

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Solid Waste. Determination of Beryllium, Nickel, Molybdenum
and Copper. Graphite Furnace Atomic Absorption Spectrometry**

固体废物 铍 镍 铜和钼的测定 石墨炉原子吸收分光光度法

(English Translation)

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I preface

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, Environment, the protection of human health, regulate solid waste in beryllium, nickel, copper and molybdenum monitoring methods, the development of this standard.

This standard specifies the atomic absorption spectrophotometric method for beryllium, nickel, copper and molybdenum in solid waste and solid waste leachate.

This standard is the first release.

Appendix A, Appendix B and Appendix C of this standard are informative.

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

The main drafting unit of this standard. Nanjing Environmental Monitoring Center Station.

The standard verification unit. Taizhou City Environmental Monitoring Center Station, Changzhou City Environmental Monitoring Center, Suzhou City Environmental Monitoring Center Station, Zhenjiang City Environmental Monitoring Center Station, Jiangsu Province, physical and chemical testing center, Nanjing Water Corporation Water Quality Monitoring Center.

The Environmental Protection Department of this standard approves on August 21, 2015.

This standard is implemented on October 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

Determination of solid waste beryllium nickel - copper and molybdenum - Graphite furnace atomic absorption spectrophotometric method Warning. The beryllium standard solution used in the experiment is a toxic chemical; perchloric acid, nitric acid, hydrogen peroxide has strong oxidizing properties and corrosive, hydrochloric acid, hydrofluoric acid with strong volatile and strong corrosive, the operation should be required to wear protective equipment, Solution preparation and sample pretreatment should be performed in a fume hood.

1 Scope of application this standard specifies the determination of solid waste leachate and solid waste in the beryllium, nickel, copper and molybdenum content of graphite furnace atomic absorption Spectrophotometric method.

This standard applies to solid waste leachate and solid waste in the determination of beryllium, nickel, copper and molybdenum.

The detection limits of beryllium, nickel, copper and molybdenum in solid waste leachate were 0.1 µg/L, 1 µg/L, 3 µg/L and 2 µg/L, the lower limit of determination was 0.4 µg/L, 4 µg/L, 12 µg/L and 8 µg/L, respectively. When the sampling volume was 0.5 g when the volume of volume is 50.0 ml, the detection limits of the total amount of beryllium, nickel, copper and molybdenum in solid waste are 0.04 Mg/kg, 0.2 mg/kg, 0.3 mg/kg and 0.2 mg/kg, respectively. The lower limit of determination was 0.16 mg/kg, 0.8 mg/kg, 1.2 mg/kg and 0.8 mg/kg.

2 normative reference documents the contents of this standard refer to the following documents or their terms. Any annotated file that does not specify the date, its valid version Applicable to 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 leachate or solid waste by acid digestion, into the graphite furnace atomizer, the sample of beryllium, nickel, Copper and molybdenum by high temperature atomization, the ground state atoms respectively beryllium, nickel, copper, molybdenum characteristic radiation line selective absorption, its absorption intensity in a certain range with the beryllium, nickel, copper and molybdenum concentration is proportional to.

4 interference and elimination 4.1 higher than 20 mg/L of Fe and 75 mg/L of Mg on the determination of beryllium, respectively, negative interference and interference, Body modifier palladium chloride solution, can eliminate interference.

4.2 less than 10 mg/L Cu, Zn, Ni, Pb, Cd, Cr, Mn, Ba, less than 50 mg/L of Ti and 100 mg/L of K, Na, Ca, Al and other coexisting elements on the determination of beryllium without interference.

4.3, less than 10 mg/L Ag, Al, B, Ba, Be, Bi, Cd, Co, Fe, Hg, Cr, Cs, Cu, Mg, Mn, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn and K, Na, Ca, Mg and the like below 100 mg/L Coexisting elements do not interfere with the determination of molybdenum.

