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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31604.37-2016

National Food Safety Standard –
Food Contact Materials and Articles –
Determination of Triethylamine and
Tributylamine

Issued on: October 19, 2016 Implemented on: April 19, 2017

Issued by: National Health and Family Planning Commission of the People's Republic of China

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National Food Safety Standard – Food Contact Materials and Articles – Determination of Triethylamine and Tributylamine

1 Scope

This Standard specifies the determination method of triethylamine and tributylamine by gas chromatography in food contact materials and articles.

This Standard is applicable to the determination of triethylamine and tributylamine in polycarbonate food contact materials and articles.

2 Principle

After the sample is dissolved in dichloromethane, acetone shall be added to precipitate the polymer resin. Triethylamine and tri-n-butylamine in solution are concentrated and weighed, then measured by gas chromatography, and quantified by external standard method.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are all analytically pure. Plastic containers and transfer equipment shall be avoided during the test.

3.1 Reagents

- **3.1.1** Dichloromethane (CH₂Cl₂).
- **3.1.2** Acetone (C₃H₆O).

3.2 Standard product

- **3.2.1** Triethylamine ($C_6H_{15}N$, CAS No.: 121-44-8): a standard substance with a purity of \geq 99% or certified by the state and granted with a standard substance certificate.
- **3.2.2** Tributylamine (C12H27N, CAS No.: 102-82-9): a standard substance with a purity of ≥99% or certified by the state and granted with a standard substance certificate.

conical flask. After the addition, shake the bottle solution. Centrifuge 10 min. Take the supernatant and evaporate to a concentration of nearly 1 mL. Completely transfer the concentrate to a plugged centrifuge tube. Dilute with dichloromethane and set volume to 2.0 mL. Obtain the sample solution for the gas chromatography determination.

NOTE 1: The solution centrifugation can be performed in several times. The supernatant after centrifugation must be merged.

NOTE 2: If there is a small amount of turbidity in the sample solution, it can be filtered by a needle-type organic phase filter and then measured by gas chromatography.

5.3 Blank test

Except that the sample is not added, use the same analysis steps, reagents and dosage same as in 5.2 for parallel operation.

5.4 Gas chromatography reference conditions

- **5.4.1** Chromatographic column: ZB-5 quartz capillary column, column length of 30m, inner diameter of 0.32mm, film thickness of $5\mu m$, or the equivalent.
- **5.4.2** Column temperature: initial temperature of 40°C, hold 5min; then increase at 20°C/min to 250°C for 5min.
- **5.4.3** Inlet temperature: 200°C.
- **5.4.4** Detector temperature: 250°C.
- **5.4.5** Carrier gas: nitrogen (purity ≥99.999%), flow of 1.0 mL/min.
- **5.4.6** Detector hydrogen flow rate: 3 mL/min.
- **5.4.7** Detector air flow: 60 mL/min.
- 5.4.8 Injection volume: 1 µL.
- **5.4.9** Injection method: split-less injection.

5.5 Making of standard working curve

Inspect the standard working solution according to the chromatographic conditions listed in 5.4. Take the concentration of triethylamine and tributylamine (mg/L) in the standard working solution as the abscissa, the corresponding chromatographic peak area as the ordinate, perform the linear regression to draw the standard working curve.

5.6 Sample solution determination

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