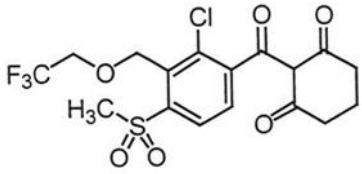
TEMBOTRIONE

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 Number	335104-84-2
Empirical formula	$C_{17}H_{16}ClF_3O_6S$
Molecular mass	440.8
m.p.	123°C
b.p.	No boiling point at atmospheric pressure; decomposition started around 150 °C
v.p	1.1×10^{-5} mPa at 20°C； 2.9×10^{-5} mPa at 25°C
d^{20}	1.56
Solubility	In water 0.22 g/L (pH 4), dichloromethane and DMSO ＞600 g/L (all at 20-25°C)
Stability	Stable at room temperature
Description	Beige powder
Formulations	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: 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 until the sample has been dissolved completely (about 5 min). 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 Agilent C_{18} 5.0 µm, or equivalent with the same selectivity.
Mobile phase	acetonitrile: 0.1% phosphoric acid aqueous solution, 60:40 (v/v)
Column temperature	30°C ± 2°C
Flow rate	1.0 ml/min
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 until the sample has been dissolved completely (about 5 min). 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.

$$f_i = \frac{s \times P}{Hs}$$

$$\text{Content of Tembotrione} = \frac{H_W \times f}{W} \text{ (g/kg)}$$

where:

fi = 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 the tembotrione reference standard (g/kg)

Repeatability r = g/kg at an active ingredient content of g/kg
Reproducibility R = g/kg at an active ingredient content of 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

2.2 Infrared. As for Tembotrione technical **790** /TC/(M)/2.2

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). And then add 50 ml acetonitrile and sonicate about 5 min. 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).

Repeatability r = g/kg at an active ingredient content of g/kg
Reproducibility R = g/kg at an active ingredient content of g/kg

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

2.2 Infrared. As for Tembotrione technical **790** /TC/(M)/2.2

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 about 5 min. 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).

Repeatability r = g/kg at an active ingredient content of g/kg
Reproducibility R = g/kg at an active ingredient content of g/kg

