

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

SN/T 5155-2019

**Test methods for food contact materials -- Polymer -
Determination of crotonic acid in food simulants -- High
performance liquid chromatography**

食品接触材料检测方法 高分子材料 食品模拟物中巴豆酸含量的测定 高效液相色谱法

(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard is proposed and managed by the General Administration of Customs of the People's Republic of China.

This standard was drafted. Tianjin Customs of the People's Republic of China, Ningbo Customs of the People's Republic of China.

1 Scope

This standard specifies the HPLC method for the determination of crotonic acid in food simulants.

This standard applies to 3% (mass concentration) acetic acid, 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol solution, 20% (volume fraction) Number) Determination

of crotonic acid in six food simulants, ethanol solution, 50% (volume fraction) ethanol solution and olive oil.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies file. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods GB/T 232966.1 Food contact materials. Restricted substances in plastics. Specific migration of substances in plastics to food and food simulants.

Guidelines for testing and content determination methods and selection of food simulant exposure conditions GB 31604.1 National Food Safety Standard General Rules for Migration Test of Food Contact Materials and Products

3 Method summary

After the sample is immersed in the food simulant, a certain amount of food simulant test solution is taken (of which the olive oil test solution needs ultrasonic-assisted liquid-liquid extraction), the content of crotonic acid was determined by high performance liquid chromatography and quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used are of analytical grade and the water is secondary water as specified in GB/T 6682.

4.1

Glacial acetic acid.

4.2

Anhydrous ethanol.

4.3

Methanol. chromatographically pure.

4.4

Formic acid. chromatographically pure.

4.5 3% (mass-volume concentration) acetic acid solution. Weigh 30.0g (accurate to 0.1g) glacial acetic acid (4.1), dilute with water and transfer to 1L in a volumetric flask, make up to volume with water.

4.6 4% (volume fraction) acetic acid solution. Measure 40mL of glacial acetic acid (4.1) in a

1L volumetric flask, and make up to volume with water.

4.7 10% (volume fraction) ethanol solution. Measure 100mL of absolute ethanol (4.2) in a 1L volumetric flask, and make up to volume with water.

4.8 20% (volume fraction) ethanol solution. Measure 200mL of absolute ethanol (4.2) into a 1L volumetric flask, and make up to volume with water.

4.9 50% (volume fraction) ethanol solution. Measure 500mL of absolute ethanol (4.2) in a 1L volumetric flask, and make up to volume with water.

4.10

Commercially available olive oil.

4.11

Crotonic acid standard, CASN O... [3724-65-0], purity $\geq 99.5\%$.

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4.12

Crotonic acid standard stock solution. Accurately weigh crotonic acid (4.11) 0.05g (accurate to 0.1mg), use methanol to dissolve and transfer to in a 500mL brown volumetric flask, make up to volume with methanol (4.3) and mix thoroughly to obtain a standard stock solution with a concentration of 100mg/L, 4 °C Save it for future use. Shelf life is 1 month.

4.13

Microporous membrane. water phase and organic phase, pore size 0.2 μm .

5 Instruments and equipment

5.1

High Performance Liquid Chromatograph (HPLC). Equipped with UV detector.

5.2

Ultrasonic extraction instrument.

5.3

Test tube with stopper. 10mL.

5.4

Syringe. 2mL.

5.5

Micropipette. 10 μL , 100 μL , 1000 μL . 5.6 Analytical balance. Sensitivity of 0.1mg and 0.01g.

6.1.1 Water-based food simulant standard working solution

Use micropipettes (5.5) to accurately measure 50 μL , 80 μL , 100 μL , 300 μL , 500 μL , 800 μL , 1000 μL of crotonic acid standard stock solution (4.12) in 7 10 mL volumetric flasks, and use migration tests respectively. Volume of water-based food simulant selected in the shaker, shake the back chamber evenly After standing at room temperature for 24 hours, the concentrations of crotonic acid in the corresponding media were 0.5 mg/L, 0.8 mg/L, 1.0 mg/L, 3.0 mg/L, Standard working solutions of 5.0 mg/L, 8.0 mg/L and 10 mg/L.

6.1.2 Standard working solution of olive oil

Using a syringe (5.4), accurately weigh 2g (accurate to 0.01g) olive oil into 6 test tubes (5.3) with stoppers, and use a micropipette (5.5) Transfer 20 μL , 40 μL , 100 μL , 160 μL , 200 μL , 400 μL of crotonic acid standard stock solution (4.12) to stoppered test tubes (5.3) to obtain concentrations of 1 mg/kg and 2 mg respectively/Kg, 5mg/kg, 8mg/kg, 10mg/kg, 20mg/kg standard working solution. 4mL of methanol (4.3) was added to each test tube. After ultrasonic extraction at 60 ° C for 60min, the layers were left to separate and aspirated with a syringe (5.4).

Take a methanol solution to obtain a standard working solution of crotonic acid with different concentrations. After filtering through a 0.2 μm filter membrane (4.13), use it for liquid chromatography determination.

6.2.1 Sample pretreatment

Wash the sample with detergent, rinse it with tap water, rinse it three times with ultrapure water, dry it, and set aside.

6.2.2 Migration experiment

The migration test shall be carried out in accordance with GB 31604.1 or GB/T 23296.1.

6.2.3 Treatment of food simulant test solution

For a water-based food simulant test solution, take an appropriate amount of the simulant solution and filter it through a 0.2 μm filter membrane (4.13) for detection by a liquid chromatograph. For olive oil test solution, weigh 2.0g (accurate to 0.01g) olive oil test solution into a 10mL stoppered test tube (5.3), add 4mL of methanol (4.4), After ultrasonic extraction at 60 ° C for 60 min, the layers were left to stand, the methanol solution was aspirated with a syringe (5.4), filtered through a 0.2 μm filter membrane (4.13), and then determined by liquid chromatography.

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