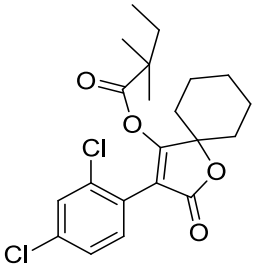
SPIRODICLOFEN

737



ISO common name	Spirodiclofen
Chemical Name	3-(2,4-dichlorophenyl)-2-oxo-1-oxaspiro[4.5]dec-3-en-4-yl 2,2-dimethylbutyrate
Empirical formula	$C_{21}H_{24}Cl_2O_4$
RMM	411.3
m.p.	94.8 °C
v.p	Less than $<3 \times 10^{-4}$ mPa (20 °C)
Log K_{ow}	5.8 (pH 4), 5.1 (pH 7) (both ambient temp.)
Solubility (g/L, 20 ℃)	In water 50 (pH 4), 190 (pH 7) (both in μg/l, 20 °C). In *n*-heptane 20, polyethylene glycol 24, *n*-octanol 44, isopropanol 47, DMSO 75, acetone, dichloromethane, ethyl acetate, acetonitrile and xylene >250 (all in g/l, 20 °C).
Description	White crystalline powder
Stability	Decomposed under alkaline conditions
Formulation	Suspension concentrates

SPIRODICLOFEN TECHNICAL

737/TC/M/-

1. Sampling. Take at least 200 g.

2. Identity tests

2.1 HPLC. Use the HPLC method below. The relative retention time of the spirodiclofen peak in the sample solution should not deviate by more than 1.5% from that of the calibration solution.

2.2 Infrared. Prepare potassium bromide disks from the sample and standard spirodiclofen (about 0.7 mg material and 400 mg potassium bromide). Scan the discs from 4000 cm^{-1} to 400cm^{-1}. The spectrum obtained from the sample should not differ significantly from that of the standard.

3. Spirodiclofen

3.1 OUTLINE OF METHOD

Spirodiclofen is determined by high performance liquid chromatography on a reversed phase column with UV detection and external standardization.

3.2 REAGENTS

Spirodiclofen standard of known purity

Water deionized, HPLC grade

Methanol HPLC grade

Mobile phase, methanol-water (90+10) (v/v)

Calibration solution. Weigh in duplicate (to the nearest 0.1mg) approximately 50mg of spirodiclofen standard (s mg) into separate volumetric flask (50ml). Add methanol to each flask (about 30 ml), place the flask in an ultrasonic bath for 5 mins. Allow the solution to attain ambient temperature and dilute to volume with methanol. Mix thoroughly (Solution C_A and C_B).

3.3 APPARATUS

High performance liquid chromatography equipped with a detector suitable for operation at 260nm, a constant-temperature column compartment and an injection system capable of injecting 20 µl.

Column stainless steel, 250mm × 4.6mm (i.d) columns, C18 packed with octadecyl silane filler, or equivalent.

Ultrasonic Bath

Electronic integrator or data system

3.4 PROCEDURE

(a) Liquid chromatographic conditions (typical)

Column: C18 (250 mm x 4.6 mm) packed with octadecyl silane filler

Mobile phase: Methanol – water, 90+10(v/v)

Column Temperature: 25℃

Flow Rate: 1 ml/min

Detector Wavelength：260nm

Injection volume: 5μl

Retention time: approximately 7.8min

(b) System equilibration

Pump sufficient mobile phase through the column to equilibrate the system. Inject 5ul portions of the calibration solution C_A and repeat the injections until peak areas obtained from two consecutive injections deviate by less than 1.5%.

(c) Preparation of sample: Prepare sample solutions in duplicate. Weigh (to the nearest 0.1mg) into separate volumetric flask (50ml) sufficient sample to contain about 50mg (w mg) spirodiclofen. Add methanol to each flask (about 30 ml), place the flask in an ultrasonic bath for 5 mins. Allow the solution to attain ambient temperature and dilute to volume with methanol, Mix thoroughly (sample solutions S_1 and S_2).

