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**General Rules for Quadrupole Inductively Coupled Plasma
Mass Spectrometry**

四级杆电感耦合等离子体质谱方法通则

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General Rules for Quadrupole Inductively Coupled Plasma Mass Spectrometry

1 Scope

This Standard specifies the methods and principles, reagents and materials, instruments and equipment, sample requirements, analytical procedures and methods, result report, and safety precautions for the detection of metallic and some non-metallic elements in samples using quadrupole inductively coupled plasma mass spectrometer (abbreviated as Q-ICP-MS).

This Standard applies to the qualitative, semi-quantitative, and quantitative analysis of elements in samples using a quadrupole inductively coupled plasma mass spectrometer and in the mode of liquid and gas injection.

This Standard does not apply to the mode of direct solid injection.

2 Normative References

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

GB/T 602 Chemical Reagent - Preparations of Standard Solutions for Impurity

GB/T 4842 Argon

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

GB/T 13966 Terminology for Analytical Instruments

GB/T 27411 Routine Methods for Evaluation and Expression of Measurement Uncertainty in Testing Laboratory

JJF 1159 Calibration Specification for Quadrupole Inductively Coupled Plasma Mass Spectrometers

3 Terms and Definitions

The terms and definitions defined in GB/T 13966, GB/T 27411 and JJF 1159, and the following terms and definitions are applicable to this document.

3.1 plasma

A conical component that combines plasma with a high-vacuum mass spectrometer. There is a circular hole approximately 1 mm in diameter at the center, which utilizes a pressure differential to draw most of the ions in the central channel of the plasma into the first-stage vacuum chamber.

3.12 skimmer cone

A conical component that connects the sampler cone to the high-vacuum mass spectrometer. There is a circular hole less than 1 mm in diameter at the center, concentric with the axial direction of the sampler cone hole, which introduces the central part of the expanded ion jet through the sampler cone hole into the mass spectrometer.

3.13 ion lens

An electric and / or magnetic field device that focuses and images the ion beam.

[GB/T 13966-2013, Definition 6.104]

3.14 ion detector

A component used to collect and / or amplify the ion stream from the mass analyzer.

[GB/T 13966-2013, Definition 6.126]

3.15 quadrupole mass analyzer

mass filter

quadrupole mass filter

A mass analyzer consisting of a quadrupole field composed of a superimposed DC and RF field. Under given field parameters, ions of different mass-to-charge ratios are in stable or unstable orbital motion within the field. Stable ions are collected, while unstable ions are filtered out.

[GB/T 13966-2013, Definition 6.123]

3.16 mass discrimination effect

In mass spectrometry, the phenomenon that separation based on mass-to-charge ratio cannot be completely achieved.

[GB/T 13966-2013, Definition 6.169]

4 Principle of Analytical Method

This is an inorganic multi-element analysis technique using inductively coupled plasma as the ion source and quadrupole mass analyzer for detection. The specimen is introduced into the plasma via a carrier gas in a certain form, where it undergoes de-solvation, vaporization, dissociation, and ionization in a high-temperature, inert atmosphere. A portion of the plasma

passes through different pressure zones and enters a vacuum system. Within the vacuum system, positive ions are extracted and separated in accordance with their mass-to-charge ratio before being detected by an ion detector. Qualitative analysis can be performed based on the mass-to-charge ratio, while quantitative analysis can be performed based on the detection signal at a specific mass-to-charge ratio.

5 Reagents and Materials

5.1 Argon

Shall comply with the requirements of GB/T 4842, with a volume fraction of $\geq 99.99\%$.

5.2 Water

Water for trace element analysis shall comply with GB/T 6682, Grade 1. Water for other tests shall comply with GB/T 6682, Grade 2.

5.3 Hydrochloric Acid

Guaranteed reagent or higher specifications shall be selected.

5.4 Nitric Acid

Guaranteed reagent or higher specifications shall be selected.

