

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

HJ 960-2018

**Soil and sediment - Determination of carbamate pesticides -
Post-column derivatization- high performance liquid
chromatography**

土壤和沉积物 氨基甲酸酯类农药的测定 柱后衍生-高效液相色谱法

(English Translation)

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Table of Contents

Foreword

1 Determination of soil and sediment carbamate pesticides

2 Normative references

3 Principle of the method

24 Interference and elimination

47.4 Preparation of samples

Foreword

To implement the "Environmental Protection Law of the People's Republic of China", protect the ecological environment, protect human health, regulate soil and sink this standard is established for the measurement method of carbamate pesticides in the product.

This standard specifies post-column derivatization-high performance liquid chromatography for the determination of 10 carbamate pesticides in soil and sediment.

law.

Appendix A of this standard is a normative appendix, and Appendix B is an informative appendix.

This standard is the first release.

This standard is formulated by the Environmental Monitoring Department and the Science and Technology Standards Department.

This standard was drafted. Zhejiang Environmental Monitoring Center.

This standard is verified by. Jiangsu Environmental Monitoring Center, Hangzhou Environmental Monitoring Center Station, Shaoxing Environmental Monitoring Center Station, Suzhou Environmental Monitoring Center Station, Zhejiang Environmental Monitoring Engineering Co., Ltd. and China Inspection Group Physical and Chemical Testing Co., Ltd.

This standard is approved by the Ministry of Ecology and Environment on July 29, 2018.

This standard has been implemented since January 1, 2019.

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

1 Determination of soil and sediment carbamate pesticides

Post-column derivatization-high performance liquid chromatography Warning. The solvents and standard solutions used in the experiment are harmful to human health. The solution preparation and sample preparation process should be Wear in a fume hood; wear protective equipment as required to avoid direct contact with skin and clothing.

1 Scope of application this standard specifies post-column derivatization-high performance liquid chromatography for the determination of 10 carbamate pesticides in soils and sediments.

law.

This standard applies to soil and sediments of aldicarb sulfoxide, aldicarb sulfone, methomyl, 3-hydroxy carbofur, aldicarb, Determination of 10 carbamate pesticides such as propoxur, carbofuran, carbaryl, isoprocarb and methylthiocarb.

When the sample volume is 10 g, the sample volume is 1.0 ml, and the injection volume is 15 μ l, 10 carbamate farmers the detection limit of the drug is 1 to 3 μ g/kg, and the lower limit of determination is 4 to 12 μ g/kg. See Appendix A for details.

2 Normative references

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

GB 17378.3 Marine monitoring specification Part 3. Sample collection, storage and transport GB 17378.5 Marine monitoring specification Part 5. Sediment analysis HJ 494 Water Quality Sampling Technical Guidance HJ 613 Determination of dry matter and moisture in soils - Gravimetric method HJ 783 Extraction of soil and sediment organic matter by pressurized fluid extraction HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Principle of the method

Carbamate pesticides in soil or sediment are extracted by organic solvent, solid phase extraction column purification, concentration, constant volume, After separation by liquid chromatography column, it is hydrolyzed under basic conditions to form methylamine (Formula 1), which reacts with the derivatizing reagent to form a strong the fluorescent substance (formula 2) was measured by a fluorescence detector. According to the retention time qualitative, the external standard method is quantitative.

24 Interference and elimination

Substances with similar retention times interfere with the determination and can be added by changing the mobile phase gradient elution procedure, standard addition Method, quantitative qualitative wavelength absorption ratio and other methods to eliminate interference.

5 reagents and materials Analytically pure reagents in accordance with national standards were used for analysis, unless otherwise stated.

The pure water of the standard.

5.1 Acetonitrile (CH_3CN). liquid chromatography grade.

5.2 Methanol (CH_3OH). liquid chromatography grade.

5.3 Dichloromethane (CH_2Cl_2). liquid chromatography grade.

5.4 Sodium hydroxide (NaOH).

5.5 Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$).

5.6 2-mercaptoethanol ($\text{HSC}_2\text{H}_4\text{OH}$) or 2-dimethylaminoethanethiol hydrochloride ($\text{C}_4\text{H}_{11}\text{NS} \cdot \text{HCl}$).

5.7 O-phthalaldehyde ($\text{C}_8\text{H}_6\text{O}_2$).

5.8 anhydrous sodium sulfate (Na_2SO_4).

The muffle furnace was fired at 450°C for 4 h, cooled slightly, and placed in a desiccator for use.

5.9 Hydrolyzate. Sodium hydroxide solution, $c(\text{NaOH}) = 0.05 \text{ mol/L}$.

Weigh 2.0 g of sodium hydroxide (5.4), dissolve in water and dilute to 1 L, and filter through a filter (5.19). Can also be purchased directly Buy commercial goods.

5.10 Sodium tetraborate solution. $c(\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}) = 0.05 \text{ mol/L}$.

19.1 g of sodium tetraborate (5.5) was weighed, dissolved in water and diluted to 1 L, and filtered through a filter (5.19). Also directly Buy commercial items.

5.11 Derivatization reagents.

Weigh 0.1 g of o-phthalaldehyde (5.7) and dissolve it in 10 ml of methanol (5.2). Pipette.200 μl 2-mercaptoethanol (5.6) or Weigh 2.0 g of 2-dimethylaminoethanethiol hydrochloride (5.6) and dissolve it in 10 ml of sodium tetraborate solution (5.10). The above two kinds the solution was mixed and diluted to 1 L with sodium tetraborate solution (5.10).

5.12 Methanol-dichloromethane mixed solvent. 12.

Mix with methanol (5.2) and dichloromethane (5.3) in a volume ratio of 1.2.

5.13 Methanol-dichloromethane mixed solvent. 19.

Mix with methanol (5.2) and dichloromethane (5.3) in a volume ratio of 1.9.

5.14 Standard stock solution of carbamate pesticide. $\rho=100$ mg/L.

Purchase commercially available certified standard solutions directly and store them in accordance with the standard solution certificate.

5.15 Standard use solution for carbamate pesticides. $\rho=10$ mg/L.

Take 500 μ l of carbamate pesticide standard stock solution (5.14) in a 5 ml volumetric flask and dilute to volume with methanol (5.2).

Mix well, place in a -18°C refrigerator, sealed, protected from light, and the shelf life is 6 months.

5.16 Solid phase extraction column. Graphitized carbon black/N-propyl ethylene diamine composite filler column (500 mg/6 ml) or other properties A similar solid phase extraction column.

35.17 Diatomaceous earth. 0.6 to 0.9 mm (30 mesh to 20 mesh).

5.18 Quartz sand. 150 to 830 μm (200 mesh to 100 mesh).

The muffle furnace was fired at 450°C for 4 h, cooled slightly, and placed in a clean dryer for later use.

5.19 Filter. 0.45 μm polyethersulfone or other equivalent material.

5.20 Filter. 0.45 μm PTFE or other equivalent material.

5.21 Nitrogen. purity $\geq 99.99\%$.

6 Instruments and equipment

6.1 High Performance Liquid Chromatograph.

6.1.1 with fluorescence detector, gradient elution function.

6.1.2 With post-column derivatization system, the derivatization reaction can be completed automatically.

6.1.3 Column. C8 column with a particle size of 5 μm , a column length of 25.0 cm and an inner diameter of 4.0 mm or other properties Similar columns.

6.2 Freeze dryer.

6.3 Extraction device. pressurized fluid extractor (with extraction tank below 50 ml), Soxhlet extraction device, automatic Soxhlet extraction Instrument or other equivalent extraction device.