

**Voluntary Report** – Voluntary - Public Distribution

**Date:** August 13, 2024

**Report Number:** CH2024-0103

**Report Name:** Draft National Food Safety Standard Food Additive Paprika Oleorein Notified to WTO

**Country:** China - People's Republic of

**Post:** Beijing

**Report Category:** Sanitary/Phytosanitary/Food Safety, FAIRS Subject Report, Trade Policy Monitoring, WTO Notifications

**Prepared By:** FAS China Staff

**Approved By:** Adam Branson

**Report Highlights:**

On July 11, 2024, China notified the draft National Food Safety Standard Food Additive Paprika Oleorein to the World Trade Organization (WTO) under G/SPS/N/CHN/1307. China's SPS Enquiry Point at [sps@customs.gov.cn](mailto:sps@customs.gov.cn) will accept comments until September 9, 2024. This report Provides an unofficial translation of the draft standard. Stakeholders should conduct their own review of the standard and provide comments as necessary.

**Report Summary:**

On July 11, 2024, China notified the draft National Food Safety Standard Food Additive Ammonium Carbonate to the World Trade Organization (WTO) under [G/SPS/N/CHN/1307](#). China's SPS Enquiry Point at [sps@customs.gov.cn](mailto:sps@customs.gov.cn) will accept comments until September 9, 2024.

The notified standard applies to food additive paprika oleorein using the fruit of *Capsicum annuum L.* or *Capsicum frutescens L.* as raw materials to extract paprika oleorein. The following solvents may be used for the extraction: methanol, ethanol, ethyl acetate, acetone, n-hexane, isopropanol, dichloromethane. It specifies the technical requirements and testing methods for the food additive paprika oleorein.

The standard is based on the revision of "National food safety standard: Food additive Paprika Oleoresin" (GB 28314-2012) and combined with the actual quality status of Chinese products.

This report provides an unofficial translation of the draft notified standard. Stakeholders should conduct their own review of [the standard](#).

**BEGIN TRANSLATION**

**National Food Safety Standard**

**Food Additive Paprika Oleoresin**

**(Draft for Comments)**

**Preface**

This standard replaces "National Food Safety Standard Food Additive Paprika Oleoresin "(GB 28314-2012).

Compared with the 2012 version, the newly drafted Standard features below changes:

1. Modified the scope of application, sensory requirements, capsaicin content and solvent residue requirements;
2. Modified the molecular formula and molecular weight of Nordihydrocapsaicin;
3. Modified the determination method of capsaicin content;
4. The determination method of solvent residue is added.

## National Food Safety Standard

### Food Additive Paprika Oleorein

#### 1. Scope of Application

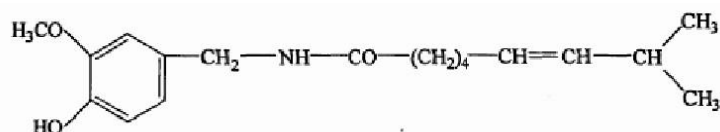
This standard applies to food additive paprika oleorein using the fruit of *Capsicum annuum L.* or *Capsicum frutescens L.* as raw materials to extract paprika oleorein, processed and refined through solvent extraction, separation, and other steps. The following solvents may be used for the extraction: methanol, ethanol, ethyl acetate, acetone, n-hexane, isopropanol, and dichloromethane.

#### 2. Molecular formula, structural formula, relative molecular mass

##### 2.1 Capsaicin

Molecular formula:  $C_{18}H_{27}NO_3$

Structural formula:

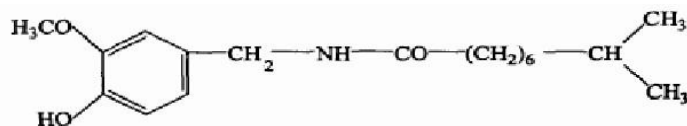


Relative molecular mass: 305.42 (based on 2022 international relative atomic mass)

##### 2.2 Dihydrocapsaicin

Molecular formula:  $C_{18}H_{29}NO_3$

Structural formula:

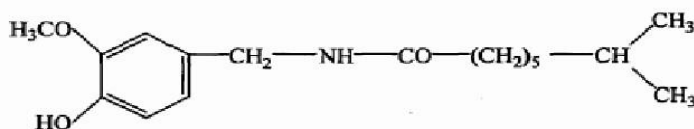


Relative molecular mass: 307.43 (based on 2022 international relative atomic mass)

##### 2.3 Nordihydrocapsaicin

Molecular formula:  $C_{17}H_{27}NO_3$

Structural formula:



Relative molecular mass: 293.41 (based on 2022 international relative atomic mass)

### 3 Technical Requirements

#### 3.1 Sensory Requirements

THIS REPORT CONTAINS ASSESSMENTS OF COMMODITY AND TRADE ISSUES MADE BY USDA STAFF AND NOT NECESSARILY STATEMENTS OF OFFICIAL U.S. GOVERNMENT POLICY

Sensory requirements shall conform to the provisions in Table 1.

