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

HJ 1055-2019

**Soil and sediment -- Determination of glyphosate -- High
performance liquid chromatography**

土壤和沉积物 草甘膦的测定 高效液相色谱法

(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 of glyphosate in soil and sediment, and formulate this standard.

This standard specifies HPLC for the determination of glyphosate in soil and sediment.

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. Nanjing Institute of Environmental Sciences, Ministry of Ecology and Environment.

Verification units of this standard. Jiangsu Environmental Monitoring Center, Jiangsu Environmental Monitoring Center, Changzhou Environmental Monitoring Center, Jiangsu Province Measurement Center, Zhenjiang Environmental Monitoring Center, Jiangsu Province, Hulunbuir Environmental Monitoring Center Station, Nanning Ecology, Guangxi Zhuang Autonomous Region Environmental Monitoring Center.

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 glyphosate in soil and sediment by high performance liquid chromatography Warning. The solvents and standard solutions used in the experiment are toxic and hazardous substances. Solution preparation and sample preparation should be performed in it should be carried out in a fume hood. Protective equipment should be worn during operation to avoid contact with skin and clothing.

1 Scope

This standard specifies HPLC for the determination of glyphosate in soil and sediment.

This standard applies to the determination of glyphosate in soil and sediment.

When the sample size is 10 g (dry weight), the detection limit of this method is 0.02 mg/kg, and the lower detection limit is 0.08 mg/kg.

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/T 91 Technical specifications for surface water and sewage monitoring
HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Method principle

Glyphosate in soil and sediment was extracted with a mixed aqueous solution of sodium phosphate and sodium citrate.

Extracted and purified by n-hexane under weak alkaline conditions. The aqueous phase was derivatized with 9-fluorenylmethylchloroformate (FMOC-Cl), and then Separation and Detection of Glyphosate-FMOC Derivatives by High Performance Liquid Chromatography with Fluorescence Detector Between qualitative and external standard method.

4 Reagents and materials

Unless otherwise specified, analytical reagents that meet national standards are used in the analysis, and the experimental water is freshly prepared distillation Water or pure water without the target compound.

4.1

Acetonitrile (CH_3CN). pure liquid chromatography.

4.2

n-hexane (C_6H_{14}).

4.3

Hydrochloric acid (HCl). $\rho(\text{HCl}) = 1.19 \text{ g/ml}$.

4.4

Hydrochloric acid solution. 1 1.

Hydrochloric acid (4.3) and water were mixed in a volume ratio of 1. 1.

4.5

Phosphoric acid (H_3PO_4). $\phi(\text{H}_3\text{PO}_4) = 85.0\%$.

4.6

Phosphoric acid solution. $\phi(\text{H}_3\text{PO}_4) = 0.2\%$.

Accurately measure 2.35 ml of phosphoric acid (4.5) into a 1 L volumetric flask, make up to volume with water, shake well, and use it immediately before use.

4.7

Sodium phosphate dodecahydrate ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$).

4.8

Sodium citrate dihydrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$).

4.9

Mixed extraction solution of sodium phosphate and sodium citrate. $c(\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}) = 0.03 \text{ mol/L}$, $c(\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}) = 0.01 \text{ mol/L}$.

Weigh accurately 11.40 g (accurate to 0.01 g) sodium phosphate dodecahydrate (4.7) and 2.94 g (accurate to 0.01 g) Hydrate trisodium citrate (4.8), dissolve in water and make up to a 1 L volumetric flask.

4.10

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

4.11

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

Accurately weigh 1.91 g (accurate to 0.01 g) sodium tetraborate decahydrate (4.10), dissolve in water and make up to 100 ml Volumetric flask.

4.12 9-fluorenyl methyl chloroformate ($C_{15}H_{11}ClO_2$, Fmoc-Cl). purity is not less than 99%, refrigerate below 4 °C, avoid Light save.

4.13 9-fluorenyl methyl chloroformate solution. $\rho(C_{15}H_{11}ClO_2) = 1.0 \text{ mg/ml}$.

Accurately weigh 50.0 mg (accurate to 0.1 mg) of 9-fluorenylmethyl chloroformate (4.12), dissolve with acetonitrile (4.1) and Make up to a 50 ml volumetric flask, shake well, transfer to a compact bottle and refrigerate below 4 °C, save from light.

4.14

Glyphosate standard stock solution. $\rho(C_3H_8NO_5P) = 100 \text{ mg/L}$.

Commercially available certified standard solutions can be used directly or formulated with standard materials. Weigh accurately 10.0 mg (to the nearest 0.1 mg) Glyphosate standard material, dissolved in water and made up to a 100 ml volumetric flask, shake well, transfer to a compact bottle and refrigerate below 4 °C Save for 2 months.

4.15

Glyphosate standard solution. $\rho(C_3H_8NO_5P) = 10.0 \text{ mg/L}$.

Transfer 1.0 ml glyphosate standard stock solution (4.14) into a 10 ml volumetric flask, dilute with water and make up to volume, shake Evenly transferred to a compact bottle and stored under refrigeration at 4 °C. The storage period is 2 months.

4.16

Quartz sand. The particle size is 0.7-0.3 mm (25-50 mesh).

4.17

Syringe filter. 0.22 μm . The material of the filter membrane is polytetrafluoroethylene or other equivalent materials.

4.18

Filter paper. medium speed filter paper, cotton fiber material.

5 Instruments and equipment

5.1

Liquid chromatograph. It has a gradient elution function, the temperature of the column oven can be controlled, and it is equipped with a fluorescence detector.

5.2

Chromatographic column. The packing is octadecylsilane bonded silica gel, the particle size is 5 μm , the column length is 250 mm, and the inner diameter is 4.6 mm.

Column or other equivalent column.