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

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**Soil and sediment. Determination of volatile organic
compounds. Headspace gas chromatography**

土壤和沉积物 挥发性有机物的测定 顶空气相色谱法

(English Translation)

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Foreword

To implement the Environmental Protection Law of the People's Republic of China, protect the environment, protect human health, regulate soil and sediment in the determination of volatile organic compounds, the development of this standard.

This standard specifies the headspace/gas chromatography of volatile organic compounds in soil and sediments.

This standard is the first release.

Appendix A of 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. Anshan City Environmental Monitoring Center Station.

The standard verification unit. Liaoning Province Environmental Monitoring Experimental Center, Shenyang City Environmental Monitoring Center Station, Dalian City Environmental Monitoring Center, Harbin Environmental Monitoring Center, Fushun City Environmental Monitoring Center Station and Liaoyang City Environmental Monitoring Center Station.

The environmental protection department of this standard approved on May 4, 2015.

This standard has been implemented since July 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

Determination of Volatile Organic Compounds in Soil and Sediments Headspace/gas chromatography WARNING. The organic reagents and standard solutions used in the test are volatile, and the procedure should be in the ventilation Cabinet to operate, should be required to wear protective equipment, to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the headspace/gas chromatographic method for the determination of 37 volatile organic compounds in soil and sediments.

This standard applies to the determination of 37 volatile organic compounds in soil and sediments. Other volatile organic compounds if verified can also be applied to this standard.

When the amount of soil and sediment samples is 2 g, the detection limits of 37 volatile organic compounds are 0.005 mg/kg ~ 0.03 Mg/kg, the determination of the lower limit of 0.02 mg/kg ~ 0.12 mg/kg, see Appendix A.

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.

Specification for ocean monitoring - Part 3. Sample collection, storage and transportation
GB 17378.3 Specification for marine monitoring - Part 5. Sediment analysis Determination of Dry Matter and Moisture in Soil Technical specification for soil environmental monitoring

3 Principle of the method

At a certain temperature, the volumetric organic matter in the sample of the headspace bottle evaporates to the liquid space, and the gas-liquid three-phase reaches the heat Mechanically dynamic equilibrium. The volatile organic compounds in the gas phase are separated by gas chromatography and detected by flame ionization detector. To protect Time qualitative, external standard method.

4 Reagents and materials

4.1

Experimental water. secondary distilled water or water prepared by pure water equipment.

Before use, subject to a blank test to confirm that the target compound in the retention time interval without interference peaks appear or its the concentration of the target compound in the method is lower than the method detection limit.

4.2

Methanol (CH_3OH). Chromatographic Purification.

Before use, subject to inspection to confirm that the target compound or target compound concentration is lower than the method detection limit.

4.3

sodium chloride (NaCl). excellent grade pure.

Baked in a muffle furnace at 400°C for 4 h, placed in a desiccator and cooled to room temperature and transferred to a glass vial.

4.4

phosphoric acid (H_3PO_4). excellent grade pure.

4.5

saturated sodium chloride solution.

500 ml of experimental water (4.1), add a few drops of phosphoric acid (4.4) to adjust $\text{pH} \leq 2$, add 180 g of sodium chloride (4.3), Dissolve and mix. Stored at 4°C , can be stored for 6 months.

24.6 standard stock solution. $\rho = 1000 \text{ mg/L} \sim 5000 \text{ mg/L}$.

Can be purchased directly to prove the standard solution can also be prepared with standard substances.

4.7

standard use of liquid. $\rho = 10 \text{ mg/L} \sim 100 \text{ mg/L}$.

The standard compound of the target compound is stored in a dense bottle for a period of 30 days or formulated with reference to the manufacturer's instructions.

4.8

quartz sand (SiO_2). analytical grade, 20 mesh to 50 mesh.

Before use, verify that the target compound or target compound concentration is below the method detection limit.

4.9

Carrier gas. high purity nitrogen ($\geq 99.999\%$), deoxidized by deoxidizer, dehydrated by molecular sieve.

4.10

Gas. high purity hydrogen ($\geq 99.999\%$), dehydrated by molecular sieve.

4.11

to help gas. air, dehydrated by silica gel, activated carbon off organic matter.

Note 1: All of the above standard solutions are methanol as solvent, the standard solution after preparation or opening should be placed in a dense bottle, below 4°C Deposit, the preservation period is generally 30 d. Before use should be restored to room temperature, mix.

5 Instruments and equipment

5.1

Gas chromatograph. with capillary split/splitless inlet, programmable temperature rise, with hydrogen flame ionization detector (FID).

5.2

Column. quartz capillary column. Column $1.60 \text{ m} \times 0.25 \text{ mm}$, film thickness 1.4 m (6% nitrile phenyl, 94% dimethyl Base polysiloxane fixative), other equivalent capillary columns may also be used. Column $2.30 \text{ m} \times 0.32 \text{ mm}$, film thickness 0.25 m (poly Diol-20M), other equivalent capillary columns may also be used.

5.3

Automatic headspace sampler. headspace bottle (22 ml), gasket (polytetrafluoroethylene/silicone material), cap (screw cap or Once used glands).

5.4

reciprocating oscillator. oscillation frequency 150 times/min, can be fixed head empty bottle.