5.5 Other Reagents

Other reagents used in the analysis shall be analytically pure or higher specifications.

5.6 Standard Solutions

5.6.1 Standard stock solutions

5.6.1.1 Single-element standard stock solution

A certified single-element standard solution (with a concentration of 1,000 $\mu\text{g/mL}$ or 100 $\mu\text{g/mL}$) may be used. Where appropriate, high-purity metals (with a purity greater than or equal to 99.99%), oxides or salts (reference or high-purity reagents) may be used to prepare the solution in accordance with GB/T 602.

5.6.1.2 Multi-element standard stock solution

A certified multi-element standard solution may be used. Where appropriate, the solution may be prepared by mixing the single-element standard stock solution (see 5.6.1.1). When mixing the single-element standard stock solution, cross-contamination of the ions being determined and the effects of anions in the solution shall be considered to avoid the generation of poorly or slightly soluble substances.

5.6.2 Standard series solutions

Dilute the single-element standard stock solution (see 5.6.1.1) or the multi-element standard stock solution (see 5.6.1.2) into standard series solutions of varying concentrations, with the final solution containing 1% ~ 5% (volume fraction) acid medium.

6 Instruments and Equipment

6.1 Overview

The main components of a quadrupole inductively coupled plasma mass spectrometer shall include sample injection system, ion source, interface, ion focusing system, quadrupole mass analyzer, vacuum system, detection system, control and data processing system, and an optional collision / reaction system. The instrument shall be equipped with a ventilation system and a power supply system.

6.2 Sample Injection System

This system includes three modes for sample injection: liquid, gas and solid. The mode of liquid injection is preferred. Its main components shall include peristaltic pump, nebulizer and spray chamber.

6.3 Ion Source

An ion source shall be provided to provide specimen ionization energy. The ion source shall consist of radio frequency generator, coupled coil, plasma torch tube and gas supply system.

6.4 Interface

There shall be two types: sampler cone and skimmer cone. An extraction lens or a second skimmer cone can be added after the skimmer cone.

6.5 Ion Focusing System

An ion lens or a 90° ion deflection system shall be used.

6.6 Quadrupole Mass Analyzer

It shall consist of four parallel metal or metal-plated rods, with two rods arranged in pairs.

6.7 Vacuum System

It shall consist of mechanical pump, molecular turbopump, various high-performance sealing valves, and vacuum pipeline, etc.

6.8 Detection System

It shall consist of ion collector and amplifier.

6.9 Control and Data Processing System

It shall consist of computer and corresponding software to realize instrument operation, adjustment and control of various parameters, determination and data processing, etc.

6.10 Collision / Reaction System

It shall utilize collisions and / or reactions between molecules and ions to eliminate polyatomic and / or molecular ion interference from the ion beam.

6.11 Power Supply System

It shall be able to provide the mass spectrometer with a regulated voltage and current power supply, quadrupole DC power supply, high-frequency AC power supply and plasma RF power supply, and good automatic control and protection for the instrument.

6.12 Instrument Type

The following types of instruments may be used:

- a) Single quadrupole inductively coupled plasma mass spectrometers with only one quadrupole capable of mass screening;
- b) Multi-quadrupole inductively coupled plasma mass spectrometers with two or more quadrupoles capable of mass screening.

6.13 Instrument Verification or Calibration

Before commissioning, equipment that may affect the accuracy or validity of the test and analysis results, as well as auxiliary measurement equipment used to measure environmental conditions, shall be verified or calibrated. Verification or calibration shall be performed in accordance with the requirements of JJF 1159.

7 Samples

7.1 Liquid Samples

7.1.1 Direct determination

7.1.1.1 Inorganic solution samples containing the component to be analyzed within the linear range of the instrument analysis may be directly injected for determination. Samples containing suspended matter, such as groundwater, tap water and surface water, shall be filtered prior to injection for determination.