**Table 1: Sensory Requirements**

Items	Requirement	Analysis method
Color	Cream-coloured, yellow, red to deep red (brown)	Take an appropriate amount of sample and place it in a clean and dry white porcelain plate. Observe its color and condition under natural light, and smell its odor.
State	Oily liquid or paste like substance	
Odor	Spicy and pungent odor	

### 3.2 Physical and Chemical Indicators

Physical and chemical indicators shall conform to the provisions in Table 2.

**Table 2: Physical and Chemical Indicators**

Items	Indicators	Testing Method
Capsaicin content, w/%	1.0~50.0	A.3 in Appendix A
Residual solvents (total of methanol, ethyl acetate, n-hexane, isopropanol, dichloromethane)/(mg/kg) ≤	50	A.4 in Appendix A
Lead (Pb)/(mg/kg) ≤	2.0	GB 5009.75 or GB 5009.12
Arsenic (As)/(mg/kg) ≤	3.0	GB 5009.76 or GB 5009.11
Note: Commercialized paprika oleorein products should be made from paprika oleorein that meets this standard as raw material, and can be supplemented with food additives such as edible vegetable oil and/or emulsifiers, antioxidants, etc. that meet the quality specifications of food additives. The capsaicin content should comply with the claimed value.		

## **Appendix A Inspection Method**

### **A.1 General Provisions**

Unless otherwise specified, the reagents and water used in this standard refer to analytical grade reagents and third grade water as specified in GB/T 6682. The standard solutions, impurity determination standard solutions, formulations, and products used in the experiment shall be prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603 unless otherwise specified. When the solvent used in the test is not specified, it refers to aqueous solution.

### **A.2 Identification Test**

#### **A.2.1 Solubility**

Almost insoluble in water, partially soluble in ethanol, insoluble in glycerol.

#### **A.2.2 Maximum Absorption Peak**

The n-hexane solution of the sample shows a maximum absorption peak near the wavelength of 445 nm.

#### **A.2.3 Color Reaction**

Take 1 drop of paprika oleorein sample, add 2 to 3 drops of chloroform and 1 drop of sulfuric acid, and the color of the solution in contact with the container should be brown to dark blue.

### **A.3 Determination of Capsaicin Content**

#### **A.3.1 Reagents and Materials**

A.3.1.1 Water: Grade I water that meets the requirements of GB/T 6682.

A.3.1.2 Acetonitrile: chromatographically pure.

A.3.1.3 Acetic acid: chromatographically pure.

A.3.1.4 95% ethanol: chromatographically pure.

A.3.1.5 Acetone: chromatographically pure.

A.3.1.6 N-vanillyl-n-nonamine (synthetic capsaicin) standard (CAS No.: 2444-46-4): purity  $\geq$  95%.

#### **A.3.2 Instruments and Equipment**

A.3.2.1 High performance liquid chromatography: equipped with ultraviolet or fluorescence detectors.

A.3.2.2 C18 solid-phase extraction column: 500 mg, 6 mL. (Before use, activate the solid-phase column with 5 mL of 95% ethanol)

A.3.2.3 Analytical Balance: with a sensitivity of 0.0001 g, and 0.001 g

A.3.3 Reference chromatographic conditions

A.3.3.1 Chromatographic Column: C18 chromatographic column ( $\Phi$  4.6 mm  $\times$  15 cm), 5  $\mu$ m, or equivalent performance chromatographic column.

