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**NATIONAL OCCUPATIONAL HEALTH STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GBZ/T 286-2016

**Method for determination of 1, 2-dichloroethane in blood by gas
chromatography-mass spectrometry**

血中1, 2-二氯乙烷的气相色谱-质谱测定方法

(English Translation)

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Foreword

This standard is formulated in accordance with the Law of the People's Republic this standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard is mainly drafted by. Shenzhen Occupational Disease Prevention and Control Institute, Guangdong Provincial Occupational Disease Prevention and Control Institute, Shenzhen Luohu District Disease Prevention and Control Heart, Shenzhen Yantian District Center for Disease Control and Prevention.

1 Scope

This standard specifies the method for the determination of 1,2-dichloroethane in blood by gas chromatography-mass spectrometry.

This standard applies to the determination of 1,2-dichloroethane in the blood of 1,2-dichloroethane occupational exposure poisoning personnel.

2.1 Principle

The 1,2-dichloroethane in the blood sample was extracted by headspace method, and the gas chromatography-mass spectrometry was used to select the ion scanning method for detection, retention time and mass spectrometry.

The relative abundance of the characteristic ions is qualitative, and the characteristic ions m/z 62 are quantitative ions, which are quantified by the standard working curve method.

2.2 Instruments

2.2.1

Anticoagulation blood collection tube, heparin sodium or EDTA anticoagulant, 5 mL.

2.2.2

Headspace bottle with Teflon silicone rubber gasket and sealing cap, 10 mL.

2.2.3

Microinjectors, 0.5 μ L, 10 μ L, 25 μ L, 50 μ L.

2.2.4

Thermostat heater.

2.2.5

Glass syringe, 1.0 mL, should be checked for air tightness before use.

2.2.6

Gas Chromatography-Mass Spectrometer.

The instrument operation reference conditions are as follows.

--Chromatographic conditions.

- a) column. 60m x 0.25mm x 0.25 μ m (5% - phenyl) - methyl polysiloxane;
- b) Column temperature. initial temperature 40 °C, hold 1.0min, then rise to 80 °C at 10 °C/min for 0.5 min;
- c) vaporization chamber temperature. 250 °C;
- d) column flow. 1.5 mL/min;
- e) Injection volume. 1.0 mL
- f) split ratio. 20:1;

--MS conditions.

- a) ion source. EI source;
- b) ionization energy. 70 eV;
- c) ion source temperature. 230 ° C;
- d) interface temperature. 280 ° C;
- e) Scan mode. select ion scan mode (SIM), select monitor ion (m/z). 49, 62, 98, quantitation ion 62, the relative abundance of the qualitative ion pair and the relative abundance of the concentration of the standard working solution are not allowed to exceed the limits specified

in Table 1.

Table 1 Maximum allowable deviation of relative ion abundance in qualitative confirmation

Relative ion abundance /%	>50	>20~50	>10~20	<=10	Maximum allowable deviation /%
	±25	±30	±50		

2.3 Reagents

2.3.1

Experimental water is secondary water.

2.3.2 1,2-Dichloroethane. Standard material or chromatographically pure reagent (>99.5%).

2.3.3

Standard solution. Add about 50 mL of water to a 100 mL volumetric flask. After weighing accurately, add an appropriate amount of 1,2-dichloroethane (about 50 mg).

Then weigh accurately. Dilute to the mark with water, calculate the concentration of the solution from the difference between the two weighings. The solution is sealed and stored in a refrigerator at 4 °C for 15 days.

Dilute to a certain concentration of standard application solution with water before use. Or use a standard solution certified by the state and awarded a standard substance certificate.

2.4 Sample collection, transportation and preservation

Collect venous blood in anticoagulation blood collection tube, about 5mL full of blood collection tube, shake well, avoid coagulation, refrigerate transportation, and storage when the condition is ≤ 4 °C, it can be stabilized for 3 days, and it can be stabilized for 7 days at or below -8 °C. If it cannot be measured immediately, it will be moved immediately after the blood sample is collected.

Take 2 mL of blood sample into the headspace bottle, seal it in the -8 °C refrigerator, and store the sample for 7 days.

2.5 Analysis steps

2.5.1

Sample processing. Shake the blood sample to be fully shaken, accurately transfer 2.0 mL of the blood sample into a 10 mL headspace bottle, and immediately close the bottle mouth.

Balance in a 60 °C constant temperature heater for 20 min, to be tested.

2.5.2

Drawing of the working curve. Take 6 10 mL headspace bottles, and add 2.0 mL of blood samples mixed with normal humans without exposure to 1,2-dichloroethane.

Different amounts of standard 1,2-dichloroethane solution were added to prepare a standard series with a concentration of 0.0 µg/L to 100.0 µg/L.

Tightly shake the bottle, shake well, equilibrate for 20 min in a 60 °C thermostat heater, and use a 1.0 mL glass syringe to extract 1.0 mL of the upper gas.

Injection gas chromatography-mass spectrometry. Characterizes the retention time of the chromatogram and the characteristic ions of the mass spectrometer, with a peak area of m/z 62 versus 1,2-two the concentration of ethyl chloride (µg/L) is plotted against a standard operating curve or a regression equation.

2.5.3

Sample determination. The ion peak area of the sample m/z 62 is measured by the operating conditions of the standard series, from the standard working curve or back.

The equation is obtained to obtain the content of 1,2-dichloroethane (µg/L).

2.6 Description

2.6.1

The minimum detection concentration of this method is 1.3 µg/L, the measurement range is 4.3 µg/L ~ 100.0 µg/L, in the actual measurement, can pass Reduce the sampling volume and increase the concentration of the standard curve to broaden the linear range. The linear range can be extended to 20mg/L, and the inter-assay precision is 6.2%~ 8.0%, the standard recovery rate was 83.2% ~ 104.0%.

2.6.2

Common organic solvents such as methanol, acetone, n-pentane, 1,1-dichloroethylene, dichloride, which may be contacted simultaneously under the conditions of the process.

Methane, n-propanol, 1,2-dichloroethylene, 1,1-dichloroethane, n-hexane, 1,2-dichloroethylene, methyl acrylate, chloroform, 1,1,1-Trichloroethane, benzene, cyclohexane, carbon tetrachloride, n-heptane, trichloroethylene, 1,2-dichloropropane, methyl methacrylate, propyl acetate, Methyl isobutyl ketone, toluene, tetrachloroethylene, ethylbenzene, xylene, cyclohexanone, 1,2,3-trichloropropane, etc. do not interfere with the measurement.

2.6.3

This method can also be determined by capillary column chromatography using equivalent mass spectrometry.