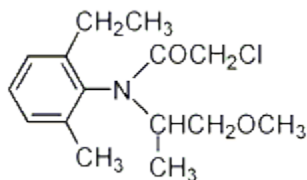


METOLACHLOR

400



<i>ISO common name</i>	Metolachlor
<i>Chemical name</i>	2-chloro-6'-ethyl-N-(2-methoxy-1-methylethyl)acet-o-toluidine (IUPAC); 2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)-acetamide (CA; 51218-45-2)
<i>Empirical formula</i>	C ₁₅ H ₂₂ ClNO ₂
<i>RMM</i>	283.8
<i>b.p.</i>	100 °C at 0.1 Pa
<i>v.p.</i>	1.7 mPa at 20 °C
<i>d²⁰</i>	1.12
<i>Solubility</i>	In water 530 mg/l at 20 °C; soluble in common organic solvents; slightly soluble in petroleum ether
<i>Description</i>	Colourless liquid
<i>Stability</i>	Hydrolysed by strong alkalis and strong mineral acids
<i>Formulations</i>	Emulsifiable concentrates and Emulsions, Oil in water

METOLACHLOR TECHNICAL

*400/TC/(M)/-

1. Sampling. Take at least 100 g.

2. Identity tests

2.1 GC. Use the GC method below. The relative retention time of metolachlor with respect to the internal standard for the sample solution should not deviate by more than 1% from that for the calibration solution.

2.2 Infrared. Prepare potassium bromide discs for the technical sample and metolachlor reference substance. A typical potassium bromide disc should contain a sample prepared in the 0.15-0.35% by weight range. Scan the discs from 4000 to 400 cm^{-1} . The spectrum from the sample should not differ significantly from that of the reference substance.

3. Metolachlor

OUTLINE OF METHOD Metolachlor is dissolved in methanol and determined by gas chromatography with flame ionization detection, using dipentyl phthalate as internal standard.

REAGENTS

Metolachlor standard of known purity

Dipentyl phthalate pure, internal standard. Should not contain any impurities that elute at the metolachlor retention time.

Methanol, HPLC grade

Internal standard solution. Weigh into a volumetric flask (250 ml) dipentyl phthalate (2.5 g). Add methanol (200 ml) into the flask, place the flask in an ultrasonic bath for 2-3 min. Allow to cool to ambient temperature. Dilute to volume with methanol. Mix thoroughly.

Calibration solutions. Weigh in duplicate (to the nearest 0.1 mg) into a volumetric flask (10 ml) about 50 mg of metolachlor standard (s mg). Add internal standard solution (5 ml) into the flask, place the flask in an ultrasonic bath for 2-3 min. Allow to cool to ambient temperature. Dilute to volume with methanol. Mix thoroughly. Filter a portion of each sample solution with a 0.22 μm filter prior to analysis (Solutions C_A and C_B).

APPARATUS

GC system with FID

Column, HP-5, capillary, 30 m x 0.32 mm x 0.25 μm , or equivalent.

Electronic integrator or data system

Ultrasonic bath

Nylon syringe filters, 0.22 µm

PROCEDURE

(a) Operating conditions (typical):

<i>Column temperature</i>	182 °C
<i>Injection port temperature</i>	250 °C
<i>Detector temperature</i>	250 °C
<i>Injection volume</i>	1.0 µl
<i>Flow rate</i>	
<i>nitrogen (carrier)</i>	2.0 ml/min
<i>hydrogen</i>	30 ml/min
<i>air</i>	300 ml/min
<i>make up</i>	25 ml/min
<i>Injection type</i>	Split
<i>Split ratio</i>	20:1
<i>Retention time</i>	Metolachlor: about 8.6 min internal standard: about 15.2 min

(b) *Preparation of sample.* Weigh in duplicate (to the nearest 0.1 mg) into a volumetric flask (10 ml) sufficient sample to contain about 50 mg of metolachlor (*w* mg). Add internal standard solution (5 ml) into the flask, place the flask in an ultrasonic bath for 2-3 min. Allow to cool to ambient temperature. Dilute to volume with methanol. Mix thoroughly. Filter a portion of each sample solution with a 0.22µm filter prior to analysis (Solutions S₁ and S₂).

