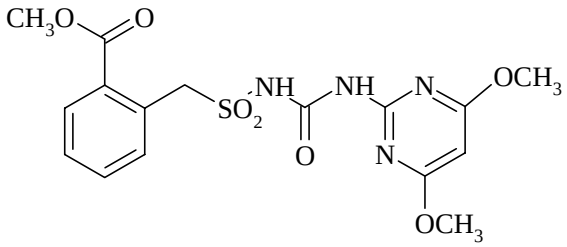
BENSULFURON-METHYL
502



ISO common name	Bensulfuron-methyl
Chemical name	Methyl 2(4,6-dimethoxypyrimidin-2-yl-3-ureido-sulphonylmethyl)benzoate (IUPAC); methyl 2-[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-amino] sulphonyl]-methyl]benzoate (CA; *83055-99-6)*
Empirical formula	$C_{16}H_{18}N_4O_7S$
RMM	410.4
m.p.	185-188 °C
v.p.	2.8×10^{-6} Pa at 25 °C
Solubility	In water: 2.9 mg/l (pH 5), 12 mg/l (pH 6), 120 mg/l (pH 7), 1.2 g/l (pH 8), all at 25 °C; acetone: 1.38 g/l; acetonitrile: 5.38 g/l; dichloromethane: 11.7 g/l; ethyl acetate: 1.66 g/l; hexane: 0.31 mg/l; xylene: 0.28 g/l, all at 20 °C
Description	White powder
Stability	DT_{50}: 11 d (pH 5), 143 d (pH 7) at 25 °C
Formulations	Water dispersible granules and wettable powders

BENSULFURON-METHYL TECHNICAL
*502/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of bensulfuron-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 bensulfuron-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 Bensulfuron-methyl

OUTLINE OF METHOD Bensulfuron-methyl is determined by reversed phase high performance liquid chromatography using a C_8 column, UV detection at 236 nm and external standardisation. The content of active ingredient is quantified using a calibration curve.

REAGENTS

Acetonitrile HPLC grade
Water HPLC grade
Bensulfuron-methyl standard of known purity
Phosphoric acid 85 %, HPLC grade
Mobile phase. Adjust water (550 ml) to pH 2.7 with phosphoric acid using a pH meter standardised at pH 4.0 and pH 2.0. Add acetonitrile (450 ml) and mix. Mixing the mobile phase may also be accomplished using a binary solvent HPLC pump. Degas and filter before use.
Calibration solution. Weigh (to the nearest 0.1 mg) into separate volumetric flasks (50 ml) 30, 40, 50 and 65 mg (± 5 mg) bensulfuron-methyl. Add to each flask acetonitrile (about 40 ml) and place the flasks in an ultrasonic bath for 10 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 Dr 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 (236 nm) and a digital integrator or other data-handling capability

Column, 150 × 4.6 mm (i.d.), packed with Zorbax SB-C_8, particle size 3.5 µm 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

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

pH meter

PROCEDURE

(a) Chromatographic conditions (typical):

Column	150 × 4.6 mm (i.d.), packed with Zorbax SB-C_8, particle size 3.5 µm
Mobile phase	acetonitrile –water (pH 2.7), 45 + 55 (v/v)
Column temperature	40 °C
Flow rate	1.0 ml/min
Injection volume	5 µl
Detector wavelength	236 nm (band width 4 nm)
Reference wavelength	406 nm (band width 80 nm)
Run time	approximately 12 min
Retention time	bensulfuron-methyl: approximately 6.6 min

(b) Preparation of sample. Grind or mill the sample prior to weighing. Weigh (to the nearest 0.1 mg) into a volumetric flask (50 ml) sufficient sample to contain 50 ± 5 mg bensulfuron-methyl (*w* mg). Add acetonitrile (about 40 ml), mix well and place the flask in an ultrasonic bath for 10 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 bensulfuron-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 bensulfuron-methyl for each sample injection.

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

where:
R = peak area of bensulfuron-methyl in the sample solutions
a = slope of calibration curve
b = intercept of calibration curve
P = purity of the bensulfuron-methyl standard (g/kg)
w = mass of the sample taken (mg)

Repeatability r = 14 g/kg at 986 g/kg active ingredient content
Reproducibility R = 19 g/kg at 986 g/kg active ingredient content

BENSULFURON-METHYL WETTABLE POWDERS
[*]**502**/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests
2.1 HPLC. As for bensulfuron-methyl technical **502**/TC/M/2.1.
2.2 Infrared. Mix sufficient sample to contain about 10 mg of bensulfuron-methyl with acetonitrile (5 to 10 ml). Mix well and filter. Evaporate the filtrate to dryness and proceed as for bensulfuron-methyl technical **502**/TC/M/2.2.

