

ICS 67.050
CCS C 53

SN

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

SN/T 5218-2019

**Determination of acetylricinoleate in exported food
(English Translation)**

Issued on: January 1, 1970

Implemented on: July 1, 2020

Issued by: General Administration of Customs

Table of Contents

Foreword
1 Scope
2 Method summary
3 Reagents and materials
4 Apparatus and equipment
5 Preparation and storage of samples
6 Measurement procedure
6.1 Extraction
6.2 Determination
7 Calculation and expression of results
8 Low limit of determination, recovery rate
8.1 Lower limit of determination
8.2 Recovery rate
9 Method summary
10 Reagents and materials
11 Apparatus and equipment
12 Preparation and storage of samples
12.1 Sample preparation
12.2 Sample storage
13 Measurement procedure
13.1 Sample handling

Foreword

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

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

Drafting organization of this standard. Gongbei Customs of the People's Republic of China.

1 Scope

This standard specifies the method for the determination of acetyl ricinoleate in food. The first method is gas chromatography, and the second method is gas chromatography-quality

Spectrum joint usage.

The first method of this standard is suitable for the determination of acetyl ricinoleate content in gum-based candies (chewing gum, bubble gum), and the second method is suitable for fruit Determination of acetyl ricinoleate content in foods such as juice, solid beverages (malted milk extract), biscuits and edible fats and oils.

Method 1 Gas Chromatography

2 Method summary

After extraction and purification, the gum-based confectionery food is separated by gas chromatograph, and detected by a hydrogen flame ionization detector.

The retention time is qualitative, and the external standard method is quantified.

3 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade (stored in glass bottles, avoid contact with plastics), and water is deionized water.

3.1

Ethyl acetate. chromatographically pure.

3.2

Anhydrous sodium sulfate (Na_2SO_4). Burn at 650 °C for 4 h, cool to room temperature in a desiccator, and store in a sealed bottle for later use.

3.3

Methyl acetylricinoleate. molecular formula $\text{C}_{21}\text{H}_{38}\text{O}_4$, CAS number 140-03-4, purity greater than or equal to 99%.

3.4

Standard stock solution of methyl acetyl ricinoleate (1 g/L). accurately weigh 0.1 g (accurate to 0.1 mg) of methyl acetyl ricinoleate standard substance (3.3), transfer to a 100 mL volumetric flask, dilute with ethyl acetate (3.1), constant volume, and store in a refrigerator at 4 °C in the dark.

3.5

Standard working solution of methyl acetyl ricinoleate. pipette 0.50 mL, 1.00 mL, 2.00 mL, 5.00 mL, 10.00 mL and 20.00 mL standard stock solution (3.4) is placed in a 100 mL volumetric flask, diluted with ethyl acetate, and the volume is constant, and the concentration is 5 mg/L, 10 mg/L, Standard series solutions of 20 mg/L, 50 mg/L, 100 mg/L and 200 mg/L should be prepared immediately before use.

4 Apparatus and equipment

4.1

Gas chromatograph (GC) with hydrogen flame ionization detector (FID).

4.2

Analytical balance. Sensitivity 0.1 mg, 0.01 g and 0.001 g.

4.3

Nitrogen blowing instrument.

4.4

Oscillator.

4.5

Vortex mixer.

4.6

Ultrasonic extractor.

4.7

Organize the masher.

4.8

Refrigerated centrifuge. the maximum speed is not less than 4 000 r/min.

5 Preparation and storage of samples

Take at least the 3 smallest complete package samples of the same batch (the total amount of samples is about 100 g), and freeze them in liquid nitrogen for about 5 minutes.

Take it out and place it in a pulverizer immediately, take it out quickly and mix it evenly, place it in a hard all-glassware, and store it in an airtight manner at 0°C~4°C.

To be weighed and processed.

6.1 Extraction

Accurately weigh about 1 g of the mixed sample (accurate to 0.001 g) into a 50 mL ground glass test tube with a stopper, add 10 mL of water to the sample Disperse the product, add sodium chloride until the water phase is saturated, add 20 mL ethyl acetate (3.1), vortex for 1 min to disperse the sample, and ultrasonically extract for 20 min.

Centrifuge at 4 000 r/min for 2 min, and collect the supernatant. Repeat the extraction once, combine the supernatants, and dilute to 50 mL with ethyl acetate (3.1).

Take an appropriate amount of the extract, place it in a refrigerated centrifuge, at 4 000 r/min at 0 °C for 10 min, take the supernatant and bottle it for GC analysis.

6.2.1 Chromatographic separation conditions

6.2.1.1

Chromatographic column. HP-5 quartz capillary column, 30 m (column length) x 0.25 mm (inner diameter) x 0.25 µm (film thickness), or equivalent.

6.2.1.2

Temperature of the injection port. 250 °C.

6.2.1.3

The carrier gas is 99.999% nitrogen, in constant flow mode, and the flow rate is 1.0 mL/min.

6.2.1.4

Detector temperature. 300 °C.

6.2.1.5

Injection volume. 1.0 µL.

6.2.1.6

Sampling mode. splitless sampling.

6.2.1.7

According to the heating program listed in Table 1, the target analyte can be separated well.

6.2.2 Quantitative determination

Inject the standard series of working fluids into the gas chromatograph to determine the chromatographic peak area of acetyl ricinoleate.

The volume concentration is the abscissa, and the corresponding peak area is the ordinate to draw a standard curve. Inject the sample solution into the instrument to obtain acetyl castor. According to the peak area of the acid ester, the concentration of acetyl ricinoleate in the test solution is obtained according to the standard curve. If the content of the analyte in the sample exceeds the linearity Range, use ethyl acetate (3.1) to dilute to a suitable concentration and analyze. Reference retention of methyl acetylrucinoleate under the above chromatographic conditions the time is 19.5 minutes, and the gas chromatogram of the standard working solution is shown in Figure A.1 of Appendix A.

6.2.3 Blank test

Except that no sample is added, all are carried out according to the above-mentioned measurement steps.