(d) Determination: Inject in duplicate **5 μl** portions of each sample solution bracketing them by injections of the calibration solutions as follows:

C_A, S_1, S_1, C_B, S_2, S_2, C_A, etc

(e) Calculation. Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the spirodiclofen contents of the bracketed sample injections.

$$f_i = \frac{s \times P}{H_s} \quad \text{……（1）}$$

$$\text{spirodiclofen content} = \frac{f \times H_w}{w} \text{ g/kg}$$

Where,

f_i = individual response factor

f = mean response factor

H_s = peak area of spirodiclofen in the calibration solution

H_w = peak area of spirodiclofen in the sample solution

s = mass of spirodiclofen reference standard in the calibration solution (mg)

w = mass of sample taken (mg)

P =purity of spirodiclofen reference standard (g/kg)

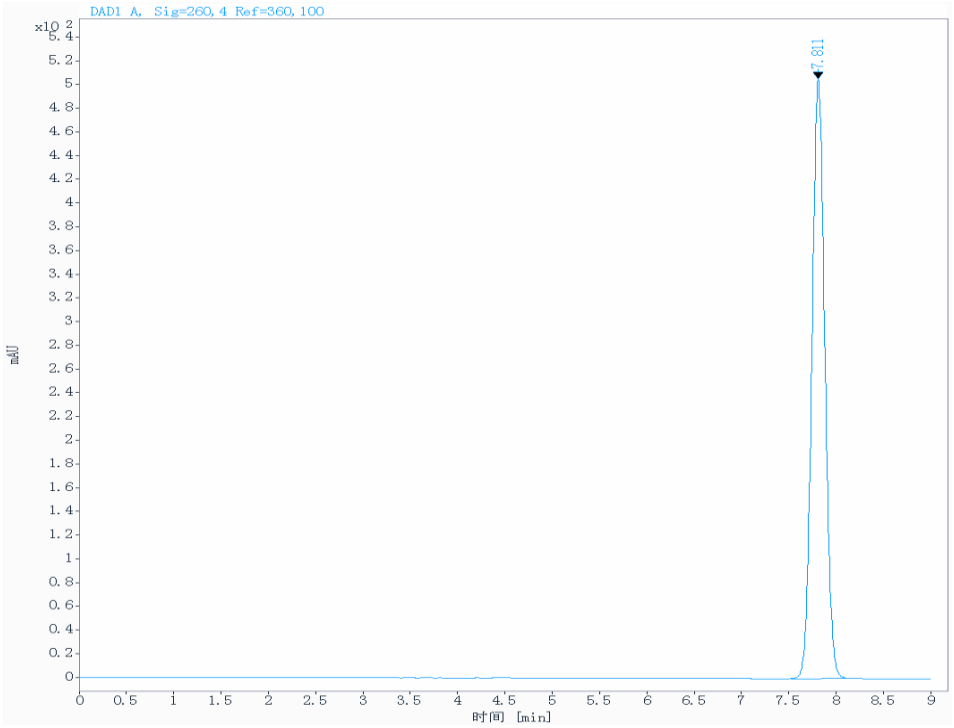


Fig 1 Chromatogram of spirodiclofen standard

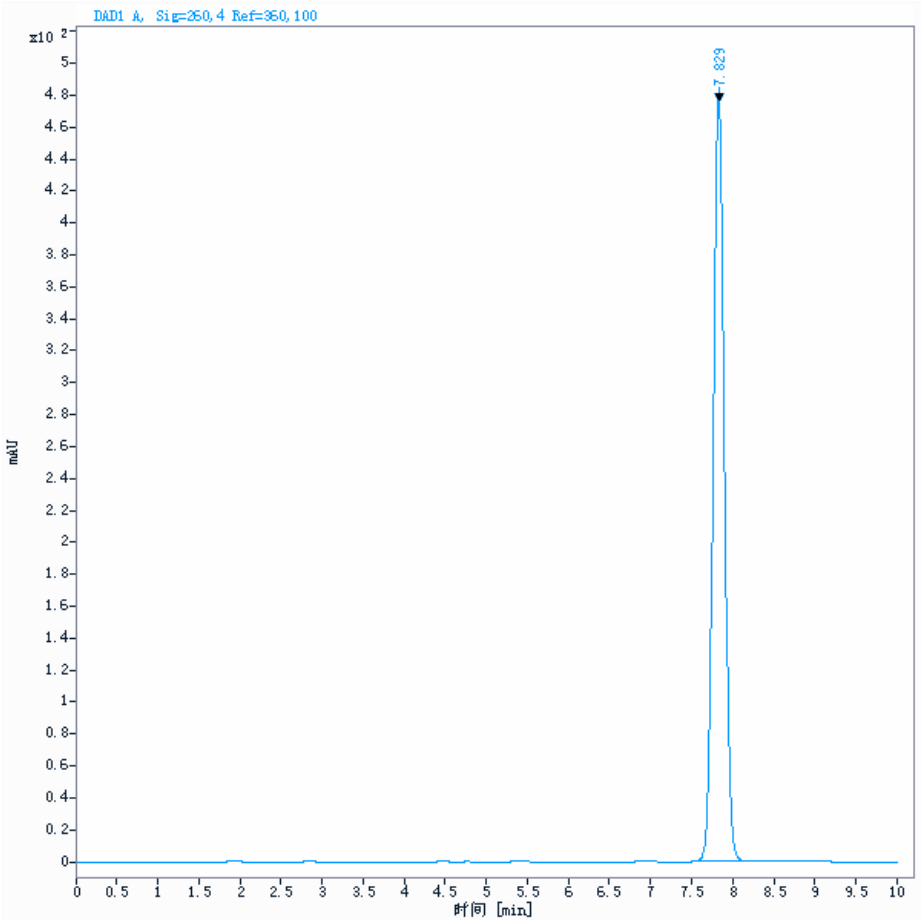


Fig 2 Chromatogram of spirodiclofen sample

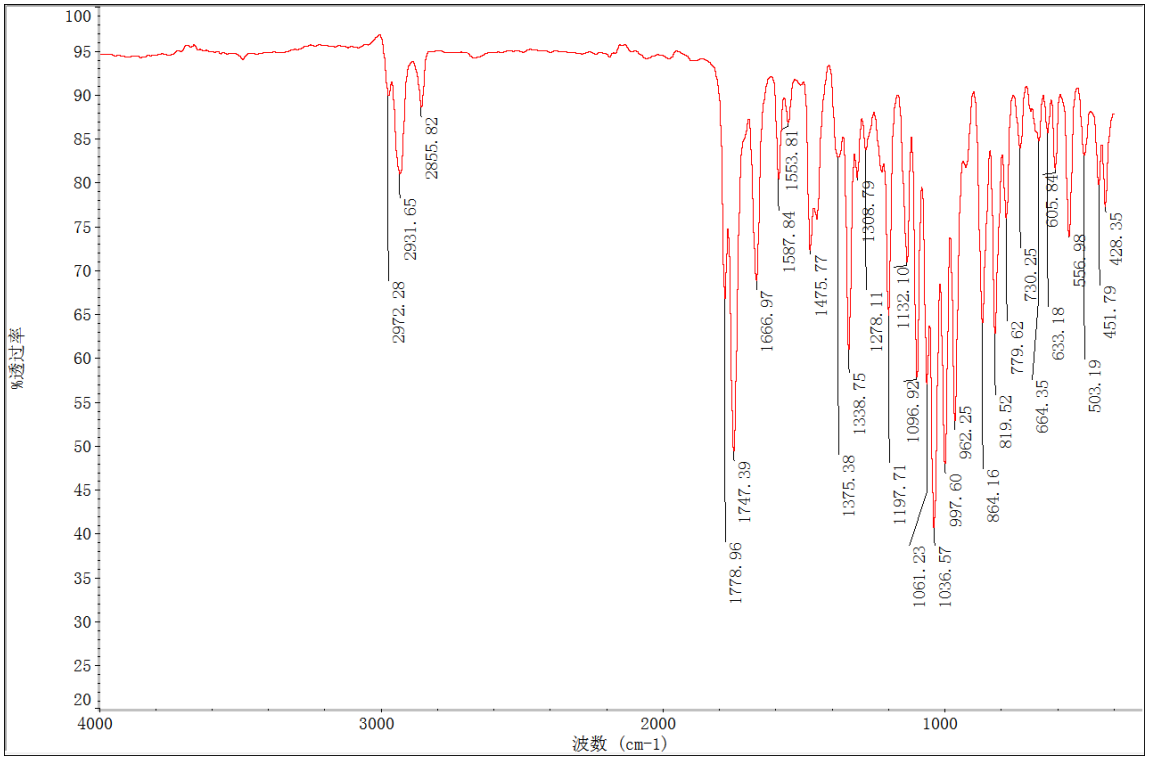


Fig 3 Infrared spectra of spirodiclofen standard

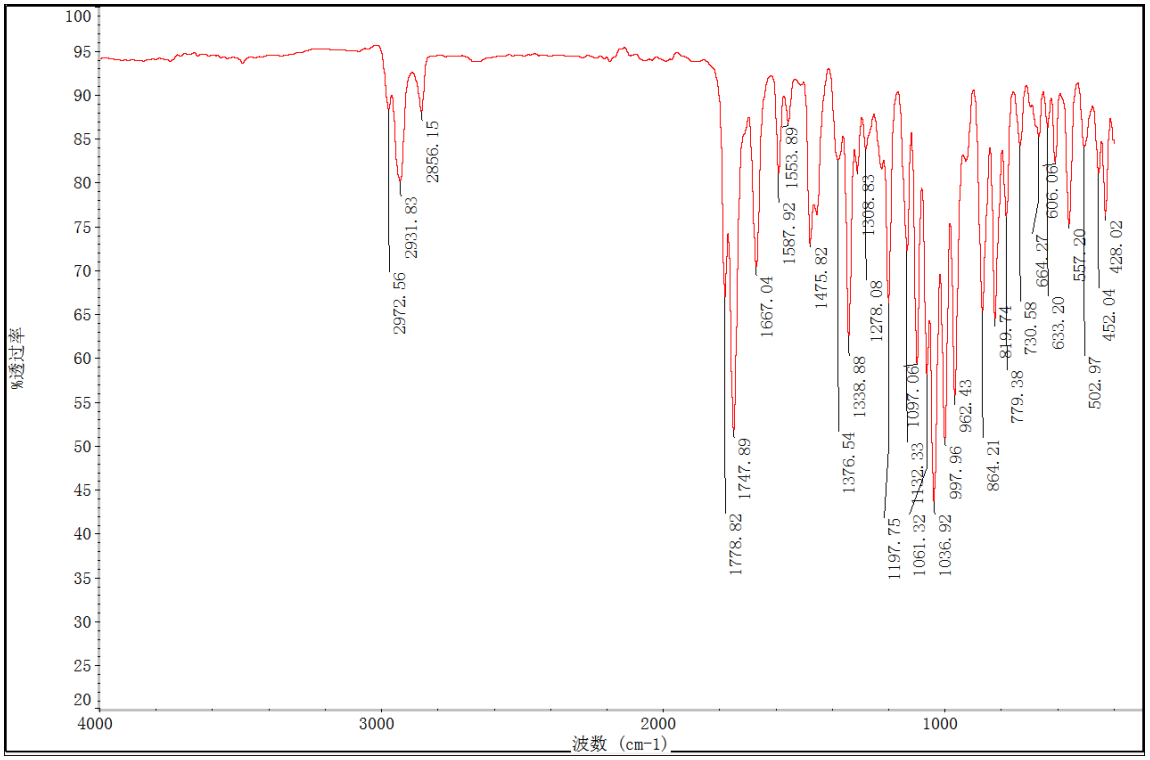


Fig 4 Infrared spectra of spirodiclofen sample

SPIRODICLOFEN SUSPENSION CONCENTRATE

737/SC/M/-

1. Sampling Take at least 500 g.

2. Identity tests

2.1 HPLC. Weigh (to the nearest 0.1mg) into volumetric flask (50ml) sufficient sample to contain about 50mg (w mg) spirodiclofen. Add methanol to flask (about 30 ml), place the flask in an ultrasonic bath for 5 mins. Allow the solution to attain ambient temperature and dilute to volume with methanol, Mix thoroughly. If necessary, centrifuge or filter the solution to obtain a clear solution. Then as for spirodiclofen technical 737/TC/M/2.1.

