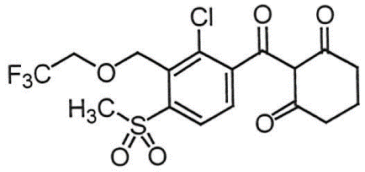
TEMBOTRIONE

790



ISO Common name	tembotrione
Chemical name	2-{2-chloro-4-mesyl-3[(2,2,2-trifluoroethoxy)methyl]benzoyl}cyclohexane-1,3-dionetrifluoroethoxy)methyl]benzoyl]-1,3-cyclohexanedione
CAS No.	335104-84-2
Empirical formula	$C_{17}H_{16}ClF_3O_6S$
RMM	440.8
m.p.	123 ℃
v.p.	1.1×10^{-5} mPa at 20 ℃； 2.9×10^{-5} mPa at 25 ℃
Solubility	In water 0.22 g/L（pH 4）, dichloromethane and DMSO＞600 g/L (all at 20-25 ℃)
Stability	Stable at room temperature
Description	Beige powder
Formulation	suspending concentrates (SC), oil-based suspension concentrates (OD)

TEMBOTRIONE TECHNICAL

790/TC/M/-

1. Sampling. Take at least 100 g.

2. Identity tests

2.1 HPLC. Use the HPLC method below. The relative retention time of the tembotrione 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 discs for the technical sample and tembotrione reference substance. Scan the discs from 4000 to 400 cm^{-1}. The spectrum from the sample should not differ significantly from that of the reference substance.

3. Tembotrione

OUTLINE OF METHOD

Tembotrione is determined by high performance liquid chromatography on a reversed phase column (C18) with UV detection at 284 nm and external standardization.

REAGENTS

Tembotrione reference standard of known purity.

Acetonitrile HPLC grade.

Water Milli-Q grade or distilled.

Phosphoric acid analytical grade .

0.1% Phosphoric acid aqueous solution Dissolve 1 ml Phosphoric acid into

1000 ml water.

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) 50 mg of tembotrione reference standard (*s* mg) into separate volumetric flasks

(100ml). And then add 50 ml acetonitrile and sonicate for 5 minutes. Allow to cool to room temperature and dilute to volume with acetonitrile. Mix thoroughly (calibration solutions C_A and C_B).

APPARATUS

High performance liquid chromatograph equipped with a UV detector capable for operation at 284 nm, a constant-temperature column compartment and an injection system capable of injecting 5µl

Column stainless steel 250 ×4.6 mm (i.d), packed with C_{18}, 5.0 µm, or equivalent with the same selectivity

Filtering apparatus disposable plastic syringes (or equivalent) fitted with 0.45 µm filters

Electronic integrator or data system

Ultrasonic bath

PROCEDURE

(a) Liquid chromatographic conditions (typical):

Column	stainless steel, 250 ×4.6 mm (i.d), packed with C_{18}, 5.0 µm, or equivalent with the same selectivity
Mobile phase	acetonitrile: *0.1%* phosphoric acid aqueous solution, 60:40 (v/v)
Flow rate	1.0 ml/min
Column temperature	30 ℃ ± 2 ℃
Detector wavelength	284 nm
Injection volume	5 µl
Retention time	approximately 6.5 min
Run time	15 min

(b) System equilibration. Inject 5 µl portions of calibration solution C_A until the response factors (*fi*) obtained for two consecutive injections differ by less than 1.5%. Then inject 5µl portions of calibration solution C_B. The response factor (fi), for two consecutive injections should not deviate by more than 1.5% from that of solution C_A, otherwise prepare new calibration solutions.

(c) Sample preparation. Prepare solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about 50 mg of tembotrione into a volumetric flask (100 ml). And then add 50 ml acetonitrile and sonicate for 5 minutes. Allow to cool to room temperature and dilute to volume with acetonitrile. 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, ...

