

ICS 13.080
CCS Z 18

HJ

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

HJ 736-2015

**Soil and sediment. Determination of volatile halohydrocarbons.
Headspace gas chromatography mass spectrometry**

土壤和沉积物 挥发性卤代烃的测定 顶空 气相色谱-质谱法

(English Translation)

Issued on: February 7, 2015

Implemented on: April 1, 2015

Issued by: Ministry of Ecology and Environment

Table of Contents

Foreword

1 Scope of application

2 Normative reference documents

3 Principle of the method

4 Reagents and materials

5 Instruments and equipment

6 Samples

6.1 Collection of samples

6.2 Storage of samples

6.3 Preparation of the sample

6.4 Preparation of blank samples

47 Analysis steps

Foreword

For the implementation of the "People's Republic of China Environmental Protection Law" to protect the environment, protect human health, regulate soil and sediment Determination of volatile halogenated hydrocarbons, the development of this standard.

This standard specifies the headspace/gas chromatography-mass spectrometry for the determination of 35 volatile halogenated hydrocarbons such as methyl chloride in soil and sediments.

This standard is the first release.

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

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The main drafting unit of this standard. Harbin Environmental Monitoring Center Station.

The standard verification unit. Heilongjiang Province Environmental Monitoring Center Station, Dalian Environmental Monitoring Center, Changchun City Environmental Monitoring Heart station, Anshan City Environmental Monitoring Center Station, Qiqihar City Environmental Monitoring Center Station and Daqing City Environmental Monitoring Center Station.

This standard is approved by the Ministry of Environmental Protection on February 7, 2015.

This standard is implemented as of April 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

Determination of volatile halohydrocarbons in soil and sediments Headspace/gas chromatography - mass spectrometry WARNING. The internal standard, substitute and standard solution used in the test are volatile, and the solution should be prepared.

Fume hood to operate; should be required to wear protective equipment, to avoid contact with skin and clothing.

1 Scope of application

This standard specifies headspace/gas chromatography-mass spectrometry for the determination of volatile halogenated hydrocarbons in soil and sediments.

This standard is applicable to the determination of 35 kinds of volatile halogenated hydrocarbons such as methyl chloride in soil and sediments. Other volatile halogenated hydrocarbons If verification is also applicable to this standard.

When the sampling amount of 2g, 35 kinds of volatile halogenated hydrocarbons detection limit of 2 ~ 3 µg/kg, the determination of the lower limit of 8 ~ 12 µg/kg. See Appendix A. for details.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

GB 17378.3 Marine Monitoring Specification Part 3 Sample Collection Storage and Transportation GB 17378.5 Marine monitoring regulations Part 5 Sediment analysis Determination of Dry Matter and Moisture in Soil Technical specification for soil environmental monitoring

3 Principle of the method

Under certain temperature conditions, the volatile halogenated hydrocarbons in the sample of the headspace bottle evaporate to the liquid space, resulting in a certain amount of steam Pressure, and to achieve gas-liquid three-phase equilibrium, take the gas sample into the gas chromatographic separation, with a mass spectrometer for testing. according to Retention time, fragment ion mass ratio and different ion abundance ratio qualitative, internal standard method quantitative.

4 Reagents and materials

4.1

Experimental water. secondary distilled water or pure water equipment to prepare water, before use to go through blank test to confirm that no target compounds or the target compound concentration is below the method detection limit.

4.2

methanol (CH₃OH). pesticide residues, before use to be tested to confirm that no target or target concentration is lower than the method of inspection Limit.

4.3

sodium chloride (NaCl). excellent grade, in the muffle furnace at 400 °C baking 4h, placed in a dryer to cool to room temperature, Stored in a brown glass bottle.

4.4

phosphoric acid (H₃PO₄). excellent grade pure.

4.5

Substrate modifier. Phosphoric acid (4.4) was added dropwise to 100 ml of experimental water to adjust the pH of the solution to less than 2;

36 g of sodium chloride (4.3). Stored at 4 °C, can be stored for 6 months.

24.6 standard stock solution. rho = .2000mg/L.

Direct purchase of commercially available standard solution. Store at or below -10 ° C, or refer to the manufacturer's product description. use should be restored to room temperature, and shake. Kaifeng in a dense bottle can be kept for a month.

4.7

standard use of liquid. rho = 20mg/L.

Take appropriate standard stock solution (4.6) and dilute with methanol (4.2). In the dense bottle -10 °C below the light protection Save, save for a week.

4.8

internal standard stock solution. rho = .2000mg/L.

Fluorobenzene, 1-chloro-2-bromopropane, 4-bromofluorobenzene as internal standard. Can be purchased directly to prove the standard solution can also be used standard Material preparation. Store in light below -10 ° C or refer to the manufacturer's product description. Use should be restored to room temperature, and shake.

Kaifeng in a dense bottle can be kept for a month.

4.9

internal standard use of liquid. $\rho = 25\text{mg/L}$.

Take appropriate internal standard stock solution (4.8) and dilute with methanol (4.2). In the dense bottle $-10\text{ }^{\circ}\text{C}$ below the light protection Save, save for a week.

4.10

Alternative stock solution. $\rho = .2000\text{mg/L}$.

Use dichloromethane- d_2 , 1,2-dichlorobenzene- d_4 as a substitute. Can be purchased directly to prove the standard solution can also be used standard Material preparation. Store in light below $-10\text{ }^{\circ}\text{C}$ or refer to the manufacturer's product description. Use should be restored to room temperature, and shake.

Kaifeng in a dense bottle can be kept for a month.

4.11

Substitute liquid. $\rho = 25\text{ mg/L}$.

Take appropriate stock solution (4.10) and dilute it with methanol (4.2). In the dense bottle $-10\text{ }^{\circ}\text{C}$ below the dark Save, save for one week.

4.12 4-bromofluorobenzene (BFB) solution. $\rho = 25\text{ mg/L}$.

Can be directly purchased certified standard solution can also be prepared with standard materials. Store at or below $-10\text{ }^{\circ}\text{C}$ or refer to the manufacturer of the product description. Use should be restored to room temperature, and shake. Kaifeng in a dense bottle can be kept for a month.

4.13

quartz sand. 20 to 50 mesh, before use to be tested to confirm that no target compound or target compound concentration below Legal limit.

4.14

helium. purity $\geq 99.999\%$, deoxidized by deoxidizer, molecular sieve dehydration.

5 Instruments and equipment

5.1

Sampling equipment. shovel and stainless steel spoon.

5.2

Sampling Bottle. 60ml threaded brown glass bottle with polytetrafluoroethylene liner.