2.2 Infrared. Weigh (to the nearest 0.01g) into breaker. Water (about 200g) is added. Mix thoroughly. And then centrifuged about 30min. Take liquid supernatant away and collect the solid at the bottom. The solid is dried with a stream of clean dry air. Then as for spirodiclofen technical 737/TC/M/2.2.

3. Spirodiclofen. As for spirodiclofen technical 737/TC/M/3，except：

Preparation of sample: Prepare sample solutions in duplicate. Weigh (to the nearest 0.1mg) into separate volumetric flask (50ml) sufficient sample to contain about 50mg (w mg) spirodiclofen. Add methanol to each flask (about 30 ml), place the flask in an ultrasonic bath for 5 mins. Allow the solution to attain ambient temperature and dilute to volume with methanol, Mix thoroughly. If necessary, centrifuge or filter the solution to obtain a clear solution. (sample solutions S_1 and S_2).

4. Suspensibility

Reagents and apparatus as for 737/TC/M and MT 184

(a) Preparation of suspension and determination of sedimentation. MT 184.

(b) Determination of Spirodiclofen in the bottom 25mL of suspension. After removal of the top 225mL suspension transfer the 25mL remaining quantitatively to a volumetric flask (100mL) and dilute to volume with methanol, sonication and filtration until the solution is transparent. Take an aliquot of the solution and determine the mass of spirodiclofen (Q g) by 737/TC/M/- .

(c) Calculation

$$\text{Suspensibility} = \frac{111(c - Q)}{c} \quad \%$$

where:

c = mass of spirodiclofen in the sample taken for the preparation of the suspension (g)

Q = mass of spirodiclofen in the bottom 25 mL of suspension (g)

