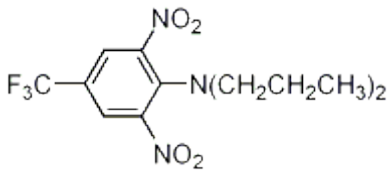
Trifluralin

183



CAS Number: 1582-09-8

IUPAC name: α , α , α -trifluoro-2,6-dinitro-N,N-dipropyl-p-toluidine

Chemical name: 2, 6-dinitro-N, N-dipropyl-4-(trifluoromethyl)benzenamine

Empirical formula: $C_{13}H_{16}F_3N_3O_4$

RMM: 335.3

M.p.: 48.5 – 49℃

Solubility: In water 0.184 (pH 5), 0.221 (pH 7), 0.189 (pH 9) (all in mg/l)

In acetone, chloroform, acetonitrile, toluene, ethyl acetate >1000, methanol 33 – 40, hexane 50 – 67 (all in g/l, 25℃).

Trifluralin Technical

183/TC/M/-

1. Sampling. Take at least 100g

2. Identity tests

2.1 HPLC. Use the reversed phase HPLC method below. The relative retention time of the Trifluralin 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 Trifluralin reference substance. A typical potassium bromide disc should contain a sample prepared in the 0.15-0.35% by weight range. Scan the discs from 4000 to 600 cm-1. The spectrum from the sample should not differ significantly from that of the reference substance.

3. Trifluralin

OUTLINE OF METHOD

Trifluralin is determined by reverse phase HPLC with DAD detector at 280nm and external standardization.

REAGENTS

Trifluralin standard of known purity.

Acetonitrile: HPLC grade；

Water: Ultra-Pure；

Calibration solutions. Weigh in duplicate about 50 mg (to the nearest 0.1 mg) of trifluralin standard (s mg) into separate volumetric flasks (50 ml). Add acetonitrile (about 40ml) and place the flask in an ultrasonic bath for 5 min. Allow to cool to ambient temperature and fill to the mark with acetonitrile. Mix thoroughly (calibration solutions C_A and C_B).

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 280 nm and an injection system capable of injecting 5ul.

Liquid chromatographic column stainless steel, 150 x 4.6 mm (i.d.), packed with C_8, 5 um, Eclipse Zorbax XDB-C8 or equivalent with the same selectivity.

PROCEDURE

(a) HPLC conditions (typical) :

Column: Eclipse Zorbax XDB-C_8 150 x 4.6 mm (i.d.), 5 um

Mobile phase: Acetonitrile: water=77:23(V/V)

Column temperature: 25℃

Injection volume: 5ul

Flow rate: 1.0ml/min

Detector wavelength: 280nm

Run time: 8.00 min

Retention time: approximately 5.2 min

(b) Equilibration of the system

Pump sufficient mobile phase through the column to equilibrate the system. Inject 5 μl portion of calibration solution C_A until the retention time and peak area obtained from two consecutive injections deviate by less than 1.0%.

(c) Preparation of sample

Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample (w mg) to contain about 50mg of trifluralin (s mg) into a volumetric flask (50 ml). Add acetonitrile (about 40ml) and place the flask in an ultrasonic bath for 5 min. Allow to cool to ambient temperature and fill to the mark with acetonitrile. Mix thoroughly (sample solutions S_1 and S_2).

(d) Determination

Inject 5 μl portion of calibration solution C_B. The response factor for this solution should not deviate by more than 1.0% from that for calibration solution C_A, otherwise prepare new calibration solutions. 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, and so on.

(e) Calculation

Determine the peak area of trifluralin and calculate the mean value of response factors from the calibration solutions bracketing the injections of the sample solutions and use this value for calculating the trifluralin content of the bracketed sample solutions. The trifluralin content is the mean value of two sample solutions.

