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

HJ 868-2017

**Ambient air. Determination of phthalate esters. High
performance liquid chromatography**

环境空气 酞酸酯类的测定 高效液相色谱法

(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 high-performance liquid chromatography of phthalates esters in the air.

This standard Appendix A normative appendix, Appendix B is an informative annex.

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 phthalates Determination of high performance liquid chromatography

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 high-performance liquid chromatography of phthalates esters in the air.

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 this method is 0.002 µg/m³ ~ 0.006 µg/m³, and the lower limit of determination is 0.008 µg/m³ ~ 0.024 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 a mixture of ether and n-hexane, the extract was concentrated, purified, with a UV detector HPLC separation test, according to the retention time of qualitative, external standard method.

4 Interference and elimination

Purification using a magnesium silicate column removes or reduces interference as detailed in 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

Acetonitrile (CH_3CN). Chromatographic purity.

5.6 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.

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

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. $\rho = .2000\text{ }\mu\text{g/ml}$.

Weigh about 0.1 g of diphenyl phthalate (5.11), accurate to 0.1 mg, dissolved in acetonitrile (5.5) in a 50 ml volumetric flask and set the volume, shake well. $4\text{ }^\circ\text{C}$ the following sealed. Can be purchased directly from the market certified standard solution, reference standard solution certificate for preservation.

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

Pipette 1.00 ml of stock solution (5.12) into a 25 ml volumetric flask, dilute to volume with acetonitrile (5.5) and mix well. $4\text{ }^\circ\text{C}$ below the dense Save sealed.

5.14 Diisobutyl phthalate Standard substance. purity 98% or more.

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

Weigh about 0.1 g of diisobutyl phthalate (5.14), accurate to 0.1 mg in a 50 ml volumetric flask, dissolve it in acetonitrile (5.5) Solution and set the volume, shake well. Can be purchased directly from the market certified standard solution. 4 °C the following sealed.

5.16 6 phthalates standard stock solution. $\rho = 2000 \mu\text{g/ml}$.

Buy a commercially available standard solution of acetonitrile or methanol as a solvent, including dimethyl phthalate, diethyl phthalate, Dibutyl phthalate, butyl benzyl phthalate, di-2-ethylhexyl phthalate and di-n-octyl phthalate. 4 °C the following seal preservation, or reference standard solution certificate to save.

5.17 8 phthalates standard solution. $\rho = 80.0 \mu\text{g/ml}$.

Pipette 1.00 ml stock solution (5.12) and 1.00 ml phthalate standard stock solution (5.15), (5.16) in 25 ml Volumetric flask, with acetonitrile (5.5) constant volume, mix well. 4 °C the following sealed.

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

5.19 Magnesium silicate solid phase extraction column. 1000 mg/6 ml, but also according to the content of impurities to select the appropriate volume of commercial solid phase extraction column.

5.20 glass (or quartz) fiber filter. According to the sampling head to select the appropriate size of the filter. The retention of the membrane on 0.3 μm standard particles the rate of not less than 99%, air flow velocity of 0.45 m/s, leaflets filter resistance of not more than 3.5 kPa.

Before use in a muffle furnace at 400 °C heating 5 h or more, cooled, wrapped with aluminum foil, stored in a filter box, to avoid direct connection with the plastic Touch, to ensure that the filter before sampling and sampling is not stained, and in the flat before the sample is not folded state.

5.21 XAD-2 resin (styrene-divinylbenzene polymer). 250 μm to 840 μm (60 mesh to 20 mesh).

Before extraction with dichloromethane (5.1) reflux 16 h, the replacement of dichloromethane (5.1) to continue reflux extraction 16 h, then ether -N-hexane mixed solution (5.7) refluxed 16 h, vacuum dried at room temperature 2 h ~ 3 h. Keep in a clean glass bottle.

5.22 Polyurethane Foam (PUF). polyether type, with a density of 22 mg/cm³ to 25 mg/cm³, cut and grown 25 mm and 50 mm in diameter 3 is a cylinder of 45 mm to 65 mm (length,