

ICS 13.080
CCS Z 18

HJ

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

HJ 1052-2019

**Soil and sediment -- Determination of 11 triazine pesticides --
High performance liquid chromatography**

土壤和沉积物 11 种三嗪类农药的测定 高效液相色谱法

(English Translation)

Issued on: January 1, 1970

Implemented on: April 24, 2020

Issued by: Ministry of Ecology and Environment

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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 of triazine pesticides in soil and sediment, and formulate this standard.

This standard specifies HPLC for the determination of 11 triazine pesticides in soil and sediment.

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

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.

This standard is verified by. Zhejiang Environmental Monitoring Center, Chongqing Ecological Environment Monitoring Center, Shaanxi Environmental Monitoring Center Xinzhan Station, Hainan Provincial Environmental Monitoring Center Station, Wuhan

Environmental Monitoring Center and Huangshi Environmental 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 11 triazine pesticides in soil and sediments by high performance liquid chromatography Warning. The organic solvents and standard materials used in the experiment are toxic and harmful substances. The solution preparation and sample preparation have been processed.

Procedures should be performed 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 HPLC for the determination of 11 triazine pesticides in soil and sediment.

This standard applies to simazine, atrazine, xicaojing, atrazine, zhongdingtong, extinguishant, Determination of eleven triazine pesticides of fenfenjing, fenfenjin, terbutin, prometryn and dicambarine.

When the sample volume is 10 g, the constant volume is 1.0 ml, and the injection volume is 10 μ l, the method of 11 kinds of triazine pesticides is checked.

The output limit is 0.02 ~ 0.08 mg/kg, and the lower limit of determination is 0.08 ~ 0.32 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 Code for Marine Monitoring 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/T 91 Technical specifications for surface water and sewage monitoring
HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Method principle

Extraction of triazine from soil or sediment with Soxhlet extraction or pressurized fluid extraction with acetone-dichloromethane as extraction agent. Pesticides, the extraction solution is purified by solid phase extraction, concentrated, and fixed volume, then separated by high performance liquid chromatography, detected by UV detector, and Qualitative retention time and external standard method.

4 Interference and cancellation

Organic compounds such as polycyclic aromatic hydrocarbons, phthalates and phenols may interfere with the determination and can be separated by chromatography or solid phase extraction purification.

5 Reagents and materials

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

5.1

Acetone (C₃H₆O). pesticide residue level.

5.2

Dichloromethane (CH₂Cl₂). pesticide residue level.

5.3

n-hexane (C₆H₁₄). pesticide residue level.

5.4

Acetonitrile (C₂H₃N). Liquid chromatography grade.

5.5

Acetone-dichloromethane mixed solvent. 1 1.

Mix with acetone (5.1) and dichloromethane (5.2) at a volume ratio of 1. 1.

5.6

Acetone-n-hexane mixed solvent. 19.

Mix with acetone (5.1) and n-hexane (5.3) in a volume ratio of 1. 9.

5.7

Triazine pesticide standard stock solution. rho = 1 000 mg/L.

You can directly purchase a commercially available certified standard solution, store it in

accordance with the requirements of the standard solution certificate, and return to room temperature during use and shake well.

5.8

Triazine pesticide standard solution. $\rho = 10.0 \sim 100 \text{ mg/L}$.

Dilute the standard stock solution (5.7) with acetonitrile (5.4) to prepare a standard use solution with a concentration of $10.0 \sim 100 \text{ mg/L}$.

Protect from light and refrigerate below 4°C for 60 days.

5.9

Anhydrous sodium sulfate (Na_2SO_4).

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.

5.10

Diatomaceous earth. particle size $150 \sim 250 \mu\text{m}$ ($100 \sim 60$ 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.

5.11

Quartz sand. The particle size is $150 \sim 250 \mu\text{m}$ ($100 \sim 60$ 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.

5.12

Solid-phase extraction column.

Commercially available magnesium silicate, silica gel, amino, or other equivalent solid-phase extraction columns with a capacity of $1\ 000 \text{ mg/6 ml}$ or greater.

5.13

Glass wool or glass fiber membrane.

Immerse in acetone-dichloromethane mixed solvent (5.5) before use. After the solvent evaporates to dryness, place it in a milled glass bottle.

Sealed.

5.14

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

The glass fiber sleeve is baked in a muffle furnace at 450°C for 4 hours before use, and then sealed in a ground glass bottle after cooling;