METSULFURON - METHYL
441

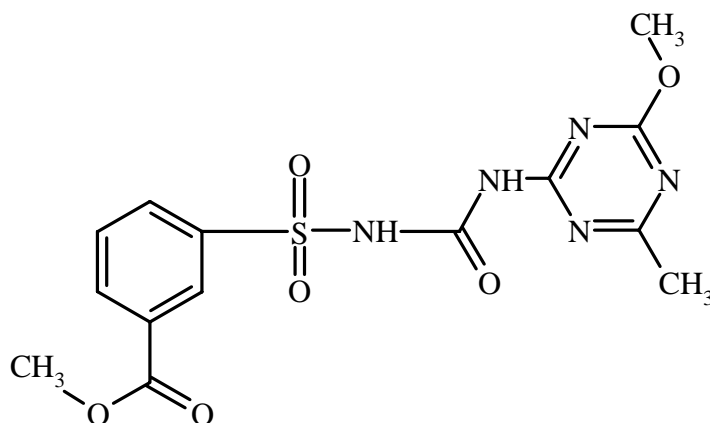|	
ISO common name	Metsulfuron-methyl
Chemical name	Methyl 2-(4-methoxy-6-methyl-1,3,5-triazin-2-yl-carbamoyl-sulfamoyl)benzoate (IUPAC); methyl 2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)-amino]carbonyl]-amino]sulfonyl]benzoate (CA; *79510-48-8*)
Empirical formula	$C_{14}H_{15}N_5O_6S$
RMM	381.4
m.p	158 °C
v.p.	3.4×10^{-10} Pa at 25 °C
Solubility	In water: 0.55 g/l (pH 5) and 2.79 g/l (pH7) at 25 °C; xylene: 0.58 g/l; hexane: 0.79 g/l; ethanol: 2.3; methanol: 7.3 g/l; acetone: 36 g/l; dichloromethane: 121 g/l; all at 20 °C
Description	Colourless crystals
Stability	Hydrolysed by strong acids and bases
Formulations	Water dispersible granules

METSULFURON-METHYL TECHNICAL
[*]441/TC/(M)/-

1 Sampling. Take at least 100 g.

2 Identity tests
2.1 HPLC. Use the HPLC method below. The retention time of metsulfuron-methyl for the sample solution should not deviate by more than 1 % from that for the calibration solution.

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

3 Metsulfuron-methyl

OUTLINE OF METHOD   Metsulfuron-methyl is determined by high performance liquid chromatography on a reversed phase column (C8) using water - acetonitrile (65 + 35 v/v at pH 3) as eluent and UV detection at 254 nm. The content of active ingredient is quantitated using a calibration curve.

REAGENTS

Acetonitrile HPLC grade
Water HPLC grade
Metsulfuron-methyl standard of known purity
Phenyl sulphone internal standard
Phosphoric acid 85 %, HPLC grade
Ammonium hydroxide solution 30 %
Ammonium hydroxide solution dilute, $c(NH_4OH) = 0.01$ mol/l. Pipette 0.8 ml of concentrated ammonia solution into water (1000 ml).
Mobile phase water - acetonitrile, 65 + 35 (v/v), adjusted to pH 3 with phosphoric acid
Sample solvent. Mix equal volumes of acetonitrile and dilute ammonia solution.
Internal standard solution. Weigh into a bottle (1 l) phenyl sulphone (9.0 g), add acetonitrile (1000 ml). Place the bottle in an ultrasonic bath for 15 min; allow to cool to room temperature (Stable for at least a month when stored at room temperature in a brown glass bottle).

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[*] CIPAC method 1997. Prepared by a Committee chaired by L Warnell Neal.Based on a method supplied by DuPont de Nemours USA.

Calibration solution. Weigh (to the nearest 0.01 mg) 75, 100 and 125 mg (± 5 mg) of metsulfuron methyl standard into three separate volumetric flasks (100 ml). Add by pipette to each flask internal standard solution (10.0 ml), add sample solvent (90 ml), and place the flasks in an ultrasonic bath for 15 min. Allow to cool to room temperature and mix well (Stable for at least 24 h).

