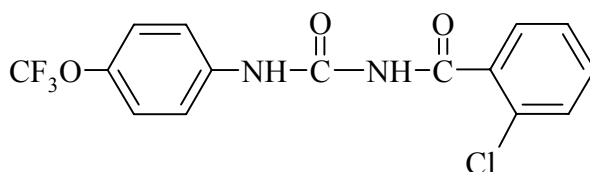


TRIFLUMURON
548



<i>ISO common name</i>	Triflumuron
<i>Chemical name</i>	1-(2-Chlorobenzoyl)-3-(4-trifluoromethoxyphenyl)urea (IUPAC); 2-chloro- <i>N</i> -{[4-(trifluoromethoxy)phenyl]amino} carbonylbenzamide (CA; 64628-44-0)
<i>Empirical formula</i>	C ₁₅ H ₁₀ ClF ₃ N ₂ O ₃
<i>RMM</i>	358.7
<i>m.p.</i>	195 °C
<i>v.p.</i>	4 × 10 ⁻⁹ Pa at 20 °C
<i>Solubility</i>	In water: 0.025 mg/l; dichloromethane: 20 - 50 g/l; propan-2-ol: 1-2 g/l; toluene: 2-5 g/l, all at 20 °C
<i>Description</i>	Colourless powder
<i>Stability</i>	Stable under normal conditions
<i>Formulations</i>	Wettable powders and suspension concentrates

TRIFLUMURON TECHNICAL
*548/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of triflumuron peak of the sample solution should not deviate by more than 2 % from that of the calibration solution. The UV spectrum of this peak should match that of the peak in the calibration solution.

2.2 Infrared. Prepare potassium bromide disks from the sample and triflumuron reference standard using 2 mg sample and 300 mg potassium bromide. Scan the discs from 4000 to 600 cm^{-1} . The spectrum obtained from the sample should not differ significantly from that of the standard (Fig. 32).

3 Triflumuron

OUTLINE OF METHOD Triflumuron is determined by reversed phase high performance liquid chromatography using a UV detection at 250 nm and external standardisation.

REAGENTS

Acetonitrile HPLC grade

Tetrahydrofuran HPLC grade

Water HPLC grade

Triflumuron standard of known purity

Mobile phase water – acetonitrile, 37 + 63 (v/v)

Solvent mixture water – tetrahydrofuran – acetonitrile, 10 + 45 + 45 (v/v)

Calibration solution. Weigh about 80 mg triflumuron standard (to the nearest 0.1 mg) into a volumetric flask (100 ml) (*s* mg). Add solvent mixture (about 80 ml) and place the flask in an ultrasonic bath for 10 min. Allow to cool to room temperature and fill to the mark with solvent mixture (solution C).

APPARATUS

High performance liquid chromatograph equipped with a UV spectrometric detector capable of measuring at 250 nm and a 5 μl injection system

Column stainless steel, 250 \times 4 mm (i.d.), packed with Lichrosphere 100, RP₁₈, 5 μm , or 150 \times 4 mm (i.d.), packed with YMC, ODS-A, 3 μm

* CIPAC method 2000. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany

Electronic integrator

Ultrasonic bath

Centrifuge

Disposable filters e.g. Millipore Millex HV

PROCEDURE

(a) *Chromatographic conditions* (typical):

<i>Mobile phase</i>	water – acetonitrile, 37 + 63 (v/v)
<i>Column temperature</i>	ambient or 40 °C
<i>Flow rate</i>	1.0 ml/min
<i>Detector wavelength</i>	250 nm
<i>Injection volume</i>	5 µl
<i>Run time</i>	approximately 15 min
<i>Retention time</i>	triflumuron: approximately 8 min

(b) *Preparation of sample.* Homogenise the sample. Weigh sufficient sample to contain about 80 mg triflumuron (to the nearest 0.1 mg) into a volumetric flask (100 ml) (*w* mg). Add solvent mixture (about 80 ml) and place the flask in an ultrasonic bath for 10 min. Allow to cool to room temperature and fill to the mark with solvent mixture (solution S).

(c) *System equilibration.* Pump sufficient eluent through the column to equilibrate the system. Inject 5 µl portions of the calibration solution and repeat the injections until retention times and peak areas vary by less than 0.5 % of the mean of three successive injections.

(d) *Determination.* Inject 5 µl portions of the calibration solution and sample solution in the following sequence:

$$C_1, S_1, S_2, C_2 \dots \text{etc.}$$

Determine the peak area of the triflumuron peaks and calculate the mean response factor (*f*) of the calibration solution injections bracketing the injections of the sample solutions.

(e) *Calculation*

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

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

where:

f = mean response factor

H_s = peak area of triflumuron in the calibration solution

H_w = peak area of triflumuron in the sample solution

s = mass of triflumuron in the calibration solution (mg)

w = mass of triflumuron in the sample solution (mg)

P = purity of triflumuron standard (g/kg)

Repeatability r = 15 g/kg at 993 g/kg active ingredient content

Reproducibility R = 15 g/kg at 993 g/kg active ingredient content

TRIFLUMURON WETTABLE POWDERS

*548/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for triflumuron technical 548/TC/M/2.1.

2.2 Infrared. Extract approximately 100 mg of the sample with 2 ml dichloromethane. Filter through a Millex filter and evaporate the solvent with a gentle stream of clean dry air. Continue as for 548/TC/M/2.2.

