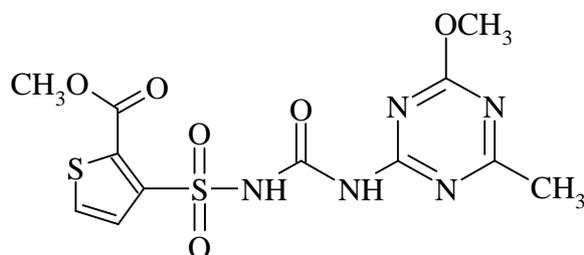


THIFENSULFURON-METHYL
452



<i>ISO common name</i>	Thifensulfuron-methyl
<i>Chemical name</i>	Methyl 3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl-carbamoylsulphamoyl)thiophen-2-carboxylate (IUPAC); methyl 3-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)-amino]carbonyl]amino]-sulphonyl]-2-thiophenecarboxylate (CA; 79277-27-3)
<i>Empirical formula</i>	C ₁₂ H ₁₃ N ₅ O ₆ S ₂
<i>RMM</i>	387.4
<i>m.p.</i>	176 °C
<i>v.p.</i>	7.5 × 10 ⁻⁹ Pa at 20 °C
<i>Solubility</i>	In water: 0.23 g/l (pH 5), 6.27 g/l (pH 7) at 25 °C; acetone: 11.9 g/l; acetonitrile: 7.9 g/l; dichloromethane: 27.5 g/l; ethanol: 0.9 g/l; hexane: < 0.9 g/l; xylene: 0.2 g/l, all at 20 °C
<i>Description</i>	Off-white crystalline solid
<i>Stability</i>	Stable at 55 °C; DT ₅₀ : 4.7 h (pH 3), 38 h (pH 5), 250 h (pH 7), 11 h (pH 9) at 45 °C
<i>Formulations</i>	Water dispersible powders

THIFENSULFURON-METHYL TECHNICAL
*452/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of thifensulfuron-methyl 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 obtained from the calibration substance.

2.2 Infrared. Prepare potassium bromide discs from the sample and the standard thifensulfuron-methyl. Scan the discs from 4000 to 400 cm^{-1} . The spectrum obtained from the sample should not differ significantly from that of the standard.

3 Thifensulfuron-methyl

OUTLINE OF METHOD Thifensulfuron-methyl is determined by reversed phase high performance liquid chromatography using a C_{18} column, UV detection at 280 nm and external standardisation. The content of active ingredient is quantified using a calibration curve.

REAGENTS

Acetonitrile HPLC grade

Water HPLC grade

Thifensulfuron-methyl standard of known purity

Phosphoric acid 85 %, HPLC grade

Mobile phase. acetonitrile – water (pH 2.5), 38 + 62 (v/v). Add to a bottle (1 l) water (620 ml) adjusted to pH 2.5 with phosphoric acid using a calibrated pH meter. Add acetonitrile (380 ml) and mix. Mixing the mobile phase may also be accomplished using a binary solvent HPLC pump. Degas and filter prior to use.

Calibration solution. Weigh (to the nearest 0.1 mg) into separate volumetric flasks (100 ml) 85, 105 and 125 mg (± 5 mg) thifensulfuron-methyl. Add to each flask acetonitrile (about 90 ml) and place the flasks in an ultrasonic bath for 15 min. Remove the flasks from the ultrasonic bath and allow to cool to room temperature for about one hour. Dilute to volume with acetonitrile and mix well. Filter a portion of each standard solution through a 0.2 μm filter prior to analysis.

* CIPAC method 2001. Prepared by a panel chaired by S W Hansen. Based on a method supplied by DuPont de Nemours, USA.