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

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

where:

f_i = individual response factor
f = mean response factor
H_s = peak area of trifluralin in the calibration solution
H_w = peak area of trifluralin in the sample solution
s = mass of trifluralin reference standard in the calibration solution (mg)

TRIFLURALIN EMULSIFIABLE CONCENTRATES

183/EC/M/-

1. Sampling. Take at least 100 ml.

2. Identity test

2.1. HPLC As for Trifluralin technical 221/TC/M2.1

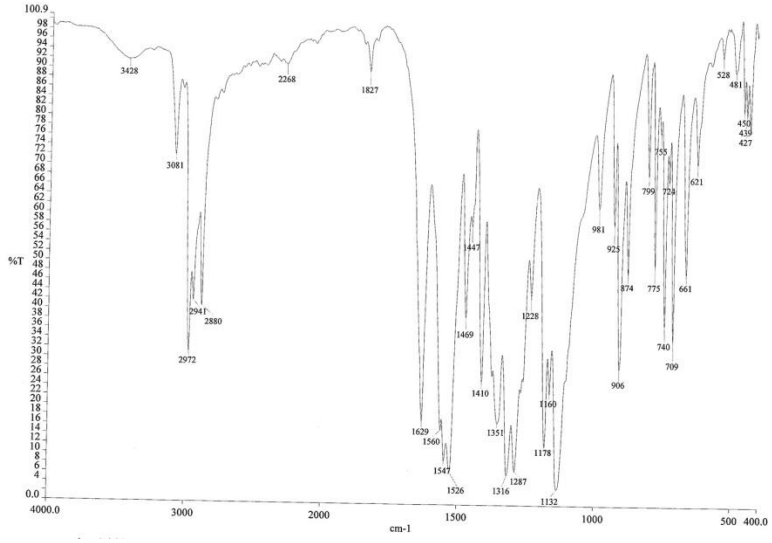
2.2 Infrared As for Trifluralin technical 221/TC/M 2.2

3. Trifluralin

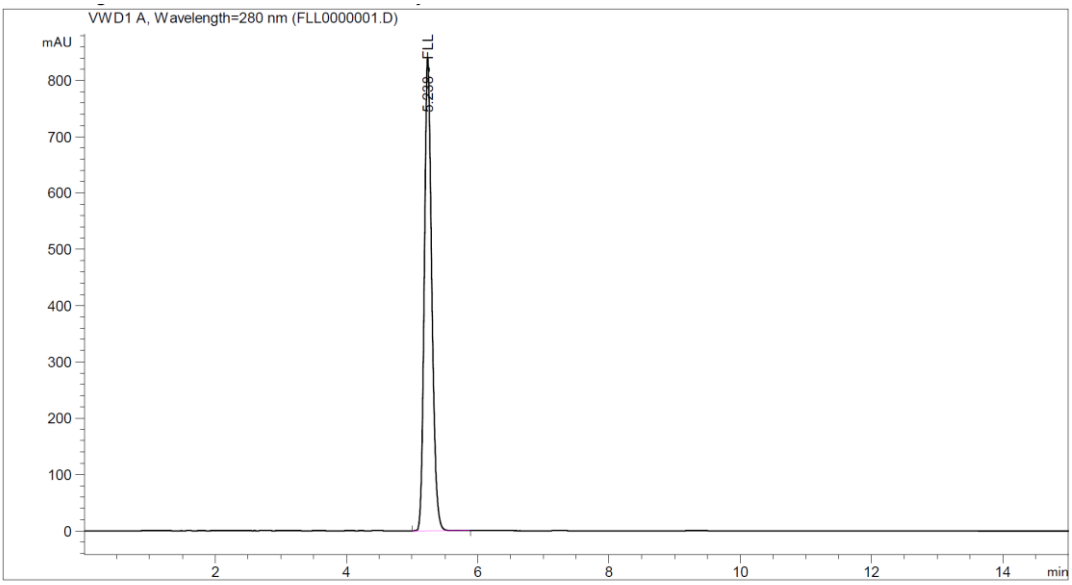
As for Trifluralin technical 221/TC/M except:

(c) Preparation of sample. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample (w mg) to contain about 50 mg of Trifluralin (s mg) into a volumetric flask (50 ml). Add acetonitrile (about 40ml) and place the flask in an ultrasonic bath