A.3.3.2 Mobile phase: 1% glacial acetic acid solution-acetonitrile, volume ratio of 60:40

A.3.3.3 Injection volume: 20  $\mu$  L

A.3.3.4 Flow rate: 1.5 mL/min

A.3.3.5 UV detection wavelength: 280 nm; Or fluorescence excitation wavelength: 280 nm, emission wavelength: 325 nm

A.3.3.6 Column temperature: 30  $^{\circ}$ C

### A.3.4 Analysis Steps

#### A.3.4.1 Preparation of standard solution

Accurately weigh 10 mg of standard substance (precision to 0.0001 g), dissolve it in 95% ethanol and dilute to 100 mL to obtain a standard solution with a concentration of 0.1 mg/mL. Seal and store it in a refrigerator at 4  $^{\circ}$ C for future use. The solution is valid for 6 months.

#### A.3.4.2 Preparation of sample solution

Accurately weigh an appropriate amount of sample (weigh 1.000 g if the capsaicin content is about 1%, 0.500 g if it is about 2%, and so on), add 5 mL of acetone to completely dissolve the sample, and dilute with 95% ethanol to a volume of 50 mL. Transfer 5 mL of the diluted solution to a C18 solid-phase extraction column and elute the column three times with 5 mL of 95% ethanol. Collect the filtrate and eluent, and dilute to 25 mL with 95% ethanol. Filter the diluted solution through a 0.45  $\mu$ m filter membrane to obtain the sample solution for future use.

#### A.3.4.3 Determination

Perform chromatographic analysis on the sample solution and standard solution under the reference chromatographic conditions in A.3.3. Based on the relative retention time of different components in the standard solution (see Table A.1), qualitatively determine the chromatographic peaks of the tested components capsaicin, dihydrocapsaicin, and nordihydrocapsaicin in the sample solution, and calculate the mass fraction of each component from the peak area.

**Table A.1: Reference Values for Relative Retention Time of Different Components in Standard Solution**

Components to be tested	Relative retention time
Capsaicin	1.02
Dihydrocapsaicin	1.52
Nordihydrocapsaicin	0.92

### A.3.5 Calculations

The mass fraction  $w_i$  of the components to be tested (capsaicin, dihydrocapsaicin, and nordihydrocapsaicin) in the sample shall be calculated according to formula (A.1):

$$w_i = \frac{A_i \times c_s}{A_s \times c_i \times R_i} \times 100\% \quad \dots\dots\dots (A.1)$$

In which:

$i$  – Take on the values 1, 2, and 3, corresponding to capsaicin, dihydrocapsaicin, and nordihydrocapsaicin respectively;

$w_i$  - the mass fraction of the component to be tested in the sample solution;

$A_i$  - peak area of the component to be tested in the sample solution;

$c_s$  - concentration of standard solution, in milligrams per milliliter (mg/mL);

$A_s$  - peak area of the component to be tested in the standard solution;

$c_i$  - concentration of the sample injection solution, in milligrams per milliliter (mg/mL);

$R_i$  - Response factors of the component to be tested in the sample solution: UV 0.89 (capsaicin), 0.93 (dihydrocapsaicin), 0.98 (nordihydrocapsaicin); Fluorescence 0.88 (capsaicin), 0.93 (dihydrocapsaicin), 0.92 (nordihydrocapsaicin).

The calculation result shall retain three significant digits.

According to equation (A.1), the contents of capsaicin, dihydrocapsaicin, and nordihydrocapsaicin are calculated as  $w_1$ ,  $w_2$ , and  $w_3$ , respectively. The sum of the three is the capsaicin content in the sample, calculated as a mass fraction  $w$  and expressed as a percentage %.

The experimental results shall be based on the arithmetic mean of the parallel measurement results. The absolute difference between two independent measurement results obtained under repetitive conditions shall not exceed 5.0% of the arithmetic mean.

## A.4 Determination of residual solvents (methanol, ethyl acetate, n-hexane, isopropanol, dichloromethane)

### A.4.1 Reagents and materials

A. 4.1.1 Water: Grade I water that meets the requirements of GB/T 6682.

A. 4.1.2 Methanol, ethyl acetate, n-hexane, isopropanol, dichloromethane standard substances: standard substances with a purity of  $\geq 99\%$ , or those certified by the state and granted with standard substance certificates.

A. 4.1.3 N, N-dimethylformamide (DMF): chromatographically pure.

### A.4.2 Instruments and Equipment

Gas chromatograph: equipped with hydrogen flame ionization detector (FID) and headspace sampler.



### **A.4.3 Reference chromatographic conditions**

A.4.3.1 Chromatographic Column: Quartz capillary column (0.32 mm × 0.25 μ m × 30 m), coated with 5% phenyl-95% dimethyl polysiloxane, or column with equivalent performance chromatographic.