(c) *Determination.* Inject into the gas chromatograph 1µl portions of the calibration solution until the area (or height) ratios (*R'*) of metolachlor to the internal standard varies by less than 1.5% for successive injections, otherwise prepare new calibration solutions. Inject in duplicate 1µl portions of each sample solution bracketing them by injections of the calibration solutions as follows: C_A, S₁, S₁, C_B, S₂, S₂, C_A, and so on. Calculate the response ratio for each injection.

(d) *Calculation.* Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the metolachlor contents of the bracketed sample injections. The metolachlor content is the mean value of two sample solutions.

$$f_i = \frac{s \times P}{R'}$$

$$\text{Metolachlor content} = \frac{R \times f}{w} \text{ g/kg}$$

where:

R = peak area (or height) ratio of metolachlor to the internal standard for the sample solution

R' = peak area (or height) ratio of metolachlor to the internal standard for the calibration solution

f_i = individual response factor

f = mean response factor

s = mass of metolachlor standard taken (mg)

w = mass of sample taken (mg)

P = purity of the metolachlor standard (g/kg)

Repeatability r = 4 to 9 g/kg at an active ingredient content of 970 to 987 g/kg

Reproducibility R = 12 to 17 g/kg at an active ingredient content of 970 to 987 g/kg

METOLACHLOR EMULSIFIABLE CONCENTRATES

***400/EC/(M)/-**

1. Sampling. Take at least 100 ml.

2. Identity tests.

2.1 GC. As for metolachlor technical 400/TC/(M)/2.1

2.2 Infrared. As for metolachlor technical 400/TC/(M)/2.2

3. Metolachlor. As for metolachlor technical 400/TC/M/3.

Repeatability r = 7 to 11 g/kg at an active ingredient content of 670 to 692 g/kg

Reproducibility R = 18 to 21 g/kg at an active ingredient content of 670 to 692 g/kg

METOLACHLOR EMULSIONS, OIL IN WATERW

***400/EW/(M)/-**

1. Sampling. Take at least 100 ml.

2. Identity tests.

2.1 GC. As for metolachlor technical 400/TC/(M)/2.1

2.2 Infrared. As for metolachlor technical 400/TC/(M)/2.2

3. Metolachlor. As for metolachlor technical 400/TC/M/3.

Repeatability r = 9 to 12 g/kg at an active ingredient content of 477 to 504 g/kg

Reproducibility R = 10 to 12 g/kg at an active ingredient content of 477 to 504 g/kg

