

CCS Z 18

HJ

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

HJ 1184-2021

**Soil and sediments Determination of 6 phthalate compounds by
gas chromatography-mass spectrometry**

土壤和沉积物 6 种邻苯二甲酸酯类化合物的测定 气相色谱-质谱法

(English Translation)

Issued on: January 1, 1970

Implemented on: January 1, 1970

Issued by: Ministry of Ecology and Environment

Table of Contents

Foreword

1 Scope

2 Normative references

3 Terms and Definitions

4 Principles of the method

5 Interference and cancellation

6 Reagents and materials

7 Instruments and equipment

8 Samples

9 Analysis steps

10 Result calculation and representation

11 Accuracy

12 Quality Assurance and Quality Control

13 Waste Disposal

14 Notes

Appendix A (normative appendix) detection limit and lower limit of determination of the method

Appendix B (informative appendix) Determination reference parameters of the target

Appendix C (informative) Accuracy of the method

1 Scope of application

This standard specifies the gas chromatography-mass spectrometry method for the determination of six phthalates in soil and sediment.

This standard applies to dimethyl phthalate (DMP), diethyl phthalate (DEP), phthalate in soil and sediments Di-n-butyl phthalate (DBP), butyl benzyl phthalate (BBP), bis(2-ethylhexyl) phthalate (DEHP) and phthalate Determination of 6 kinds of phthalate compounds such as di-n-octyl dicarboxylate (DNOP). Other phthalates are verified can also be determined by this method.

When the sampling volume is 10.0 g, the extraction volume is 20.0 ml, and the injection volume is 1.0 μ l, this standard method for the determination of phthalates the detection limit is 0.02 mg/kg~0.05 mg/kg, and the lower limit of determination is 0.08 mg/kg~0.20 mg/kg. See Appendix A for details.

2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to this standard.

GB 17378.3 Marine Monitoring Specification Part 3: Sample Collection, Storage and Transportation GB 17378.5 Marine Monitoring Specification Part 5: Sediment Analysis HJ/T 166 Technical Specification for Soil Environment Monitoring HJ 494 Water Quality Sampling Technical Guide HJ 613 Gravimetric Method for Determination of Soil Dry Matter and Moisture HJ 834 Determination of Semivolatile Organic Compounds in Soil and Sediments by Gas Chromatography-Mass Spectrometry

3 Terms and Definitions

The following terms and definitions apply to this standard.

Phthalates phthalate esters Also known as phthalate esters (PAEs), it is a general term for ester compounds formed by phthalic acid, generally referring to phthalates.

Esters of dicarboxylic acid and alcohols of 4 to 15 carbons.

4 Principles of the method

Phthalates in soil and sediment were extracted with ethyl acetate, separated by gas chromatography and detected by mass spectrometry. According to retention Time, mass-to-charge ratio of fragment ions and their abundance were qualitative, and internal standard method was used for quantification.

5 Interference and cancellation

5.1

In the process of sample collection and preservation, use metal sampling utensils, brown ground-mouth jars with stoppers, and wrap them in aluminum foil (6.17).

Wrap to eliminate distractions.

5.2

When weighing the sample, remove the sample on the surface of the bottle mouth to eliminate interference.

5.3

During the injection process, the injection septum of the gas chromatograph will cause positive interference to the measurement results, which cannot be eliminated by the interference of the instrument itself.

Note: Try to choose septa with good quality and low blank.

5.4

The bottle gasket of the sample bottle (7.4) will interfere with the measurement results, which can be avoided by placing a layer of aluminum foil (6.17) between the sample bottle and the bottle cap.

method to eliminate interference.

6 Reagents and Materials

Unless otherwise specified, analytical reagents that meet national standards were used in the analysis. The experimental water was freshly prepared distilled water with clean glass container.

6.1

Dichloromethane (CH_2Cl_2). pesticide residue grade.

6.2

Acetone (CH_3COCH_3). pesticide residue grade.

6.3

Ethyl acetate ($\text{CH}_3\text{COOC}_2\text{H}_5$). pesticide residue grade.

6.4

Phthalates mixed standard stock solution. $\rho = 1\,000\text{ mg/L}$.

Commercially available certified standard solutions can be purchased directly, stored in the refrigerator below 4°C , protected from light, or stored with reference to the standard solution certificate. use should be returned to room temperature and shaken.

6.5

Phthalate mixed standard intermediate solution. $\rho = 100\text{ mg/L}$.

Pipette 1.00 ml of phthalate mixed standard stock solution (6.4) into a 10 ml brown volumetric flask, dilute with ethyl acetate (6.3) and make up to volume to the marking line, shake well, transfer to a compact bottle (7.10), refrigerate below 4°C , and store in the dark.

6.6

Phthalate mixed standard solution. $\rho = 20.0\text{ mg/L}$.

Pipette 2.00 ml of phthalate mixed standard intermediate solution (6.5) into a 10 ml brown volumetric flask, dilute with ethyl acetate (6.3) and make up to volume to the marking line, shake well, transfer to a compact bottle (7.10), refrigerate below 4°C, store in the dark, and store for no more than 7 days.

6.7

Substitute stock solution. $\rho = 5\,000\text{ mg/L}$.

Benzyl benzoate (BBZ) was chosen as an alternative. Commercially available certified standard solutions can be directly purchased, refrigerated below 4°C, and stored in the dark.

Or refer to the standard solution certificate for preservation. Return to room temperature and shake well before use.

6.8

Substitute intermediate solution. $\rho = 100\text{ mg/L}$.

Pipette 200 μl of substitute stock solution (6.7) into a 10 ml brown volumetric flask, dilute with ethyl acetate (6.3) and make up to the mark, shake Homogenize, transfer to a compact bottle (7.10), refrigerate below 4°C, and store in the dark.

6.9

Substitute liquid. $\rho = 20.0\text{ mg/L}$.

Pipette 2.00 ml of substitute intermediate solution (6.8) into a 10 ml brown volumetric flask, dilute with ethyl acetate (6.3) and dilute to the mark, shake Homogenize, transfer to a compact bottle (7.10), refrigerate below 4°C, and store in the dark. Return to room temperature and shake well before use.

6.10

Internal standard stock solution. $\rho = 100\text{ mg/L}$.

Diamyl phthalate-d4 (DPP-d4) and bis(2-ethylhexyl) phthalate-d4 (DEHP-d4) were selected as internal standards.

Commercially available certified standard solutions can be purchased directly, stored in the refrigerator below 4°C, protected from light, or stored with reference to the standard solution certificate. should be used Return to room temperature and shake.

6.11

Internal standard solution. $\rho = 20.0\text{ mg/L}$.

Pipette 2.00 ml of internal standard stock solution (6.10) into a 10 ml brown volumetric flask, dilute with ethyl acetate (6.3) and dilute to the mark, shake Homogenize, transfer to a compact bottle (7.10), refrigerate below 4°C, and store in the dark. Return to room