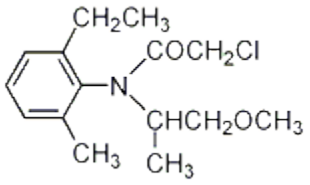
METOLACHLOR

400



ISO common name	Metolachlor
Chemical name	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
CAS No.	51218-45-2
Empirical formula	$C_{15}H_{22}ClNO_2$
RMM	283.8
b.p.	100 ℃ at 0.1 Pa
v.p.	1.7 mPa at 20 ℃
d^{20}	1.12
Solubility	In water 530 mg/l at 20 ℃; soluble in common organic solvents; slightly soluble in petroleum ether
Description	Colourless liquid
Stability	Hydrolysed by strong alkalis and strong mineral acids
Formulations	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 ×0.25 μm, or equivalent.

Electronic integrator or data system

Ultrasonic bath

Nylon syringe filters, 0.22 μm

PROCEDURE

(a) Operating conditions (typical):

Column temperature		Heating rate (℃/min)	Temperature (℃)	Hold time (min)
	Initial	/	180	1.0
	Ramp	5.0	230	1.0
Injection port temperature	250 ℃			
Detector temperature	250 ℃			
Injection volume	1.0 μl			
Flow rate				
nitrogen (carrier)	2.0 ml/min			
Detector				
hydrogen	30 ml/min			
air	300 ml/min			

make up	25 ml/min
Injection type	Split
Split ratio	30:1
Run time	12 min
Retention time	Metolachlor: about 6.6 min
	Internal standard: about 8.7 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 S_1 and S_2).

(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, S_1, S_1, C_B, S_2, S_2, 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.

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

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

where:

f_i = individual response factor

f = mean response factor

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

for the calibration solution

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

s = mass of metolachlor standard taken (mg)

w = mass of sample taken (mg)

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

Repeatability r = g/kg at an active ingredient content of g/kg

Reproducibility R = g/kg at an active ingredient content of 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 = g/kg at an active ingredient content of g/kg
Reproducibility R = g/kg at an active ingredient content of 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 = g/kg at an active ingredient content of g/kg
Reproducibility R = g/kg at an active ingredient content of g/kg