[*] CIPAC method 2002. Prepared by a panel chaired by Dr S W Hansen. Based on a method supplied by DuPont de Nemours, USA

3 Bensulfuron-methyl. As for bensulfuron-methyl technical **502**/TC/M/3, except:

Repeatability r = 11 g/kg at 610 to 594 g/kg active ingredient content
Reproducibility R = 27 g/kg at 610 to 594 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for **502**/TC/M/3and MT 15 except:
Calibration solutions. As for **502**/TC/M/3, *Calibration solutions*, but use other concentrations if needed.

PROCEDURE

(a) Preparation of suspension. M T 15.1 *(i)*.

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

(c) Determination of bensulfuron-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 bensulfuron-methyl (*Q* g) by **502**/TC/M/3.

(d) Calculation

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

where:
c = mass of bensulfuron-methyl in the sample taken for the preparation of the suspension (g)
Q = mass of bensulfuron-methyl in the bottom 25 ml of suspension (g)

BENSULFURON-METHYL WATER DISPERSIBLE GRANULES
[*]**502**/WG/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for bensulfuron-methyl technical **502**/TC/M/2.1.

2.2 Infrared. Mix sufficient sample to contain about 10 mg of bensulfuron-methyl with acetonitrile (5 to 10 ml). Mix well and filter. Evaporate the filtrate to dryness and proceed as for bensulfuron-methyl technical **502**/TC/M/2.2.

3 Bensulfuron-methyl. As for bensulfuron-methyl technical **502**/TC/M/3.

Repeatability r = 11 g/kg at 610 to 594 g/kg active ingredient content
Reproducibility R = 27 g/kg at 610 to 594 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for and MT 168 except:
Calibration solutions. As for **502**/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 bensulfuron-methyl in the bottom 25 ml of suspension. As for **502**/WP/M/*4(c).*

