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

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**Soil and sediment. Determination of acrolein, acrylonitrile,
acetonitrile. Headspace-Gas chromatography method**

土壤和沉积物 丙烯醛、丙烯腈、乙腈的测定 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, and regulate soil and sediment this standard is formulated for the determination of acrolein, acrylonitrile and acetonitrile.

This standard specifies headspace-gas chromatography for the determination of acrolein, acrylonitrile and acetonitrile in soils and sediments.

This standard is the first release.

Appendix A of this standard is an informative annex.

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard is mainly drafted by. Ningbo Environmental Monitoring Center.

This standard is verified by. Zhoushan Marine Ecological Environment Monitoring Station, Taizhou Environmental Monitoring Center Station, Hangzhou Environment Monitoring Center Station, Jiaxing Environmental Protection Monitoring Station, Huzhou Environmental Protection Monitoring Center Station and Jinhua Environmental Monitoring Center Station.

This standard was approved by the Ministry of Environmental Protection on November 21, 2013.

This standard has been implemented since February 1, 2014.

This standard is explained by the Ministry of Environmental Protection.

Determination of acrolein, acrylonitrile and acetonitrile in soils and sediments

Headspace-gas chromatography Warning. The reagents and standard solutions used in the test are volatile and toxic compounds. They should be well ventilated during preparation.

Carry out in the environment; wear protective equipment as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies headspace-gas chromatography for the determination of acrolein, acrylonitrile and acetonitrile in soils and sediments.

This standard applies to the determination of acrolein, acrylonitrile and acetonitrile in soil

and sediment.

When the sampling amount is 2.0g, the detection limit of acrolein is 0.4mg/kg, the lower limit of determination is 1.6mg/kg; the detection of acrylonitrile the limit is 0.3 mg/kg, the lower limit of determination is 1.2 mg/kg; the detection limit of acetonitrile is 0.3 mg/kg, and the lower limit of determination is 1.2 mg/kg.

2 Normative references

The contents of this standard refer to the following documents or their terms. For undated references, the valid version is appropriate.

Used in 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 Specifications for Soil Environmental Monitoring
HJ 613 Determination of dry matter and moisture in soils - Gravimetric method

3 Principle of the method

The sample sealed in the headspace bottle, under certain temperature conditions, the acrolein, acrylonitrile, acetonitrile contained in the sample volatilize to the upper space, and achieve thermodynamic dynamic balance in gas-liquid-solid three-phase. Take a certain amount of gas-phase gas injection zone in the headspace bottle Separation and determination were carried out in a gas chromatograph with a hydrogen flame detector. Characterize by retention time and quantify by external standard method.

4.1 Experimental water

Freshly prepared organic-free deionized or distilled water. A blank test is required before use to confirm that there is no target compound or the target compound concentration is below the method detection limit.

4.2 Sodium chloride (NaCl). excellent grade pure. Bake at 400 ° C for 4 h to remove possible interfering substances, cool and store in the grinding the glass bottle is sealed and stored.

4.3 Methanol (CH₃OH). Chromatographic grade. Test before use to confirm that there is no target compound or low concentration of target compound the method detection limit.

4.4 Phosphoric acid (H₃PO₄). excellent grade pure.

4.5 Matrix modifier

Measure 500 ml of experimental water (4.1), add a few drops of phosphoric acid (4.4) to adjust $\text{pH} \leq 2$, and then add 180 g of sodium chloride (4.2).

Dissolve and mix. Sealed and stored below 4 °C in an environment free from organic interference. The shelf life is 6 months.

4.6 Standard solution of acrolein, acrylonitrile and acetonitrile in methanol. $\rho = 2000 \text{ mg/L}$

Prepare with a standard solvent such as acrolein, acrylonitrile or acetonitrile with methanol as solvent, or directly purchase a commercially available certified standard solution.

The standard solution is stored at -18 °C or less in the dark. The solution was returned to room temperature before use and shaken. Use a dense bottle to protect from light after opening. Save and use within 1 month.

4.7 Quartz sand (SiO_2). 20~50 mesh. Test before use to confirm that there is no target compound or low concentration of target compound the method detection limit.

4.8 Carrier gas. nitrogen, purity $\geq 99.999\%$.

4.9 Combustion gas. hydrogen, purity $\geq 99.99\%$.

4.10 Gas. Air, need to dehydrate and de-organize.

5 Instruments and equipment

5.1

Gas Chromatograph. Capillary split/splitless inlet, programmable temperature, with hydrogen flame ionization detector.

5.2

Capillary column. 30m (length) x 0.53mm (inside diameter) x 1.0 μm (film thickness), polyethylene glycol fixative or other Effective capillary column.

5.3

Headspace Sampler. with headspace bottle, gasket (silicone rubber lined with Teflon) and sealed cap. Headspace bottle, cap If you need to reuse it, you should first clean it with detergent, then rinse it with tap water and distilled water, and then seal it at 105 °C.

Save for backup.

5.4

Reciprocating Oscillator. The oscillation frequency is 150 times/min, which can fix the