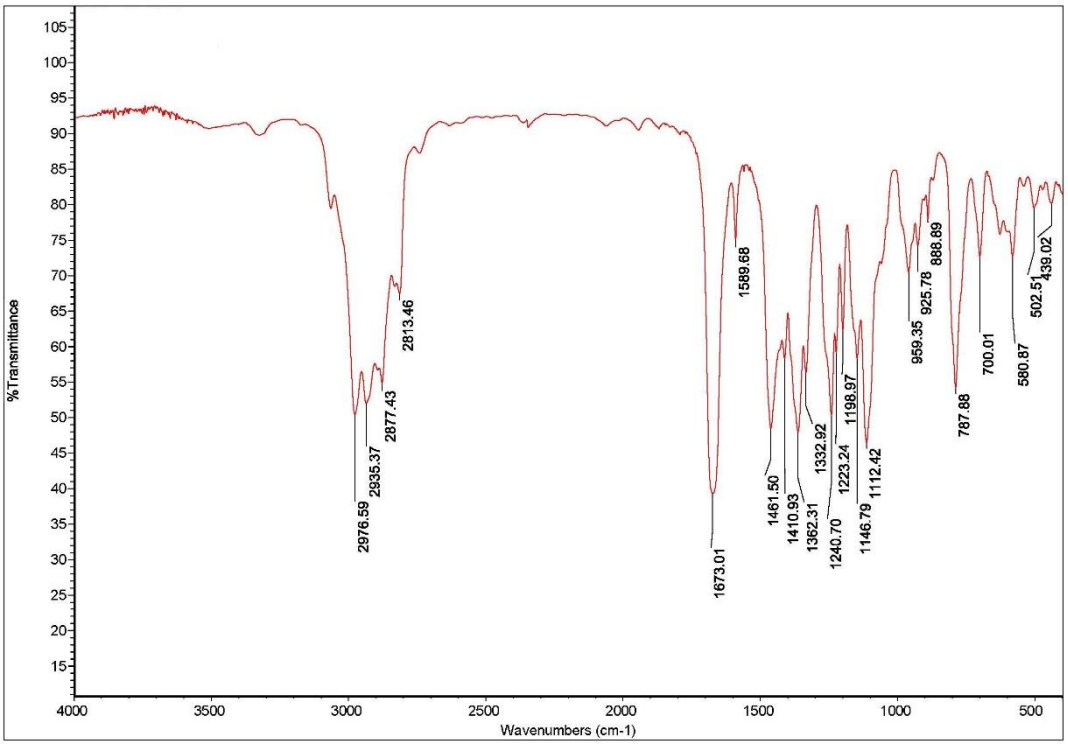


Fig. 1 FTIR spectrum of metolachlor

g

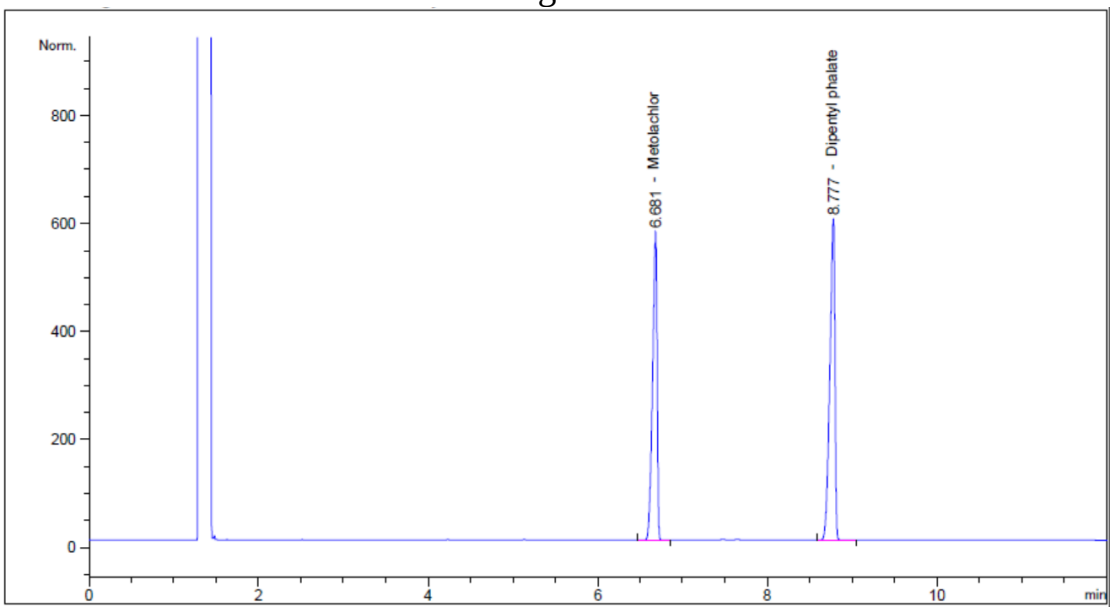


Fig. 2 GC Chromatogram of metolachlor standard

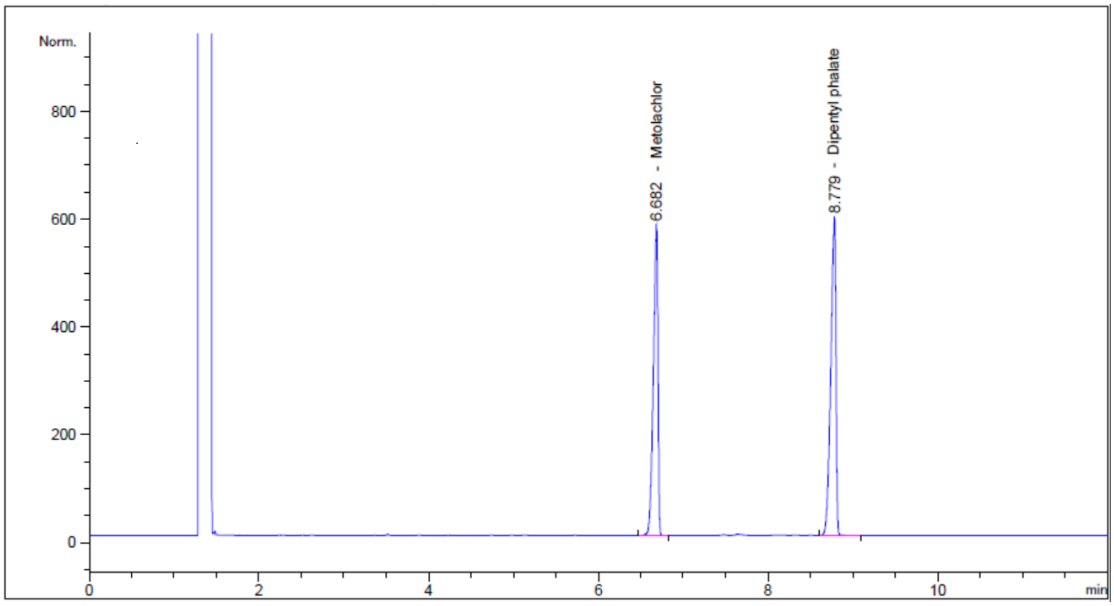


