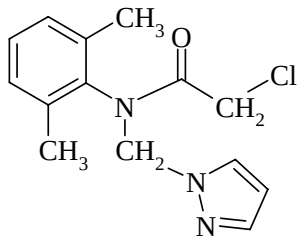
METAZACHLOR 411



ISO common name	Metazachlor
Chemical name	2-Chloro-*N*-(pyrazol-1-ylmethyl)acet-2,6-xylidide (IUPAC); 2-chloro-*N*-(2,6-dimethylphenyl)-*N*-(1*H*-pyrazol-1-ylmethyl)acetamide(CA; *67129-08-2*)
Empirical formula	$C_{14}H_{16}ClN_3O$
RMM	277.8
m.p.	85 °C
v.p.	1.3×10^{-5} Pa at 20 °C
Solubility	In water: 0.43 g/kg at 20 °C; in ethanol: 200 g/kg; in ethyl acetate: 590 g/kg; in acetone: more than 1000 g/kg; in chloroform: more than 1000 g/kg; in ether: 143 g/kg; in toluene: 770 g/kg; in cyclo-hexane: 13 g/kg at 20 °C
Description	The pure material is a colourless and odourless crystalline solid
Stability	At least 2 years at 40 °C
Formulations	Suspension concentrate

METAZACHLOR TECHNICAL
*411/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 Infrared. Prepare potassium bromide discs from the sample and from pure metazachlor using 1.3 to 1.5 mg material and 300 mg potassium bromide. Scan the discs from 4000 to 400 cm^{-1}. The spectrum produced from the sample disc should not differ significantly from that from the standard.

2.2 HPLC. Use the HPLC method below. The retention time of metazachlor for the sample solution should not deviate by more than 1 % from that for the calibration solution.

3 Metazachlor

OUTLINE OF METHOD The active ingredient is chromatographed by HPLC on a reversed phase column (C_{18}, methanol/water) and quantitatively determined by UV detection with external calibration.

REAGENTS

Methanol HPLC grade, e. g. methanol LiChrosolv® from Merck
Water HPLC grade
Mobile phase methanol/water 3 + 2 (v/v)
Metazachlor standard of known purity
Calibration solution. Weigh (to the nearest 0.1 mg) about 270 mg of metazachlor standard (*s* mg) into a 50 ml volumetric flask and dissolve quantitatively in 6 ml methanol. Make up to volume with mobile phase. Adjust the amount of metazachlor (*s* mg) such that the peak height is not more than 80 % of full scale. In case of doubt, check the linearity of the detector. Prepare two calibration solutions of similar concentrations. Do not use the same calibration solutions for periods longer than one week.

APPARATUS

HPLC system consisting of:
- high precision HPLC pump
- injection valve with 10 µl loop, e. g. Reodyne 7125
- stainless steel column 250 × 4 mm, packed with LiChrosorb/ RP 18, 7 µm, from Merck or equivalent

* CIPAC method 1992. Prepared by the German Committee (DAPA), Chairman: Dr W Dobrat. Based on a method supplied by BASF (FRG)

- variable wavelength UV detector with linear response at high absorbence values, e. g. Perkin Elmer LC 75
- data system with recorder for peak evaluation

PROCEDURE

(a) Preparation of sample solution. Weigh (to the nearest 0.1 mg) into a 50 ml volumetric flask enough sample (*w* mg) to contain about 270 mg pure metazachlor. Dissolve in 6 ml methanol and make up to volume with mobile phase. The concentration of metazachlor should be similar to its concentration in the calibration solutions. Prepare three solutions for each sample.

(b) Chromatographic conditions (typical):

Column stainless steel column	250 × 4 (i.d.)mm
Stationary phase	LiChrosorb® RP 18, 7 m, Merck e.g. Hibar® pre-packed column from Merck
Mobile phase	methanol/water, 3 + 2 (v/v)
Injection volume	10 µl
Flow rate	1.1 ml/min
Detector wavelength	263 nm
Detector sensitivity	1 absorbence unit full scale
Chart speed	1 cm/min
Retention time	metazachlor: about 7 min
Temperature	ambient.

Do not use elevated column temperatures, as this may lead to deterioration of metazachlor.

(c) Determination of metazachlor. Inject 10 µl portions of both the calibration solutions. Inject each calibration solution at least two times and calculate the average peak area to mass ratio. The individual values should not deviate from the mean by more than ± 0.8 %, otherwise repeat the calibration. Then inject in duplicate 10 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows: calibration solution I, sample solution 1, sample solution 1, calibration solution II, sample solution 2, sample solution 2, calibration solution I, sample solution 3, sample solution 3, calibration solution II, and so on for other sample solutions. Measure the relevant peak areas. Calculate the mean value of each pair of calibration factors (*d*) bracketing two sample injections and use this value for evaluating the two bracketed sample runs.

(d) Calculation

Calibration factor:

$$f = \frac{H_s}{s}$$

where:

H_s = area of the metazachlor peak in the calibration solution
s = mass of metazachlor standard taken (mg)

$$\text{Metazachlor content} = \frac{H_w \times P}{f \times w}$$

where:

H_w = area of the metazachlor peak in the sample solution
P = purity of metazachlor standard (g/kg)
w = mass of the sample taken (mg)

The content of metazachlor is the mean value of the results of the three sample solutions.

Repeatability r = 16 at 967 g/kg active ingredient content
Reproducibility R = 26 at 967 g/kg active ingredient content

Based on a study with 19 participants and 95 values.

METAZACHLOR SUSPENSION CONCENTRATES
[*]**411**/SC/M/-

1 Sampling. Take at least 1 l.

2 Identity tests

2.1 Infrared. Weigh about 0.1 g of sample into a 50 ml centrifuge tube, add 50 ml of acetone, stir for 1 h and centrifugate for 20 min at about 3000 min^{-1}. Transfer the supernatant, clear solution into a 100 ml round-bottomed flask and evaporate to dryness under reduced pressure. Dry the residue for 2 h at 60 °C in vacuo. Proceed as described for **411**/TC/M/2.1.

2.2 HPLC. As for 411/TC/M/2.2

[*] CIPAC method 1992. Prepared by the German Committee (DAPA), Chairman: Dr W Dobrat. Based on a method supplied by BASF (FRG)

3 Metazachlor. As for **411**/TC/M/3 except:
(a) Preparation of sample solution. Thoroughly shake the sample container to assure that the suspension is homogeneous. Immediately weigh into a 50 ml volumetric flask enough sample (*w* mg) to contain about 270 mg of pure metazachlor. Dissolve in mobile phase and continue according to **411**/TC/M/3a.

Repeatability r = 6.7 at 450 g/kg active ingredient content
Reproducibility R = 14 at 450 g/kg active ingredient content

Based on a study with 19 participants and 95 values.

4 Suspensibility (Draft Method)
(a) Preparation of suspension and determination of sedimentation. MT 161 CIPAC F, *p. 394*

(*b) Determination of metazachlor in the bottom 25 ml of suspension*. After removal of the top 225 ml of suspension proceed as for **411**/SC/M/3.