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HJ

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

HJ 540-2016

Replacing HJ 540-2009

**Stationary source emission - Determination of arsenic- Silver
diethyldithiocarbamate spectrophotometric method**

固定污染源废气 砷的测定 二乙基二硫代氨基甲酸银分光光度法（发布稿）

(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 the Prevention and Control of Atmospheric Pollution, Environment, the protection of human health, regulate the fixed source of waste gas in the form of particulate matter and the determination of arsenic and its compounds, Develop this standard.

This standard specifies the determination of fixed sources of waste gas in the form of particulate matter and its compounds of diethyldisulfide Substituted carbamate spectrophotometric method.

This standard is for the determination of "ambient air and waste arsenic, silver diethyldithiocarbamate spectrophotometric (provisional)" (HJ 540-2009).

This standard was first published in 2009, the original standard drafting unit for the Beijing Environmental Protection Monitoring Center. This is the first the main contents of the amendment are as follows.

- According to the scope of application of the standard, the original standard name "Ambient air and arsenic determination of diethyldithioamino Formic acid silver spectrophotometric method (tentative) "modified to" fixed source of waste gas arsenic determination of diethyldithiocarbamate silver Spectrophotometry";
- the contents of the ambient air and so on have been removed from the original standard;
- Based on the experimental results, the detection limit was revised from 0.00 µg/m³ to 0.004 mg/m³ and the lower limit of determination was 3.5 µg/m³ Revised to 0.016 mg/m³;
- the collection of samples and the preparation of samples and other content has been revised;
- increased precision and accuracy and other content;
- improve the quality assurance and quality control content;
- Increased waste disposal.

Since the implementation of the standard date, the original standard "Determination of ambient air and arsenic diethyldithiocarbamate Photometric method (provisional) "(HJ 540-2009) abolished.

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The drafting of this standard. Beijing Environmental Protection Monitoring Center, Beijing Xicheng District Environmental Protection Monitoring Station.

The standard verification unit. Yunnan Honghe State Environmental Monitoring Station, Beijing Changping District Environmental Protection Monitoring Station, Beijing Feng Taiwan Environmental Protection Monitoring Station, Beijing Xicheng District Environmental Protection Monitoring Station, Beijing China measured North Detection Technology Co., Ltd.

The environmental protection department of this standard approved on July 26, 2016.

This standard has been implemented since October 1, 2016.

This standard is explained by the Ministry of Environmental Protection.

Determination of arsenic in fixed source Silver dimethyldithiocarbamate spectrophotometric method Warning. arsine is a highly toxic gas, before the test must check whether the arsine generator is connected to prevent leakage or reaction the bottle was disintegrated. The reaction shall be carried out in a fume hood, and the operator shall wear protective equipment.

1 Scope of application

This standard specifies the determination of fixed sources of waste gas in the form of particulate matter arsenic and its compounds diethyldithio Silver phthalate spectrophotometric method.

This standard is applicable to the determination of arsenic and its compounds in the form of particulate matter in fixed waste gas.

When the sampling volume is 0.4 m³ (standard state), the volume of the volume is 50.0 ml, the detection limit of this method is 0.004 mg/m³, the lower limit of determination is 0.016 mg/m³ (both in As).

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. For undated references, the valid version is applicable in this standard.

Determination of particulate matter and sampling method of gaseous pollutants in exhaust

gas of fixed pollution sources GB/T 16157 HJ/T 373 fixed pollution source monitoring quality assurance and quality control technical specifications (Trial) HJ/T 48 smoke and dust sampler technical conditions HJ 77.2 Ambient air and exhaust dioxins Determination of isotope dilution High resolution gas chromatography - High resolution mass spectrometry

3 Principle of the method

Using quartz fiber filter cartridge to collect fixed sources of waste gas containing arsenic particles, nitric acid, sulfuric acid, hydrogen peroxide digestion after preparation the solution of pentavalent arsenic in this solution was reduced to trivalent arsenic with potassium iodide (KI) and stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) Zinc particles and acid, resulting in new ecological hydrogen, the trivalent arsenic further reduced to gaseous arsine (AsH_3), and dissolved in trichloromethyl (CH_2Cl_2) in the alkylene chloride (CHCl_3) to form a magenta complex at 510 nm Absorbance measured at the wavelength, within a certain range of its absorbance and arsenic content is proportional to.

4 Interference and elimination

Mercury, manganese, copper, nickel, cobalt, lead and iron, 100 μg or less of cadmium and antimony below 50 μg , bismuth below 30 μg , 20 μg or less of chromium, 10 μg of the following selenium, no significant interference with the determination. When the content of antimony in the sample is greater than 50 μg , the interference Arsenic determination, adding 3 ml of stannous chloride and 5 ml of potassium iodide solution, can inhibit 300 μg of antimony interference. Sulfide interference, can be removed with lead acetate cotton.

5 Reagents and materials

Unless otherwise stated, analytical reagents conforming to national standards are used for analysis. The experimental water is a freshly prepared deionized water or distilled water.

5.1

Arsenic free zinc tablets. 10 mesh to 20 mesh.

Note 1: The rate of occurrence of arsine is affected by the size, surface state and dosage of the zinc particles, the reaction acidity and the temperature. Zinc tablets to 10 to 20 the purpose of the surface rough zinc particles to restore the high efficiency, the amount of 3 g ~ 5 g between the effect is better.

5.2

Potassium iodide (KI).

5.3

Stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$).

5.4

Lead acetate $[\text{Pb}(\text{CH}_3\text{COO})_2]$.

5.5

Diethyldithiocarbamate ($\text{C}_5\text{H}_{10}\text{AgNS}_2$).

5.6

Arsenic trioxide (As_2O_3). Reference reagent.

Before use, in $105\text{ }^\circ\text{C} \sim 110\text{ }^\circ\text{C}$ bake 2 h, stored in the dryer cooling, spare.

5.7

Sodium hydroxide (NaOH).

5.8

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

5.9

sulfuric acid. $\rho(\text{H}_2\text{SO}_4) = 1.84\text{ g/ml}$, excellent grade pure.

5.10

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

5.11

Trichloromethane (CHCl_3).

5.12

Triethanolamine $[(\text{HOCH}_2\text{CH}_2)_3\text{N}]$.

5.13

Hydrogen peroxide. $\omega(\text{H}_2\text{O}_2) = 30\%$, excellent grade pure.

5.14

sulfuric acid solution. 1 1.

Prepared with sulfuric acid (5.9).

5.15 Potassium iodide solution. $\rho(\text{KI}) = 150\text{ g/L}$

Weigh 15 g of potassium iodide (5.2), dissolved in water and diluted to 100 ml beaker.

5.16 Stannous chloride solution. $\rho(\text{SnCl}_2 \cdot 2\text{H}_2\text{O}) = 400\text{ g/L}$.

Weigh 48 g of stannous chloride (5.3), add 50 ml of hydrochloric acid (5.10), dissolve it by heating, and then diluted with water to 100 ml Beaker, ready for use.

5.17 lead acetate solution. $\rho[\text{Pb}(\text{CH}_3\text{COO})_2] = 100\text{ g/L}$.