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HJ

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

HJ 869-2017

**Stationary source emission. Determination of phthalate esters.
Gas chromatography**

固定污染源废气 酞酸酯类的测定 气相色谱法
(English Translation)

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1 Scope of application

This standard specifies the determination of fixed sources of emissions of organized emissions of phthalates ester gas chromatography.

This standard applies to fixed sources of emissions of organized emissions of dimethyl phthalate, diethyl phthalate, two Butyl phthalate, butyl benzyl phthalate, di-2-ethylhexyl phthalate and di-n-octyl phthalate were measured.

If verified, this standard also applies to the determination of other phthalates.

The detection limit of the method is $0.4 \mu\text{g}/\text{m}^3 \sim 0.8 \mu\text{g}/\text{m}^3$ when the sampling volume is 1 m^3 (dry sampling volume in the standard state) the limit is $1.6 \mu\text{g}/\text{m}^3 \sim 3.2 \mu\text{g}/\text{m}^3$. 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.

Determination of Particulate Matter and Gaseous Pollutants in Exhaust of Fixed Pollution Sources GB/T 16157 HJ/T 48 dust sampler technical conditions HJ/T 373 fixed pollution source monitoring quality assurance and quality control technical specifications (Trial) HJ/T 397 fixed source exhaust gas monitoring technical specifications

3 Method principle

Constant-speed sampling mode, so that fixed sources of exhaust through the heated sampling tube, cooling device and the resin column, particulate and gas phase of the phthalates were collected on glass (or quartz) fiber filters (or cartridges) and XAD-2 resin, respectively, while the condensate was collected.

The filter (or cartridge) and XAD-2 resin were extracted with ether-n-hexane mixed solution, the condensate was extracted with n-hexane, the extract was concentrated, the net After purification, with hydrogen flame ionization detector gas chromatography detection, according to retention time qualitative, internal standard method or 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, the analysis of analytical reagents are used in line with national standards of analytical chemistry, experimental water is freshly prepared pure water.

25.1 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°C for 4 h before use, cool, and seal in a milled glass jar.

5.6

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

5.7

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

5.8

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

5.9

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

5.10

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

Alternatives may also be used.

5.11

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

Weigh diphenyl phthalate (5.10) 0.1 g (accurate to 0.1 mg), in a 50 ml volumetric flask, add a little acetone (5.4) Dissolve and dilute to volume with n-hexane (5.2) and mix well. 4°C the following sealed.

5.12

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

Pipette 1.00 ml of stock solution (5.11) into a 25 ml volumetric flask, dilute with n-hexane (5.2) and mix well. 4 °C below the dense Save sealed.

5.13

Benzyl benzoate ($\text{C}_{14}\text{H}_{12}\text{O}_2$). purity of 98% or more.

Internal standard, can also use other analogues.

5.14

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

Weigh benzyl benzoate (5.13) 0.1 g (accurate to 0.1 mg), in a 50 ml volumetric flask, add a small amount of methylene chloride (5.1) dissolved Solution and n-hexane (5.2) constant volume, mix well. 4 °C the following sealed.

5.15

phthalate 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 benzyl phthalate, di (2-ethylhexyl) phthalate and di-n-octyl phthalate, the solvent is n-hexane, the following 4 °C dense Seal preservation, or reference standard solution certificate preservation.

5.16

phthalate mixed standard solution. $\rho = 80.0 \mu\text{g/ml}$.

Remove phthalate standard stock solution (5.15) and replacement stock solution (5.11) 1.00 ml in 25 ml volumetric flask with n-hexane (5.2) constant volume, mix well. 4 °C the following sealed.

5.17

Magnesium silicate. 150 μm to 250 μm (100 mesh to 60 mesh).

Before use, weighed 100 g $\pm$ 10 g into the evaporating dish, heated at 140 °C for 16 h, while hot transfer to 500 ml grinding mouth reagent bottle, Capped, cooled to room temperature, add 3.0 ml $\pm$ 0.1 ml experimental water, sealed, shake thoroughly mixed 10 min, allowed to stand for 2 h before use.

5.18

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 magnesium silicate SPE column.