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

HJ 735-2015

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

土壤和沉积物 挥发性卤代烃的测定 吹扫捕集 气相色谱-质谱法

(English Translation)

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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 determination of 35 kinds of volatile halogenated hydrocarbons such as methyl chloride in soil and sediments. Purge and capture/gas chromatography Spectrum method.

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 Purge and trap/gas chromatography - mass spectrometry WARNING. The internal standards, substitutes and standard solutions used in the tests are volatile, toxic chemicals.

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 the Purge and Trap/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 such as the validation is also applicable to this standard.

When the sampling amount of 5g, 35 kinds of volatile halogenated hydrocarbons detection limit of 0.3 ~ 0.4 µg/kg, the determination of the lower limit of 1.2 ~ 1.6 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

The volatile halogenated hydrocarbons in the sample are purged with high purity helium (or nitrogen) and adsorbed in the trap, Heat and re-blow with helium (or nitrogen), the volatile halogenated hydrocarbons in the trap are desorbed by heat, and the components are passed into the gas chromatographic fraction After the separation, with a mass spectrometer for testing. According to the 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 water equipment prepared by water. Before use to be blank test, to confirm no target or the target concentration is lower than 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 detection limit.

4.3

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. Should be used when used Revert to room temperature and shake. Kaifeng in a dense bottle can be kept for a month.

4.4

standard use of liquid. rho = 2.5 mg/L.

2 Take appropriate standard stock solution (4.3) and dilute it with methanol (4.2). In the dense bottle -10 °C below the dark preservation, can save for a week.

4.5

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 Quality preparation. Store in light below -10 ° C or refer to the manufacturer's product description. Use should be restored to room temperature, and shake. open Sealed in a dense bottle can be kept for a month.

4.6

internal standard use of liquid. rho = 2.5mg/L.

Take appropriate internal standard stock solution (4.5) and dilute it with methanol (4.2). In the dense bottle -10 °C below the dark preservation, can save for a week.

4.7

Substitute stock solution. rho = .2000mg/L.

Use dichloromethane-d₂,1,2-dichlorobenzene-d₄ as a substitute. Can be purchased directly to prove the standard solution can also be used standard Quality preparation. Store in light below -10 ° C or refer to the manufacturer's product description. Use should be restored to

room temperature, and shake. open Sealed in a dense bottle can be kept for a month.

4.8

Substitute liquid. $\rho = 2.5\text{mg/L}$.

Take appropriate amount of the stock solution (4.7) and dilute it with methanol (4.2). In the dense bottle $-10\text{ }^{\circ}\text{C}$ below the light protection Save, save for a week.

4.9 4-bromofluorobenzene (BFB) solution. $\rho = 25\text{mg/L}$, can be directly purchased certified standard solution can also be prepared using standard materials. in $-10\text{ }^{\circ}\text{C}$ or less in the dark to save or refer to the manufacturer's product description. Use should be restored to room temperature, and shake. After opening in the dense the bottle can be kept for one month.

4.10

quartz sand. 20 ~ 50 mesh. Before use, pass the test to confirm that no target or target is below the method detection limit.

4.11

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. Polytetrafluoroethylene Silicone Tape Screw Cover 60ml wide-mouth glass bottle.

5.3

Vials. 40ml brown glass bottles and colorless glass bottles with PTFE cover screw cap.

5.4

Gas Chromatography-Mass Spectrometer. EI Ionization Source.

5.5

Column. quartz capillary column, length 30 m, inner diameter 0.25 mm, film thickness 1.4 μm , stationary phase 6% nitrile phenyl/94% Dimethylpolysiloxane, and other equivalent capillary columns may also be used.

5.6

Purge and trap device. suitable for soil sample determination. The capture tube was mixed with 1/3 Tenax, 1/3 silica gel, 1/3 activated carbon Adsorbents or other equivalent adsorbents.