

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

HJ 702-2014

**Solid Waste-Determination of Mercury, Arsenic, Selenium,
Bismuth, Antimony. MicrowaveDissolution/AtomicFluorescence
Spectrometry**

固体废物 汞、砷、硒、铋、锑的测定 微波消解原子荧光法

(English Translation)

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Foreword

To implement the "People's Republic of China Environmental Protection Law" and "People's Republic of China Solid Waste Pollution Prevention Law" Protecting the environment, safeguarding human health, regulate solid waste and solid waste leaching solution of mercury, arsenic, selenium, bismuth, antimony monitoring methods, Development of this standard.

This standard provides solid waste and solid waste leaching solution of mercury, arsenic, selenium, bismuth, antimony microwave digestion/Atomic Fluorescence Spectrometry test methods.

This standard is the first release.

Appendix A of this standard is an informative catalog.

This standard is developed by the Ministry of Environmental Protection Science, Technology organization.

This standard was drafted. Ningbo Municipal Environmental Monitoring Center.

The standard verification. Zhejiang Zhoushan Marine Ecological Environmental Monitoring Station, Jiaxing Municipal Environmental Protection Monitoring Station, Huzhou City Environment Conservation Monitoring Centre Station, Jinhua Municipal Environmental Monitoring Center, Taizhou City Environmental Monitoring Center, Ningbo Beilun District Environmental Protection supervisor ST.

This standard MEP September 3, 2014 for approval.

This standard since November 1, 2014 implementation.

The standard explanation by the Ministry of Environmental Protection.

Solid waste mercury, arsenic, selenium, bismuth, antimony determination Microwave Digestion/Atomic Fluorescence Spectrometry Warning. nitric acid used in the experiments, volatile and corrosive hydrochloric acid, the operation should be required to required to wear protective equipment, Solution preparation and sample pre-treatment process should be performed in a fume hood.

1 Scope

This standard specifies the microwave solid waste and solid waste leaching solution of mercury, arsenic, selenium, bismuth, antimony digestion/atomic fluorescence measurement Setting Method.

This standard applies to solid waste mercury, arsenic, selenium, bismuth, antimony and determination of solid waste leaching solution. When the solid waste taken the sample size is 0.5g, the present method of mercury detection limit of 0.002 μ g/g, detection limit 0.008 μ g/g; arsenic, selenium, bismuth and antimony inspection the limit was 0.010 μ g/g, detection limit 0.040 μ g/g. When the solid waste leachate sampling volume of 40ml, the mercury detection limit it was 0.02 μ g/L, detection limit 0.08 μ g/L; arsenic, selenium, bismuth, antimony detection limit is 0.10 μ g/L, detection limit 0.40 μ g/L.

2 Normative references

This standard refers to the contents of the following documents or the terms. For undated references, the effective version suitable for this standard.

GB/T 21191 atomic fluorescence spectrometer HJ/T 20 industrial solid waste sampling sample preparation Technical Specifications HJ/T 298 Identification of Hazardous Waste Technical Specifications HJ/T 299 methods for leaching toxicity of solid waste sulfuric acid nitric acid method HJ/T 300 solid waste leaching toxicity Acetic acid buffer solution method HJ 557 solid waste leaching toxicity level oscillation method

3 PRINCIPLE OF THE METHOD

Solid waste and leachate samples after microwave digestion, atomic fluorescence into the instrument, wherein the arsenic, bismuth, antimony, selenium and mercury Element in the solution of potassium borohydride reduction to produce arsine, hydrogen bismuth, antimony, hydrogen, selenide and mercury vapor atoms.

These gases are excited by the formation of ground-state atoms in an argon hydrogen flame, the light elements (mercury, arsenic, selenium, bismuth, antimony) emitting light production Health atomic fluorescence spectrometry, atomic fluorescence intensity is

proportional to the elements in the sample.

4 Reagents and materials

Unless otherwise indicated, the use of line with national standards for gifted class pure reagents analysis, test water is distilled water.

4.1

HCl. rho (HCl) = 1.19 g/ml.

Nitrate 4.2. rho (HNO₃) = 1.42 g/ml.

4.3

Potassium hydroxide (KOH).

4.4

potassium borohydride (KBH₄).

4.5

hydrochloric acid solution. 5 95 (v/v).

Measure 25ml of hydrochloric acid (4.1) diluted to 500ml with distilled water.

Hydrochloric acid solution was 4.6. 1 1 (v/v).

Measure 500ml of hydrochloric acid (4.1) diluted to 1000ml with distilled water.

4.7

thiourea (CH₄N₂S). AR.

4.8

ascorbic acid (C₆H₈O₆). AR.

4.9

reductant.

4.9.1

potassium borohydride (KBH₄) Solution A. = rho 10g/L.

Weigh 0.5g of potassium hydroxide (4.3) containing 100 ml of distilled water into a beaker, stirring until completely dissolved and then the glass rod Join the weighed 1.0g potassium borohydride (4.4), stirring to dissolve. This solution preparation day, for the determination of mercury.

4.9.2

potassium borohydride (KBH₄) solution B. = rho 20g/L.

Weigh 0.5g of potassium hydroxide (4.3) containing 100 ml of distilled water into a beaker, stirring until completely dissolved and then the glass rod Join the weighed 2.0g potassium borohydride (4.4), stirring to dissolve. This solution preparation day, for the determination of arsenic, selenium, bismuth, antimony.

1 Note. You can also use sodium hydroxide, sodium borohydride, sodium borohydride solution configuration.

4.10

thiourea and ascorbic acid mixed solution.

Weigh thiourea (4.7), ascorbic acid (4.8) each 10g, with 100ml distilled water, mix, formulated the day.

4.11

mercury (Hg) standard solution.

4.11.1

the mercury standard fixative (fixative for short).

0.5g of potassium dichromate was dissolved in 950ml of distilled water, then add 50ml of nitric acid (4.2) and mix.

4.11.2

the mercury standard stock solution. = rho 100.0mg/L.

Purchase a commercially available certified reference materials/certified reference materials, or weighed on a silica gel dryer stand overnight mercuric chloride (HgCl_2) 0.1354g, with the amount of dissolved in distilled water to move 1000ml volumetric flask, and finally with fixative (4.11.1) volume to the mark, Mix well.

4.11.3

mercury intermediate standard solution. = rho 1.00mg/L.

Pipette mercury stock standard solution (4.11.2) 5.00ml, placed in 500ml volumetric flask, add 50ml hydrochloric acid solution (4.6), with fixative (4.11.1) volume to the mark, and mix.

4.11.4

mercury standard solution. = rho 10.0 $\mu\text{g/L}$.

Pipette mercury intermediate standard solution (4.11.3) 5.00ml, placed 500ml volumetric flask, add 50ml hydrochloric acid solution (4.6), with fixative (4.11.1) volume to the mark, and mix. The time is now equipped with.

4.12

Arsenic (As) standard solution.