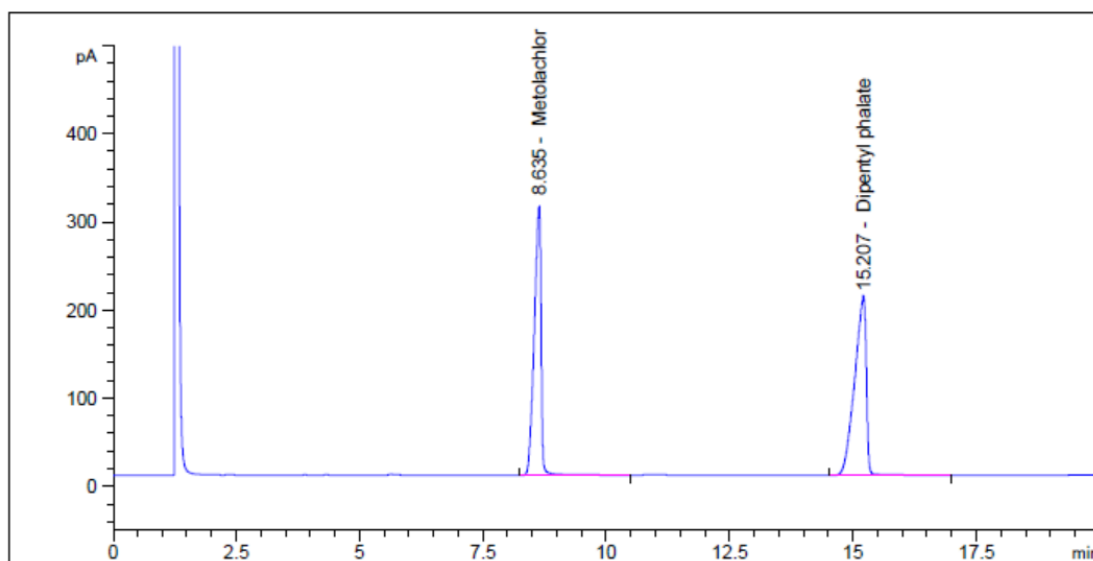


Fig 1 Example of GC Chromatogram of Metolachlor standard

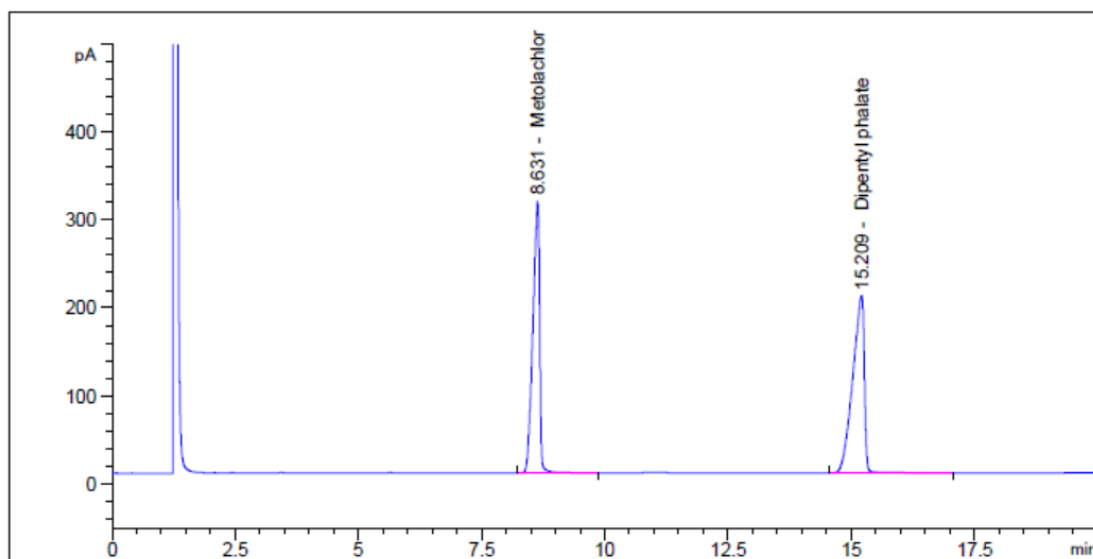


Fig 2 Example of GC Chromatogram of Metolachlor TC