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

$$f_i = \frac{S \times P}{H_s}$$

$$Tembotrione\ content\ (g/kg) = \frac{H_w \times f}{W}$$

where:

f_i = individual response factor
f = mean response factor
H_s = peak area of tembotrione in the calibration solution
H_w = peak area of tembotrione in the sample solution
S = mass of tembotrione reference standard in the calibration solution (mg)
W = mass of sample taken (mg)
P = purity of tembotrione reference standard (g/kg)

TEMBOTRIONE SUSPENDING CONCENTRATE

790/SC/M/-

1. Sampling. Take at least 1000 g .

2. Identity tests.

2.1 HPLC. As for tembotrione technical 790/TC/M/2.1

3. Tembotrione. As for tembotrione technical 790 /TC/M/3.

TEMBOTRIONE OIL-BASED SUSPENSION CONCENTRATE

790/OD/M/-

1. Sampling. Take at least 1000 g .

2. Identity tests.

2.1 HPLC. As for tembotrione technical 790/TC/M/2.1

3. Tembotrione. As for tembotrione technical 790 /TC/M/3 except:

(c) Sample preparation. Weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about 50 mg of tembotrione into a volumetric flask (100 ml). Add 2 ml water and shake well. And then add 50 ml acetonitrile and sonicate for 5 minutes. Allow to cool to room temperature and dilute to volume with acetonitrile. Mix thoroughly and filter through a 0.45 μm filter membrane prior to analysis (sample solutions S_1 and S_2).