A.4.3.2 Carrier gas: Nitrogen.

A.4.3.3 Carrier gas flow rate: 0.7 mL/min.

A.4.3.4 Column temperature: Maintain at 60 °C for 10 minutes.

A.4.3.5 Injection port temperature: 200°C.

A.4.3.6 Detector temperature: 300°C.

A.4.3.7 Injection volume: 1.0 mL, separation ratio 80:1.

### **A.4.4 Reference Headspace Injection Conditions.**

A.4.4.1 Sample heating temperature: 70 °C.

A.4.4.2 Sample heating time: 30 minutes.

A.4.4.3 Quantitative ring temperature: 80 °C.

A.4.4.4 Transmission line temperature: 80 °C.

### **A.4.5 Analysis Steps**

#### **A. 4.5.1 Preparation of blank solution**

Accurately measure 5.0 mL of N, N-dimethylformamide (DMF), transfer it into a headspace vial, and seal it; heat it at 70 °C for 30 min and shake vigorously, mix well.

#### **A.4.5.2 Preparation of standard solution**

Standard stock solution: Accurately weigh 0.05 g (precision to 0.0001 g) of each of methanol, ethyl acetate, n-hexane, isopropanol, and dichloromethane standard substances (A.4.1.2), dissolve them in N, N-dimethylformamide, and dilute with N, N-dimethylformamide to a 50 mL volumetric flask to the mark. 1.0 mL of this solution is equivalent to 1.0 mg of methanol, ethyl acetate, n-hexane, isopropanol, and dichloromethane. Store in a refrigerator at 4°C for future use.

Standard working solution: Accurately draw 1.0 mL of standard stock solution into a 100 mL volumetric flask, dilute to the mark with N, N-dimethylformamide, and mix thoroughly. 1.0 mL of this solution is equivalent to 10 μg of methanol, ethyl acetate, n-hexane, isopropanol, and dichloromethane.

#### **A.4.5.3 Drawing of standard curve**

Draw 0.25 mL, 0.5 mL, 1 mL, 2 mL, and 5 mL of standard working solution into headspace bottles and dilute to 5.0 mL with N, N-dimethylformamide, respectively. Seal with an aluminum cap containing a

PTFE coated gasket. Put it into the automatic headspace analyzer, and automatically import the balanced sample into the gas chromatograph for analysis. Using the chromatographic peak height (peak area) as the vertical axis and the corresponding sample solvent content as the horizontal axis, create a standard curve or perform linear regression to obtain the standard curve equation.

#### A.4.5.4 Determination of sample solution

Weigh 0.5g of the sample, precision to 0.0001 g, and place it in a headspace vial. Add 5.0 mL of N, N-dimethylformamide (DMF) and seal with an aluminum cap containing a PTFE coated gasket. After dissolution and mixing, place the sample in an automatic headspace analyzer, and automatically import the equilibrated sample into a gas chromatograph for analysis.

#### A.4.5.5 Calculation of results

The content  $w_i$  of the components to be tested (methanol, ethyl acetate, n-hexane, isopropanol, dichloromethane) in the sample is measured in milligrams per kilogram (mg/kg) and calculated according to formula (A.2):

$$w_i = \frac{m_0}{m} \dots\dots\dots (A.2)$$

In which:

$m_0$  - the mass of each solvent obtained from the standard curve, in micrograms ( $\mu\text{g}$ );

$m$  - the mass of the sample, in grams (g);

The contents of methanol, ethyl acetate, n-hexane, isopropanol, and dichloromethane calculated from equation (A.2) are  $w_1$ ,  $w_2$ ,  $w_3$ ,  $w_4$ , and  $w_5$ . The sum of the five is the residual solvent content in the sample. The calculation result shall retain three significant digits.

