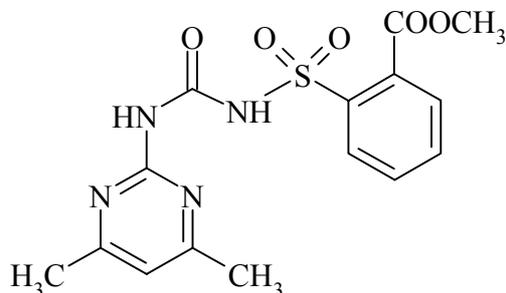# SULFOMETURON-METHYL
610

ISO common name	Sulfometuron-methyl
Chemical name	Methyl 2-(4,6-dimethylpyrimidin-2-yl-carbamoyl-sulfamoyl)-benzoate (IUPAC); methyl 2-[[[[(4,6-dimethyl-2-pyrimidinyl)amino]carbonyl]amino]-sulfonyl]benzoate (CA; *74222-97-2*)
Empirical formula	$C_{15}H_{16}N_4O_5S$
RMM	364.4
m.p	203-205 °C
v.p.	7.73×10^{-14} Pa at 25 °C
Solubility	In water: 8 mg/l (pH 5) and 70 mg/l (pH7); acetone: 2.4 g/kg; acetonitrile: 1.5 g/kg; diethyl ether: 32 mg/kg; ethanol: 137 mg/kg; xylene: 37 mg/kg (all at 25 °C)
Description	White powder
Stability	Stable to hydrolysis at pH 7 to pH 9
Formulations	Water dispersible granules

SULFOMETURON-METHYL TECHNICAL
[*]610/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of sulfometuron-methyl for the sample solution should not deviate by more than 5 % from that for the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from sulfometuron-methyl standard using 1 to 2 mg material and 200 mg potassium bromide. Scan the discs from 400-4000 cm^{-1}. The absorbance maximum should be between 0.6 and 1.0 absorbance units. If the absorbance is outside this range, adjust the mixture accordingly. The spectrum from the sample should not differ significantly from that of the standard (Fig. 29).

3 Sulfometuron-methyl

OUTLINE OF METHOD Sulfometuron-methyl is determined by high performance liquid chromatography on a reversed phase column (C$_8$) using water at pH 3 - acetonitrile 60 + 40 (v/v) as eluent, UV detection at 234 nm, and internal standardisation (benzanilide). The content of active ingredient is quantified using a calibration curve.

REAGENTS

Acetonitrile HPLC grade
Water HPLC grade
Sulfometuron-methyl standard of known purity
Benzanilide internal standard
Phosphoric acid 85 %, HPLC grade
Mobile phase Adjust water (600 ml) to pH 3.0 with phosphoric acid using a pH meter standardised at pH 7.0 and pH 2.0. Add acetonitrile (400 ml) and mix. Mixing the mobile phase may also be accomplished using a binary solvent HPLC pump. Degas before use.
Internal standard solution. Weigh benzanilide (5.0 g) into a bottle (1 l) and add acetonitrile (500 ml). Place the bottle in an ultrasonic bath until the benzanilide has dissolved (about 10 min).

Calibration solution. Weigh (to the nearest 0.1 mg) 65, 75 and 85 mg (± 2 mg) of sulfometuron-methyl standard into three separate bottles (150 ml). Tare and cap the bottles. Uncap and add internal standard solution (10.0 ml) to each bottle. Recap each bottle and determine (to the nearest 0.1 mg) the mass of internal standard solution added. Take care to minimise solvent evaporation during addition and weighing of the internal standard solution. Add acetonitrile (90 ml) to each bottle, place the bottles in an ultrasonic bath for 15 min. and mix well (Label as solutions C_1, C_2, and C_3 respectively). Filter a portion of each calibration solution through a 0.2 μm filter prior to analysis.

Note: Due to the low solubility of sulfometuron-methyl, sample dissolution is critical to this method. No more than four samples should be sonicated at any one time. If results are erratic or excessively poor calibration curve linearity is observed, poor dissolution is a likely source of error.

APPARATUS

High performance liquid chromatograph equipped with a constant-temperature column compartment, a 5 μl loop injection valve, and a UV spectrophotometric detector capable of operating at 234 nm

Column stainless steel, 150 mm × 4.6 mm (i.d.) packed with YMC ODS-AQ (5 μm), with in-line filter with replaceable 0.5 μm frit (Upchurch Scientific Inc model A-102X, A-318, or equivalent). Substitution of alternate columns must be accompanied by demonstrated equivalency and/or method revalidation.