APPARATUS

High performance liquid chromatograph equipped with a constant-temperature compartment, a 5 µl loop injection valve, and a UV spectrophotometric detector capable of operating at 254 nm
Column stainless steel, 150 × 4.6 (i. d.) mm packed with Zorbax SB-C_8 (5 mm), with in-line filter
Filtering apparatus with 0.2 µm filter (Acrodisc-CR or equivalent)
Integrator or *electronic data system*

PROCEDURE

(a) Operating conditions (typical):

Eluent flow rate	2.0 ml/min
Column temperature	40 °C
Injection volume	5 µl. Do not use larger injection volumes, because this will result in bad peak shapes due to ionisation of the active ingredient.
Detector wavelength	254 nm (bandwith at least 4 nm)
Retention time	metsulfuron methyl: about 3 min
	phenyl sulphone: about 6 min

(b) Sample preparation. Weigh (to the nearest 0.01 mg) into a volumetric flask (100 ml) sufficient sample to contain about 100 mg (*w* mg) of metsulfuron-methyl. Add by pipette internal standard solution (10.0 ml), add sample solvent (90 ml), and place the flask in an ultrasonic bath for 15 min. Allow to cool to room temperature and mix well.

(c) Determination. Filter all solutions through a 0.2 µm filter before injecting. Equilibrate the column by pumping the mobile phase through the column until a stable baseline has been obtained. Then make duplicate injections of 5 µl of the calibration and the sample solutions.

Calculate the average metsulfuron-methyl to phenyl sulphone peak area ratio for each injection of each calibration and sample solution. Prepare a calibration curve by plotting the average peak area ratio for each calibration solution versus the mass of metsulfuron-methyl in the calibration solutions. Using the least-squares method calculate the line that best fits the experimental data. The correlation coefficient should be 0.998 or better. If not, repeat the calibration. Determine the mass of metsulfuron-methyl in the sample solution using the equation of the calibration curve.

(d) Calculation

$$\text{Metsulfuron-methyl content} = \frac{(R - b) \times P}{a \times w} \, 1\text{g/kg}$$

where:

R = average metsulfuron-methyl to phenyl sulphone ratio of the sample solutions

a = slope of calibration curve

b = intercept of calibration curve

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

w = mass of sample taken (mg)

Repeatability r = 58 to 42 g/kg at 987 g/kg active ingredient content

Reproducibility R = 58 to 42 g/kg at 987 g/kg active ingredient content

METSULFURON-METHYL WATER DISPRSIBLE GRANULES
[*]441/WG/M/-

1 Sampling. Take at least 500 g.

2 Identity tests.
2.1 HPLC. As for **441**/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 **441**/TC/M/2.2.

3 Metsulfuron-methyl. As for metsulfuron-methyl **441**/TC/M/3.

Repeatability r = 15 to 19 g/kg at 607 g/kg active ingredient content
= 6 to 9 g/kg at 212 g/kg active ingredient content
Reproducibility R = 19 to 20 g/kg at 607 g/kg active ingredient content
= 6 to 9 g/kg at 212 g/kg active ingredient content

4 Suspensibility (Draft method)

REAGENTS AND APPARATUS As for MT 168 and **441**/TC/M/3, except:

Calibration solutions. Weigh (to the nearest 0.1 mg) into three separate volumetric flasks 20, 50 and 80 mg (± 5 mg) metsulfuron-methyl. Add by pipette to each flask internal standard solution (10.0 ml) and sample solvent (90 ml). Place the flasks in an ultrasonic bath for 15 min. Allow to cool to room temperature and mix well. Prepare in the same way standard solutions at other concentrations, if needed. Filter a small portion of each solution through a 0.2 µm (or 0.45 µm) PTFE filter prior to analysis.

PROCEDURE

(a) Preparation of suspension determination of sedimentation. MT 168.
(b) Determination of metsulfuron-methyl in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension add to the remaining 25 ml sample solvent (65 ml) and by pipette internal standard solution (10 ml). Place the cylinder in an ultrasonic bath for 5 min. Allow to cool to room temperature and take a suitable aliquot of the solution for the determination of the mass of metsulfuron-methyl (Q g). Proceed as for **441**/TC/M/3.
(c) Calculation

