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

HJ 1047-2019

**Water quality - Determination of antimony - Graphite furnace
atomic absorption spectrometry**

水质 锑的测定 石墨炉原子吸收分光光度法

(English Translation)

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Foreword

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 Water Pollution Control", protect the ecology Environment, protect human health, standardize the determination method of antimony in water, and formulate this standard.

This standard specifies graphite furnace raw materials for the determination of antimony in domestic sewage, industrial wastewater, certain contaminated surface water and groundwater Atomic absorption spectrophotometry.

Appendix A and Appendix B of this standard are normative appendixes, and Appendix C is an informative appendix.

This standard is issued for the first time.

This standard is formulated by the Department of Eco-Environmental Monitoring, Laws and Standards Department of the Ministry of Ecology and Environment.

This standard was drafted. Shanghai Pudong New Area Environmental Monitoring Station.

Verification units of this standard. Shanghai Environmental Monitoring Center, Shanghai Minhang District Environmental Monitoring Station, Baoshan District, Shanghai Monitoring Station, Shanghai Jiading District Environmental Monitoring Station, Shanghai Qingpu District Environmental Monitoring Station, and Shanghai Huateng Label Testing Technology Limited the company.

This standard was approved by the Ministry of Ecology and Environment on October 24, 2019.

This standard will be implemented from April 24, 2020.

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

Water quality-Determination of antimony-Graphite furnace atomic absorption spectrophotometry Warning. The antimony and its compounds used in the experiment are more toxic, hydrochloric acid is highly corrosive and volatile, and nitric acid is Strongly corrosive and oxidizing, reagent preparation and sample preparation should be carried out in a fume hood, and should be worn as required during operation Protective equipment to avoid contact with skin and clothing.

1 Scope

This standard specifies the graphite furnace atomic absorption spectrophotometry for the determination of antimony in water.

This standard is applicable to the determination of antimony in domestic sewage, industrial wastewater, certain contaminated surface water and groundwater.

When the injection volume is 20 μL , the detection limits of this standard for soluble antimony and total antimony are both 2 $\mu\text{g/L}$.

The limits are all 8 $\mu\text{g/L}$.

2 Normative references

This standard refers to the following documents or clauses therein. For undated references, the valid version applies to this standard.

HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 164
Technical Specifications for Groundwater Environmental Monitoring

3 Terms and definitions

The following terms and definitions apply to this standard.

Soluble antimony

Refers to antimony measured after a non-acidified sample is filtered through a 0.45 μm filter.

Total quantity of antimony Refers to antimony measured after acid filtration of an unfiltered sample.

4 Method principle

The sample is filtered or digested and injected into a graphite furnace atomizer. The antimony element is dried, ashed, and atomized to form Selective atomic vapor of antimony in the ground state produces selectivity to the 217.6 nm characteristic line emitted by a light source (hollow cathode lamp or other light source) Absorption, within a certain range, its absorbance value is proportional to the mass concentration of antimony.

5 Interference and cancellation

5.1

When the mass concentrations of lead, zinc, cadmium, calcium, and aluminum are less than 200 mg/L, 200 mg/L, 250 mg/L, at 1000 mg/L and 1000 mg/L, it does not interfere with the determination of antimony.

5.2

When the mass concentration of nickel and iron is greater than 60 mg/L, 800 mg/L and SO_4^{2-} , Cl^- are greater than 160 mg/L, at 250 mg/L, it negatively interferes with the determination of antimony. Inspection of matrix interference is shown in Appendix A; it can be offset by standard addition method See Appendix B for interference; see Appendix C for applicability of the standard addition method.

6 Reagents and materials

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

Water or water of equivalent purity.

6.1

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

6.2

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

6.3

Palladium nitrate.

6.4

Magnesium nitrate.

6.5

Nitric acid solution. 1 + 1.

6.6

Nitric acid solution. 1 + 99.

6.7

Matrix modifier. $\rho [\text{Pd} (\text{NO}_3)_2] = 6.5 \text{ g/L}$, $\rho [\text{Mg} (\text{NO}_3)_2] = 2.0 \text{ g/L}$.

Weigh 650 mg of palladium nitrate (6.3) into a beaker, add 1 ml of nitric acid (6.2) and 0.1 ml of hydrochloric acid (6.1) Dissolve, weigh.200 mg of magnesium nitrate (6.4), place in a beaker, add a small amount of water to dissolve, and mix the two reagents in one Transfer to a 100 ml volumetric flask, dilute with water to the mark, shake well, transfer to a polyethylene bottle, seal, and save 12 months.

6.8

Tartaric acid. $w \geq 99.5\%$.

6.9

Metal antimony. spectrally pure, $w \geq 99.99\%$.

6.10

Standard antimony stock solution. $\rho (\text{Sb}) = 1000 \text{ mg/L}$.

Accurately weigh 1.0 g (to the nearest 0.0001 g) metal antimony (6.9), place it in a beaker, and add 10 ml of hydrochloric acid (6.1) and 20 ml of nitric acid solution (6.5); add 100 ml of water and 1.5 g of tartaric acid (6.8) and warm to completely dissolve. cool down Then transfer it to a 1000 ml volumetric flask, dilute with water to the mark, shake well, transfer to a polyethylene bottle, and seal it.

Months. Or use a commercially available certified antimony standard solution.

6.11

Standard antimony solution. $\rho (\text{Sb}) = 100.0 \text{ mg/L}$.

Accurately transfer 10.00 ml of standard antimony stock solution (6.10) into a 100 ml volumetric flask and dilute with a nitric acid solution (6.6) Make up to the mark, shake well, transfer to a polyethylene bottle and seal, and it can be stored for 6 months.

6.12

Standard antimony solution. $\rho (\text{Sb}) = 10.0 \text{ mg/L}$.

Accurately transfer 10.00 ml of standard antimony solution (6.11) into a 100 ml volumetric