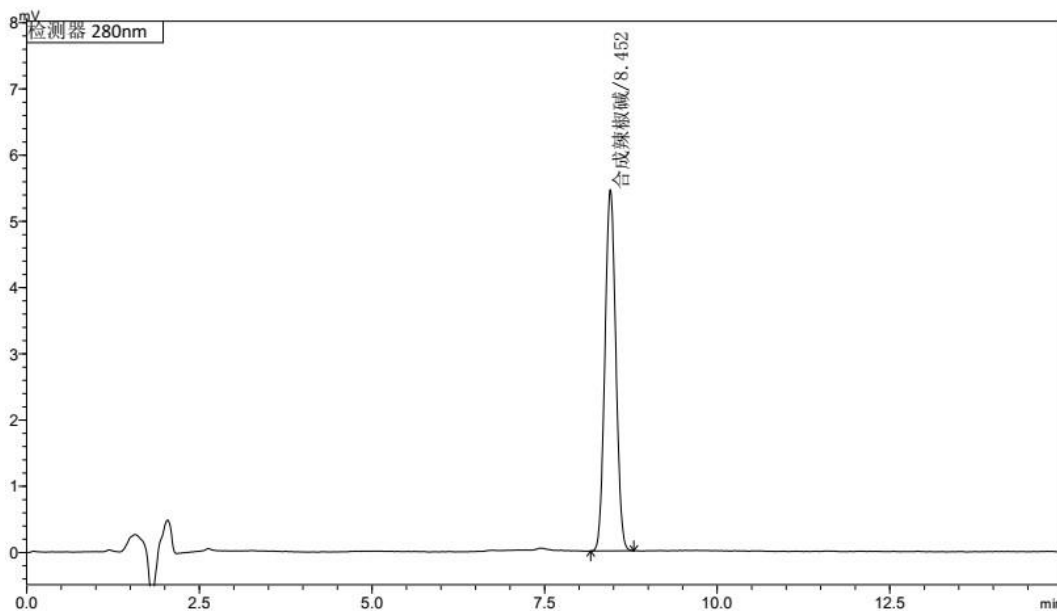
The experimental results shall be based on the arithmetic mean of the parallel measurement results. The absolute difference between two independent measurement results obtained under repetitive conditions shall not exceed 10 % of the arithmetic mean. The sample size is 0.5 g, the detection limit of this method is 3.0 mg/kg, and the quantification limit is 10.0 mg/kg.

## Appendix B

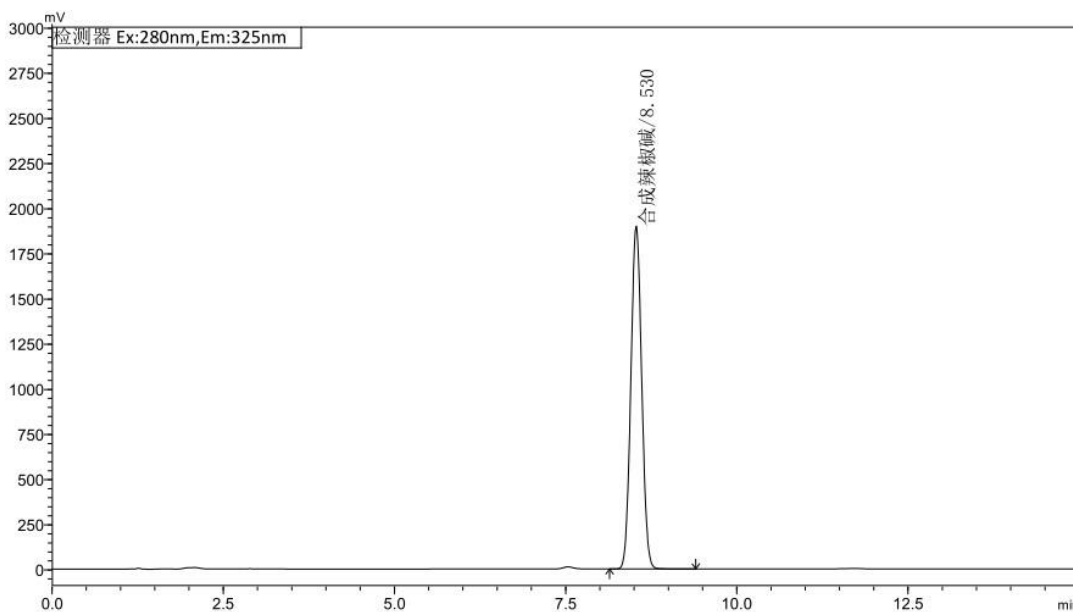
### Standard Solution Liquid Chromatogram for the Determination of Capsaicin Content

#### B. 1. Liquid chromatogram of synthesized capsaicin standard solution

The liquid chromatogram of the synthesized capsaicin standard solution is as shown in Figure B.1 and Figure B.2.