$$\text{Suspensibility} = \frac{111 \, (c - Q)}{c} \, 2\%$$

where:
c = mass of active ingredient in sample actually taken
Q = mass of active ingredient in the 25 ml remaining in the cylinder
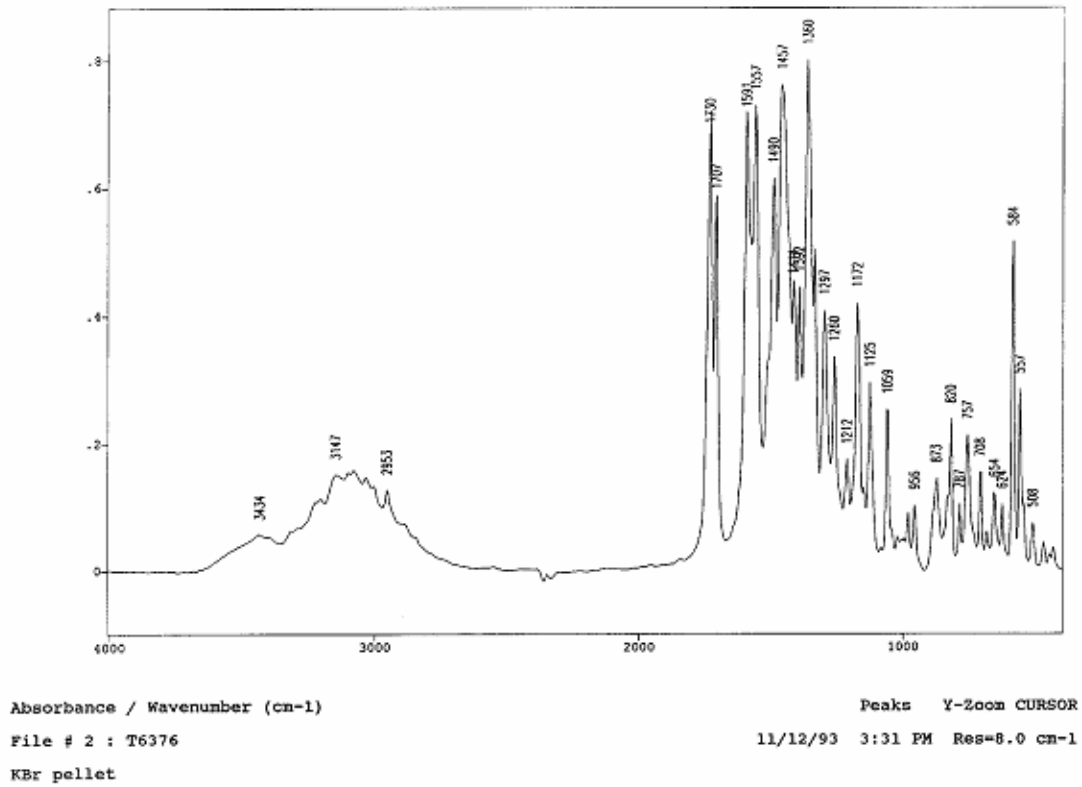
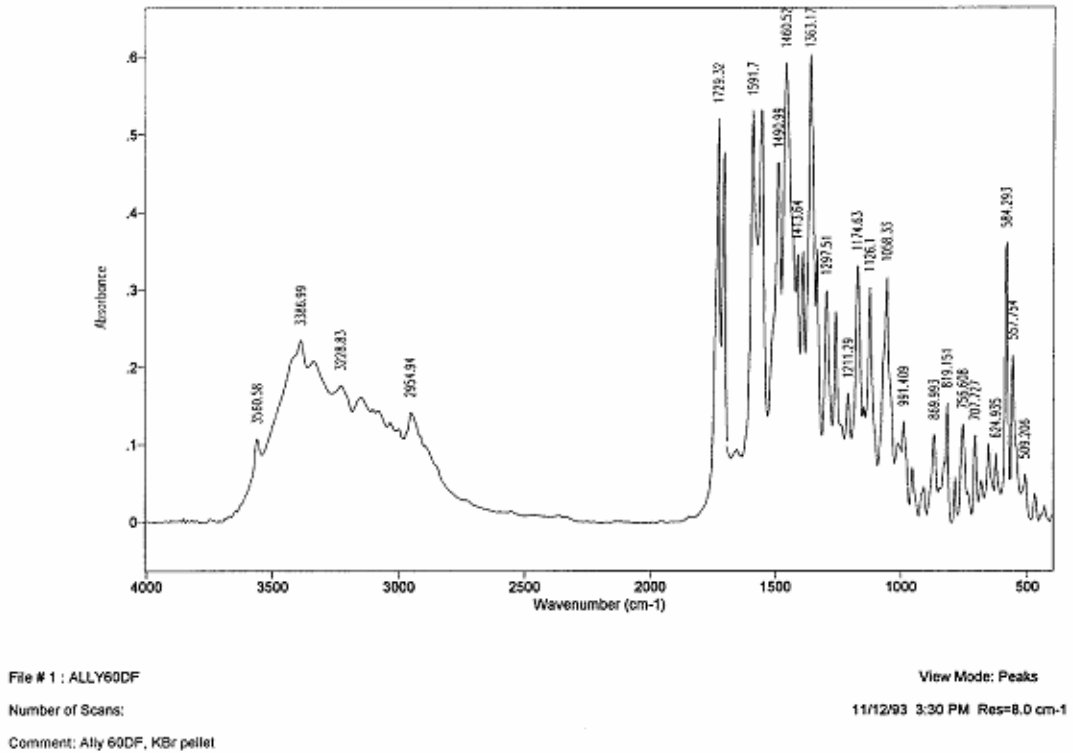**Fig. 18**. Infrared spectrum of metsulfuron-methyl reference standard