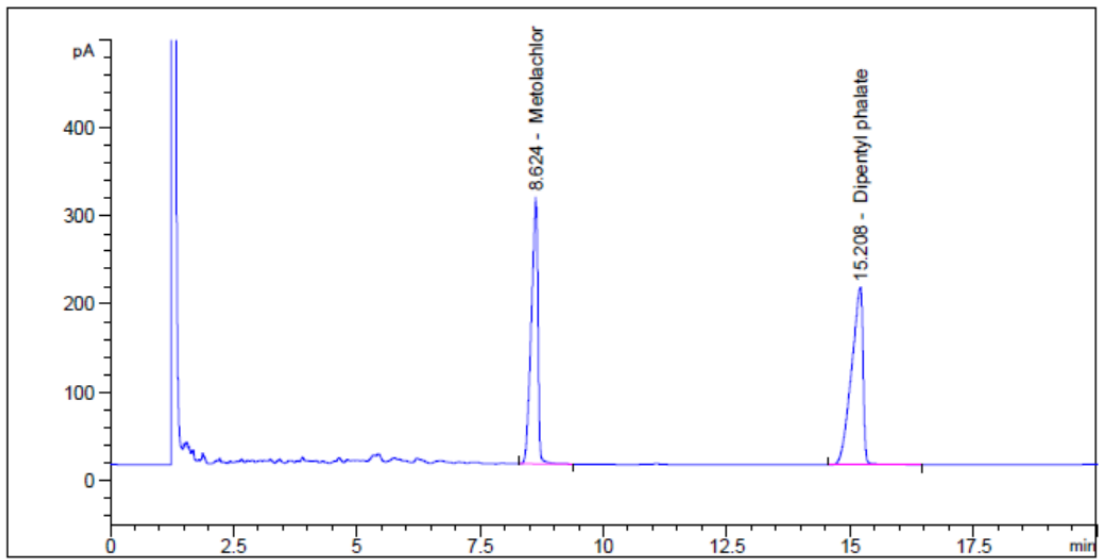


Fig 3 Example of GC Chromatogram of Metolachlor EC

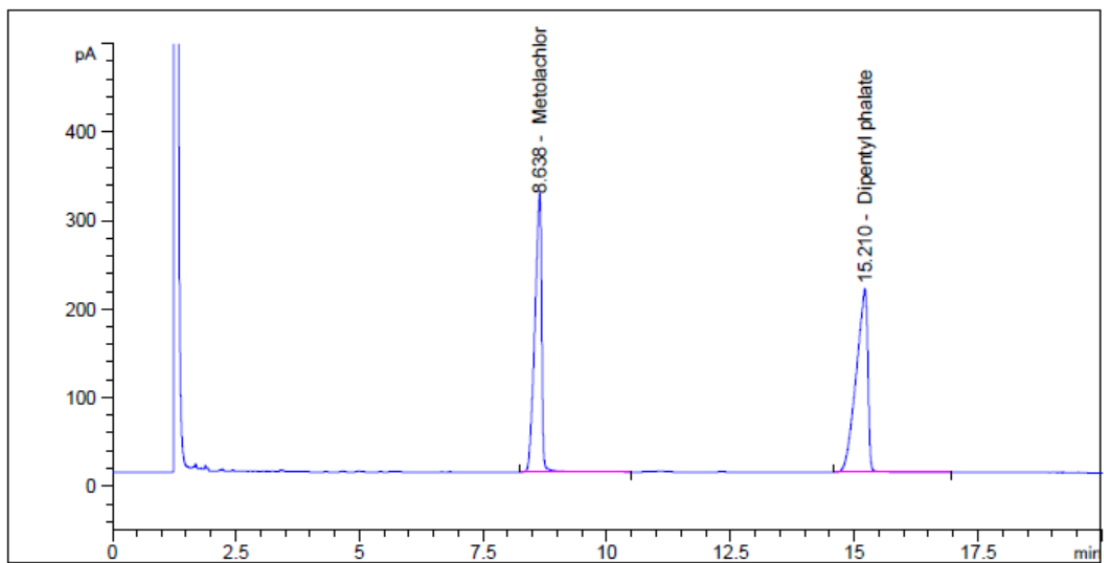


Fig 4 Example of GC Chromatogram of Metolachlor EW