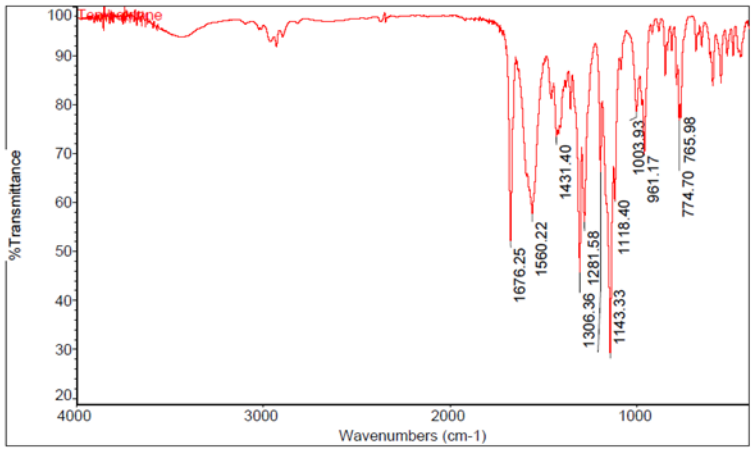


Fig. 1 FTIR spectrum of tembotrione standard

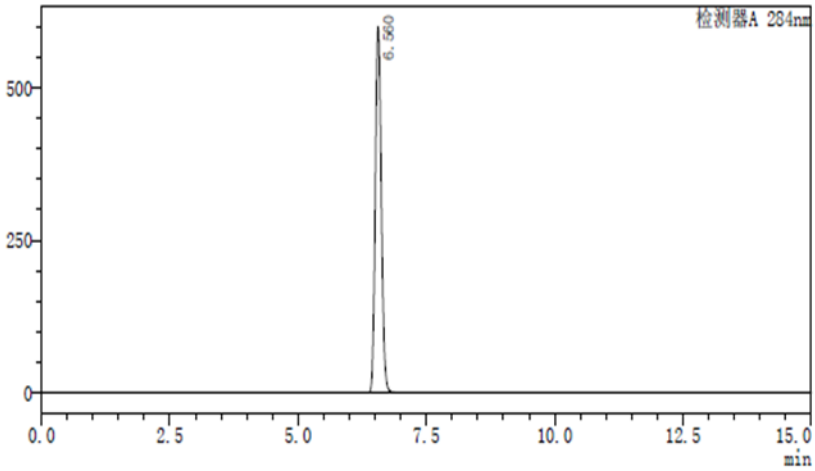


Fig. 2 HPLC Chromatogram of tembotrione standard

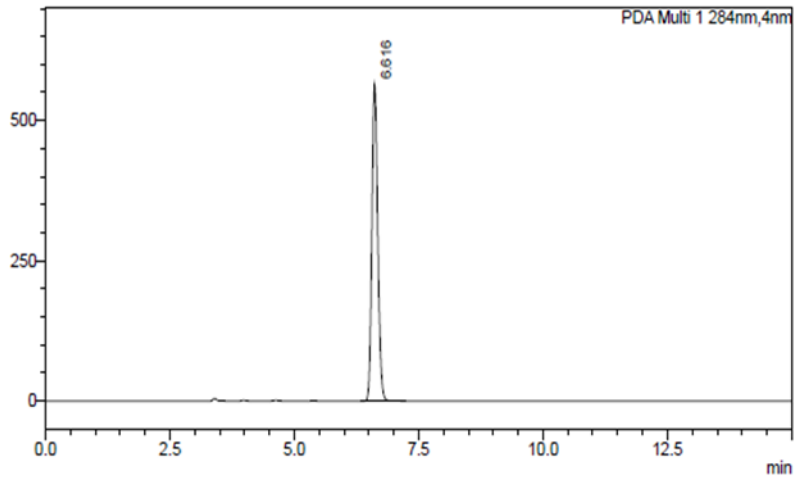


Fig. 3 HPLC Chromatogram of tembotrione TC

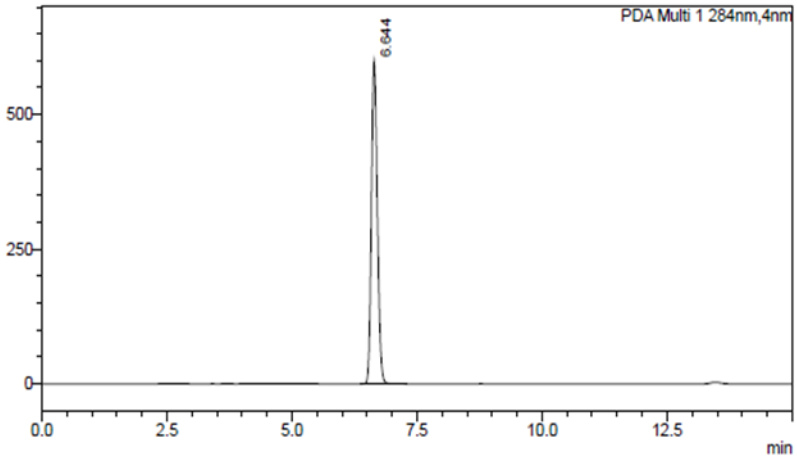


Fig. 4 HPLC Chromatogram of tembotrione SC

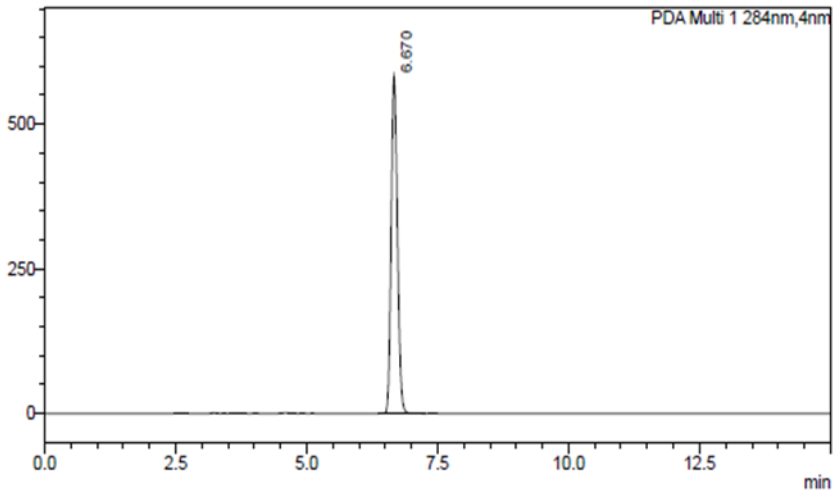


Fig. 5 HPLC Chromatogram of tembotrione OD