Filtering apparatus disposable plastic syringes (3 ml) fitted with 0.2 μm filter Acrodisc-CR or equivalent

Integrator or *electronic* data *system*

Ultrasonic bath

PROCEDURE

(a) Operating conditions (typical):

Column	YMC ODS-AQ, 150 × 4.6 mm
Mobile phase	water at pH 3 - acetonitrile 60 + 40 (v/v)
Eluent flow rate	1.5 ml/min
Column temperature	40 °C
Injection volume	5 μl
Detection wavelength	234 nm (bandwidth 4 nm)
Reference wavelength	350 nm (bandwidth 80 nm)
Retention time	sulfometuron-methyl: about 4.7 min
	benzanilide: about 6.9 min
Run time	10 min

(b) Sample preparation. Mill or grind all samples prior to weighing. Weigh (to the nearest 0.1 mg) sufficient sample to contain 75 ± 5 mg of sulfometuron-methyl (*w* mg) into a bottle (150 ml). Cap and tare the bottle. Uncap and add internal standard solution (10.0 ml) to the bottle. Recap the bottle and determine (to the nearest 0.1 mg) the mass of internal standard solution added. Take care to minimise solvent evaporation during addition and weighing of the internal standard solution. Add acetonitrile (90 ml) to the bottle, place the bottles in an ultrasonic bath for 15 min and mix. Filter a portion of the sample solution through a 0.2 μm filter prior to analysis.

Note: Due to the low solubility of sulfometuron-methyl, sample dissolution is critical to this method. No more than four samples should be sonicated at anyone time. If results are erratic, poor dissolution is a likely source of error.

(c) Determination. Equilibrate the column by pumping the mobile phase through the column until a stable baseline has been obtained. Make duplicate injections of 5 μl of each calibration and sample solution. Peak response must be within the linear range of the detector. If necessary, perform additional dilutions after the addition of the internal standard to obtain appropriate detector response.

Calculate the sulfometuron-methyl to benzanilide peak area ratio for each injection of the calibration and sample solutions. Prepare a calibration curve by plotting the average peak area ratio for each calibration solution (Solutions C_1, C_2, and C_3) versus the corresponding average sulfometuron-methyl to benzanilide mass ratio. Using the least-squares method calculate the line that best fits the experimental data. The correlation coefficient should be 0.999 or better. If not, repeat the calibration.

(d) Calculation

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

where:
  R = sulfometuron-methyl to benzanilide area ratio of the sample solutions
  a = slope of calibration curve
  b = intercept of calibration curve
  P = purity of the sulfometuron-methyl standard (g/kg)
  w = sulfometuron-methyl to benzanilide mass ratio of the sample

Repeatability r   =   13 g/kg at 991 to 1000 g/kg active ingredient content
Reproducibility R =   19 g/kg at 991 to 1000 g/kg active ingredient content

SULFOMETURON-METHYL WATER DISPERSIBLE GRANULES
[*]**610**/WG/M/-

1 Sampling. Take at least 500 g.

2 Identity tests.
2.1 HPLC. As for **610**/TC/M/2.1.
2.2 Infrared. Extract the sample with acetonitrile, filter and evaporate the solvent in a stream of clean dry air. Continue as for **610**/TC/M/2.2.

3 Sulfometuron-methyl. As for sulfometuron-methyl **610**/TC/M/3.

Repeatability r = 16 g/kg at 747 g/kg active ingredient content
Reproducibility R = 18 g/kg at 747 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for **610**/TC/M/3 and MT 168.

