

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 961-2018

**Soil and sediment - Determination of carbamate pesticides -
High performance liquid chromatography-triple quadrupole
mass spectrometry**

土壤和沉积物 氨基甲酸酯类农药的测定 高效液相色谱-三重四极杆质谱法
(English Translation)

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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 high performance liquid chromatography-triple quadrupole for the determination of 15 carbamate pesticides in soil and sediment.

Mass spectrometry.

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, Zhejiang Zhoushan Marine Ecological Environment Monitoring Station, Hangzhou Environment Monitoring Center Station, Suzhou Environmental Monitoring Center, Shaoxing Environmental Monitoring Center Station and Jiaxing Environmental Protection Monitoring Station.

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

High performance liquid chromatography-triple quadrupole mass spectrometry 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 high performance liquid chromatography-triple quadrupole for the determination of 15 carbamate pesticides in soil and sediment.

Mass spectrometry.

This standard applies to soil and sediments such as chlorpyrifos, methomyl, dioxin, aldicarb, chlorpyrifos, carbofuran, and 15 kinds of ammonia such as chlorpyrifos, carbaryl, ethyl thiophene, anti-??, isoprocarb, zhongdingwei, methylthiocarb, chlorpyrifos and cotton ring Determination of carbamate pesticides.

When the sample volume is 10 g, the sample volume is 1.0 ml, and the injection volume is 1.0 μ l, 15 carbamate farmers the detection limit of the drug is 1 to 2 μ g/kg, and the lower limit of determination is 4 to 8 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

The carbamate pesticide in the soil or sediment is extracted by organic solvent, purified by solid phase extraction column, concentrated, and made up to volume.

It was determined by high performance liquid chromatography-triple quadrupole mass spectrometry and quantified by internal standard method according to retention time and characteristic ion characterization.

4 Interference and elimination

Substances with similar retention times and molecular ion peaks interfere with the determination by changing the mobile phase and changing the quantification ion pairs or high resolution mass spectrometry to eliminate interference.

25 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 Methanol (CH₃OH). liquid chromatography grade.

5.2 Dichloromethane (CH₂Cl₂). liquid chromatography grade.

5.3 Ammonium acetate (CH₃COONH₄). excellent grade pure.

5.4 anhydrous sodium sulfate (Na₂SO₄).

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

5.5 Methanol-dichloromethane mixed solvent. 1 2.

Mix with methanol (5.1) and dichloromethane (5.2) in a volume ratio of 1.2.

5.6 Methanol-dichloromethane mixed solvent. 19.

Mix with methanol (5.1) and dichloromethane (5.2) in a volume ratio of 1.9.

5.7 Ammonium acetate solution. c(CH₃COONH₄) = 5 mmol/L.

Take 192.5 mg of ammonium acetate (5.3) and dissolve to a volume of 500 ml with water.

5.8 Methanol-ammonium acetate mixed solution. 2 3.

Mix with methanol (5.1) and ammonium acetate solution (5.7) in a 2.3 volume ratio.

5.9 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.

5.10 Standard use solution for carbamate pesticides. rho=10 mg/L.

Take 500 µl of carbamate pesticide standard stock solution (5.9) in a 5 ml volumetric flask and dilute to volume with methanol (5.1).

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

5.11 Standard internal standard stock solution. $\rho=100$ mg/L.

Directly purchase commercially available carbaryl-D7 and methomyl-D3 certified standard solutions, and store them according to the standard solution certificate.

5.12 Internal standard use solution. $\rho=10$ mg/L.

Take 500 μ l of internal standard stock solution (5.11) in a 5 ml volumetric flask, dilute to volume with methanol (5.1), mix and place.

-18 ° C refrigerator, sealed, protected from light, the shelf life is 6 months.

5.13 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.

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

5.15 Quartz sand. 150 ~ 830 μ m (200 ~ 100 mesh).

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

5.16 Filter. 0.22 μ m PTFE or other equivalent material.

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

5.18 Argon. purity $\geq 99.99\%$.

6 Instruments and equipment

6.1 High Performance Liquid Chromatography - Triple Quadrupole Mass Spectrometer.

36.1.1 Mass spectrometer. Triple quadrupole mass spectrometer with electrospray ion source with multiple reaction monitoring.

6.1.2 Chromatograph. Gradient elution function.

6.1.3 Column. Low-bond octadecylsilane-bonded silica gel column with a particle size of 1.7 μ m and a column length of 50 mm.

The inner diameter is 2.1 mm. Other columns with similar properties that meet the analytical requirements can also be used.

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.

6.4 Concentrator. Nitrogen blown concentrator, rotary evaporator or other equivalent equipment.