3 Triflumuron. As for triflumuron technical 548/TC/M/3, except:

(b) *Preparation of sample.* Weigh sufficient sample to contain about 80 mg triflumuron (to the nearest 0.1 mg) into a volumetric flask (100 ml) (w mg). Add solvent mixture (about 80 ml) and place the flask in an ultrasonic bath for 10 min. Shake the flask several times, allow to cool to room temperature and fill to the mark with solvent mixture (solution S). If necessary, centrifuge or filter the solution to obtain a clear solution.

Repeatability r = 15 g/kg at 409 g/kg active ingredient content

Reproducibility R = 20 g/kg at 409 g/kg active ingredient content

Repeatability r = 10 g/kg at 400g/kg active ingredient content

Reproducibility R = 15 g/kg at 400 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for 548/TC/M/3 and MT 15.

* CIPAC method 2000. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany

PROCEDURE

(a) *Preparation of suspension.* MT 15.1(i).

(b) *Determination of sedimentation.* MT 15.1(ii).

(c) *Determination of triflumuron in the bottom 25 ml of suspension.* After removal of the top 225 ml of suspension transfer the remaining 25 ml of the suspension to a volumetric flask (100 ml) and fill up to the mark with solvent mixture (water/acetonitrile/tetrahydrofuran). Homogenise and determine the mass of triflumuron (Q g) by 548/TC/M/3.

(d) *Calculation*

$$Q = \frac{H_w \times f}{1000000}$$

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

where:

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

Q = mass of triflumuron in the bottom 25 ml of suspension (g)

TRIFLUMURON SUSPENSION CONCENTRATES

*548/SC/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for triflumuron technical 548/TC/M/2.1.

2.2 Infrared. As for triflumuron wettable powders 548/WP/M/2.2.

3 Triflumuron. As for triflumuron technical 548/TC/M/3, except:

(b) *Preparation of sample.* Homogenise the sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) sufficient sample to contain about 80 mg triflumuron (w mg). Add water (about 10 ml) and suspend thoroughly. Then add solvent mixture (about 80 ml) and homogenise. Place the flask in an ultrasonic

* CIPAC method 2000. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany

bath for 10 min. Shake the flask several times, allow to cool to room temperature and fill to the mark with solvent mixture (solution S). If necessary, centrifuge or filter the solution to obtain a clear solution.

Repeatability r = 6.2 to 8.6 g/kg at 251 g/kg active ingredient content
Reproducibility R = 8.5 to 11.3 g/kg at 251 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for **548/TC/M/3** and MT 161.

PROCEDURE

(a) Preparation of suspension and determination of sedimentation. MT 161.

(b) Determination of triflumuron in the bottom 25 ml of suspension. As for **548/WP/M/4** (c).

(c) Calculation

As for **548/WP/M/4** (d).

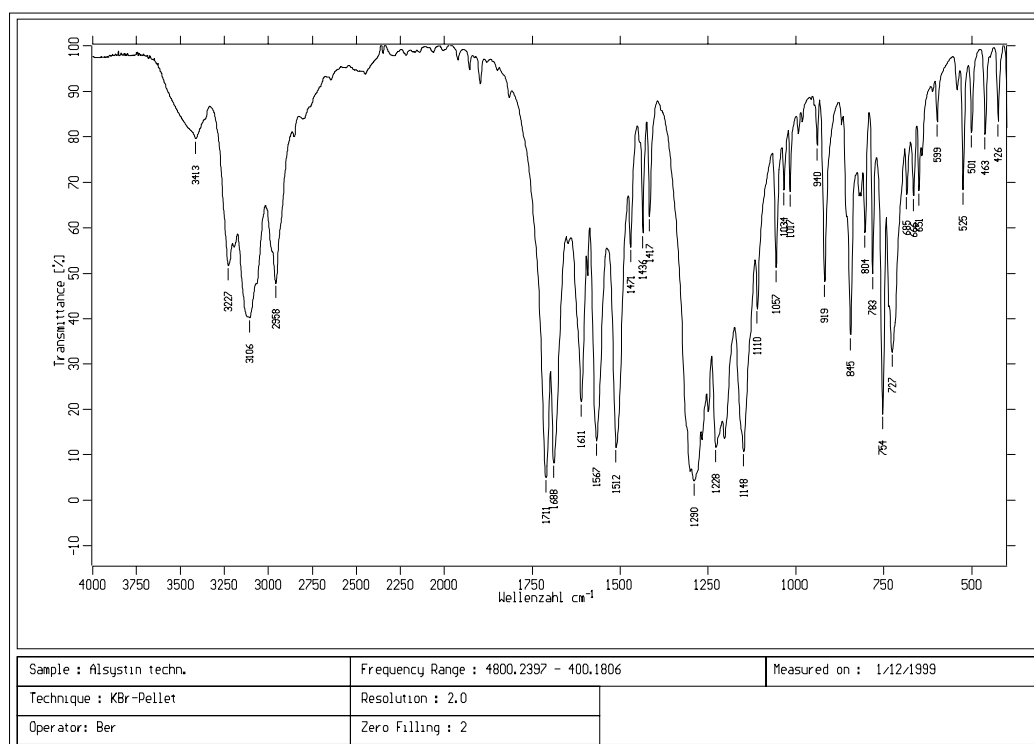


Fig. 32 IR Spectrum of Triflumuron