

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 766-2015

**Solid Waste. Determination of metals. Inductively coupled
plasma mass spectrometry (ICP-MS)**

固体废物 金属元素的测定 电感耦合等离子体质谱法
(English Translation)

Issued on: November 20, 2015

Implemented on: December 15, 2015

Issued by: Ministry of Ecology and Environment

Table of Contents

Preface	.ii
1 Scope of application	
2 Normative reference documents	
3 Principle of the method	
4 Interference and elimination	
5 Reagents and materials	
6 Instruments and equipment	
7 Sample	
8 Analysis steps	

Foreword

In order to implement the Environmental Protection Law of the People's Republic of China and the Law of the People's Republic of China on the Prevention and Control of Environmental Pollution by Solid Wastes, Protect the environment, protect human health, regulate the solid metal waste metal monitoring methods, the development of this standard.

This standard specifies the inductively coupled plasma mass spectrometry for the determination of metal elements in solid waste and solid waste leachate.

This standard is the first release.

Appendix A to this standard is a normative appendix, Appendix B, Appendix C is an informative appendix.

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The main drafting unit of this standard. Qingdao Environmental Monitoring Center Station the standard verification unit. Heilongjiang Province Environmental Monitoring Center Station, Jinan City Environmental Monitoring Center Station, Harbin Environmental Monitoring Measuring center station, Yantai City Environmental Monitoring Center Station, Zaozhuang City Environmental Monitoring Station, Qingdao Economic and Technological Development Zone Water Supply and Drainage Monitoring Station.

The Environmental Protection Department of this standard approves on November 20, 2015.

This standard is implemented as of December 15, 2015.

This standard is explained by the Ministry of Environmental Protection.

Solid waste - Determination of metallic elements - Inductively coupled plasma mass spectrometry Warning. Preparation of thallium, beryllium and other highly toxic substances in the standard solution, should avoid direct contact with the skin. Nitric acid used in the experiment, Hydrochloric acid, hydrofluoric acid is volatile and corrosive, the operation should be required to wear protective equipment, the entire experimental process must Operate in a fume hood.

1 Scope of application

This standard specifies the inductively coupled plasma mass spectrometry for the determination of metal elements in solid waste and solid waste leachate.

This standard applies to the solid waste and solid waste leaching solution of silver (Ag), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), manganese (Mn), molybdenum (Mo), nickel (Ni), lead (Pb) Determination of 17 kinds of metal elements such as selenium (Se), thallium (Tl), vanadium (V) and zinc (Zn). This standard is also applicable if verified Determination of other metal elements.

When the sampling volume of solid waste leaching solution is 25 ml, the detection limit of 17 kinds of metal elements is 0.7 µg/L to 6.4 µg/L, the lower limit of determination was 2.8 µg/L ~ 25.6 µg/L. The limit of detection for each element is given in Appendix A. When the amount of solid waste sample is 0.1 g , The detection limit of 17 kinds of metal elements was 0.4 mg/kg ~ 3.2 mg/kg, and the determination limit was 1.6 mg/kg ~ 12.8 mg/kg.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

Technical specification for SJ sample preparation for industrial solid waste Technical Specification for Hazardous Waste Identification HJ/T 299 Solid waste leaching toxicity leaching method sulfuric acid nitric acid method HJ/T 300 solid waste leaching toxicity leaching method acetic acid buffer solution method HJ 557 Solid waste leaching toxicity leaching method Horizontal oscillation method

3 Principle of the method

Solid waste or solid waste leaching solution by microwave digestion pretreatment, the use of inductively coupled plasma mass spectrometer for inspection Measured, according to the elements of the spectrum or characteristic ions for qualitative, internal standard method quantitative.

4.1 Mass spectrum interference

Mass spectrometry interference mainly includes isotope interference, polyatomic ion interference, oxide and double charge interference. The same amount Isotope interference can be corrected using the interference correction equation or by chemical separation of the sample before analysis except. Commonly used mass count interference correction equation is shown in Appendix B-1.

Polyatomic ion interference is the most important source of interference for ICP-MS. It can use the interference correction equation, instrument optimization and Collision reaction pool technology to eliminate. Common polyatomic ion disturbances are given in Appendix B-2.

Oxide interference and double charge interference can reduce the degree of interference by adjusting the instrument parameters.

4.2 Non-mass spectral interference

Non-mass spectral interference mainly includes matrix suppression interference, space charge effect interference, physical effect interference and so on. Non-mass spectrometry the degree of interference is related to the nature of the sample matrix and can be eliminated by means of internal standard method, instrument condition optimization or standard addition method.

5 Reagents and materials

Unless otherwise stated, the use of the use of national standards in line with the excellent grade pure chemical reagents, experimental water for the new preparation Ion water.

5.1

hydrochloric acid. $\rho(\text{HCl}) = 1.19 \text{ g/ml}$, excellent grade pure or high purity.

5.2

nitric acid. $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$, excellent grade pure or high purity.

5.3

hydrofluoric acid. $\rho(\text{HF}) = 1.49 \text{ g/ml}$.

5.4

Hydrogen peroxide. $\omega(\text{H}_2\text{O}_2) = 30\%$.

5.5

Nitric acid solution. 2 98.

5.6

nitric acid solution. 5 95.

5.7

single element standard stock solution. $\rho = 1.00 \text{ mg/ml}$.

Can be prepared with high purity metal (purity greater than 99.99%) or metal salts (reference or high purity reagent) formulated 1.00 mg/ml A standard stock solution containing nitric acid solution (5.5), or a commercially available standard solution.

5.8

multi-element standard stock solution. $\rho = 100 \text{ mg/L}$.

Use a nitric acid solution (5.5) to dilute the single element standard stock solution (5.7), or purchase a multi-element mixed standard solution directly.

5.9

Multi-element standard solution. $\rho = 1.00 \text{ mg/L}$.

The standard stock solution (5.7 or 5.8) was diluted with nitric acid solution (5.5).

5.10

Internal standard stock solution. $\rho = 10.0 \text{ mg/L}$.

Should choose ^6Li , ^{45}Sc , ^{74}Ge , ^{89}Y , ^{103}Rh , ^{115}In , ^{185}Re , ^{209}Bi for the internal standard elements. Can be purchased directly to prove the standard solution was prepared and the medium was nitric acid solution (5.5).

5.11

mass spectrometer tuning solution. $\rho = 10.0 \text{ }\mu\text{g/L}$.

A solution containing Li, Y, Be, Mg, Co, In, Tl, Pb and Bi elements should be used as the tuning solution for the mass spectrometer.

Can be purchased directly to prove the standard solution preparation.

Note 1: All elements of the standard solution should be prepared in sealed polyethylene or polypropylene bottles.

5.12

argon. purity of not less than 99.99%.

6 Instruments and equipment

6.1

Inductively Coupled Plasma Mass Spectrometer (ICP-MS). The mass range that can be scanned is 6 amu to 240 amu at 10% Peak height should be between 0.6 amu ~ 0.8 amu .