Fig. 3 GC Chromatogram of metolachlor TC

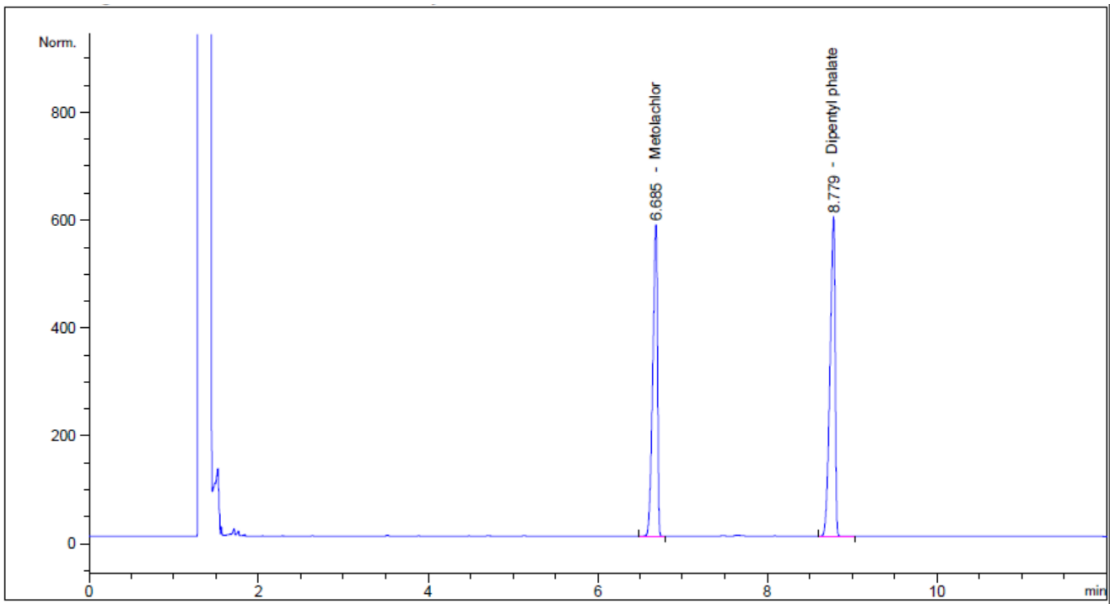


Fig. 4 GC Chromatogram of metolachlor EC

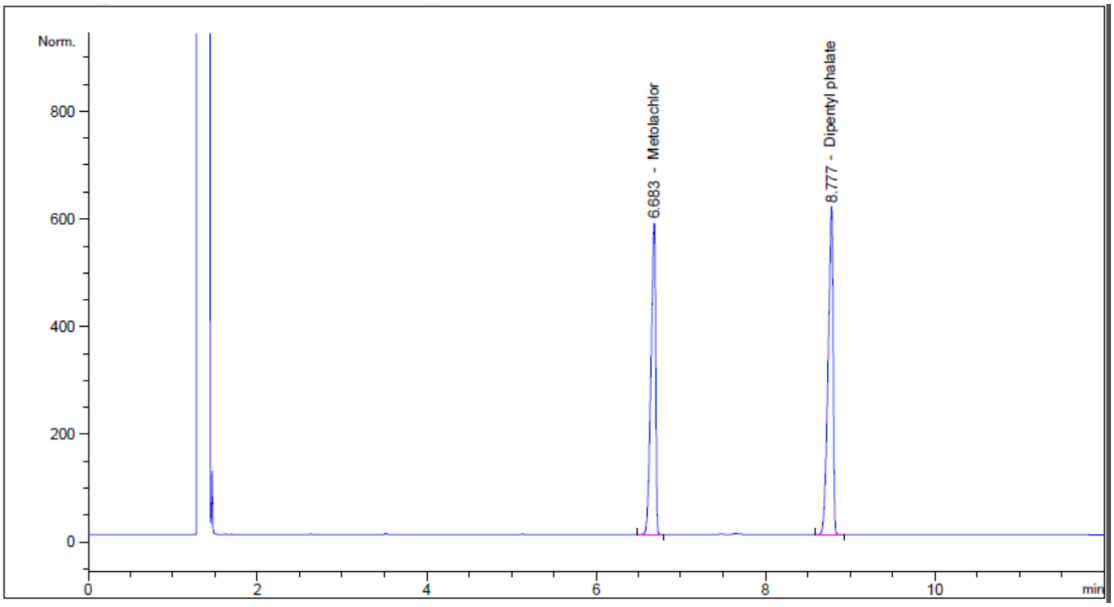


Fig. 5 GC Chromatogram of metolachlor EW