



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

GB 31604.12-2016

**National food safety standard - Food contact materials and
products - Determination of 1,3-butadiene and migration
(English Translation)**

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Foreword

This Standard replaces GB/T 23296.2-2009 "Food contact materials - Polymer - Determination of 1,3-butadiene in food simulants - Gas chromatography" and GB/T 23296.3-2009 "Food contact materials - Determination of 1,3-butadiene in plastics - Gas chromatography".

Compare this Standard with GB/T 23296.3-2009, the main changes are as follows.

- The standard name is revised to "National food safety standard - Food contact materials and products - Determination of 1,3-butadiene and migration";
- ADD the determination of 1,3-butadiene migration in food contact materials.

National food safety standard -

Food contact materials and products -

Determination of 1,3-butadiene and migration

1 Scope

This Standard specifies the determination method for 1,3-butadiene in food contact materials and products and the determination method for migration.

This Standard applies to the determination of 1,3-butadiene in food contact materials and products and the determination of migration.

Determination of 1,3-butadiene

2 Principle

Food contact materials and products are dissolved or dispersed in the headspace bottle with N, N-dimethylformamide. HEAT to bring the components to be determined to gas-liquid equilibrium. The headspace gas is then quantitatively taken for determination by gas chromatography. QUALIFY according to the retention time, using n-pentane as the internal standard; QUANTIFY by internal standard method.

3 Reagents and materials

The water used in this method is Grade I water specified in GB/T 6682. Containers and transfer apparatus in the test shall avoid the use of plastic materials.

3.1 Reagents

N, N-Dimethylformamide (DMF). Analytical grade, with no interference peak at 1,3-butadiene and n-pentane retention times.

3.2 Standards

3.2.1 1,3-butadiene (C₄H₆, CAS number. 106-99-0). 2000 µg/mL, or standard substances certified by the state and awarded the standard substance certification.

3.2.2 n-pentane (C₅H₁₂, CAS number. 106-66-0). with the purity greater than 99.5 %,

or standard substances certified by the state and awarded the standard substance
WEIGH 0.5 g (to the nearest 0.01 g) of sample into a headspace bottle; ADD 5.0 mL

of N, N-dimethylformamide; immediately COVER to seal; USE a 100 μ L micro syringe to ADD 100 μ L of n-pentane standard solution to the headspace bottle; SHAKE well, so that the sample in the bottle is completely dissolved or fully dispersed for determination. At the same time, carry out the blank test.

5.3.1 Headspace sampler conditions

Headspace sample injector conditions are listed below.

- a) balance time. 30 min;
- b) furnace temperature. 80 °C;
- c) pressure. 138 kPa;
- d) pressurizing time. 2 min;
- e) sample injection time. 0.04 min;
- f) transmission line temperature. 85 °C.

5.3.2 Gas chromatographic conditions

Gas chromatographic conditions are listed below.

- a) chromatographic column. polystyrene-divinylbenzene quartz capillary column with column length of 30 m, inner diameter of 0.32 mm, membrane thickness of

10 μ m, or equivalent column;

- b) column temperature program. maintain at 80 °C for 12 min, rise to 120 °C at a rate of 10 °C/min and maintain for 10 min;
- c) inlet temperature. 220 °C;
- d) detector temperature. 250 °C.
- e) carrier gas nitrogen flow rate. 50 mL/min;
- f) hydrogen flow rate. 10 mL/min;
- g) air flow rate. 400 mL/min;
- h) inlet constant-pressure injection. 100 kPa.

5.4 Preparation of calibration solution and plotting of calibration curve

10.4.1 1,3-butadiene standard stock solution. PIPETTE 1.0 mL of 1,3-butadiene standard and TRANSFER into a 10 mL volumetric flask; ADD N, N-dimethylformamide to DILUTE to the mark, MIX. The concentration of 1,3-butadiene in this solution is 200 µg/mL. The solution shall be stored in the dark, which is valid for 3 months at -20 °C, or valid for one week at 4 °C.

10.4.2 1, 3-butadiene standard use solution. PIPETTE 0.1 mL, 0.2 mL, 0.3 mL, 0.4 mL,

0.5 mL of 1, 3-butadiene standard stock solution; TRANSFER into 10 mL volumetric

flasks, respectively. ADD N, N-dimethylformamide respectively to DILUTE to the mark, MIX (corresponding to 2 µg, 4 µg, 6 µg, 8 µg, 10 µg of 1,3-butadiene per milliliter). Prepare at the day when it is to be used.

10.5 Preparation of n-pentane standard solution

Following the procedure similar to that of 3.3, use N, N-dimethylformamide to prepare a portion of n-pentane standard solution at a concentration of about 10 µg/mL.

11.2 Headspace auto sample injector.

11.3 20 mL headspace gas measuring bottle, with fluorinated vinyl silicone coated rubber cap and aluminum cap.

11.5 Analytical balance. with a division of 0.1 mg.

11.6 10 mL volumetric flask.

11.7 50 µL micro syringe.

12.1.1 Water-based, acidic, alcohol-based, oil-based food simulants

In this Standard, the food simulation test uses water-based, acidic, alcohol-based, oil-based food simulants. The required sample solutions are obtained by the migration test, which may be stored in the refrigerator at 4 °C keeping away from light for 1 week.