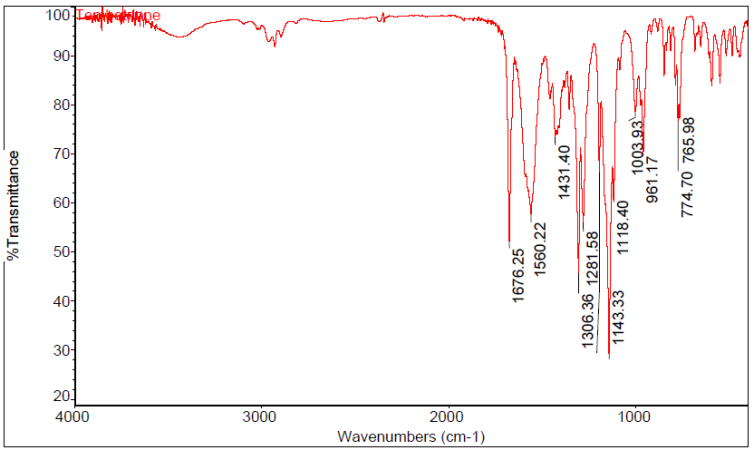


Fig.1 FTIR spectrum of tembotrione standard

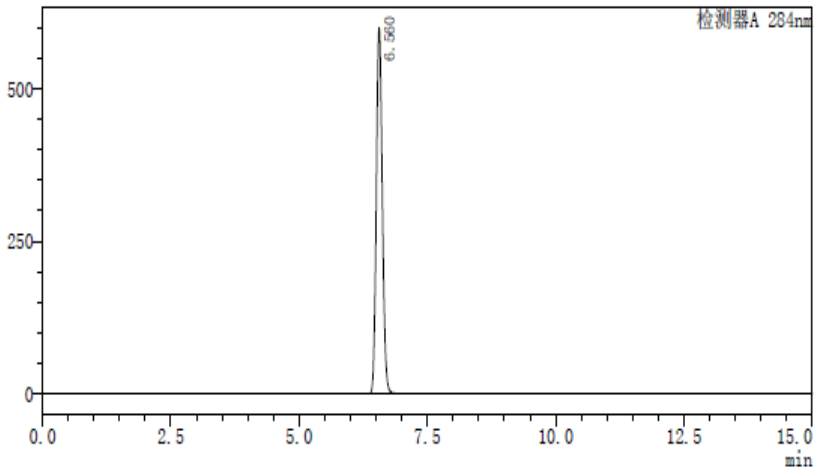


Fig. 2 HPLC Chromatogram of tembotrione standard

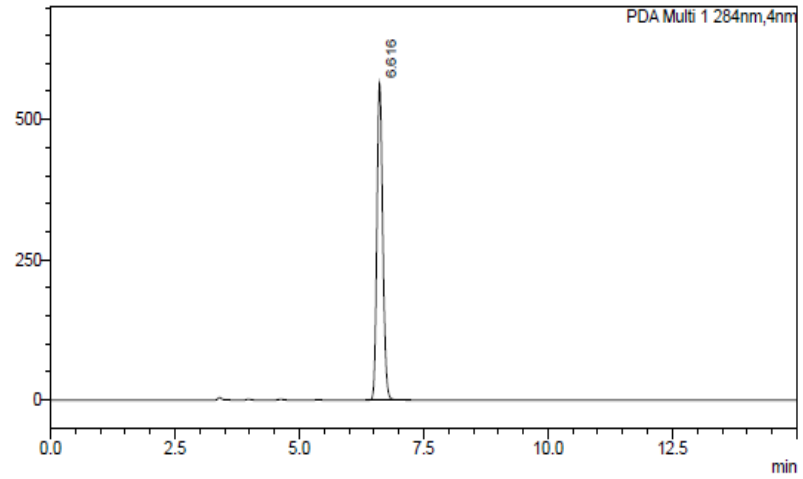


Fig. 3 HPLC Chromatogram of tembotrione TC

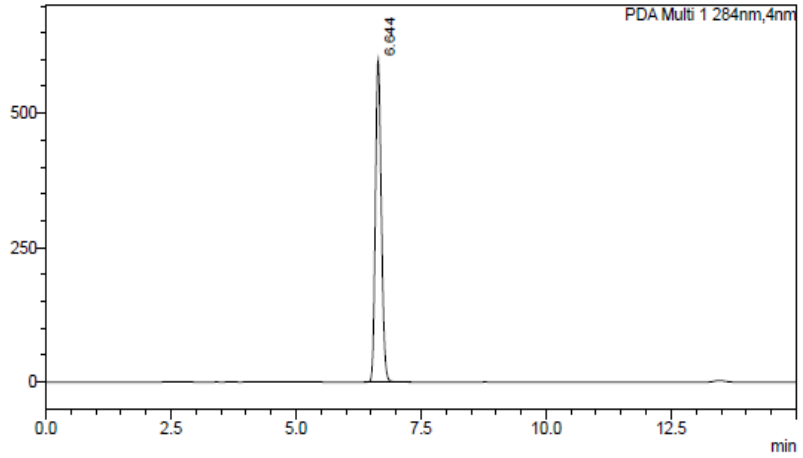


Fig. 4 HPLC Chromatogram of tembotrione SC

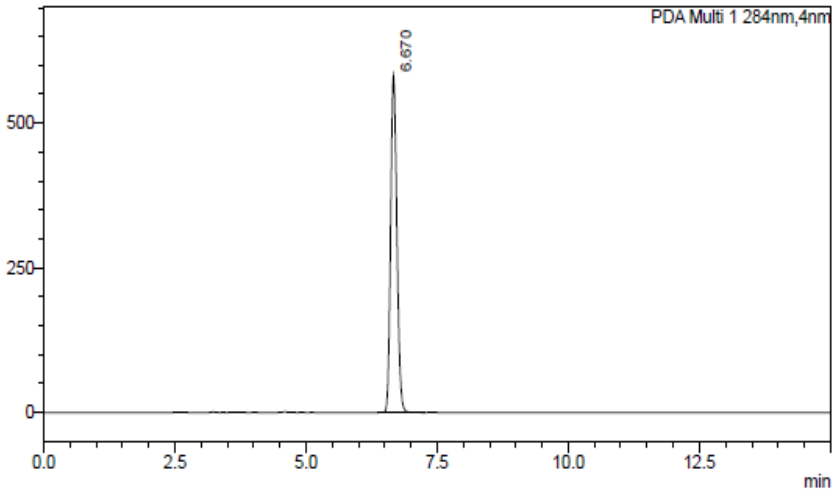


Fig. 5 HPLC Chromatogram of tembotrione OD