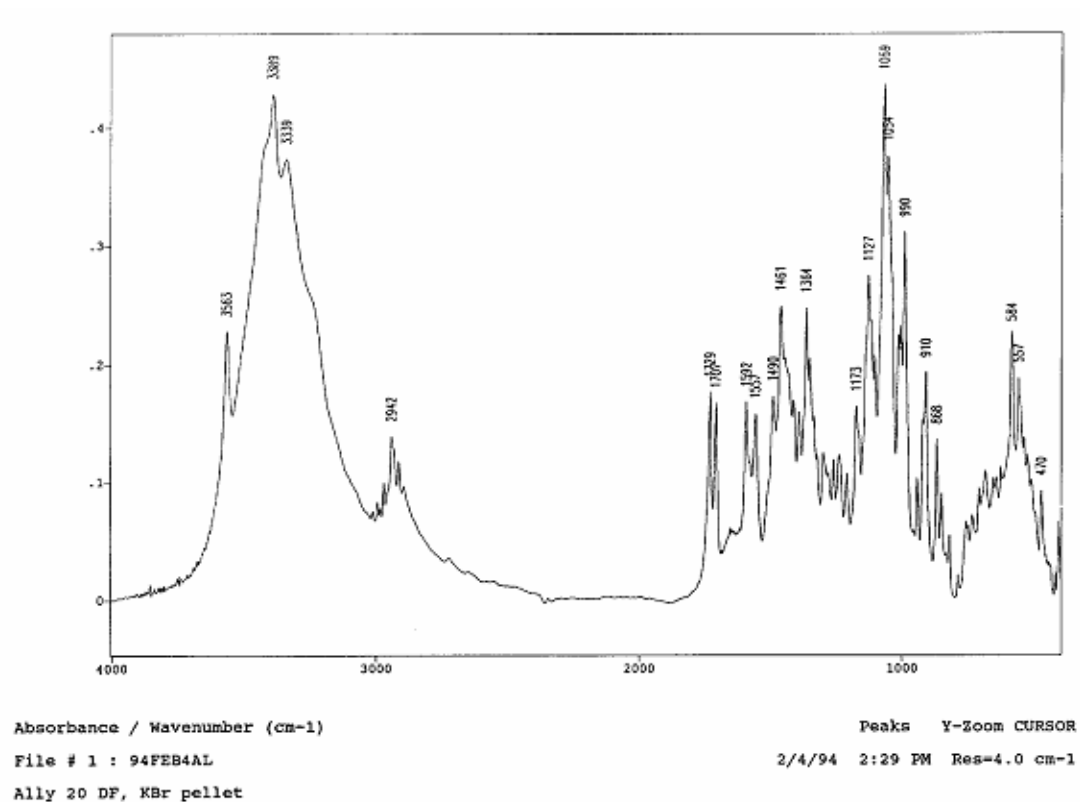**Fig. 19**. Infrared spectrum of metsulfuron-methyl 60% WG

