

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

HJ 1133-2020

**Ambient air and waste gas - Determination of arsenic, selenium,
bismuth, antimony in ambient particle matter - Atomic
fluorescence spectrometry**

环境空气和废气 颗粒物中砷、硒、铋、锑的测定 原子荧光法
(English Translation)

Issued on: May 15, 2020

Implemented on: August 15, 2020

Issued by: Ministry of Ecology and Environment

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Foreword

To implement the Environmental Protection Law of the People's Republic of China and the Air Pollution Prevention and Control Law of the People's Republic of to ensure human health, regulate the determination of arsenic, selenium, bismuth, and antimony in ambient air and waste gas particles, and formulate this standard.

This standard specifies the atomic fluorescence method for the determination of arsenic, selenium, bismuth, and antimony in ambient air and exhaust particles.

Appendix A of this standard is a normative appendix, and Appendix B is an informative appendix.

This standard is issued for the first time.

This standard was formulated by the Department of Ecological Environment Monitoring and the Department of Regulations and Standards of the Ministry of Ecology and Environment.

Drafting organization of this standard. China Environmental Monitoring Center.

The verification unit of this standard. Qinghai Province Ecological Environment Monitoring

Center, Liaoning Province Ecological Environment Monitoring Center, Inner Mongolia Autonomous Region Environmental Monitoring Center Station, Taiyuan Ecological Environment Monitoring Center of Shanxi Province, Yangzhou Environmental Monitoring Center of Jiangsu Province, and Zhanjiang Environmental Protection Monitoring Station.

This standard was approved by the Ministry of Ecology and Environment on May 15, 2020.

This standard will be implemented on August 15, 2020.

This standard is interpreted by the Ministry of Ecology and Environment.

1 Determination of arsenic, selenium, bismuth and antimony in ambient air and waste gas particles Atomic fluorescence Warning. Nitric acid and hydrochloric acid are highly corrosive and oxidizing. The sample digestion process should be carried out in a fume hood. The experimenters should pay attention to wearing protective equipment.

Avoid breathing into the respiratory tract or contact with skin and clothing.

1 Scope of application

This standard specifies the atomic fluorescence method for the determination of arsenic, selenium, bismuth, and antimony in ambient air and exhaust particles.

This standard applies to ambient air, unorganized emission monitoring point air and fixed pollution sources organized emission of exhaust particles Determination of arsenic (As), selenium (Se), bismuth (Bi), and antimony (Sb).

When the ambient air sampling volume is 150 m³ (actual state) and the sample pretreatment constant volume is 50.0 ml, the method is the detection limit is 0.2 ng/m³~2 ng/m³, the lower limit of determination is 0.8 ng/m³~8 ng/m³; air sampling at monitoring points for fugitive emissions when the volume is 50 m³ (standard state) and the sample pretreatment constant volume is 50.0 ml, the method detection limit is 0.4 ng/m³ to 4 ng/m³, the lower limit of determination is 1.6 ng/m³~16 ng/m³; the sampling volume of organic emission from fixed pollution sources is 0.600 m³ (standard state Dry flue gas), when the sample pretreatment constant volume is 100.0 ml, the detection limit of the method is 0.1 g/m³~0.7 g/m³.

The limit is 0.4 g/m³~2.8 g/m³. See Appendix A for details.

2 Normative references

This standard quotes the following documents or their clauses. For undated reference documents, their valid versions are applicable to this standard.

GB/T 16157 Determination of particulate matter in exhaust from stationary sources and sampling method for gaseous pollutants GB/T 21191 Atomic Fluorescence Spectrometer HJ/T 48 Technical requirements for soot sampler HJ/T 55 Technical Guidelines for

Monitoring Unorganized Emissions of Air Pollutants HJ 93 Ambient air particulate matter (PM₁₀ and PM_{2.5}) sampler technical requirements and detection methods HJ 194 Technical specification for manual monitoring of ambient air quality HJ/T 374 Technical requirements and testing methods for total suspended particulate matter sampler HJ/T 397 Fixed source exhaust gas monitoring technical specification HJ 664 Technical specification for the layout of ambient air quality monitoring points (for trial implementation)

3 Principles of the method

Use a filter membrane or a filter cartridge to collect particulate matter in the air or waste gas. After the sample is digested by a mixed acid of nitric acid and hydrochloric acid, it enters the atomic fluorescence Optical spectrometer, the arsenic, selenium, bismuth, and antimony in the sample undergo oxidation-reduction with potassium borohydride (or sodium borohydride) under acidic conditions the reaction produces arsine, hydrogen selenide, hydrogen bismuth, and hydrogen antimonide gas. The hydride forms ground-state atoms in the argon-hydrogen flame.

2 element lamps (arsenic, selenium, bismuth, antimony) emit atomic fluorescence under excitation, and the intensity of atomic fluorescence within a certain concentration range it is proportional to the content of the elements in the test solution.

4 Interference and elimination

Transition metal elements such as copper may interfere with the determination. Adding thiourea-ascorbic acid mixed solution (5.17) can basically Eliminate interference. Under the experimental conditions of this standard, the sample contains Cu₂ below 100 mg/L, Fe₃ below 50 mg/L, Co₂ below 1 mg/L, Pb₂ below 10 mg/L and Mn₂ below 150 mg/L do not affect the determination.

5 Reagents and materials

Unless otherwise specified, analytical reagents that meet national standards are used for analysis, and the experimental water is newly prepared deionized water or distilled water.

5.1

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

5.2

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

5.3

Potassium hydroxide (KOH).

5.4

Sodium hydroxide (NaOH).

5.5

Potassium borohydride (KBH₄).

5.6

Thiourea (CH₄N₂S).

5.7

Ascorbic acid (C₆H₈O₆).

5.8

Arsenic trioxide (As₂O₃). pure superior grade.

5.9

Selenium powder. high purity (mass fraction above 99.99%).

5.10

Bismuth. high purity (above 99.99% mass fraction).

5.11

Antimony trioxide (Sb₂O₃). pure superior grade.

5.12 Hydrochloric acid solution. 5 95

Measure 50 ml of hydrochloric acid (5.1) and dissolve in about 500 ml of water, then dilute with water and dilute to 1 L.

5.13 Hydrochloric acid solution. 1 1

Measure 500 ml of hydrochloric acid (5.1) and dissolve it in about 400 ml of water, then dilute with water and dilute to 1 L.

5.14 Hydrochloric acid solution. c(HCl)=1 mol/L

Measure 41.6 ml of hydrochloric acid (5.1), dissolve it in a certain amount of water, and dilute with water to 500 ml.

5.15 Nitric acid-hydrochloric acid mixed solution

Measure 55.5 ml of nitric acid (5.2) and 166.5 ml of hydrochloric acid (5.1) respectively, dissolve in about 500 ml of water, and dilute with water Relieve and dilute to 1 L.