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

HJ 541-2009

**Yellow phosphorus production emission. Determination of
gaseous arsenic. Silver diethyldithiocarbamate
spectrophotometric method**

黄磷生产废气 气态砷的测定 二乙基二硫代氨基甲酸银分光光度法(暂行)

(English Translation)

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Foreword

To protect the environment and protect people 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 Air Pollution this standard is formulated for the health monitoring of arsenic and its compounds in the exhaust gas from yellow phosphorus production.

This standard specifies the silver diethyldithiocarbamate spectrophotometric method for the determination of arsenic and its compounds in the waste gas from yellow phosphorus production.

This standard was formulated by the Science and Technology Standards Department of the

Ministry of Environmental Protection.

This standard was drafted. Beijing Environmental Protection Monitoring Center.

This standard was approved by the Ministry of Environmental Protection on December 30, 2009.

This standard has been implemented since April 1, 2010.

This standard is explained by the Ministry of Environmental Protection.

Determination of gaseous arsenic in waste gas from yellow phosphorus production Silver diethyldithiocarbamate spectrophotometry (interim) Warning. Arsine is a highly toxic gas. Before the experiment, it is necessary to check whether the hydrogen arsenide generator is connected to prevent air leakage or reverse.

The bottle should be broken. The reaction should be carried out in a fume hood.

1 Scope of application

This standard specifies the silver diethyldithiocarbamate spectrophotometric method for the determination of gaseous arsenic and its compounds in the exhaust gas from yellow phosphorus production.

This standard applies to the determination of arsenic and its compounds in gaseous form in the exhaust gas of yellow phosphorus production.

The detection limit of this method is 1.25 µg/5 ml silver diethyldithiocarbamate absorption. When the gas volume is 15 L, the detection limit of arsenic is 0.08 mg/m³, the lower limit of determination is 0.32 mg/m³ (both in As).

2 Normative references

The contents of this standard refer to the terms in the following documents. For undated references, the valid version applies to this standard.

GB/T 16157 Determination of particulate matter in fixed pollution source exhaust gas and sampling method of gaseous pollutants HJ/T 373 Technical Specifications for Quality Assurance and Quality Control of Fixed Pollution Source Monitoring (Trial) GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle of the method

The gaseous arsenic and its compounds in the waste gas from the production of yellow phosphorus are absorbed by the brominated nitric acid absorption liquid, and the brominated nitric acid oxidizes the low-priced arsenide into pentavalent arsenic.

Compound, in an acidic solution, using potassium iodide and stannous chloride (KI-SnCl₂)

to reduce pentavalent arsenic to trivalent arsenic, adding zinc particles and acid, producing new ecological hydrogen, further reducing trivalent arsenic to gaseous arsine (AsH_3), and diethylamino group dissolved in chloroform (CHCl_3) the action of silver dithiocarbamate ($\text{Ag}\cdot\text{DDC}$) to form a purple-red complex, the absorbance is measured at 510 nm, and the yellow phosphorus can be calculated.

The content of arsenic and its compounds in the production of exhaust gas.

4 Interference and elimination

Hydrogen sulfide, hydrogen halide, and phosphine have similar color reaction with $\text{Ag}\cdot\text{DDC}$, which produces positive interference with the determination of arsenic. When the sample is digested, Sulfur and phosphorus have been oxidized and decomposed by nitric acid, no longer affecting. Hydrogen sulfide produced by a small amount of sulfide present in the reagent can be removed by lead acetate absorbent cotton.

can be masked with $\text{KI}\cdot\text{SnCl}_2$ below 300 μg .

5 Reagents and materials

Unless otherwise stated, analytically pure reagents that meet national standards were used for the analysis. Water, GB/T 6682, third grade.

5.1

Arsenic-free zinc particles. 10 to 20 mesh.

5.2

Bromine water. $\rho(\text{Br}_2) = 3.12 \text{ g/ml}$.

5.3

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

5.4

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

5.5

Hydrochloric acid solution. $c(\text{HCl}) = 1 \text{ mol/L}$.

Measure 83 ml of hydrochloric acid (5.3) and dilute to 1 000 ml with water.

5.6

Hydrochloric acid solution. 3 2.

5.7

Nitric acid solution. 1 1.

5.8

Sodium hydroxide solution. $c(\text{NaOH}) = 0.1 \text{ mol/L}$.

Weigh 4 g of sodium hydroxide (analytical grade) dissolved in water and dilute to 1000 ml.

5.9 Bromide nitric acid absorption solution

Nitric acid (5.4) plus bromine water (5.2) to saturation, or 60 ml of nitric acid solution (5.7), add 20 ml of bromine water (5.2). 0 to 4 ° C Store in cold storage.

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

Weigh 15 g of potassium iodide and dissolve in 100 ml of water.

5.11 Stannous chloride solution. $\rho(\text{SnCl}_2) = 400 \text{ g/L}$.

Weigh 48 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$), dissolve in 50 ml of hydrochloric acid (5.3), and add water to 100 ml.

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

Weigh 10 g of lead acetate and dissolve in 100 ml of water.

5.13 Silver diethylaminodithiocarbamate ($\text{Ag} \cdot \text{DDC}$) absorption liquid. $w(\text{Ag} \cdot \text{DDC}) = 0.25\%$.

Weigh 0.25 g of silver diethylaminodithiocarbamate ($\text{Ag} \cdot \text{DDC}$), add 1.0 ml of triethylamine, dissolve in 100 ml of trichloromethane.

Shake well in alkane (CHCl_3), stand overnight, filter, and store in the dark.

5.14 Standard stock solution of arsenic. $\rho(\text{As}) = 1.00 \text{ mg/ml}$.

Weigh 0.132 0 g arsenic trioxide (As_2O_3 , benchmark reagent, pre-baked at 105-110 ° C for 2 h), dissolved in 2.0 ml of hydroxide in the sodium solution (5.8), add 50 ml of water and 2.0 ml of hydrochloric acid solution (5.6). Transfer to a 100 ml volumetric flask and dilute with water to the score line. This solution the solution contains 1.00 mg of arsenic per ml.

A standard solution for arsenic can also be used as a standard solution.

5.15 Arsenic standard use solution. $\rho(\text{As}) = 1.00 \text{ } \mu\text{g/ml}$.

When used, the standard stock solution of arsenic (5.14) is diluted with water to a standard solution containing 1.00 μg of arsenic per ml.

5.16 Lead acetate cotton

Immerse 10 g of cotton wool in 100 ml lead acetate solution (5.12), remove it after 30 min, dry at room temperature, and store in the dark.