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HJ

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

HJ 867-2017

**Ambient air. Determination of phthalate esters. Gas
chromatography/mass spectrometry**

环境空气 酞酸酯类的测定 气相色谱—质谱法

(English Translation)

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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, protect the environment and protect people Health, standardize the determination of phthalates in ambient air, the development of this standard.

This standard specifies the determination of phthalates in ambient air by gas chromatography - mass spectrometry.

Appendix A of this standard is a normative appendix, Appendix B and Appendix C are informative appendices.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. Shenyang Environmental Monitoring Center Station.

This standard verification unit. Liaoning Province Environmental Monitoring Experimental Center, Dalian City Environmental Monitoring Center, Anshan City Environmental Monitoring Center Station, Fushun City Environmental Monitoring Center Station, Shenyang City Center for Disease Control and Prevention and Shenyang Environmental Protection Bureau Tiexi Branch Environmental Monitoring Station.

This standard MEP approved on November 28, 2017.

This standard since January 1, 2018 come into operation.

This standard is interpreted by the MEP.

1 Ambient air - Determination of phthalates - Gas chromatography - mass spectrometry

Warning. The solvents and reagents used in this method have some toxicity. The sample preparation process should be carried out in a fume hood and worn as required Protective equipment to avoid contact with skin and clothing.

1 scope of application this standard specifies the determination of phthalates in ambient air by gas chromatography - mass spectrometry.

This standard applies to the ambient air in the gas phase and particulate matter dimethyl phthalate, diethyl phthalate, phthalic acid two different Butyl, dibutyl phthalate, butyl benzyl phthalate, di (2-ethylhexyl) phthalate and di-n-octyl phthalate A total of 7 phthalates determination. If verified, this standard also applies to the determination of other phthalate compounds.

When the sampling volume is 144 m³ (standard state) and the volume of concentrated volume is 1.0 ml, the detection limit of the method is 0.003 µg/m³ to 0.004 µg/m³, and the lower limit of determination is 0.012 µg/m³ to 0.016 µg/m³. See Appendix A for details.

2 Normative references

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

HJ 691 Environmental airborne semi-volatile organic compounds sampling techniques

3 Method principle

Phthalates in ambient air are collected on glass (or quartz) fiber filters using a medium- or high-flow sampler. Polyurethane foam (PUF) and XAD-2 resin, extracted with ether-n-hexane mixed solution, the extract was concentrated, purified, and analyzed by gas

chromatography-mass spectrometry Instrument separation test. According to the retention time and characteristic ion abundance ratio qualitative, internal standard method.

4 Interference and elimination

4.1

Disturbances with the same retention time At the onset of an interference, the qualifier ions can be detected by mass spectrometry to distinguish them.

4.2

Purification using magnesium silicate column can remove or reduce interference, see 7.3.3.

5 Reagents and materials

Unless otherwise specified, analytical grade analytical reagents used in accordance with national standards were used. Experimental water is freshly prepared ultrapure water.

5.1

Dichloromethane (CH_2Cl_2). Chromatographic purity.

5.2

n-Hexane (C_6H_{14}). Chromatographic purity.

5.3

ether ($\text{C}_4\text{H}_{10}\text{O}$). Chromatography.

5.4

Acetone ($\text{C}_3\text{H}_6\text{O}$). Chromatography pure.

5.5 Anhydrous sodium sulfate (Na_2SO_4)

Bake in a muffle furnace at $450\text{ }^\circ\text{C}$ for 4 h before use, cool, and store in sealed glass jar.

25.6 decafluorotriphenylphosphine (DFTPP). $\rho = 50\text{ }\mu\text{g/ml}$, can be purchased directly from the certified public standard solution, or with a high concentration of standard solution with system.

5.7 ether - n-hexane mixed solution. 1 9, Pro use now.

5.8 ether - n-hexane mixed solution. 2 8, Pro use now.

5.9 acetone - n-hexane mixed solution. 1 9, Pro use now.

5.10 methylene chloride - n-hexane mixed solution. 2 8, Pro use now.

5.11 diphenyl phthalate ($\text{C}_{20}\text{H}_{14}\text{O}_4$). purity of 98% or more.

Alternatives may also be used.

5.12 Substitute stock solution. $\rho = 2000 \mu\text{g/ml}$.

Weigh diphenyl phthalate (5.11) about 0.1 g, accurate to 0.1 mg, in 50 ml volumetric flask, dissolved in a small amount of acetone, with n-hexane (5.2) constant volume, mix well. 4°C the following sealed. Can also be purchased directly from the market certified standard solution.

5.13 Alternative solution. $\rho = 80.0 \mu\text{g/ml}$.

Pipette 1.00 ml of stock solution (5.12) into a 25 ml volumetric flask, dilute with n-hexane (5.2) and mix well. 4°C below the dense Save sealed.

5.14 Internal standard stock solution. $\rho = 4000 \mu\text{g/ml}$.

Direct purchase of a certified public standard solution, with γ -D10, Philippines-D10, -D12, reference standard solution certificate.

5.15 Internal standard solution. $\rho = 1000 \mu\text{g/ml}$.

The internal standard stock solution (5.14) was diluted to $1000 \mu\text{g/ml}$ with dichloromethane (5.1). 4°C below the sealed dark save.

5.16 Diisobutyl phthalate Reference material. purity 98% or more.

5.17 Diisobutyl phthalate stock solution. $\rho = 2000 \mu\text{g/ml}$.

Weigh 0.1 g of diisobutyl phthalate standard (5.16), accurate to 0.1 mg in a 50 ml volumetric flask with n-hexane (5.2) Dissolve and set the volume, mix well. 4°C the following sealed. Can also be purchased directly from the market certified standard solution.

5.18 6 phthalates standard stock solution. $\rho = 2000 \mu\text{g/ml}$.

Direct purchase of a certified commercial standard solution, including dimethyl phthalate, diethyl phthalate, dibutyl phthalate, o Butyl phthalate, di-2-ethylhexyl phthalate and di-n-octyl phthalate in n-hexane.

4°C below the preservation, or reference standard solution certificate preservation.

5.19 8 phthalates standard solution. $\rho = 80.0 \mu\text{g/ml}$.

Remove phthalate standard stock solution (5.17), (5.18) and replacement stock solution (5.12) each 1.00 ml, in 25 ml capacity Volume flask with n-hexane (5.2) constant volume, mix well. 4°C the following sealed.

5.20 Magnesium silicate. $150 \mu\text{m}$ to $250 \mu\text{m}$ (100 mesh to 60 mesh). Before use, weigh $100 \text{ g} \pm 10 \text{ g}$ into an evaporating dish at 140°C Heated 16 h, while hot transferred to 500 ml grinding mouth reagent bottle, capped cooled to room temperature, add 3.0 ml $\pm 0.1 \text{ ml}$ experimental water, sealed, Shake well for 10 min and let stand for 2 h.