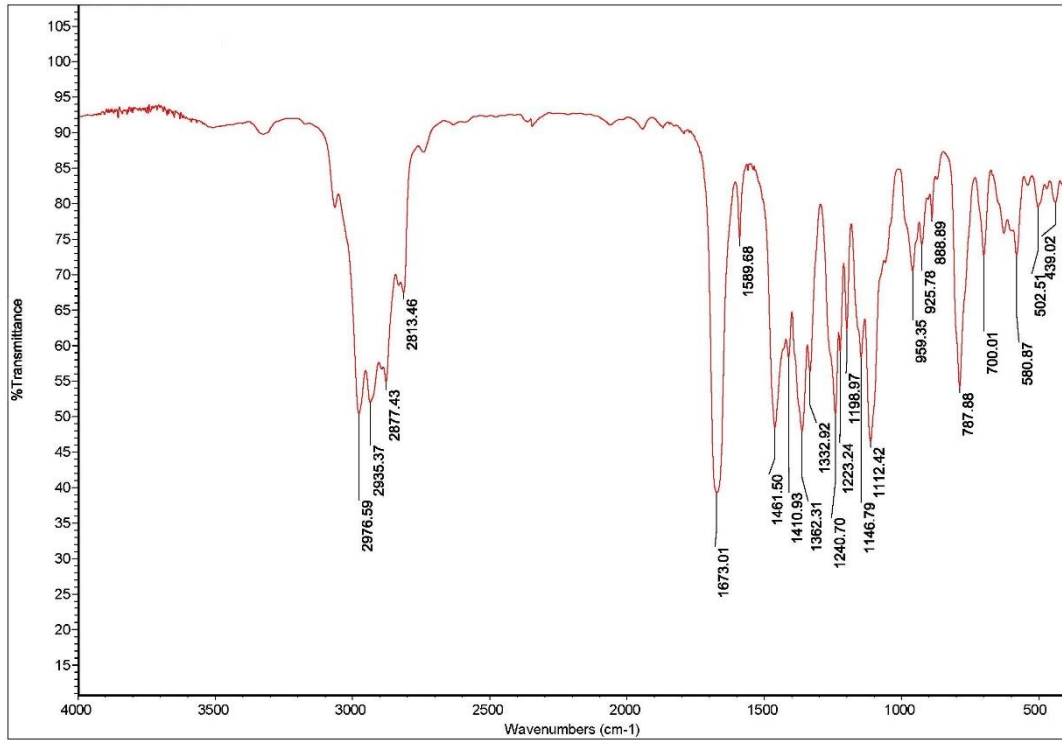


Fig 5 Example of FTIR spectrum of Metolachlor Standard

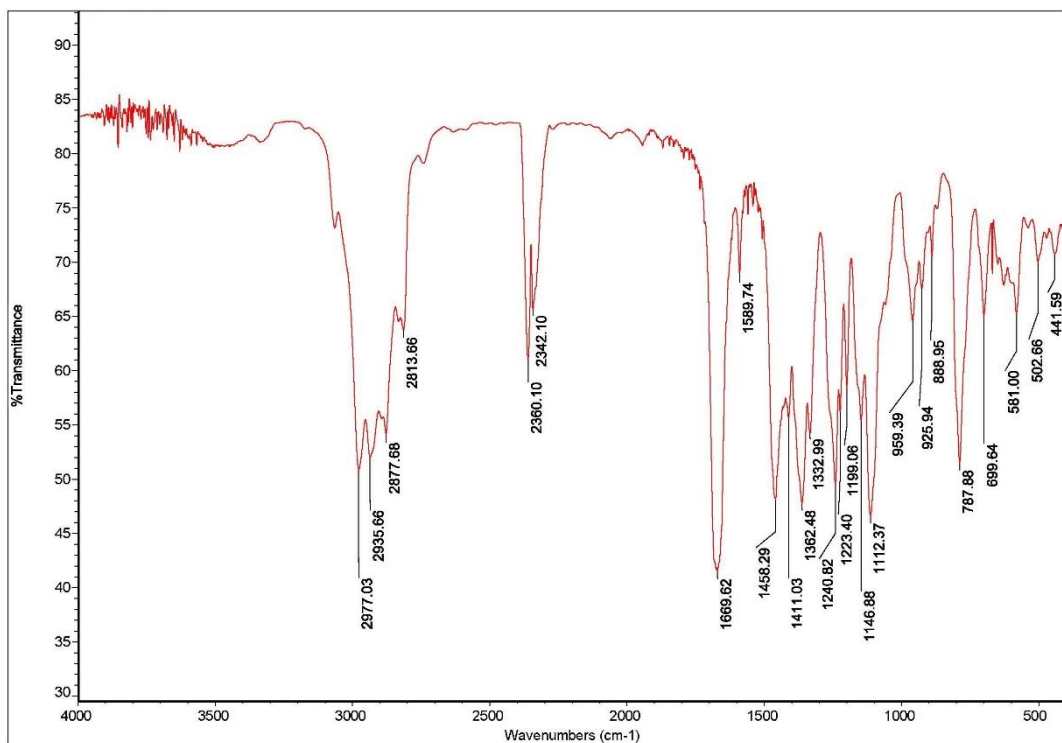


Fig 6 Example of FTIR spectrum of Metolachlor TC