7.1.1.2 For organic solutions, if the instrument is equipped with a sample injection system for organic matter, direct injection for determination is permitted. If the instrument is not equipped with the sample injection system for organic matter, the sample shall be treated before being

injected for determination.

7.1.2 Determination after dilution or concentration

If the content of the element to be determined in the sample is excessively high and exceeds the calibration curve range, the sample shall be appropriately diluted before determination. If the content of the element to be determined in the sample is below the quantification limit, it can be concentrated and enriched for determination.

7.1.3 Determination after digestion

When determining the element to be determined in liquid samples containing organic matter, other special media, or suspended matter, the sample shall be digested using wet, dry or microwave digestion methods before determination.

7.2 Solid Samples

7.2.1 When determining the content of soluble elements, the sample shall be immersed or extracted with a certain volume of an appropriate solvent. After such treatment, take the supernatant and carry out the determination in accordance with the requirements of 7.1.

7.2.2 When determining the total amount of the element to be determined, the sample shall be digested using an appropriate method before analysis. Given the diversity of solid sample types, appropriate treatment methods shall be selected based on the sample's characteristics, such as wet digestion, dry digestion or microwave digestion. The sample size shall be determined based on the salt tolerance of the instrument, the precision of the analytical method and the accuracy requirements of the analysis results.

7.3 Gaseous Samples

Direct sample injection for determination, absorption or enrichment by an absorption liquid or gas sampling membrane may be selected for determination. When using an absorption liquid or gas sampling membrane, the absorption liquid or membrane shall be treated in accordance with the requirements of 7.1 or 7.2.

7.4 Experimental Procedure Blank

It shall be identical to the sample treatment process, using the same reagents, volumes and treatment steps. The corresponding experimental procedure blank value shall be subtracted from the sample analysis results.

8 Analytical Procedures and Methods

8.1 Analytical Procedures

8.1.1 Power-on pre-heating

After powering on the instrument, turn on the power supply to the host machine and computer, enable vacuum, and switch the instrument from shutdown to standby mode. Before starting the plasma, it shall be ensured that there is sufficient argon gas for continuous operation and that the waste collection barrel has sufficient space for waste liquid collection. Open the ventilation system and adjust the argon gas pressure output to meet the instrument's normal operation requirements. Open the water-cooling circulation system, start the plasma, and after the plasma stabilizes, start the determination.

8.1.2 Selection of analytical conditions

8.1.2.1 Before sample analysis, an analytical method appropriate for the sample to be analyzed shall be established. In accordance with the analytical demands, select parameters such as ion lens parameters, ICP power, gas flow rates (coolant gas, auxiliary gas and carrier gas), peristaltic pump speed, integration time, analytical element and its mass-to-charge ratio, internal standard element, torch tube position, sampling depth, mass spectrometry measurement mode, and instrument resolution, etc. The principle of selection is to ensure strong signals for most elements simultaneously measured, with high precision and minimal interference. Instrument signal-to-background ratio test or method detection limit test may be performed in accordance with the requirements of the instrument instruction manual to determine the optimal operating conditions of the instrument.

8.1.2.2 When using a multi-quadrupole inductively coupled plasma mass spectrometer for analysis, the parameters of the first-stage quadrupole, collision / reaction cell, and second-stage quadrupole also need to be set.

8.1.3 Sample analysis

In accordance with the analytical method in 8.2, analyze the sample. During the analysis, interference elimination and quality control steps shall be added.

8.1.4 Completion of analysis

After the analysis is completed, use 5% (volume fraction) nitric acid solution to inject the sample for approximately 5 minutes, followed by water (see 5.2) for approximately 5 minutes. In accordance with the instrument operating manual, place the instrument on standby or shutdown mode.

8.2 Analytical Method

8.2.1 Interference elimination

8.2.1.1 Physical interference

Physical interference can be corrected using the internal standard method, matrix matching method, or standard addition method.

8.2.1.2 Isobaric interference

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