Fig. 20. Infrared spectrum of metsulfuron-methyl 20% WG
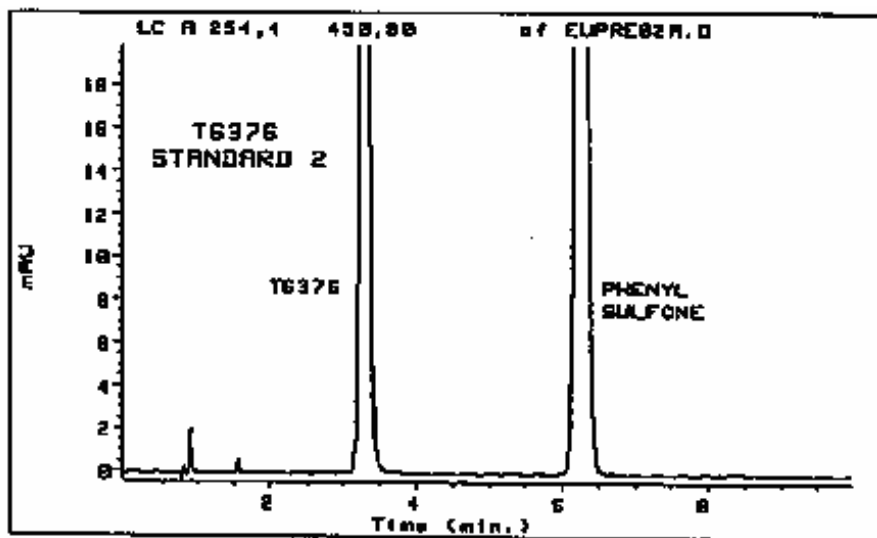

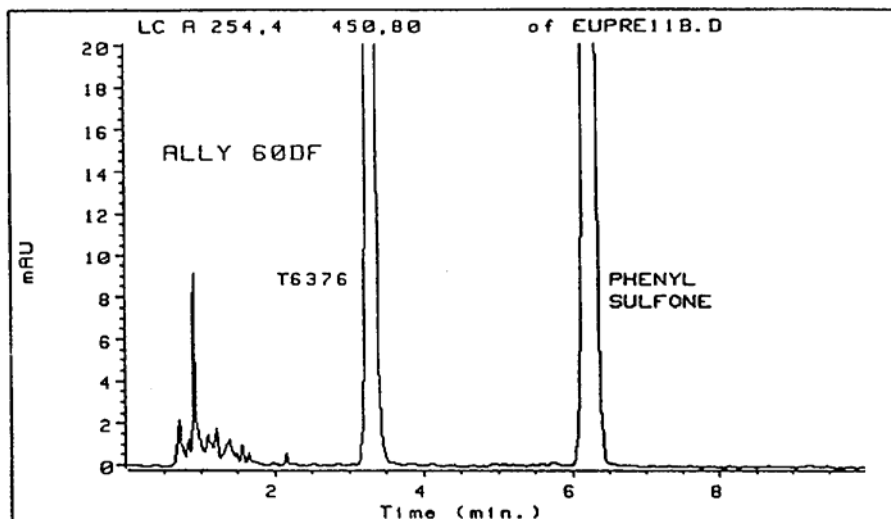**Fig. 21**. Chromatogram of metsulfuron-methyl standard
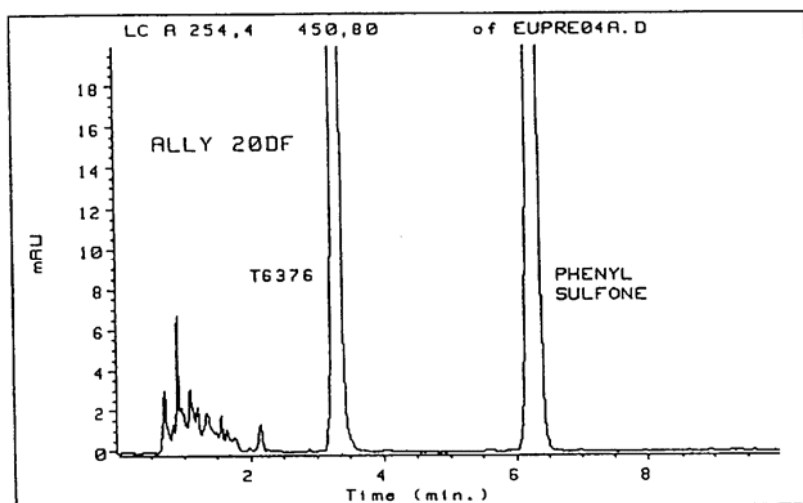
Fig. 22. Chromatogram of metsulfuron-methyl 60% WG

Fig. 23. Chromatogram of metsulfuron-methyl 20%WG