PROCEDURE

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

(b) Determination of sulfometuron-methyl in the bottom 25 ml of suspension.
After removal of the top 225 ml of suspension transfer the 25 ml remaining quantitatively to a 100 ml volumetric flask and dilute to volume with acetonitrile. Place the volumetric flask in an ultrasonic bath for 15 min. Allow to cool to room temperature and take a suitable aliquot of the solution for the determination of sulfometuron-methyl. Proceed as for **610**/TC/M/3.
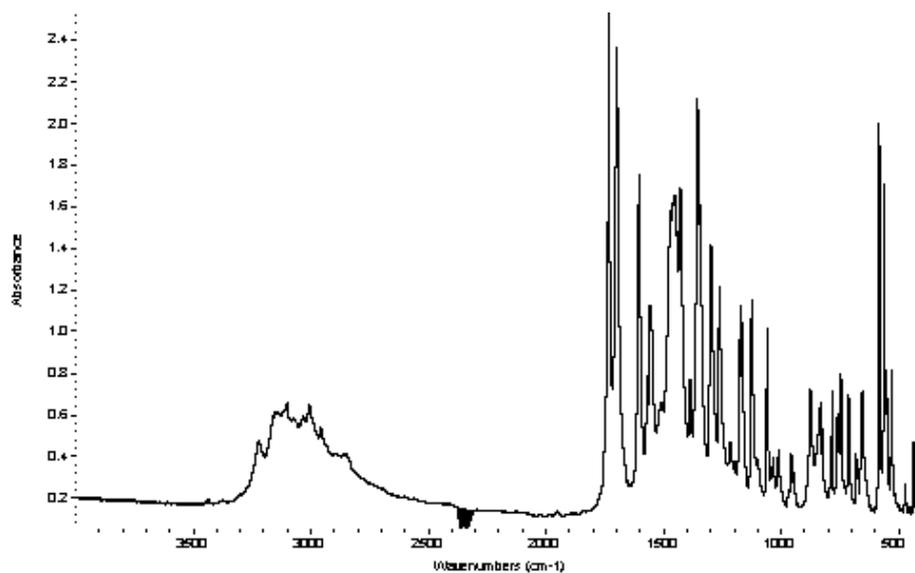
(d) Calculation

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

where:
c = mass of active ingredient in sample actually taken
Q = mass of active ingredient in the 25 ml remaining in the cylinder

Reference IR Spectrum of Sulfometuron-methyl Standard

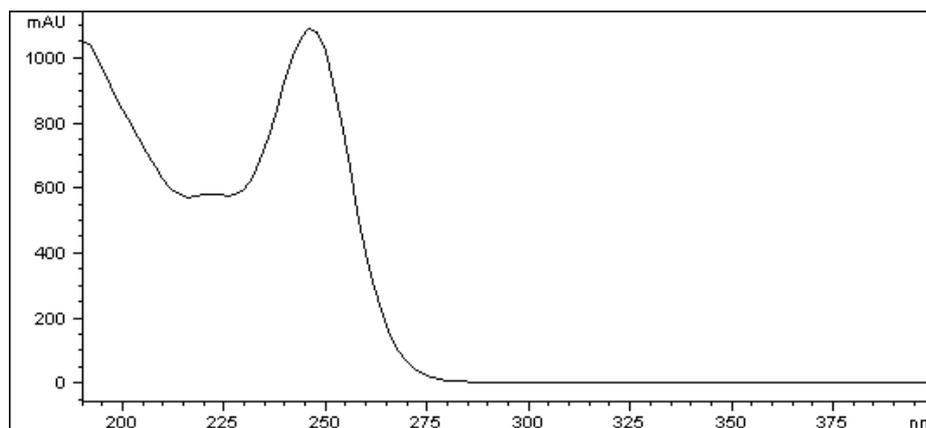**Fig. 29** IR spectrum sulfometuron-methyl standard

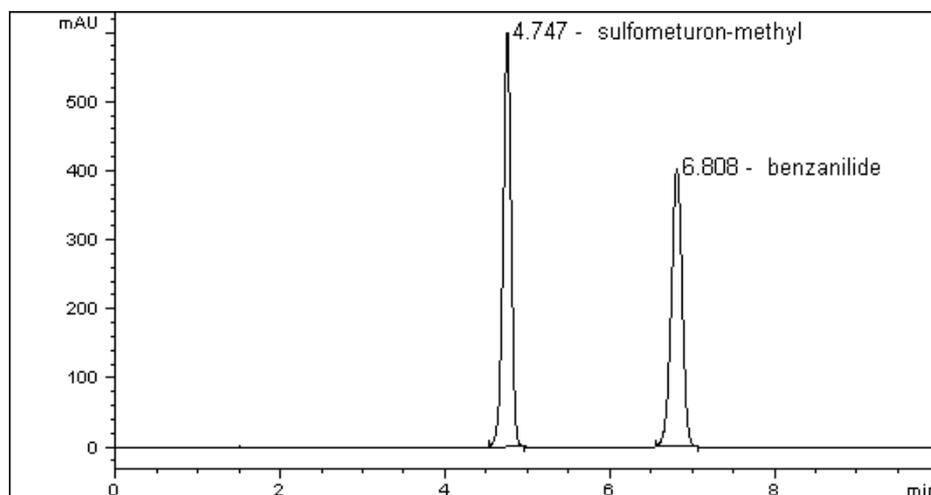
Fig. 30 UV spectrum sulfometuron-methyl standard

Fig. 31 Chromatogram of sulfometuron-methyl standard

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