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HJ

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

HJ 697-2014

**Water quality. Determination of acrylamide. Gas
chromatography method**

水质 丙烯酰胺的测定 气相色谱法

(English Translation)

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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, to ensure human health, standardize the determination method of acrylamide in water, and formulate this standard.

This standard specifies gas chromatography for the determination of acrylamide in water.

This standard is the first release.

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 method verification unit. Jiangsu Environmental Monitoring Center, Wuxi Environmental Monitoring Center Station, Suzhou Environmental Monitoring Central Station, Changzhou Environmental Monitoring Center, Zhenjiang Environmental Monitoring Center Station and Taizhou 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 acrylamide - Gas chromatography 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; protective equipment should be worn as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies gas chromatography for the determination of acrylamide in water.

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

When the sample amount is 100ml, the detection limit of this standard is 0.07µg/L, and the lower limit of determination is 0.28µg/L.

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

3 Principle of the method

Under the condition of pH 1~2, acrylamide reacts with bromine to form alpha,beta-dibromopropionamide. Acetic acid Ethyl ester extraction of alpha,beta-dibromopropionamide, the extract is dried over anhydrous sodium sulfate, concentrated, and made up to volume, with electron capture detection the gas

chromatograph of the instrument is separated and detected, and the external time method is used for quantification according to the retention time.

4 Interference and elimination

Due to its high sensitivity, the electron capture detector is prone to interference due to a large number of impurity peaks. When the target compound is detected, Apply column 2 to aid in qualitative confirmation to eliminate interference.

5 Reagents and materials

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

5.1

Ethyl acetate. pesticide residue grade.

5.2

Acrylamide standard solution ($\text{CH}_2\text{CHCONH}_2$). $\rho = 100 \text{ mg/L}$, the solvent is methanol.

5.3

alpha,beta-dibromopropionamide standard solution. $\rho = 100 \text{ mg/L}$, the solvent is ethyl acetate.

5.4

Sulfuric acid solution. $c(\text{H}_2\text{SO}_4) \sim 3 \text{ mol/L}$.

Take about 167 ml of concentrated sulfuric acid, slowly add a small amount of water, and dilute to 1L.

5.5 Potassium Bromide (KBr)

5.6 Potassium bromate solution. $c(\text{KBrO}_3) = 0.1 \text{ mol/L}$.

Weigh 1.67 g of potassium bromate dissolved in a small amount of water and dilute to 100 ml.

5.7 Sodium thiosulfate solution. $c(\text{Na}_2\text{S}_2\text{O}_3) = 1.0 \text{ mol/L}$.

Weigh 15.8 g of sodium thiosulfate in a small amount of water and dilute to 100 ml.

5.8 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.

5.9 Silicon Magnesium Adsorbent

60-100 mesh, fired at 300 ° C for 2 h, naturally cooled, placed in a ground glass bottle, and stored in a desiccator.

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

6 Instruments and equipment

6.1

Gas Chromatograph. With an electron capture detector.

6.2

Column 1. Quartz capillary column, 30m x 0.32mm, coated with polyethylene glycol, film thickness 0.25 μ m. Or other equivalent hair Thin tube column.

6.3

Column 2. Quartz capillary column, 30m x 0.32mm, coated with 35% phenyl-methyl polysiloxane, film thickness 0.25 μ m.

Or other equivalent capillary column.

6.4

Concentration device. Rotary evaporation device, or equivalent equipment such as KD concentrator and concentrator.

6.5

Oscillator.

6.6

Magnetic stirrer (with magnetic stirrer).

6.7

Drying column. glass column with a length of 250 mm and an inner diameter of 20 mm and a glass piston without oil. At the lower end of the column, put A small amount of glass wool or fiberglass filter paper. Or use other types of drying equipment.

An electronic balance of 6.8 million.

6.9

Separating funnel. 250 ml.

6.10

Bottle with stoppered iodide. 250ml.