

CCS Z 16

HJ

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

HJ 699-2014

**Water quality. Determination of organochlorine pesticides and
chlorobenzenes. Gas chromatography mass spectrometry**

水质 有机氯农药和氯苯类化合物的测定 气相色谱-质谱法

(English Translation)

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68 Strength less than 69 Fragments 2% 275 Strength 198 Fragments 10-30%

70 Strength less than 69 fragments 2% 365 intensity greater than 198 fragments 1%

Foreword

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 Prevention and Control, to protect the environment, this standard is formulated to ensure human health and to regulate the determination methods of organochlorine pesticides and chlorobenzenes in water.

This standard specifies gas chromatography-mass spectrometry for the determination of organochlorine pesticides and chlorobenzenes in water.

This standard is the first release.

Appendix A of this standard is a normative appendix, and Appendix B and Appendix C are informative appendices.

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard is mainly drafted by. Taizhou Environmental Monitoring Center Station.

This standard is verified by. Jiangsu Environmental Monitoring Center, Hunan Environmental Monitoring Center Station, Nanjing Environmental Monitoring Center Station, Wuxi Environmental Monitoring Center Station, Suzhou Environmental Monitoring Center Station and Changzhou Environmental Monitoring Center Station.

This standard was approved by the Ministry of Environmental Protection on March 31, 2014.

This standard has been implemented since July 1, 2014.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of organochlorine pesticides and chlorobenzene compounds - Gas chromatography - mass spectrometry Warning. The reagents and standard solutions used in this method are volatile toxic compounds. The preparation process should be in the fume hood.

Carry out the operation; wear protective equipment as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies liquid-liquid extraction or solid phase extraction/gas chromatography for the determination of organochlorine pesticides and chlorobenzenes in water.

Mass spectrometry.

This standard applies to surface water, groundwater, domestic sewage, industrial wastewater and seawater organochlorine pesticides and chlorobenzene compounds Determination of matter.

The detection limit and the lower limit of determination of the target and its method determined by the method are shown in Appendix A.

2 Normative references

The contents of this standard refer to the terms in the following documents. For undated references, the valid version applies to this standard.

HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring HJ 442 Code for Environmental Monitoring of Coastal Waters

3 Principle of the method

The liquid chlorine extraction or solid phase extraction method is used to extract organochlorine pesticides and chlorobenzene compounds in the sample, and the extract is

dehydrated.

After concentration, purification and constant volume, separation and detection were carried out by gas chromatography mass spectrometry. According to retention time, fragment ion mass-to-charge ratio and different the ion abundance is qualitative and the internal standard method is used for quantification.

4 Reagents and materials

Unless otherwise stated, analytically pure reagents and distilled water in accordance with national standards were used for the analysis.

4.1

n-hexane (C₆H₁₄). pesticide residue grade.

4.2

Dichloromethane (CH₂Cl₂). pesticide residue grade.

4.3

Methanol (CH₃OH). pesticide residue grade.

4.4

Ethyl acetate (C₄H₈O). pesticide residue grade.

4.5

Acetone (C₃H₆O). pesticide residue level.

4.6

Standard solution of organochlorine pesticide. rho=10.0mg/L, the solvent is n-hexane.

4.7

Chlorobenzene compound standard solution. rho = 10.0 mg/L, the solvent is methanol.

4.8

Internal standard stock solution (deuterated 1,4-dichlorobenzene, deuterated phenanthrene, deuterated -). rho = 4000mg/L, the solvent is methanol.

4.9

Internal standard use solution. rho=40.0mg/L.

Pipette 100.0μl of internal standard stock solution (4.8) into a 10ml volumetric flask with a micro-syringe and dilute to volume with n-hexane (4.1).

Mix well.

4.10

Alternative (tetrachloromethane, decabromobiphenyl) standard solution. $\rho = 10.0 \text{ mg/L}$, the solvent is methanol.

4.11

Decafluorotriphenylphosphine (DFTPP) solution. $\rho = 1000.0 \text{ mg/L}$, the solvent is methanol.

4.12 Decafluorotriphenylphosphine solution

Pipette 500.0 μl of decafluorotriphenylphosphine solution (4.11) into a 10ml volumetric flask with a micro-syringe, using n-hexane (4.1) Make up to the mark and mix. The standard solution should be sealed after use and stored in a dark place below 4 °C.

4.13 Hydrochloric acid solution (HCl). 1 1.

4.14 Sodium Chloride (NaCl)

After burning at 400 ° C for 4 h, it was cooled and placed in a ground glass bottle and stored in a desiccator.

4.15 Anhydrous sodium sulfate (Na_2SO_4)

After burning at 400 ° C for 4 h, it was cooled and placed in a ground glass bottle and stored in a desiccator.

4.16 Solid phase extraction cartridge. The filler is C18 or equivalent type or combined packing, commercially available, according to the organic matter in the sample the content determines the amount of filler used.

Note 1: If it is confirmed by experiments that the performance requirements of the method can be met, solid phase extraction cartridges or solid phase extraction discs with other fillers can also be used.

4.17 Helium. Purity $\geq 99.999\%$.

4.18 Nitrogen. purity $\geq 99.999\%$.

5 Instruments and equipment

5.1

Gas Chromatography-Mass Spectrometer. EI source.

5.2

Column. quartz capillary column, length 30m, inner diameter 0.25mm, film thickness 0.25 μm , stationary phase 35% phenylmethyl Polysiloxane.