

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

HJ 545-2017

Replacing HJ 545-2009

**Stationary source emission?Determination of total gaseous
phosphorus -- Quimociac volumetric analysis**

固定污染源废气 气态总磷的测定 喹钼柠酮容量法

(English Translation)

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Table of Contents

Foreword .ii
1 Scope of application .1
2 Normative references .1
3 Terms and definitions
4 Method principle .1
Interference and elimination
6 Reagents and Materials
7 Instruments and equipment
8 Sample
9 Analysis steps .6
10 Results Calculation and Presentation .7
11 Precision and accuracy .7
12 Quality Assurance and Quality Control .8
13 Waste treatment .8
14 Matters needing attention .8

Foreword

In order to implement the Law of the People's Republic of China on Environmental Protection and the Law of the People's Republic of China on Prevention and Control of Atmospheric Pollution, Environment, protect human health, standardize the determination of gaseous total phosphorus in the exhaust gas of fixed sources of pollution, to develop this standard.

This standard specifies the determination of fixed sources of emissions of gaseous total phosphorus Quimociac volume method.

This standard is "a fixed source of emissions of gaseous total phosphorus Determination Quimociac Volume Act (Interim)" (HJ 545-2009) amendment.

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

- Revised method limits of detection, reagents and materials, apparatus and equipment, sample collection and analysis step clauses.

- Supplement and improve the quality assurance and quality control part of the content.
- Added warning, interference and elimination, precision and accuracy, waste disposal and precautions.

From the date of implementation of this standard, "fixed source of emissions of gaseous total phosphorus Determination Quimociac Volume Act (Interim)" (HJ 545-2009) Repealed.

This standard is revised by the Ministry of Environmental Protection Environmental Monitoring Department and Science and Technology Standards Division.

This standard was drafted. Beijing Environmental Protection Monitoring Center, Sichuan Institute of Industrial Environmental Monitoring.

This standard verification unit. Sichuan Institute of Industrial Environmental Monitoring, Chaoyang District, Beijing Environmental Protection Monitoring Station, Beijing Dongcheng District Environmental Monitoring Station, Haidian District, Beijing Environmental Protection Agency monitoring station, Beijing North China Measurement North Detection Technology Co., Ltd.

And Beijing Environmental Protection Monitoring Center.

This standard MEP approved on December 29, 2017.

This standard since April 1, 2018 come into operation.

This standard is interpreted by the MEP.

Stationary sources - Determination of gaseous total phosphorus - Quinocetone volumetric method Warning. The bromine water and nitric acid used in the experiment are oxidizing and corrosive, and must be handled in a fume hood when formulated Take care when handling samples and laboratories to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the determination of fixed sources of emissions of gaseous total phosphorus Quimociac volume method.

This standard applies to the determination of gaseous total phosphorus in fixed sources of exhaust gas.

When the sample volume is 18 L, the detection limit of the method is 2 mg/m³ and the lower limit of determination is 8 mg/m³. When the sample volume is

4.5 L, the detection limit of the method is 7 mg/m³ and the lower limit of determination is 28 mg/m³.

2 Normative references

This standard references the following documents or the terms. For undated references, the effective version applies to this standard.

Determination of Particulate Matter and Gaseous Pollutants in Exhaust of Fixed Pollution Sources GB/T 16157 HJ/T 373 fixed pollution source monitoring quality assurance and quality control technical specifications (Trial)

3 Terms and definitions

The following terms and definitions apply to this standard.

Total gaseous phosphorus Fixed pollution sources of gaseous single-phase phosphorus and phosphorus compounds in general.

4 Method principle

The gaseous total phosphorus is absorbed by brominated nitric acid absorption liquid and oxidized to orthophosphate. In acidic medium, orthophosphate and quinalocin Ketone to form a yellow quinoline phosphomolybdate $((C_9H_7N)_3H_3[PO_4 \cdot 12MoO_3] \cdot H_2O)$ precipitate. The precipitate is filtered and washed After dissolving in a known excess sodium hydroxide solution, after the completion of the reaction titration with hydrochloric acid standard solution of the remaining sodium hydroxide, the gaseous total phosphorus content is calculated based on the amount of sodium hydroxide standard solution consumed.

The reaction is as follows.

Absorption reaction.

$P_4 + 16H_2O + 10Br_2 = 4H_3PO_4 + 20HBr + 3P_4 + 20HNO_3 + 8H_2O = 12H_3PO_4 + 20NO + 8H_2O + 2Br_2 = 4H_3PO_4 + 4HBr + PH_3 + 4H_2O + 4Br_2 = H_3PO_4 + 8HBr$ Precipitation reaction.

$H_3PO_4 + 3C_9H_7N + 12Na_2MoO_4 + 24HNO_3 = (C_9H_7N)_3H_3[PO_4 \cdot 12MoO_3] \cdot H_2O + 11H_2O + 24NaNO_3$ Dissolution reaction.

$(C_9H_7N)_3H_3[PO_4 \cdot 12MoO_3] \cdot H_2O + 26NaOH = Na_2HPO_4 + 12Na_2MoO_4 + 3C_9H_7N + 15H_2O$ Titration reaction.

$NaOH + HCl = NaCl + H_2O$

5 Interference and elimination

Hydrogen sulfide has an impact on the sampling process, discoloration of brominated nitric acid absorption solution, thereby reducing the oxidation of brominated nitric acid, and

Sulfate and elemental sulfur are produced. Increase the first absorption of brominated bromine absorption of the amount of liquid nitrogen so that hydrogen sulfide in the first absorption bottle is fully absorbed within, eliminating the interference of hydrogen sulfide on the sampling process. Sulfate does not react with Quimociac because of simple substance Sulfur can not be dissolved in sodium hydroxide, it does not affect the measurement results. Fluoride, arsenic and their compounds have no interference with this method.

6 Reagents and materials

Unless otherwise specified, all analytically pure reagents that meet the national standards were used for the analysis. Water used for the experiment was prepared using 6.1.

6.1

Water. No carbon dioxide water, prepared by one of the following methods.

6.1.1

Boiling method.

Take appropriate deionized or distilled water into beakers as needed, boil for at least 10 minutes, or allow water to evaporate 10% on the cover cold.

6.1.2

aeration method.

Pass inert gas or pure nitrogen into deionized or distilled water to saturation.

6.2

Sodium molybdate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$).

6.3

Citric acid ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$).

6.4

Sodium hydroxide (NaOH). excellent grade pure.

6.5

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

6.6

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

6.7

Absolute ethanol. $\rho(\text{CH}_3\text{CH}_2\text{OH}) = 0.79 \text{ g/ml}$.