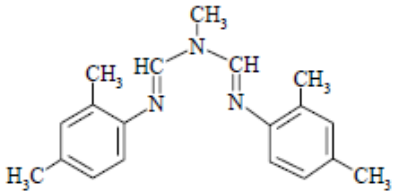
AMITRAZ
362



ISO common name	Amitraz
Chemical name	*N*-Methyl-bis(2,4-xylyliminomethyl)amine(IUPAC); *N'*-(2,4-dimethylphenyl)-*N*-[[(2,4-dimethylphenyl)-imino]-methyl]-*N*-methylmethanimidamide (CA; *33089-61-1*)
Empirical formula	$C_{19}H_{23}N_3$
RMM	293.4
m.p.	86-87 °C
v.p.	5.1×10^{-5} Pa at 20 °C
Solubility	In water about 1 mg/l at room temperature; soluble in common organic solvents
Description	Colourless crystals
Stability	Unstable in acidic solution, slow deterioration on prolonged storage under moist conditions
Formulations	Emulsifiable concentrates and wettable powders

AMITRAZ TECHNICAL
*362/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 GLC. Use the GLC method below. The relative retention time of amitraz 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 TLC. Use the following conditions:

TLC plate	Whatman K6F or Whatman PE SIL G/UV
Eluting solvent	ethyl acetate - hexane, 1 + 4(v/v)
Loading	100 µg
Visualising method	UV at 254 nm

The major spot from the sample should have the same R_F value as that from the standard.

3 Amitraz

OUTLINE OF METHOD Amitraz is separated from the other components present by gas chromatography, using a 3% OV-101 packed column, and quantified using squalane as internal standard.

REAGENTS

Methyl or ethyl acetate

Amitraz of known purity

Squalane internal standard. Must not show a peak with the same retention time as amitraz.

Internal standard solution. Dissolve 5.0 g squalane in methyl or ethyl acetate in a volumetric flask (500 ml) and make up to the mark with the same solvent. This solution is stable for at least one month.

Calibration solution. Weigh (to the nearest 0.1 mg) into a vial or stoppered flask (20 ml) between 90 and 110 mg pure amitraz (*s* mg). Add by pipette internal standard solution (10.0 ml), cap and shake to dissolve completely.

* CIPAC method 1993. Prepared by the Amitraz Panel of PAC-UK. Chairman: D S Wooldridge. Based on a method supplied by Schering Agrochemicals Ltd, UK.

APPARATUS

Gas chromatograph fitted with a flame ionisation detector and an integrator or data system

Column glass, 1.5 to 2 m × 3 mm (i.d.) packed with 3% OV-101 on Gas Chrom Q (0.18 to 0.15 mm) or equivalent high performance support

Microfiltration apparatus to remove particles larger than 0.5 µm

PROCEDURE

(*a*) *Operating conditions* (typical):

Oven temperature	220°C
Injection temperature	230°C. A higher temperature may cause amitraz decomposition.
Detector temperature	300°C
Injection volume	2 µl
Flow rate carrier gas	Nitrogen, 45 ml/min
Flow rates other gases	As described in the user's manual
Retention time	amitraz: about 14 to 18 min. Adjust flow rate of carrier gas such that amitraz elutes between these limits.

(*b*) *Preparation of sample*. Weigh (to the nearest 0.1 mg) into a vial or stoppered flask (20 ml) enough sample to contain between 90 and 110 mg amitraz (*w* mg). Add by pipette, using the same pipette as for the preparation of the calibration solution, internal standard solution (10.0 ml), cap and shake to dissolve completely.

(*c*) *Determination*. Inject into the gas chromatograph 2 µl portions of the calibration solution. Determine the response factor. Continue to inject calibration solutions until three consecutive response factors agree to within 1% of their mean. Then make duplicate 2 µl injections of the sample solution, followed by another injection of the calibration solution. Average the response factors of the injections following and preceding the sample injections and record the areas of the relevant sample peaks.

(*d*) *Calculation*

$$f = \frac{I_r \times s \times P}{H_s}$$

$$\text{Content of amitraz} = \frac{f \times H_w}{I_q \times w} \text{ g/kg}$$

where:

f = average response factor
H_s = area of the amitraz peak in the calibration solution
H_w = area of the amitraz peak in the sample solution
I_r = area of the internal standard in the calibration solution
I_q = area of the internal standard peak in the sample solution
s = mass of amitraz in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of the standard amitraz (g/kg)

Repeatability r = 11 g/kg at 953 g/kg active ingredient content
Reproducibility R = 25 g/kg at 953 g/kg active ingredient content

AMITRAZ WETTABLE POWDERS
*362/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 GLC. As for amitraz technical **362**/TC/M/2.1.

2.2 TLC. Shake 200 mg of sample with hexane (10 ml). Filter a small portion and continue as for amitraz technical **362**/TC/M/2.2.

* CIPAC method 1993. Prepared by the Amitraz Panel of PAC-UK. Chairman: D S Wooldridge. Based on a method supplied by Schering Agrochemicals Ltd, UK.

3 Amitraz. As for amitraz technical **362**/TC/M/3, except:
(*b*) *Preparation of sample*. Weigh (to the nearest 0.1 mg) into a vial or stoppered flask (20 ml) enough sample to contain between 90 and 110 mg amitraz (*w* mg). Add by pipette, using the same pipette as for the preparation of the calibration solution, internal standard solution (10.0 ml), cap and shake to dissolve the amitraz. Filter a suitable portion of the solution through a suitable micro-filter.

Repeatability r = 9.4 g/kg at 530 g/kg active ingredient content
Reproducibility R = 14 g/kg at 530 g/kg active ingredient content

4 Suspensibility (Draft method)
(*a*) *Preparation of suspension*. MT 15.1 (*i*).
(*b*) *Determination of sedimentation*. MT 15.1 (*ii*).
(*c*) *Determination of amitraz in the bottom 25 ml of suspension*. After removal of the top 225 ml of suspension, transfer the remaining 25 ml to a separating funnel. Add anhydrous calcium chloride (0.5 g) and extract with dichloromethane (2 × 25 ml). Dry the dichloromethane extracts by filtering through a pad of anhydrous sodium sulphate. Evaporate to dryness using a steam bath in a fume hood. Weigh the residue and determine its amitraz content using the method for amitraz technical **362**/TC/M/3.

(*d*) *Calculation*

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

where:
c = mass of amitraz in the sample taken for the preparation of the suspension (g).
Q = mass of in the bottom 25 ml of suspension (g)

AMITRAZ EMULSIFIABLE CONCENTRATES
*362/EC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests

2.1 GLC. As for amitraz technical **362**/TC/M/2.1.

2.2 TLC. Dilute 0.5 g of sample with hexane (10 ml). Continue as for amitraz technical **362**/TC/M/2.2.

3 Amitraz. As for amitraz technical **362**/TC/M/3.

Repeatability r	=	8.1 g/kg at 200 g/kg active ingredient content
Reproducibility R	=	13 g/kg at 200 g/kg active ingredient content

* CIPAC method 1993. Prepared by the Amitraz Panel of PAC-UK. Chairman: D S Wooldridge. Based on a method supplied by Schering Agrochemicals Ltd, UK.