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HJ

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

HJ 621-2011

Replacing GB/T 17131-1997

**Water quality. Determination of chlorobenzenes. Gas
chromatography
(English Translation)**

Issued on: September 1, 2011

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Foreword

To protect the environment and protect the human body 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 Prevention and Control this standard is established for the determination of healthy, standardized chlorinated compounds in water.

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

This standard is for the determination of 1,2-dichlorobenzene, 1,4-dichlorobenzene, 1,2,4-trichlorobenzene by gas chromatography (GB 17131-1997) Revision.

This standard was first published in 1997. The original drafting unit was the China Environmental Monitoring Center. This is the first revision. Major within the revision the capacity is as follows.

-- Separating the packed column by capillary column separation;

-- Increased target compound components.

Gas chromatographic method for the determination of 1,2-dichlorobenzene, 1,4-dichlorobenzene and 1,2,4-trichlorobenzene from the date of implementation of this standard (GB/T 17131-1997) abolished.

Appendix A 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. China Environmental Monitoring Center, Liaoning Provincial Environmental Monitoring Experimental Center.

This standard is verified by. Zhejiang Environmental Monitoring Center, Jilin Province Environmental Monitoring Center Station, Shenyang Environmental Monitoring Center Station, Dalian City Environmental Monitoring Center, Anshan Environmental Monitoring Center Station and Liaoyang Environmental Monitoring Station.

This standard was approved by the Ministry of Environmental Protection on September 1,

2011.

This standard has been implemented since November 1, 2011.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of chlorobenzene compounds - Gas chromatography
Warning. The standard samples of chlorobenzene compounds used in this method, carbon disulfide, concentrated sulfuric acid, etc. are harmful to human health.

Wear protective gear to avoid contact with skin and clothing.

1 Scope of application

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

This standard applies to the determination of chlorobenzene compounds in surface water, groundwater, drinking water, seawater, industrial wastewater and domestic sewage. With the body components include. chlorobenzene, 1,4-dichlorobenzene, 1,3-dichlorobenzene, 1,2-dichlorobenzene, 1,3,5-trichlorobenzene, 1,2,4-trichloro Benzene, 1,2,3-trichlorobenzene, 12 kinds of 1,2,4,5-tetrachlorobenzene, 1,2,3,5-tetrachlorobenzene, 1,2,3,4-tetrachlorobenzene, pentachlorobenzene and hexachlorobenzene.

When the water sample is 1L and the volume is set to 1.0mL, the method detection limit and the lower limit of measurement are shown in Table 1.

Table 1 Detection limit and lower limit of determination of chlorobenzene compounds

No.	Compound name	CAS	Detection limit (μg/L)	Lower limit of determination (μg/L)
1.	Chlorobenzene	108-90-7	12	48
2.	1,4-Dichlorobenzene	106-46-7	0.23	0.92
3.	1,3-Dichlorobenzene	541-73-1	0.35	1.4
4.	1,2-dichlorobenzene	95-50-1	0.29	1.2
5.	1,3,5-trichlorobenzene	108-70-3	0.11	0.44
6.	1,2,4-trichlorobenzene	120-82-1	0.08	0.32
7.	1,2,3-trichlorobenzene	87-61-6	0.08	0.32
8.	1,2,4,5-tetrachlorobenzene	95-94-3	0.01	0.05
9.	1,2,3,5-tetrachlorobenzene	634-90-2	0.02	0.06
10.	1,2,3,4-tetrachlorobenzene	634-66-2	0.02	0.07
11	PeCB	608-93-5	0.003	0.012
12	Hexachlorobenzene	118-74-1	0.003	0.012

2 Principle of the method the chlorobenzene compound in the water sample is extracted with carbon disulfide, and the extract is purified, concentrated, and made up to volume, and then used with an electron capture detector.

(ECD) gas chromatograph was analyzed and characterized by retention time and quantified by external standard method.

3 interference and elimination Under the analytical conditions, organochlorine pesticides

commonly found in environmental waters can be separated from chlorobenzene compounds without interfering with the determination. Hexachlorobutadiene to interfere with the determination of 1,2-dichlorobenzene, non-polar column separation can be selected to eliminate interference. When organic halides or organic nitrations may be present when the compound (see Appendix C) interferes, it can be confirmed by gas chromatography-mass spectrometry or separated by different polar columns to eliminate interference.