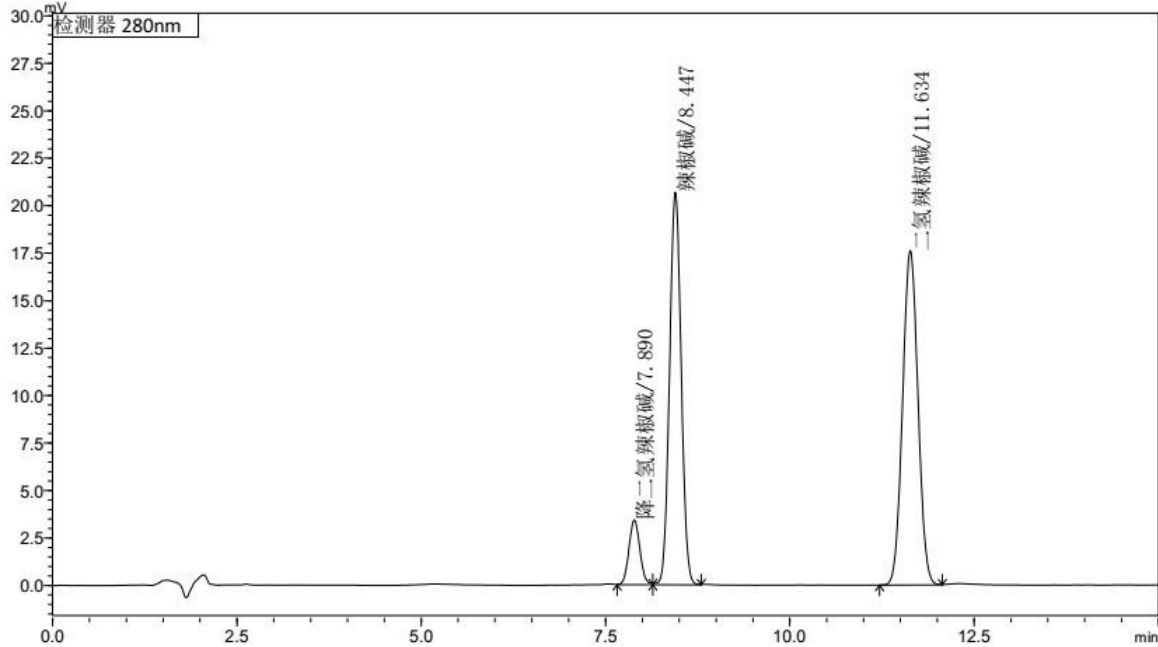
**Figure B. 1. Liquid Chromatogram of Synthesized Capsaicin Standard Solution (UV detection wavelength: 280 nm)**



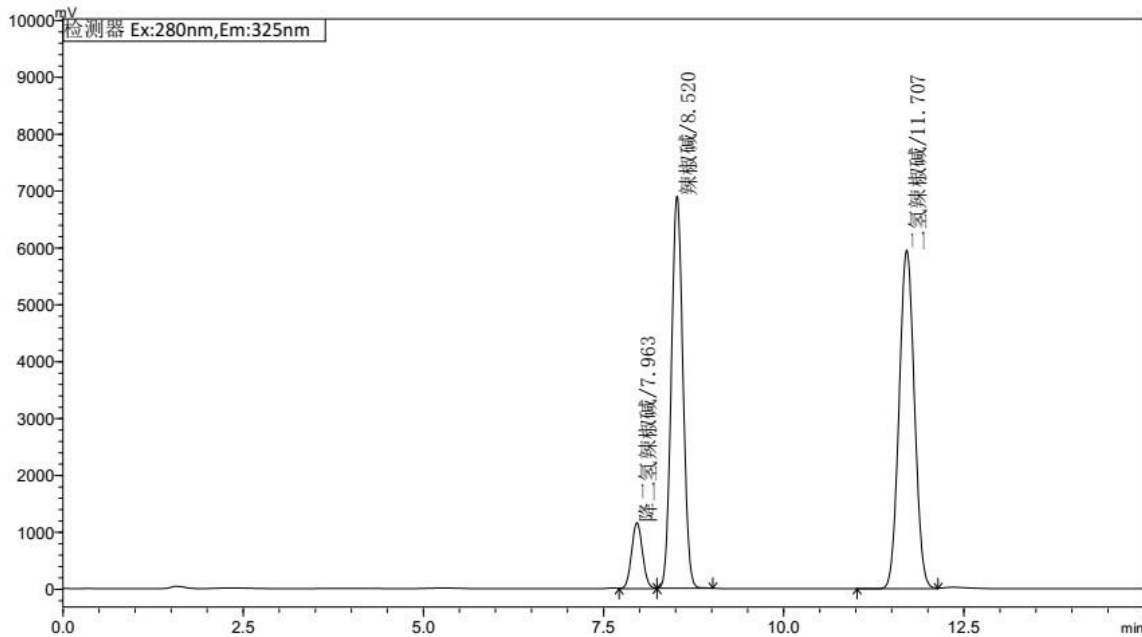
**Figure B.2. Liquid Chromatogram of Synthesized Capsaicin Standard Solution (Fluorescence excitation wavelength: 280 nm, emission wavelength: 325 nm)**