APPARATUS

High performance liquid chromatograph equipped with a constant-flow pump, constant temperature column compartment, a sample injector capable of injecting 5 µl aliquots, a UV spectrometric detector (280 nm) and a digital integrator or other data-handling capability

Column, 150 × 4.6 mm (i.d.), packed with Zorbax SB-C₁₈, particle size 3.5 µm with in-line filter with replaceable frit. Substitution of alternative columns should be accompanied by demonstrated equivalency and/or method validation. The filter frit should be Upchurch Scientific, Inc. Model A-102X, A-318 or equivalent (0.5 µm frit)

Ultrasonic bath

pH meter

Filtering apparatus (for sample and standard solutions) disposable plastic 3 ml syringes fitted with 0.2 µm Acrodisc-CR (PFTE) filters or equivalent

PROCEDURE

(a) *Chromatographic conditions* (typical):

<i>Column</i>	150 × 4.6 mm (i.d.), packed with Zorbax SB-C ₁₈ , particle size 3.5 µm
<i>Mobile phase</i>	acetonitrile – water (pH 2.5), 38 + 62 (v/v)
<i>Column temperature</i>	40 °C
<i>Flow rate</i>	1.0 ml/min
<i>Injection volume</i>	5 µl
<i>Detector wavelength</i>	280 nm (band width 4 nm)
<i>Reference wavelength</i>	450 nm (band width 100 nm)
<i>Run time</i>	approximately 11 min
<i>Retention time</i>	thifensulfuron-methyl: approximately 4.4 min

(b) *Preparation of sample*. Grind or mill the sample prior to weighing. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) sufficient sample to contain 105 ± 5 mg thifensulfuron-methyl (*w* mg). Add acetonitrile (about 90 ml), mix well and place the flask in an ultrasonic bath for 15 min. Remove the flask from the ultrasonic bath and allow to cool to room temperature for about one hour. Dilute to volume with acetonitrile and mix well. Filter a portion of each standard solution through a 0.2 µm filter prior to analysis.

(c) *Determination*. Equilibrate the column by pumping the mobile phase through the column until a stable baseline is obtained. Inject in duplicate 5 µl each, of a

solvent blank (acetonitrile), standards and samples bracketing the samples with calibration solutions.

(d) *Preparation of a calibration curve.* Prepare a calibration curve for thifensulfuron-methyl by plotting peak areas versus the mass of the standards (mg). Using the method of least-squares, calculate the equation for the straight line that best fits the experimental data. The correlation coefficient should be 0.999 or better. If not, repeat the calibration.

(d) *Calculation.* Determine the concentration of thifensulfuron-methyl for each sample injection.

$$\text{Thifensulfuron-methyl content} = \frac{(R - b) \times P}{a \times w} \text{ g/kg}$$

where:

R = peak area of thifensulfuron-methyl in the sample solutions

a = slope of calibration curve

b = intercept of calibration curve

P = purity of the thifensulfuron-methyl standard (g/kg)

w = mass of the sample taken (mg)

Repeatability r = 14 g/kg at 983 g/kg active ingredient content

Reproducibility R = 19 g/kg at 983 g/kg active ingredient content

THIFENSULFURON-METHYL WATER DISPERSIBLE GRANULES *452/WG/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for thifensulfuron-methyl technical 452/TC/M/2.1.

2.2 Infrared. Mix sufficient sample to contain about 10 mg of thifensulfuron-methyl with acetonitrile (5 to 10 ml). Mix well and filter. Evaporate the filtrate to dryness and proceed as for thifensulfuron-methyl technical 452/TC/M/2.2.

* CIPAC method 2001. Prepared by a panel chaired by S W Hansen. Based on a method supplied by DuPont de Nemours, USA.

3 Thifensulfuron-methyl. As for thifensulfuron-methyl technical **452/TC/M/3**.

Repeatability r = 14 to 17 g/kg at 751 g/kg active ingredient content

Reproducibility R = 23 to 27 g/kg at 751 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for **452/TC/M/3** and MT 168 except:

Calibration solutions. As for **452/TC/M/3**, *Calibration solutions*, but use other concentrations if needed.

PROCEDURE

(a) *Preparation of suspension and determination of sedimentation.* MT 168.

