

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

HJ 923-2017

**Soil and sediment--Determination of total mercury--Catalytic
pyrolysis-cold atomic absorption spectrophotometry**

土壤和沉积物 总汞的测定 催化热解-冷原子吸收分光光度法

(English Translation)

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Foreword

In order to carry out "Environmental Protection Law of the People's Republic of China", protect the environment, protect human health, regulate soil and sediment Determination of total mercury, the development of this standard.

This standard specifies the catalytic pyrolysis - cold atomic absorption spectrophotometry for the determination of total mercury in soils and sediments.

Appendix A of this standard is an informative annex.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. Jiangsu Provincial Environmental Monitoring Center.

This standard verification unit. Zhenjiang City Environmental Monitoring Center Station, Lianyungang City Environmental Monitoring Center Station, Jiangsu Province, disease prevention Control Center, China Geological Survey Nanjing Geological Survey Center, Ministry of Land Resources Nanjing Mineral Resources Supervision and Testing Center and River Suzhou Provincial Environmental Monitoring Center.

This standard MEP approved on December 28, this standard since April 1, 2018 come into operation.

This standard is interpreted by the MEP.

Determination of total mercury in soils and sediments - Catalytic pyrolysis - Cold atoms Absorption spectrophotometry Warning. The nitric acid used in the experiment is highly volatile and corrosive. The standard solution preparation process should be done in a fume hood ; Operation should be required to wear protective equipment to avoid inhalation of the respiratory tract and direct contact with the skin, clothing.

1 Scope of application

This standard specifies the catalytic pyrolysis - cold atomic absorption spectrophotometry for the determination of total mercury in soils and sediments.

This standard applies to the determination of total mercury in soils and sediments.

When the sample volume is 0.1 g, the detection limit of this standard method is 0.2 µg/kg and the determination range is 0.8 to 6.0 x 10³ µg/kg.

2 Normative references

This standard references the following documents or the terms. For undated references, the effective version applies to this standard.

GB 17378.3 Marine monitoring code - Part 3. Sample collection, storage and transport GB 17378.5 Marine monitoring code - Part 5. Sediment analysis HJ 494 water sampling technical guidance HJ 613 Determination of soil dry matter and moisture Gravimetric method HJ/T 91 Technical Specifications for Surface Water and Sewerage Monitoring HJ/T 166 Soil Environmental Monitoring Technical Specifications

3 Method principle

After the sample is introduced into the combustion catalytic converter, the various forms of mercury are reduced to elemental mercury through the processes of drying, thermal decomposition and catalytic reaction, Mercury into the kerosene generated gold amalgam, rapid chemical vaporization of mercury in the amalgam mercury released as a vapor form, mercury vapor was the carrier gas was taken into a cold atomic absorption spectrophotometer, and mercury vapor absorbed the 253.7 nm characteristic line. At a certain concentration range, Absorption intensity is proportional to the concentration of mercury.

4 Reagents and materials

Unless otherwise specified, the analysis of the use of analytical reagents in line with national standards, experimental water for the newly prepared deionized Water or distilled water.

4.1

Nitric acid (HNO_3). $\rho = 1.42 \text{ g/ml}$, excellent grade pure.

4.2

potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$). excellent grade pure.

4.3

Mercury chloride (HgCl_2). excellent grade pure.

Pro use the dryer when fully dry.

4.4

Fixative.

Dissolve 0.5 g potassium dichromate (4.2) in 950 ml distilled water, add 50 ml nitric acid (4.1) and mix well.

4.5

Mercury standard stock solution. $\rho (\text{Hg}) = 100 \text{ mg/L}$.

Weigh 0.1354 g mercury chloride (4.3), with a fixed solution (4.4) dissolved, transferred to 1000 ml volumetric flask, and then solid Set the liquid (4.4) to the marking line, and shake it well. Can also be purchased directly from the market certified standard solution.

4.6

Mercury standard solution. $\rho (\text{Hg}) = 10.0 \text{ mg/L}$.

Pipette mercury standard stock solution (4.5) 10.0 ml, placed in 100 ml volumetric flask, with a fixed solution (4.4) to the mark, Mix well. Pro use now with.

4.7

carrier gas. high purity oxygen (O_2), purity $\geq 99.999\%$.

4.8

quartz sand. $75 \mu\text{m} \sim 150 \mu\text{m}$ (200 mesh $\sim$ 100 mesh).

Placed in a muffle furnace 850°C 2h, cooled and filled into the stopper sealed glass jar sealed.

5 Instruments and equipment

5.1

Mercury analyzer. Equipped with a sample boat (nickel boat or magnetic boat), a catalytic combustion furnace, a homogeneous pipe, a desorption furnace and a cold atomic absorption spectrophotometer.

Refer to the working flow chart, see Figure 1.

1 - sample boat 2 - combustion catalyst 3 - homogeneous tube 4 - desorption furnace 5 - cold atomic absorption spectrophotometer 6 - low concentration detection cell;

7-high concentration detection cell; C-carrier gas; W-exhaust gas.

Figure 1 reference workflow

5.2

Analytical Balance. Feel 0.0001 g.

5.3

General Laboratory common equipment and equipment.

6.1 Sample Collection and Storage

Soil samples were collected and stored according to the relevant requirements of HJ/T 166, and samples of marine sediments were collected according to the phase of GB 17378.3 Off Requirements Collection and storage, surface water sediment samples collected according to HJ/T 91 and HJ 494 requirements. After sample collection, Store in a glass bottle below 4 °C for storage for 28 days.

6.2 Sample Preparation

According to HJ/T 166 and GB 17378.3, the collected samples were air-dried in the laboratory, crushed, sieved and stored for future use.

6.3 Determination of moisture

The dry matter content of the soil sample (6.2) was determined according to HJ 613 and the sediment sample (6.2) was determined according to GB 17378.5 Moisture content.

7.1 Instrument reference conditions

According to the instruction manual of the instrument, connect the gas path of the instrument, and check the gas path before use. Reference instrument Instructions, select the best analysis conditions. Instrument reference conditions shown in Table 1.