## B.2 Liquid chromatograms of standard solutions of capsaicin, dihydrocapsaicin, and nordihydrocapsaicin

The liquid chromatograms of standard solutions of capsaicin, dihydrocapsaicin, and nordihydrocapsaicin are as shown in Figure B.3 and Figure B.4, respectively



**Figure B.3: Liquid Chromatography of Standard Solutions of Capsaicin, Dihydrocapsaicin, and Nordihydrocapsaicin (UV detection wavelength: 280 nm)**



**Figure B.4: Liquid Chromatography of Standard Solutions of Capsaicin, Dihydrocapsaicin, and Nordihydrocapsaicin (Fluorescence excitation wavelength: 280 nm, emission wavelength: 325 nm)**

## Appendix C

### Gas Chromatograms of Five Solvent Residue Standard Solutions

The gas chromatograms of five solvent residue standard solutions are as shown in Figure C.1.

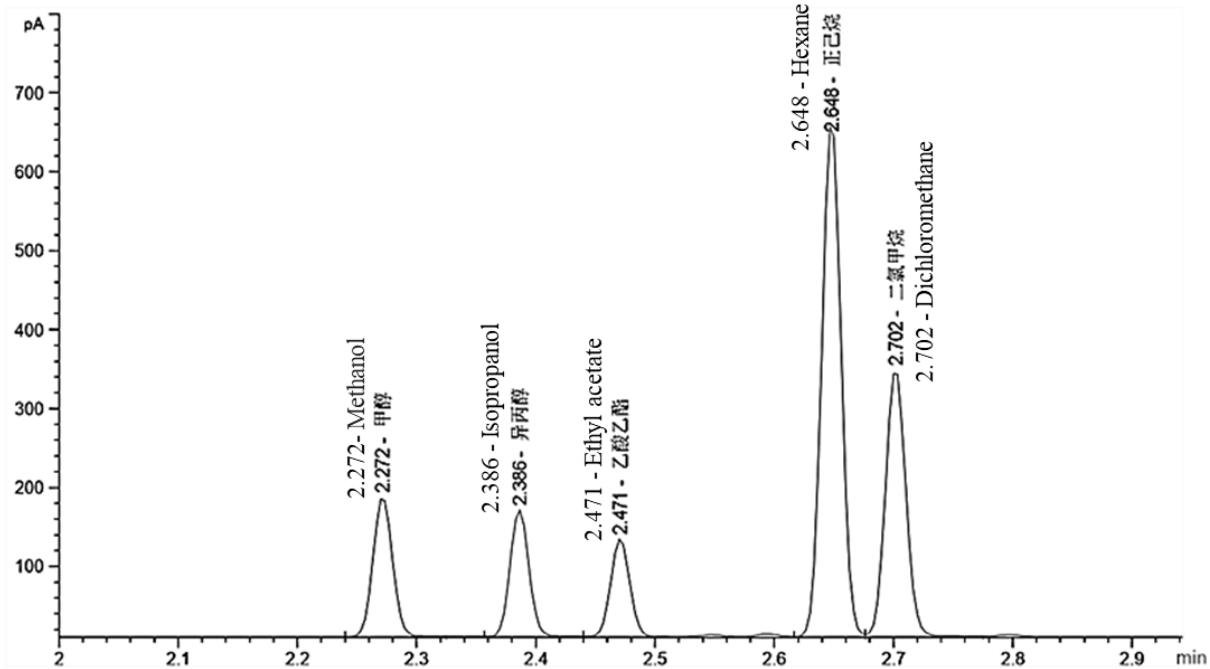


Figure C.1: Gas Chromatogram of Standard Solutions of 5 Solvents Residual

END TRANSLATION

**Attachments:**

No Attachments.