

ICS 13.060
CCS Z 16

HJ

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

HJ 1071-2019

**Water quality--Determination of glyphosate--High performance
liquid chromatography**

水质 草甘膦的测定 高效液相色谱法

(English Translation)

Issued on: January 1, 1970

Implemented on: June 30, 2020

Issued by: Ministry of Ecology and Environment

Table of Contents

Foreword
1 Scope
2 Normative references
3 Methodology
4 Interference and cancellation
5 Reagents and materials
6 Instruments and equipment
7 Sample
8 Analysis steps
9 Results calculation and representation
10 Precision and accuracy
11 Quality Assurance and Quality Control
12 Waste disposal

Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China" and the "Law of the People's Republic of China on Water Pollution Control", protect the ecology Environment, protect human health, standardize the determination method of glyphosate in water, and formulate this standard.

This standard specifies the HPLC method for the determination of glyphosate in surface water, groundwater, domestic sewage and industrial wastewater.

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.

This standard is verified by. Jiangsu Environmental Monitoring Center, Anhui Ecological Environment Monitoring Center, Nanjing Environmental Monitoring Center, Jiangsu Province Measurement center, Jiangsu Changzhou Environmental Monitoring Center, Jiangsu Wuxi Environmental Monitoring Center and Jiangsu Taizhou Environmental Monitoring Center.

This standard was approved by the Ministry of Ecology and Environment on December 31, 2019.

This standard will be implemented from June 30, 2020.

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

Water quality-Determination of glyphosate-High performance liquid chromatography
Warning. Some solvents and standards used in the experiment have certain toxicity. The reagent preparation and sample preparation process should be 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 water.

This standard applies to the determination of glyphosate in surface water, groundwater, domestic sewage and industrial wastewater.

When the injection volume is 20 μL , the detection limit of the method is 2 $\mu\text{g/L}$, and the lower detection limit is 8 $\mu\text{g/L}$.

2 Normative references

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

HJ 91.1 Technical Specifications for Sewage Monitoring HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring

3 Method principle

Trisodium citrate dihydrate was added to the sample at a pH of 4-9, and the sample was purified by filtration or solid phase extraction and filtered with 9-the methyl chloroformate (Fmoc-Cl) was subjected to derivatization reaction, and the generated fluorescent product was extracted and purified by dichloromethane to remove the derivatization.

After the by-products are converted, they are separated and detected by high performance liquid chromatography with a fluorescence detector. Characterized by retention time and characteristic wavelength, external Standard quantification.

4 Interference and cancellation

The metal ions in the sample will form a stable complex with glyphosate and interfere with the determination of glyphosate.

Hydrated trisodium citrate eliminates interference.

5 Reagents and materials

Unless otherwise specified, analytical reagents that meet national standards are used in the analysis.

Out of deionized water.

5.1

Acetonitrile (CH_3CN). chromatographically pure.

5.2

Methanol (CH_3OH). chromatographically pure.

5.3

Dichloromethane (CH_2Cl_2). chromatographically pure.

5.4

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

5.5

Phosphoric acid. $\rho(\text{H}_3\text{PO}_4) = 1.69 \text{ g/ml}$, excellent purity.

5.6

Sodium hydroxide (NaOH).

5.7

Hydrochloric acid solution. 1 1.

Measure 50 ml of hydrochloric acid (5.4) and slowly add to 50 ml of water.

5.8

Sodium hydroxide solution. $c(\text{NaOH}) = 0.1 \text{ mol/L}$.

Weigh 0.4 g of sodium hydroxide (5.6) in a small amount of water and make up to 100 ml.

5.9

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

Take 2.0 ml of phosphoric acid (5.5) into a 1000 ml volumetric flask, dilute with water to the mark, and mix.

5.10

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

5.11

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

5.12

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

Weigh 1.91 g of sodium tetraborate decahydrate (5.11) in a small amount of water and make up to 100 ml.

5.13 9-fluorenyl methyl chloroformate ($\text{C}_{15}\text{H}_{11}\text{ClO}_2$) standard. purity not lower than 99.0%, refrigerated below 4°C , protected from light.

5.14 9-fluorenyl methyl chloroformate acetonitrile solution. $\rho(\text{C}_{15}\text{H}_{11}\text{ClO}_2) = 1000 \text{ mg/L}$.

Weigh 50 mg of 9-fluorenyl methyl chloroformate standard (5.13), dissolve with a small amount of acetonitrile (5.1), and transfer to a 50 ml container.

In a measuring flask, dilute to the mark with acetonitrile (5.1) and mix. Refrigerate and protect from light below 4°C , the shelf life is 3 months.

5.15

Glyphosate ($\text{C}_3\text{H}_8\text{NO}_5\text{P}$) Standard. The purity is not less than 99.0%, and refrigerated under 4°C , protected from light.

5.16

Glyphosate standard stock solution. $\rho(\text{C}_3\text{H}_8\text{NO}_5\text{P}) = 1000 \text{ mg/L}$.

Accurately weigh 50.0 mg of glyphosate standard (5.15), dissolve in a small amount of water, transfer to a 50 ml volumetric flask, and dilute with water. Release the volume to the mark and mix well. Refrigerate below 4°C , protect from light, shelf life is 6 months, or purchase certified standard solution directly.

5.17

Standard glyphosate solution. $\rho(\text{C}_3\text{H}_8\text{NO}_5\text{P}) = 10.0 \text{ mg/L}$.

Take an appropriate amount of glyphosate standard stock solution (5.16), dilute with water, and prepare a glyphosate standard at a concentration of 10.0 mg/L.

With liquid. Refrigerate and protect from light below 4°C , the shelf life is 2 months.

5.18

Filter membrane. $0.45 \mu\text{m}$, hydrophilic polypropylene, glass fiber, hydrophilic PTFE or other equivalent materials.