4.4 When the sample matrix interference is serious, the standard addition method can be used for determination, see Appendix B.

25 Reagents and materials

Unless otherwise stated, analytical reagents conforming to national standards are used in the analysis, and the test water is freshly prepared Sub-water.

5.1 hydrochloric acid. $\rho(\text{HCl}) = 1.19 \text{ mg/L}$, excellent grade pure.

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

5.3 hydrofluoric acid. $\rho(\text{HF}) = 1.49 \text{ g/ml}$, excellent grade pure.

5.4 perchloric acid. $\rho(\text{HClO}_4) = 1.68 \text{ g/ml}$, excellent grade pure.

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

5.6 beryllium sulfate ($\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$), excellent grade pure.

5.7 Metal Nickel (Spectrum Pure).

5.8 Metallic copper (pure spectrum).

5.9 Ammonium molybdate $\{(NH_4)_6 [MOO_4] \cdot 4H_2O\}$, excellent grade pure.

5.10 palladium chloride ($PdCl_2$).

5.11 magnesium nitrate $Mg(NO_3)_2$, excellent grade pure.

5.12 Nitric acid solution. 1:1 (v/v) Measure 50 ml nitric acid (5.2) with deionized water to 100 ml.

5.13 Nitric acid solution. 1:99 (v/v) Measure 10 ml of nitric acid (5.2) Dilute to 1000 ml with deionized water.

5.14 Substrate modifier I. palladium chloride solution Weigh 0.34 g of palladium chloride (5.10), add a small amount of water and 1 ml of nitric acid (5.2), dissolved in 50 ° C, 100 ml.

5.15 Substrate modifier II. palladium chloride - magnesium nitrate mixed solution Weigh 0.25 g of palladium chloride (5.10), add a small amount of water and 1 ml of nitric acid (5.2), 50 ° C dissolved;

Magnesium nitrate (5.11) is dissolved in a small amount of water. Mix the two solutions and dilute to 100 ml with water.

5.16 beryllium standard stock solution. $\rho(Be) = 100 \text{ mg/L}$ Use a commercially available standard solution (beryllium single element or a multi-element mixed standard solution containing beryllium); or accurately weigh 1.9660 G beryllium sulfate (5.6), with a small amount of water dissolved after the full transfer into the 1 000 ml volumetric flask, add 10 ml of nitric acid (5.2), Water volume to the mark, shake. Stored in a polyethylene bottle, refrigerated, can be used for two years.

5.17 beryllium standard use of liquid. $\rho(Be) = 50 \text{ } \mu\text{g/L}$ with nitric acid solution (5.13) step by step dilution beryllium standard Huaihu solution (5.16) prepared.

5.18 nickel standard stock solution. $\rho(Ni) = 1\,000 \text{ mg/L}$ Use a commercially available standard solution (nickel single element or nickel-containing multi-element mixed standard solution); or accurately weigh 1.000 G metal nickel (5.7) dissolved in 10 ml nitric acid (5.2), transferred into a 1 000 mL volumetric flask, Line, shake well. Stored in a polyethylene bottle, refrigerated, can be used for two years.

5.19 nickel standard use of liquid. $\rho(Ni) = 250 \text{ } \mu\text{g/L}$ with nitric acid solution (5.13) step by step dilution of nickel standard Huaihu solution (5.18) from the preparation.

5.20 copper standard stock solution. $\rho(Cu) = 1\,000 \text{ mg/L}$ Use commercially available standard solution (copper single element or copper-containing multi-element mixed standard solution); or accurately weigh 1.000 3g of copper (5.8) dissolved in 10 ml of nitric acid (5.2), transferred into a 1 000 mL volumetric flask, Line, shake well. Stored in a polyethylene bottle, refrigerated, can be used for two years.

5.21 copper standard use of liquid. $\rho(Cu) = 250 \text{ } \mu\text{g/L}$ with nitric acid solution (5.13) by