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

GBZ/T 318.1-2018

**Determination of trimethyltin chloride (TMT) in blood -- Part 1:
Gas chromatographic method**

血中三甲基氯化锡的测定 第1部分：气相色谱法
(English Translation)

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Table of Contents

Foreword
1 Scope
2 Normative references
3 Principle
4 Instruments
5 Reagent
6 Sample collection, transportation and storage
7 Analysis steps
8 Calculation
9 Description

Foreword

This standard is formulated in accordance with the Law of the People's Republic GBZ/T 318 "Determination of Trimethyltin Chloride in Blood" is divided into two parts.

-- Part 1. Gas chromatography;

-- Part 2. Gas chromatography-mass spectrometry.

This part is the first part of GBZ/T 318.

This part is drafted in accordance with the rules given in GB/T 1.1-2009.

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1 Scope

This part of GBZ/T 318 specifies gas chromatography for the determination of trimethyltin chloride in blood.

This section applies to the determination of trimethyltin chloride in the blood of occupational contacts.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this article.

Pieces. For undated references, the latest edition (including all amendments) applies to this document.

GBZ/T 295 General rules for occupational health biological monitoring

3 Principle

Trimethyltin chloride in the blood is derivatized with sodium tetraethylborate to trimethylethyltin, extracted with n-hexane, gas chromatographic flame photometric examination the detector (sulfur filter) is detected and characterized by retention time and peak area is quantified.

4 Instruments

4.1

Heparin sodium anticoagulation blood collection tube, 5mL.

4.2

Centrifuge, 0r/min ~ 3000r/min.

4.3

Microinjector, 1 μ L, 10 μ L, 50 μ L, 100 μ L.

4.4

Plug glass tube. 15mL with PTFE sealing cap.

4.5

vortex oscillator.

4.6

Gas Chromatograph, Flame Photometric Detector (Sulfur Filter).

Instrument operation reference conditions.

- Column. 30 m x 0.32 mm x 0.25 μ m diphenyl-95% dimethyl siloxane copolymer (HP-5);
- column temperature. initial temperature 50 ° C, maintained for 7.0 min, raised to.200 ° C at 20 ° C/min;
- inlet temperature. 250 ° C;
- Detector temperature..200 ° C;

- hydrogen flow. 50mL/min;
- Air flow. 60mL/min;
- carrier gas (nitrogen) flow rate. 1.0mL/min;
- Split ratio. 5.1.

5 Reagent

5.1

The experimental water is deionized water.

5.2

Trimethyltin chloride, excellent grade (>99.8%).

5.3

sodium tetraethyl borate, excellent grade pure.

5.4

Tetrahydrofuran, analytically pure.

5.5

Sodium acetate, analytically pure.

5.6

Glacial acetic acid, analytically pure.

5.7

n-hexane, chromatographically pure.

5.8

Acetonitrile, chromatographically pure.

5.9

Derivatizing agent. Weigh 0.1 g of sodium tetraethyl borate and dissolve it in 10 mL of tetrahydrofuran to prepare 1% sodium tetraethylborate-tetrahydrofuran.

The solution is stored in a refrigerator at 4 ° C in the dark and stable for 1 month.

5.10

Buffer (pH 4.0 ± 0.2). Add 0.6 g of sodium acetate to 120 mL of water, followed by 1.4 mL of glacial acetic acid.

5.11

Standard solution. Add a small amount of water to a 10mL volumetric flask. After weighing

accurately, add a certain amount of trimethyltin chloride.

Weighing, add water to the scale; calculate the concentration of this solution from the difference of 2 weighings, which is the standard stock solution (concentration is about 5mg/mL), this solution is at 4°C it can be stored in the refrigerator for 6 months. Dilute to 50.0 µg/mL Trimethyltin chloride standard solution with water before use. Or purchase certified by the state and A standard solution substance that is awarded a standard substance certificate.

6 Sample collection, transportation and storage

Use heparin sodium anticoagulant blood collection tube to collect 5 mL of blood sample of workers exposed to trimethyltin chloride, transport at room temperature or refrigerated, and place it at 4 ° C as soon as possible.

Stored in the box, the sample can be stored for 15d.

7 Analysis steps

7.1

Sample Handling. Remove the blood sample from the refrigerator, set it back to room temperature, and shake well. Accurately draw 1.0mL blood sample in 15mL Add 2.0 mL of buffer solution to the stoppered glass tube, shake well, add 7.0 mL of acetonitrile, seal and shake for 1 min to fully precipitate the protein, then Centrifuge at 3000r/min for 1min, transfer the supernatant to another 15mL glass tube, add a small amount of acetonitrile to wash the precipitate, and combine the washing solution was placed in a 15 mL stoppered glass tube, 0.2 mL of the derivatizing agent was added, and 1.0 mL of n-hexane was accurately added after mixing, and the mixture was shaken for 5 minutes after sealing.

After standing or centrifuging, the upper organic phase was analyzed by 1.0 µL. If the concentration of trimethyltin chloride in the sample exceeds the measurement range, n-hexane can be used.

Determined after dilution and multiplied by the dilution factor.

7.2

Reagent blank treatment. Replace the blood sample with 1.0mL water, and treat the sample as the reagent blank.

7.3

Preparation and determination of standard series of solutions. Add 1.0 mL of water to a 15 mL stoppered glass tube, and then add a standard solution to prepare the content.

For the standard solution of trimethyltin chloride from 0.0g to 2.5g, add 2.0mL of buffer solution to each standard series solution, shake and add Into 7.0mL of acetonitrile and