standard, prepare a sample solution, a blank test solution and three or more standard series solutions of different concentrations (all shall be prepared in the same matrix, generally using 1% ~ 5% dilute nitric acid as the medium); under the specified instrument conditions, measure the corresponding emission intensity values, respectively. Draw a standard curve with the mass concentration of the standard solution ($\mu\text{g/mL}$) as the abscissa and the corresponding emission intensity value as the ordinate. Find the mass concentration of the element to be measured in the sample solution on the standard curve, and then calculate the content of the element to be measured in the sample.

7.4.3.2 Internal standard calibration standard curve method

This method uses one element as a reference point to calibrate the measurement of another element or multiple elements. Add internal standard (ISTD) elements of the same concentration (generally using 1% ~ 5% dilute nitric acid as the medium) to the standard solution, sample solution and blank test solution of the element to be measured; under the specified instrument conditions, respectively measure the emission intensity values of corresponding concentrations. Taking the ratio of the emission intensity value of the standard solution of the element to be measured and the emission intensity value of the internal standard element as the ordinate, and the corresponding mass concentration ($\mu\text{g/mL}$) as the abscissa, draw the internal standard calibration standard curve and calculate the regression equation. Using the ratio of the emission intensity value of the element to be measured and the emission intensity value of the internal standard element in the test solution, after subtracting the reagent blank, find the mass concentration of the element to be measured in the sample solution from the internal standard calibration standard curve, and then calculate the content of the element to be measured in the sample.

7.4.3.3 Standard addition method

Prepare the test solution and blank test solution according to the provisions for the element to be measured in the product standard. Use a pipette to pipette 5 portions of test solution of the same volume and place them in 5 volumetric flasks of the same volume. Except for the first volumetric flask, use a pipette to pipette proportioned standard solutions of the element to be measured into the other 4 volumetric flasks (usually using 1% ~ 5% dilute nitric acid as the medium); dilute to the mark respectively; shake well. Under the specified instrument conditions, use the blank test solution for zero setting, and respectively measure the corresponding emission intensity values. Taking the mass concentration ($\mu\text{g/mL}$) of the added standard solution as the abscissa and the corresponding emission intensity value as the ordinate, draw a working curve; extend the curve in the opposite direction and intersect the horizontal axis. The distance

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