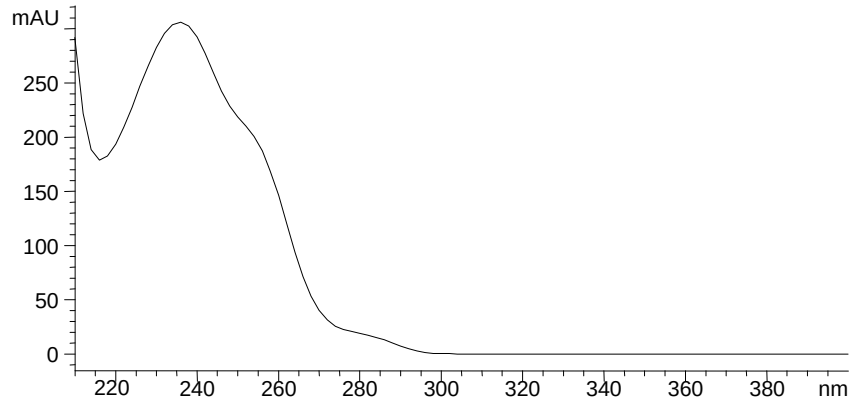


Fig. 1 UV spectrum of bensulfuron-methyl standard

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

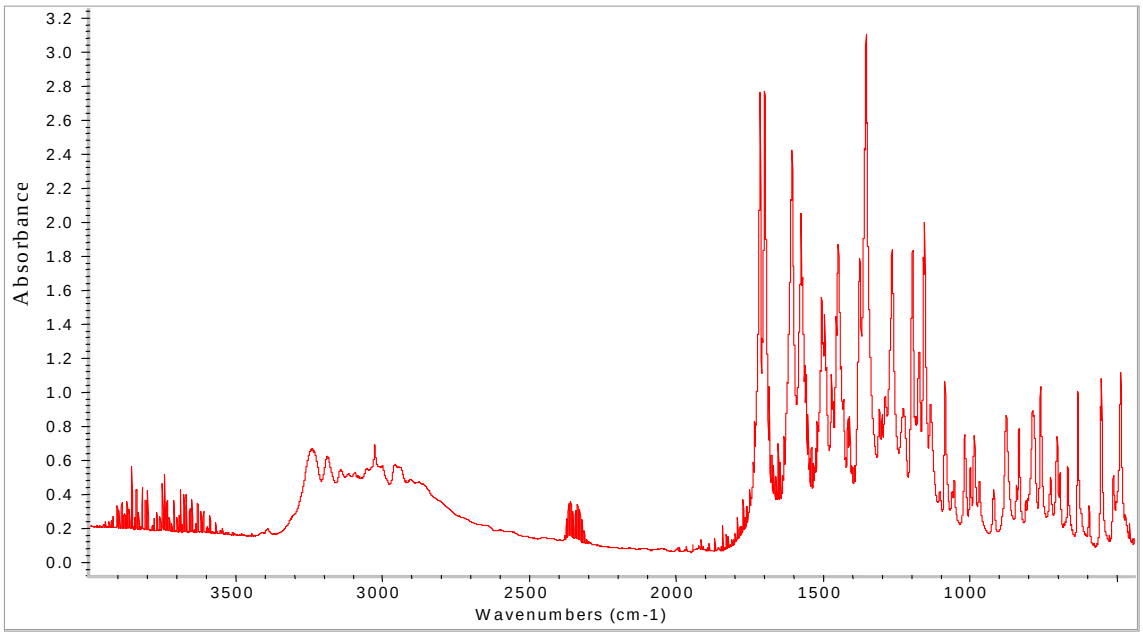


Fig. 2 IR spectrum of bensulfuron-methyl standard

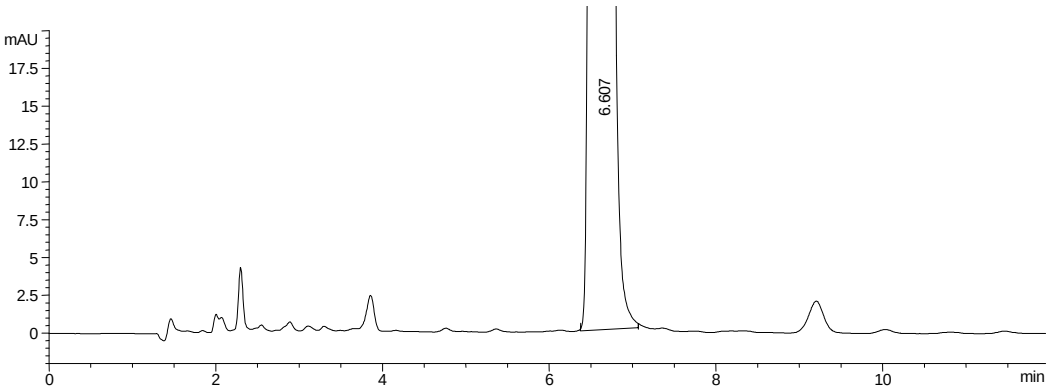


Fig. 3 Chromatogram of bensulfuron-methyl technical

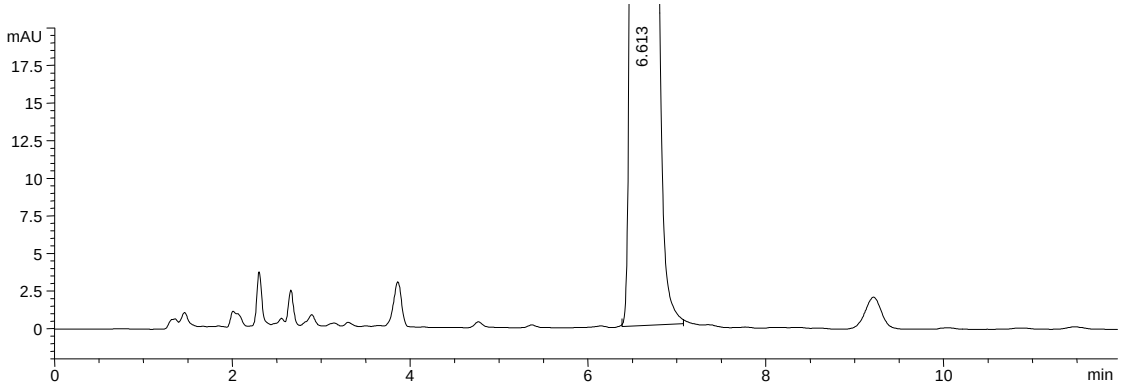


Fig. 4 Chromatogram of bensulfuron-methyl WG