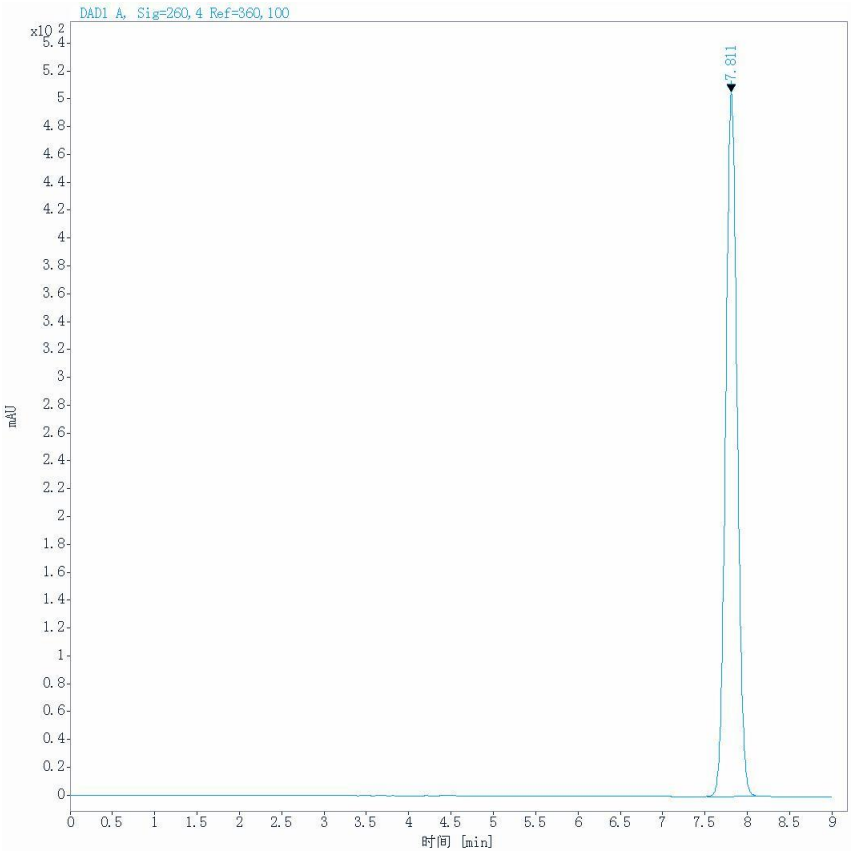


Fig 5 Chromatogram of spirodiclofen standard

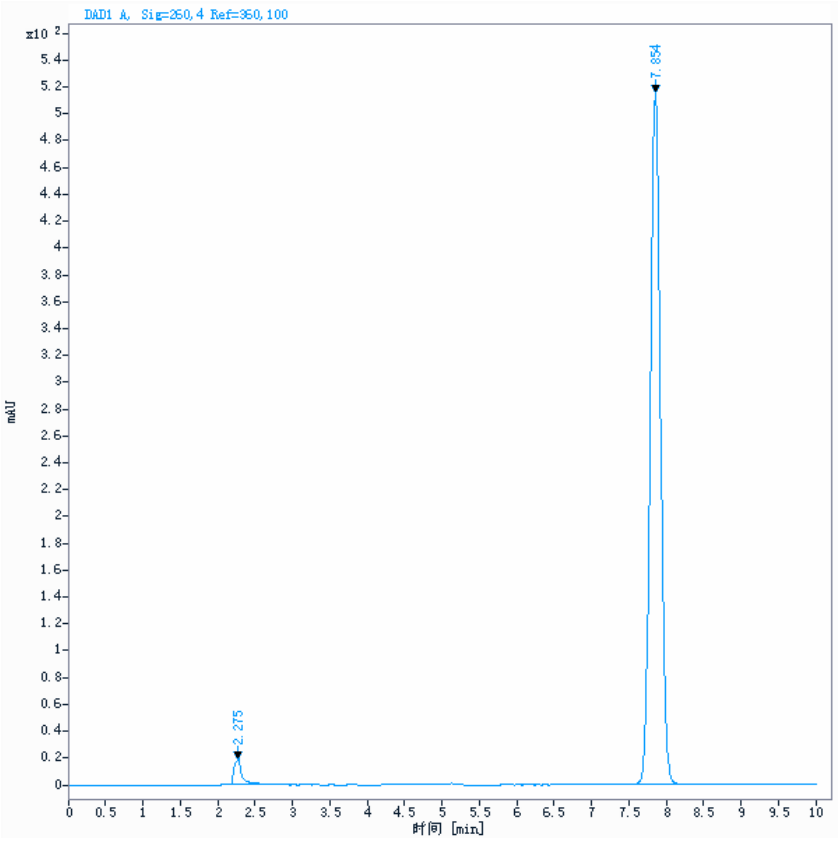


Fig 6 Chromatogram of spirodiclofen SC sample

