

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

HJ 914-2017

**Water quality--Determination of paraquat and diquat--Solid
phase extraction high performance liquid chromatography**

水质 百草枯和杀草快的测定 固相萃取-高效液相色谱法

(English Translation)

Issued on: December 28, 2017

Implemented on: April 1, 2018

Issued by: Ministry of Ecology and Environment

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Foreword

In order to implement the "Law of the People's Republic of China on Environmental Protection" and the "Law of the People's Republic of China on Prevention and Control of Water Pollution", protect the environment, Protection of human health, regulating paraquat and grass quick determination of water methods, the development of this standard.

This standard specifies the determination of surface water, groundwater and wastewater paraquat and grass fast solid phase extraction - high performance liquid chromatography.

This standard Appendix A, Appendix B is an informative annex.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. Zhejiang Provincial Environmental Monitoring Center.

This standard verification unit. Jiangsu Provincial Environmental Monitoring Center,

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

This standard MEP approved on December 28, this standard since April 1, 2018 come into operation.

This standard is interpreted by the MEP.

Water quality - Determination of paraquat and benzalkonium bromide - Solid phase extraction - High performance liquid chromatography Warning. The solvents and standard solutions used in the experiment are harmful to human health. Solution preparation and sample preparation should be performed at Fume hood, and wear protective equipment as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the determination of paraquat in water and herbicides fast solid phase extraction - high performance liquid chromatography.

This standard applies to the surface water, groundwater and waste water paraquat and grass quick determination.

When the sample volume is 500 ml, the sample volume is 1.0 ml and the injection volume is 50 μ l, the detection limit of paraquat is 0.3 μ g/L, the lower limit of determination was 1.2 μ g/L; the fast limit of detection was 0.4 μ g/L and the lower limit of determination was 1.6 μ g/L.

2 Normative references

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

HJ/T 91 Technical Specifications for Surface Water and Wastewater Monitoring HJ/T 164 Groundwater Environmental Monitoring Technical Specifications

3 Method principle

Paraquat and herbicides in water samples are enriched in weak cation-exchange SPE cartridges and eluted with formic acid in acetonitrile After separation by liquid chromatography, UV detector detection, according to retention time qualitative, external standard method.

4 Interference and elimination

4.1

For water samples with high salinity, in order to avoid penetration of solid phase extraction column, the sampling volume can be reduced, and the water samples can be diluted or collected Enrich with larger capacity solid phase extraction cartridge.

4.2

Determination of material interference with similar retention time, by changing the mobile phase to improve the resolution; can also be used standard solution Liquid addition method, the absorption ratio under different wavelengths, UV absorption spectroscopy to assist the qualitative comparison.

5 Reagents and materials

Unless otherwise specified, the analysis of the use of analytical reagents in line with national standards, experimental water for the absence of the target Ionized water.

5.1

Acetonitrile (CH_3CN). Liquid chromatography pure.

5.2

methanol (CH_3OH). liquid chromatography pure.

5.3

Formic acid (HCOOH). liquid chromatography pure.

5.4

Formic acid ammonium (HCOONH_4). excellent grade pure.

5.5

Sulfuric acid (H_2SO_4). $\rho = 1.84 \text{ g/ml}$.

5.6

Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$). excellent grade pure.

5.7

Sodium hydroxide (NaOH).

5.8

sulfuric acid solution. 1 1.

Take 50 ml of sulfuric acid (5.5) and slowly add 50 ml of water.

5.9

Sodium hydroxide solution. $\rho = 0.4 \text{ g/ml}$.

Weigh 40 g sodium hydroxide (5.7) dissolved in water, set the volume to 100 ml.

5.10

methanol solution. 1 L.

Separately take 50 ml of methanol (5.2) and 50 ml of water, and mix well.

5.11

ammonium formate solution. $\text{pH} = 2 \sim 3$.

Weigh 12.6 g of ammonium formate (5.4) dissolved in water, add 20 ml of formic acid (5.3), and dilute to 1000 ml with water.

5.12

containing 2% formic acid in acetonitrile.

Take 2 ml of formic acid (5.3) and 98 ml of acetonitrile (5.1) respectively, and mix well.

5.13

standard stock solution. $\rho = 100 \text{ mg/L}$.

Direct purchase of commercial paraquat ($\text{C}_{12}\text{H}_{14}\text{Cl}_2\text{N}_2$, CAS NO.1910-42-5) and quick kill grass ($\text{C}_{12}\text{H}_{12}\text{Br}_2\text{N}_2$, CAS NO.85-00-7) certified standard solution, according to the standard solution certificate requirements.

5.14

standard solution. $\rho = 10.0 \text{ mg/L}$.

Take 500 μL standard stock solution (5.13) in a 5 ml volumetric flask (polypropylene or polyvinyl chloride), dissolve in methanol Liquid (5.10) constant volume, mix, placed in -18°C refrigerator, sealed, protected from light, shelf life of 6 months.

5.15

SPE Columns. Weak Cation Exchange Columns (150 mg/6.0 ml or 500 mg/6.0 ml) or other similar properties Solid phase extraction column.

5.16

Nitrogen. Purity $\geq 99.999\%$.

5.17

Filter. $0.45 \mu\text{m}$ nylon membrane.