4 reagents and materials Reagents used in this standard use analytically pure reagents that meet national standards unless otherwise stated. Experimental water is not included in the new preparation Pure water of the organism.

4.1 Chlorobenzene compound mixed standard solution.

Chlorobenzene $\rho = 100,000 \mu\text{g/ml}$, 1,4-dichlorobenzene $\rho = 1000 \mu\text{g/ml}$,
1,3-dichlorobenzene $\rho = 1000 \mu\text{g/ml}$, 1,2-dichlorobenzene $\rho = 1000 \text{ Gg/ml}$,
1,3,5-trichlorobenzene $\rho = .200 \mu\text{g/ml}$, 1,2,4-trichlorobenzene $\rho = .200 \mu\text{g/ml}$,
1,2,3-trichlorobenzene $\rho = .200 \mu\text{g/ml}$, 1,2,4,5-tetrachlorobenzene $\rho = 50.0 \mu\text{g/ml}$,
1,2,3,5-tetrachlorobenzene $\rho = 50.0 \mu\text{g/ml}$, 1,2,3,4-tetrachlorobenzene $\rho = 50.0 \mu\text{g/ml}$,
pentachlorobenzene $\rho = 20.0 \mu\text{g/ml}$, Hexachlorobenzene $\rho = 20.0 \mu\text{g/ml}$.

Purchase different concentrations of certified reference materials or standard solutions as needed. The opened standard solution is sealed under freezing and dark conditions save.

4.2 Sodium chloride

Bake at 300°C for 4 h, cool to room temperature in a desiccator, and store in a ground glass bottle.

4.3 anhydrous sodium sulfate Bake at 300°C for 4 h, cool to room temperature in a desiccator, and store in a ground glass bottle.

4.4 Concentrated sulfuric acid. excellent grade, $\rho_{20} = 1.84 \text{ g/ml}$.

4.5 Carbon disulfide. chromatographically pure, 100 times concentrated and chromatographically detected without interference peaks. Analytically pure reagents can be purified according to Appendix B.

4.6 Methanol. pesticide residue level.

4.7 n-hexane. pesticide residue level.

4.8 Sodium sulfate solution. $\rho(\text{Na}_2\text{SO}_4) = 20 \text{ g/L}$.

20 g of anhydrous sodium sulfate (4.3) was weighed and dissolved in water containing no organic matter and diluted to 1000 ml.

4.9 carrier gas. nitrogen, purity $\geq 99.999\%$.

4.10 Glass wool

In the presence of interference, it can be soxed in n-hexane for 4 h and stored in a closed container.

5 Instruments and equipment

5.1 Gas chromatograph with electron capture detector (ECD).

5.2 Column. Quartz capillary column, 30m (length) x 0.25mm (inside diameter) x 0.25 μ m (film thickness), the stationary phase is nitro pair Phthalic acid modified polyethylene glycol or other equivalent stationary phase.

5.3 Separating funnel. 125ml,.2000ml, several.

5.4 Round bottom flask. 100 ml, several.

5.5 Volumetric flask. 50ml, several.

5.6 Rotate the evaporator.

5.7 Nitrogen blowing instrument.

5.8 Measuring cylinder. 1000ml.

5.9 Oscillator, 300 times/minute.

5.10 Brown threaded bottle. 1ml with push-on bonnet.

5.11 Vial. 2ml.

5.12 Microinjector. 10.0 μ l, 50.0 μ l.

5.13 Other instruments and equipment commonly used in laboratories.

6 samples

6.1 Sample Collection Samples were taken in brown glass bottles to fill the sample bottles. Sealed with a cap lined with a Teflon silicone pad (or aluminum foil pad), Prevent bubbles.

6.2 Sample storage

Samples collected should be analyzed as soon as possible. If it cannot be analyzed on the day, add 1.0ml concentrated sulfuric acid (4.4) per liter of water sample at the time of sampling, at 2~ Store at 5 ° C and complete sample analysis within 7 days.

6.3 Sample preparation

6.3.1 Extraction

Take 1000ml water sample from the measuring cylinder, place in.2000ml separatory funnel, add 30g sodium chloride (4.2), use 20ml, 10mL respectively Carbon disulfide (4.5) was extracted twice. Start shaking gently, and pay attention to deflation. After deflation is complete, fully oscillate on the oscillator.