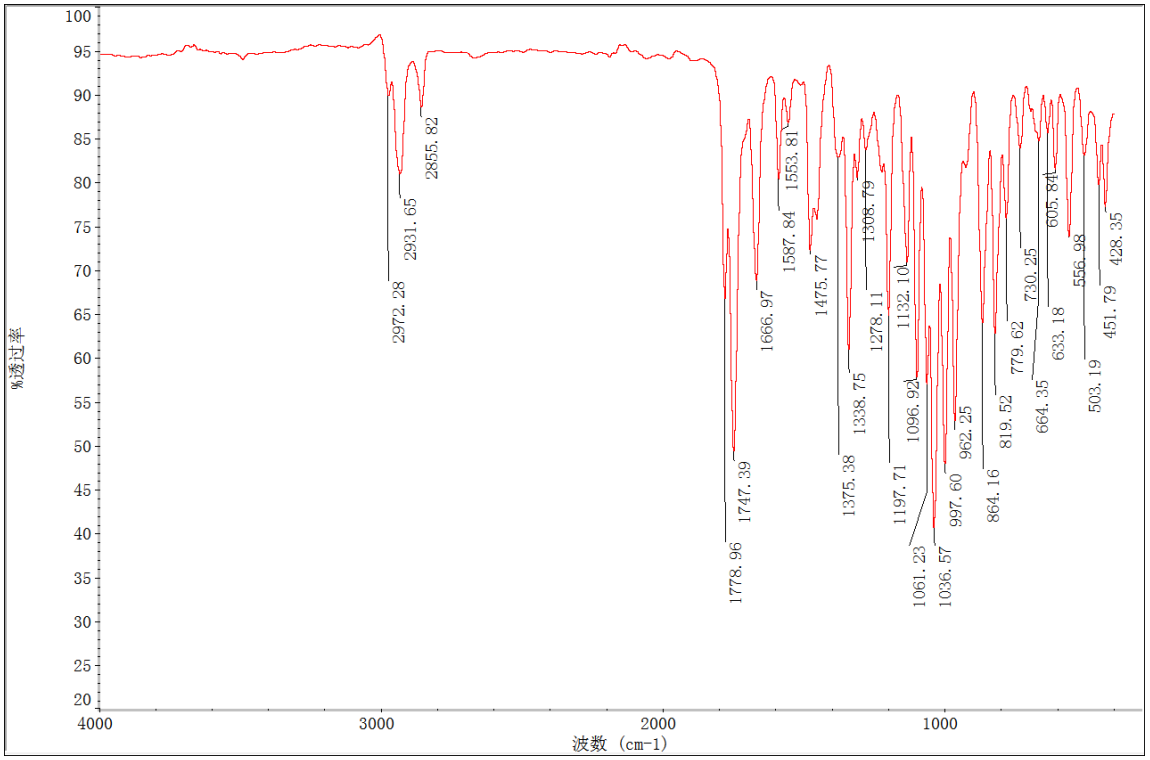


Fig 7 Infrared spectra of spirodiclofen standard

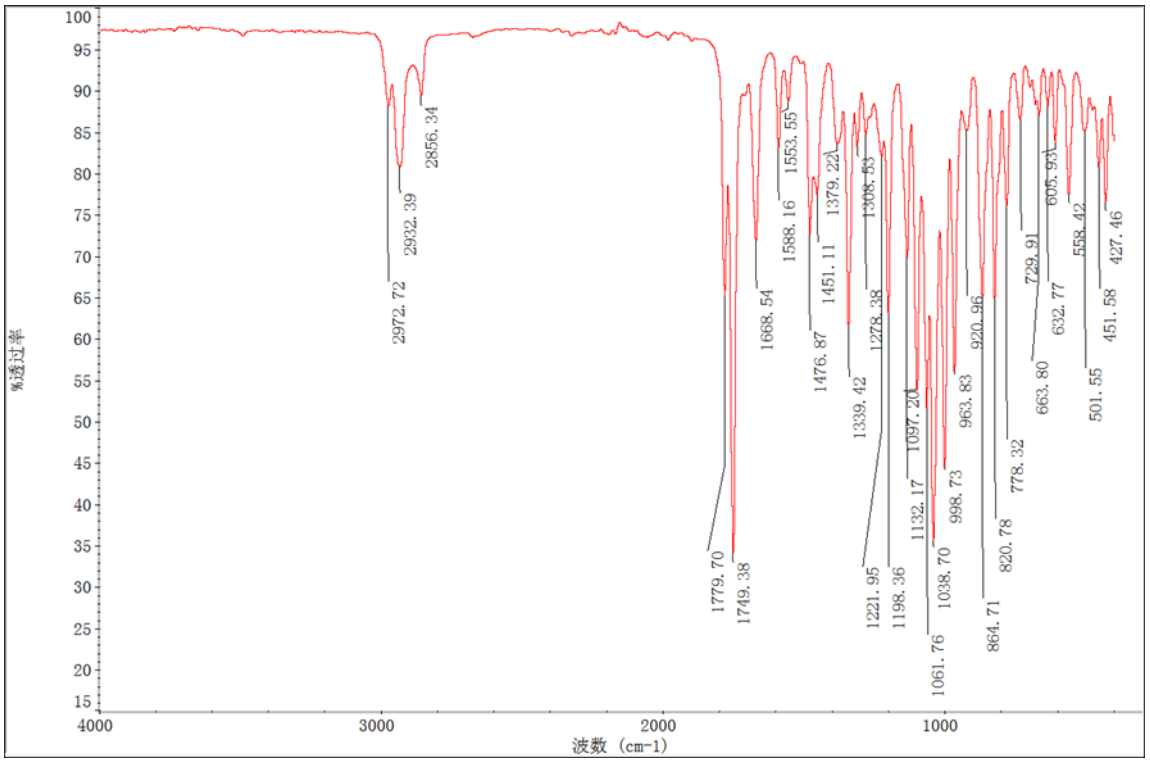


Fig 8 Infrared spectra of spirodiclofen SC sample