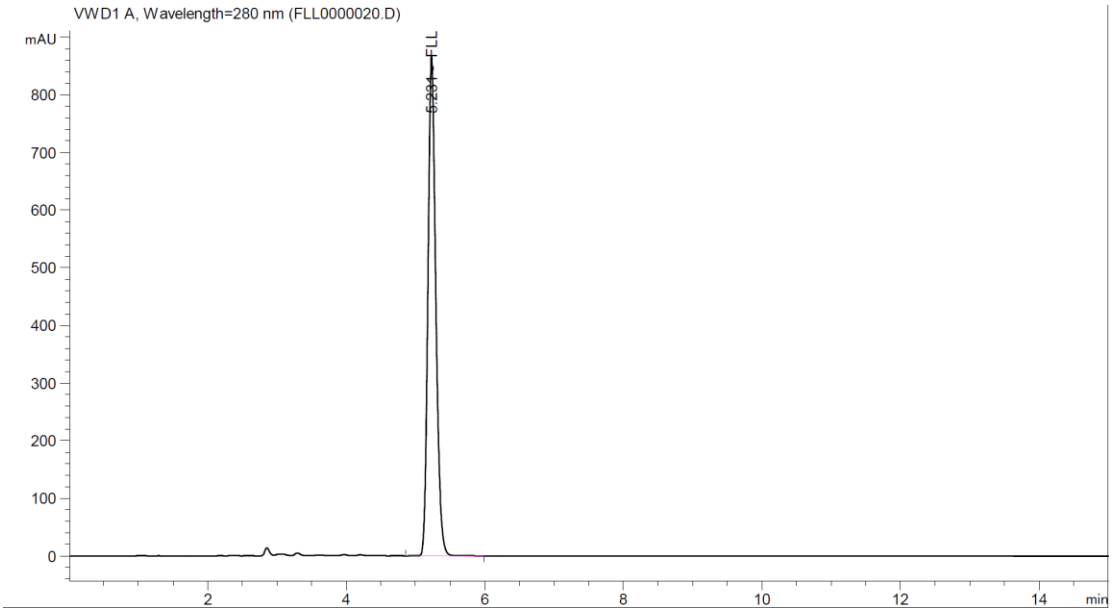
for 5 min. Allow to cool to ambient temperature and fill to the mark with acetonitrile. Mix thoroughly (sample solutions S_1 and S_2).



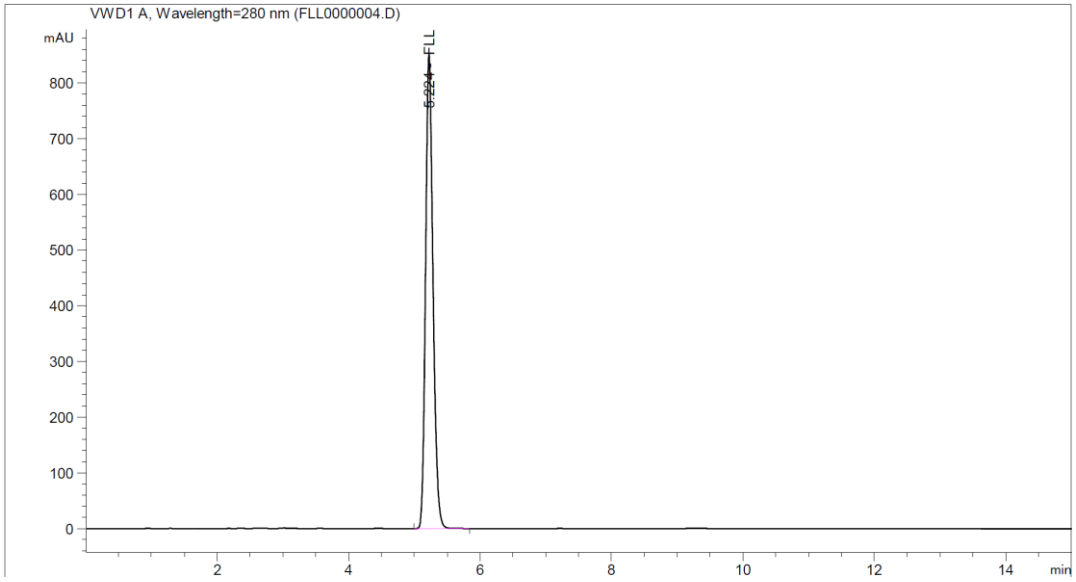
IR spectrum of Trifluralin



Typical HPLC-chromatogram of trifluralin standard



Typical HPLC-chromatogram of trifluralin TC



Typical HPLC-chromatogram of trifluralin EC