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

HJ 1053-2019

**Soil and sediment -- Determination of 8 amide pesticides -- Gas
chromatography mass spectrometry**

土壤和沉积物 8 种酰胺类农药的测定 气相色谱-质谱法

(English Translation)

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Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China" Environment, protect human health, standardize the determination method of amide pesticides in soil and sediment, and formulate this standard.

This standard specifies gas chromatography-mass spectrometry for the determination of eight amide pesticides in soil and sediment.

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

This standard is issued for the first time.

This standard is formulated by the Department of Eco-Environmental Monitoring, Laws and Standards Department of the Ministry of Ecology and Environment.

This standard was drafted. Hubei Provincial Environmental Monitoring Center Station.

Verification units of this standard. Zhejiang Environmental Monitoring Center, Chongqing Ecological Environment Monitoring Center, Hainan Environmental Monitoring Center Xinzhan Station, Wuhan Environmental Monitoring Center, Huangshi Environmental

Monitoring Station and Tai'an Environmental Protection Monitoring Station.

This standard was approved by the Ministry of Ecology and Environment on October 24, 2019.

This standard will be implemented from April 24, 2020.

This standard is explained by the Ministry of Ecology and Environment.

Determination of 8 amide pesticides in soil and sediments by gas chromatography-mass spectrometry Warning. The organic solvents and standard materials used in the experiment are toxic and hazardous substances. Solution preparation and sample preparation the process should be carried out in a fume hood, and protective equipment should be worn in accordance with regulations to avoid inhalation and contact with skin and clothing.

1 Scope

This standard specifies gas chromatography-mass spectrometry for the determination of eight amide pesticides in soil and sediment.

This standard applies to acetochlor, isopropachlor, alachlor, diazepam, methachlor, fenitrothion, Determination of Butachlor and Pretilachlor 8 Amides when the sample volume is 10 g and the constant volume is 1.0 ml, the detection limit of the method is as follows.

0.01 ~ 0.02 mg/kg, the lower limit of measurement is 0.04 ~ 0.08 mg/kg. See Appendix A for details.

2 Normative references

This standard refers to the following documents or clauses therein. For undated references, the valid version applies to this standard.

GB 17378.3 Marine Monitoring Code Part 3. Sample Collection, Storage and Transportation
GB 17378.5 Marine Monitoring Code Part 5. Sediment Analysis HJ 494 Water Quality Sampling Technical Guide HJ 495 Water Quality Sampling Scheme Design Technical Regulations HJ 613 Soil dry matter and moisture determination by gravimetric method HJ 783 Soil and sediment organic matter extraction Pressurized fluid extraction method HJ 911 Soil and sediment organic matter extraction ultrasonic extraction method HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Method principle

Using acetone-n-hexane as an extractant, extract the soil by Soxhlet extraction, pressurized fluid extraction or ultrasonic extraction or the amide pesticides in the sediment are purified by solid phase extraction, concentrated and fixed to volume, then separated by gas

chromatography and detected by mass spectrometry. Under warranty Retention time, fragment ion mass-to-charge ratio and abundance ratio were qualitative and quantified by internal standard method.

4 Reagents and materials

Unless otherwise specified, analytical reagents that meet national standards are used. Experimental water is freshly prepared ultrapure water or distilled water.

4.1

Hydrochloric acid. ρ (HCl) = 1.19 g/ml.

4.2

Hydrochloric acid solution. 1 5.

Measure 5 ml of hydrochloric acid (4.1), slowly add to 25 ml of water, and mix well.

4.3

Copper particles (Cu). Purity $\geq 99.5\%$.

Activate with hydrochloric acid solution (4.2) before use to remove surface oxides. Wash away the residual acid with distilled water, and then use propylene Ketone cleaning, and finally drying under a stream of nitrogen, gives the copper particles a shiny surface. Handle before each use.

4.4

Acetone (C₃H₆O). pesticide residue level.

4.5

n-hexane (C₆H₁₄). pesticide residue grade.

4.6

Dichloromethane (CH₂Cl₂). pesticide residue level.

4.7

Methanol (CH₃OH). chromatographically pure.

4.8

Acetone-n-hexane mixed solvent. 1 1.

Acetone (4.4) and n-hexane (4.5) were mixed in a 1. 1 volume ratio.

4.18

Diatomaceous earth. The particle size is 150 ~ 250 μ m (100 ~ 60 mesh).

Bake in a muffle furnace at 450 ° C for 4 hours before use. After cooling, place it in a

ground glass bottle and seal it. Store in a desiccator.

4.19

Quartz sand. The particle size is 150 ~ 250 μm (100 ~ 60 mesh).

Bake in a muffle furnace at 450 ° C for 4 hours before use. After cooling, place it in a ground glass bottle and seal it. Store in a desiccator.

4.20

Solid-phase extraction column.

Commercially available Flory silica, gel, amino, or other equivalent solid-phase extraction columns, 1 000 mg/6 ml or larger capacity specifications.

4.21

Glass wool or glass fiber membrane.

Dip with dichloromethane (4.6) before use. After it has completely evaporated, place it in a ground glass bottle and keep it sealed.

4.22

Soxhlet extraction sleeve. glass fiber or natural fiber material sleeve.

Glass fiber sleeves are placed in a muffle furnace at 450 ° C for 4 hours before use. Natural fiber material sleeves should be used and sampled before use.

The product was extracted with the same solvent and washed.

4.23

Nitrogen. purity $\geq 99.99\%$.

4.24

Helium. Purity $\geq 99.999\%$.

5 Instruments and equipment

5.1

Gas chromatograph-mass spectrometer. Capillary column and split/splitless inlet, programmable temperature, mass spectrometer with electron bomb Shock ionization (EI) source.

5.2

Chromatographic column. the stationary phase is 35% phenyl-methyl polysiloxane, the column length is 30 m, the inner diameter is 0.25 mm, and the film thickness is 0.25 μm Fused silica capillary columns can also be used with other similar performance columns to meet analytical requirements.