

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 680-2013

**Soil and sediment - Determination of mercury, arsenic,
selenium, bismuth, antimony - Microwave dissolution/Atomic
Fluorescence Spectrometry**

土壤和沉积物 汞、砷、硒、铋、锑的测定 微波消解/原子荧光法

(English Translation)

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1 Scope of application

This Standard specifies the microwave dissolution/atomic fluorescence spectrometry for the determination of mercury, arsenic, selenium, bismuth, and antimony in soil and sediment.

This Standard applies to the determination of mercury, arsenic, selenium, bismuth, and antimony in soil and sediment.

When the sample size is 0.5 g, for mercury, the detection limit of this method is 0.002 mg/kg, and the lower limit of determination is 0.008 mg/kg; for arsenic, selenium, bismuth, and antimony, the detection limit is 0.01 mg/kg, and the lower limit of determination is 0.04 mg/kg.

2 Normative references

This Standard refers to the following documents or their clauses. For undated references, the latest edition applies to this Standard.

GB 17378.3 The specification for marine monitoring - Part 3. Sample collection storage and transportation

GB 17378.5 The specification for marine monitoring - Part 5. Sediment analysis

GB/T 21191 Atomic fluorescence spectrometer

HJ/T 166 Technical specification for soil environmental monitoring

HJ 613 Soil - Determination of dry matter and water content - Gravimetric method

3 Principle of the method

After the sample is dissolved by microwave, the test solution enters the atomic fluorescence spectrometer; under the reducing action of potassium borohydride solution, arsine, bismuth hydrogen, antimony hydrogen and hydrogen selenide gas are generated, and mercury is reduced to atomic state. Ground-state atoms are formed in the hydrogen- hydrogen flame, and atomic fluorescence is generated under the excitation of light emitted by element lamps (mercury, arsenic, selenium, bismuth, antimony). The atomic fluorescence intensity is proportional to the element content in the test solution.

4 Reagents and materials

Unless otherwise specified, the analysis is performed using guarantee reagents that comply with national standards, and the experimental water is freshly prepared distilled water.

4.1

Hydrochloric acid (HCl), $\rho = 1.19$ g/ml.

4.2

Nitric acid (CHNO₃), $\rho = 1.42$ g/ml.

4.3

Potassium hydroxide (KOH).

4.4

Potassium borohydride (KBH₄).

4.5 Hydrochloric acid solution

5 + 95.

PIPETTE 25 ml of hydrochloric acid (4.1) and DILUTE to 500 ml with experimental water.

4.6 Hydrochloric acid solution

1 + 1.

PIPETTE 500 ml of hydrochloric acid (4.1) and DILUTE to 1000 ml with experimental water.

4.7 Thiourea (CH₄N₂S)

analytical reagent.

4.8 Ascorbic acid (C₆H₈O₆)

analytical reagent.

4.9

Reducing agents.

4.9.1

Potassium borohydride solution A. $\rho = 10 \text{ g/L}$.

WEIGH 0.5 g of potassium hydroxide (4.3); PUT it in a beaker containing 100 ml of experimental water; STIR with a glass rod until completely dissolved; ADD 1.0 g of potassium borohydride (4.4); STIR to dissolve. This solution is prepared on the day of use for the determination of mercury.

4.9.2 Potassium borohydride solution B

$\rho = 20 \text{ g/L}$.

WEIGH 0.5 g of potassium hydroxide (4.3); PUT it in a beaker containing 100 ml of experimental water; STIR with a glass rod until completely dissolved; ADD 2.0 g of potassium borohydride (4.4); STIR to dissolve. This solution is prepared on the day of use for the determination of arsenic, selenium, bismuth, and antimony.

4.13.3 Arsenic standard use solution

$\rho = 100.0 \text{ } \mu\text{g/L}$.

PIPETTE 10.00 ml of arsenic standard intermediate solution (4.13.2); PLACE it in a 100 ml volumetric flask; ADD 20 ml of hydrochloric acid solution (4.6); DILUTE to the marked line with experimental water; MIX well. Prepare before use.

4.14

Selenium (Se) standard solution.

4.14.1 Selenium standard stock solution

$\rho = 100.0 \text{ mg/L}$.

PURCHASE commercially available certified reference materials/certified reference

samples, or WEIGH 0.1000 g of high-purity selenium powder; PLACE it in a 100 ml beaker; ADD 20 ml of nitric acid (4.2); HEAT at low temperature until dissolved;

COOL to room temperature; TRANSFER to a 1000 ml volumetric flask; DILUTE to the marked line with experimental water; MIX well.

4.14.2 Selenium standard intermediate solution

$\rho = 1.00 \text{ mg/L}$.

PIPETTE 5.00 ml of selenium standard stock solution (4.14.1); PLACE it in a 500 ml volumetric flask; DILUTE to the marked line with experimental water; MIX well.

4.14.3 Selenium standard use solution

$\rho = 100.0 \text{ } \mu\text{g/L}$.

PIPETTE 10.00 ml of selenium standard intermediate solution (4.14.2); PLACE it in a 100 ml volumetric flask; DILUTE to the marked line with experimental water; MIX well. Prepare before use.

4.15

Bismuth (Bi) standard solution.

4.15.1 Bismuth standard stock solution

$\rho = 100.0 \text{ mg/L}$.

PURCHASE commercially available certified reference materials/certified reference samples, or WEIGH 0.100 0 g of high-purity metal bismuth; PLACE it in a 100 ml beaker; ADD 20 ml of nitric acid (4.2); HEAT at low temperature until completely dissolved; COOL; TRANSFER to a 1000 ml volumetric flask; DILUTE to the marked line with experimental water; MIX well.

4.15.2 Bismuth standard intermediate solution

$\rho = 1.00 \text{ mg/L}$.

PIPETTE 5.00 ml of bismuth standard stock solution (4.15.1); PLACE it in a 500 ml volumetric flask; ADD 100 ml of hydrochloric acid solution (4.6); DILUTE to the mark line with experimental water; MIX well.

4.15.3 Bismuth standard use solution

$\rho = 100.0 \text{ } \mu\text{g/L}$.