(b) *Determination of thifensulfuron-methyl in the bottom 25 ml of suspension.* After removal of the top 225 ml of suspension add to the remaining 25 ml acetonitrile (75 ml). Place the cylinder in an ultrasonic bath for 5 min. Allow to cool to room temperature, mix well by inverting the cylinder several times. Take a suitable aliquot of the solution and determine the mass of thifensulfuron-methyl (Q g) by **452/TC/M/3**.

(c) *Calculation*

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

where:

c = mass of thifensulfuron-methyl in the sample taken for the preparation of the suspension (g)

Q = mass of thifensulfuron-methyl in the bottom 25 ml of suspension (g)

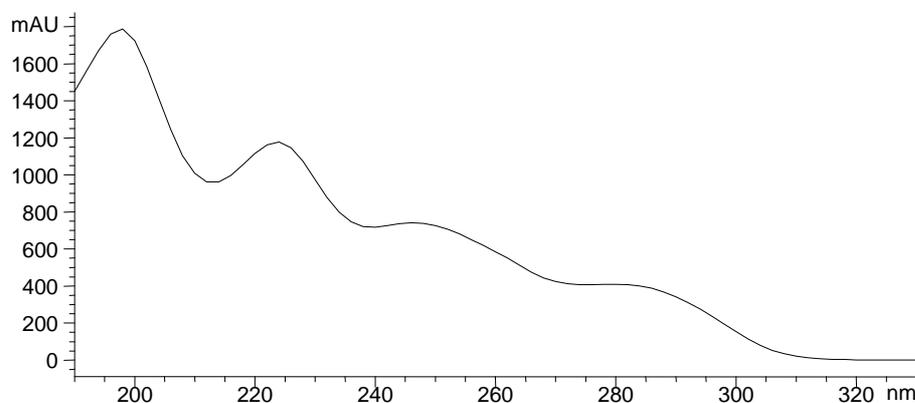


Fig. 39 UV spectrum of thifensulfuron-methyl standard

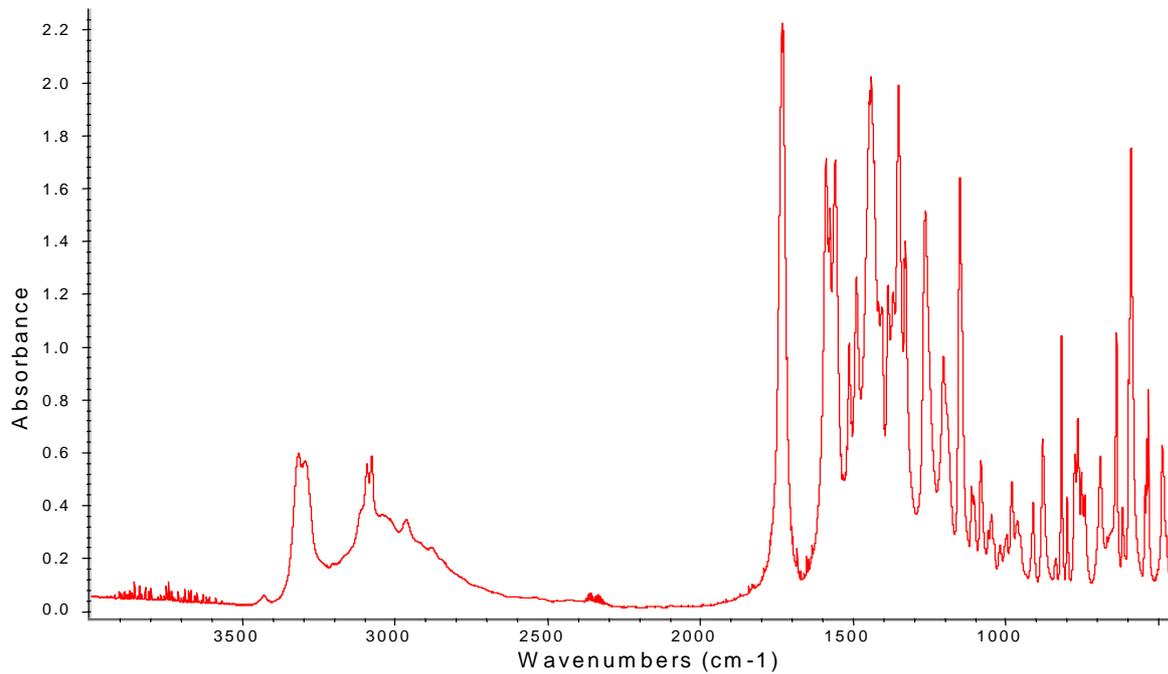


Fig. 40 IR spectrum of thifensulfuron-methyl standard

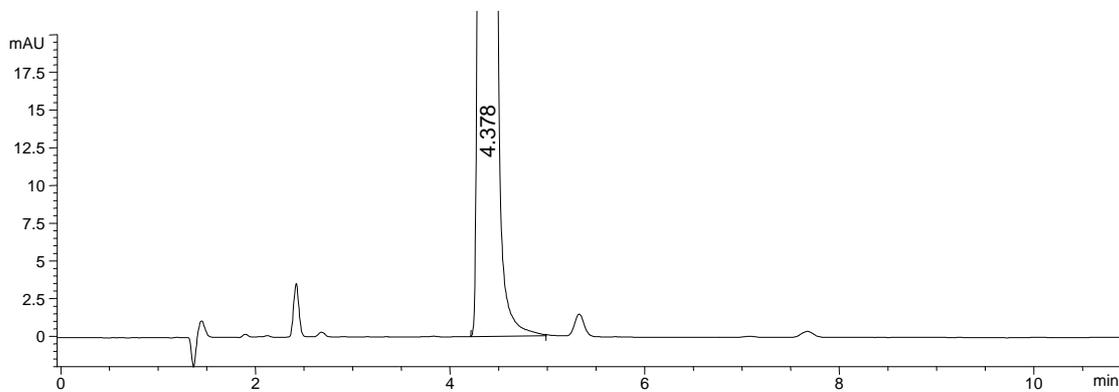


Fig. 41 Chromatogram of thifensulfuron-methyl technical

THIFENSULFURON-METHYL 452

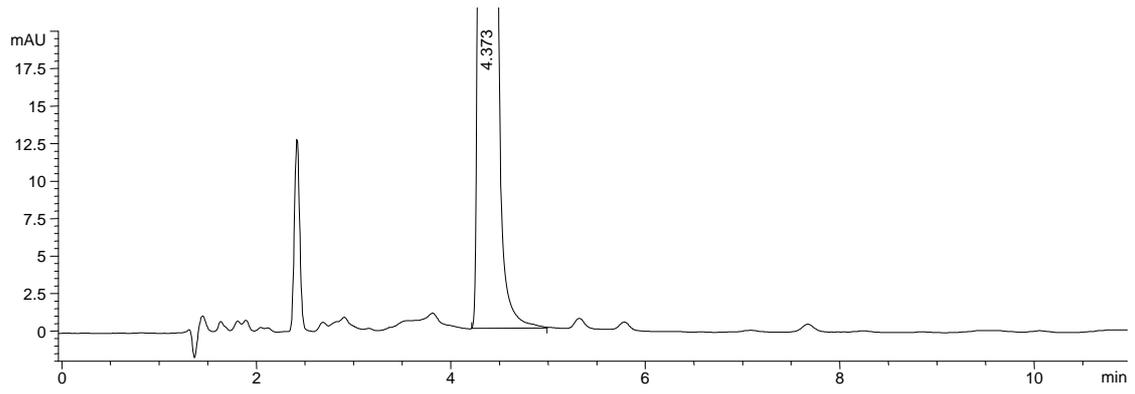


Fig. 42 Chromatogram of thifensulfuron-methyl wettable granules