

CCS X 04



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

**GB 31657.1-2021**

---

**National food safety standard - Determination of Tau-fluvalinate  
residue in honey and royal jelly by gas chromatography method**

**Issued on: September 16, 2021**

**Implemented on: February 1, 2022**

---

**Issued by: National Health Commission of the People's Republic of  
China, State Administration for Market Regulation**

## Table of Contents

Foreword

1 Scope

2 Normative reference documents

3 Terms and definitions

4 Principles

5 Reagents and materials

6 Instruments and equipment

8 Measurement steps

9 Calculation and presentation of results

10 Sensitivity, accuracy and precision of detection methods

### Foreword

This document complies with the provisions of GB/T 1:1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents"

Drafting:

This document is published for the first time:

National food safety standards

Determination of fluvalinate residues in honey and royal jelly by gas chromatography

### 1 Scope

This document specifies the sample preparation and gas chromatographic determination methods for the detection of fluvalinate residues in honey and royal jelly:

This document is applicable to the determination of fluvalinate residues in honey and royal jelly:

### 2 Normative reference documents

The contents of the following documents constitute essential provisions of this document through normative citations in the text: Among them, the cited documents with dates are:

Only the version corresponding to the date applies to this document; for undated referenced documents, the latest version (including all amendments) applies to this document:

document:

GB/T 6682 Specifications and test methods for water used in analytical laboratories

### 3 Terms and definitions

There are no terms or definitions that need to be defined in this document:

### 4 Principles

The remaining fluvalinate in the sample was dissolved in water, extracted with n-hexane-acetone solution, and purified with a Florisil solid-phase extraction column, using the configuration

Determination by gas chromatography with electron capture detector and quantification by external standard method:

### 5 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade and the water is first-grade water that complies with GB/T 6682:

#### 5.1 Reagents

5.1.1 Acetone ( $\text{CH}_3\text{COCH}_3$ ): chromatographically pure:

5.1.2 n-hexane ( $\text{C}_6\text{H}_{14}$ ): chromatographically pure:

#### 5.2 Solution preparation

5.2.1 n-hexane-acetone solution (1:1): Take equal volumes of n-hexane and acetone and mix well:

5.2.2 n-hexane-acetone solution (95:5): Take 5mL of acetone and 95mL of n-hexane and mix well:

#### 5.3 Standard products

Fluvalinate (tau-fluvalinate,  $\text{C}_{26}\text{H}_{22}\text{ClF}_3\text{N}_2\text{O}_3$ , CAS number: 102851-06-9): content  $\geq 98.0\%$ :

#### 5.4 Preparation of standard solution

5.4.1 Standard stock solution: Take 10 mg of fluvalinate reference substance, weigh it accurately, dissolve it with n-hexane and dilute it to a volume of 100 mL:

bottle, prepare a standard stock solution with a concentration of 100  $\mu\text{g/mL}$ : Store at  $0^\circ\text{C} \sim 5^\circ\text{C}$ , and is valid for 6 months:

5.4.2 Standard working solution: Precisely measure an appropriate amount of standard

stock solution, dilute it with n-hexane, and prepare a concentration of 10 µg/L, 20 µg/L, 50µg/L, 100µg/L, 200µg/L and 500µg/L series standard working solutions: Ready for use:

#### 5:5 Materials

Florisil solid phase extraction column: 1g/6mL, or equivalent:

## 6 Instruments and equipment

6:1 Gas chromatograph: equipped with electron capture detector:

6:2 Analytical balance: sensitivity 0:00001g and 0:01g:

6:3 Vortex mixer:

6: Centrifuge:

6:5 Rotary evaporator:

6:6 Solid phase extraction device:

6:7 Nitrogen blower:

### 7: Preparation and preservation of samples

#### 7:1 Preparation of samples

Honey: Stir the test sample evenly; for crystallized samples, place them in a water bath not exceeding 60°C to dissolve them in a sealed state, and stir

Mix evenly and quickly cool to room temperature: Separate about 150g as a sample, place it in a sample bottle, seal it, and mark it:

Royal jelly: Stir the test sample evenly; for frozen test samples, stir evenly after thawing completely at room temperature: Separate about 150g:

As a test material, place it in a sample bottle, seal it, and mark it:

#### 7:2 Preservation of samples

Honey: Store at room temperature:

Royal jelly: Store below -18 degrees C:

## 8 Measurement steps

### 8:1 Extraction

Take 5g of honey (accurate to +/-0:02g) [2g of royal jelly (accurate to +/-0:02g)], put it into a 50mL stoppered polypropylene centrifuge tube, and add water

10 mL, vortex and mix for 1 min to dissolve: Add 20 mL of n-hexane-acetone solution (1:1),

vortex for 3 min, and centrifuge at 3000 r/min for 5 min:

Take the upper layer into a 150 mL chicken heart bottle: Repeat the extraction once with 20 mL of n-hexane-acetone solution (1:1), combine the upper layers, and decompress in a 40°C water bath:

Concentrate to nearly dryness, add 3 mL of n-hexane to dissolve, and set aside:

## 8:2 Purification

The Florisil solid-phase extraction column was activated with n-hexane-acetone solution (95:5) and 5 mL of n-hexane in sequence: Pass the reserve solution through the column, and then use normal hexane:

Wash the chicken heart bottle with 3 mL of hexane, pass the washing liquid through the column, and discard this part of the effluent: Elute with 10 mL of n-hexane-acetone solution (95:5), and collect the washing liquid:

Deliquidate, blow to dryness with nitrogen in a 40°C water bath, redissolve with 1:0 mL of n-hexane, and use for gas chromatography measurement:

## 8:3 Preparation of standard working curve

Accurately draw an appropriate amount of fluvalinate standard working solution and inject samples respectively: Take the concentration of the standard working solution as the abscissa and the peak area as the vertical axis:

mark and draw the standard working curve:

## 8: Measurement

### 8:4:1 Gas Chromatography Reference Conditions

a) Chromatographic column: VF?1MS (100% dimethylpolysiloxane) quartz capillary column (30mx0:0:25mmx0:025m), or equivalent

By;

b) Carrier gas: nitrogen, purity  $\geq 99.999\%$ ;

c) Inlet temperature: 280 degrees C;

d) Detector temperature: 300 degrees C;

e) Column temperature: initial temperature is 80°C, hold for 1 minute, increase temperature to:200°C at 30°C/min, hold for 1 minute, and then increase temperature at 10°C/min:

to 280 degrees C, keep for 6 minutes;

f) Carrier gas flow rate: 1:0mL/min;

g) Injection volume: 1μL;