DIAZINON
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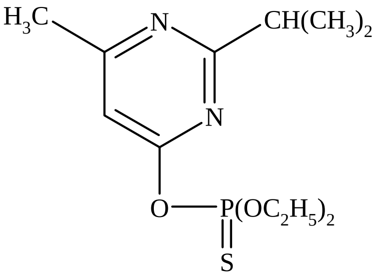

ISO common name	Diazinon
Chemical name	*O,O*-diethyl *O*-2-isopropyl-6-methylpyrimidin-4-yl phosphorothioate(IUPAC); *O,O*-diethyl *O*-{2-(1-methylethyl)-6-methyl-4-pyrimidinyl} phosphorothioate (CA; *333-41-5*)
Empirical formula	$C_{12}H_{21}N_2O_3PS$
RMM	304.3
b.p	83 - 84 °C at 0.3 Pa
v.p.	1.9×10^{-2} Pa at 20 °C
Solubility	In water: 40 mg/l at 20 °C; miscible with ethanol, acetone, and xylene; soluble in petroleum oils
Description	Colourless oil
Stability	Stable under alkaline conditions; slowly hydrolysed by water and dilute acids
Formulations	Wettable powders, emulsifiable concentrates, solutions, and dustable powders

DIAZINON TECHNICAL

[*] **15**/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

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

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

3 Diazinon

OUTLINE OF METHOD Diazinon is determined by gas chromatography on a DC 200 column using flame ionisation detection and internal standardisation.

REAGENTS

Diazinon standard of known purity

HHDN (aldrin) internal standard. Should not contain any impurities that elute at the retention time of diazinon.

Acetone

Internal standard solution. Weigh into a beaker (600 ml) 4.0 ± 0.1 g of HHDN. Slurry with acetone (400 ml) to dissolve and filter through paper into a volumetric flask (1000 ml), washing with several portions of acetone (100 ml). Dilute to volume and mix well.

Calibration solution. Weigh (to the nearest 0.1 mg) into a glass-ground stoppered round bottomed flask (100 ml) about 125 mg (*s* mg) diazinon standard. Add by pipette internal standard solution (50.0 ml), stopper, and shake mechanically for 30 min.

APPARATUS

Gas chromatograph fitted with a flame ionisation detector

Column glass, 1.8 m × 4 mm (i.d.) packed with 10 % DC 200 on 80 to 100 mesh Gas Chrom Q. Condition the column at 240 °C for 24 h using carrier gas at about 40 ml/min.

Electronic integrator or *data system*

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[*] AOAC-CIPAC method 1972.

Mechanical shaker

PROCEDURE

(a) Operating conditions (typical):

Oven temperature	100 ± 10 °C	
Injection port temperature	240 °C	
Detector temperature	240 °C	
Injection volume	3 µl	
Number of theoretical plates	at least 2000	
Flow rate carrier gas	nitrogen or helium, 80 to 100 ml/min	
Flow rates other gases	as recommended for the particular detector	
Retention times	diazinon:	5 to 6 min
	internal standard:	10 to 12 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) into a ground-glass stoppered round bottomed flask (100 ml) enough sample to contain about 250 mg diazinon (*w* mg). Add by pipette internal standard solution (50.0 ml), stopper and shake mechanically for 30 min. Allow any insoluble material to settle, or centrifuge a portion of the solution to obtain a clear solution.

(c) Determination. Inject into the gas chromatograph 3 µl portions of the calibration solution until the peak height ratio of diazinon : HHDN varies by less than 1 % for successive injections. Then make duplicate 3 µl injections of the sample solution followed by duplicate injections of the calibration solution. Peak height ratios must be within 1 % of the first accepted standard values or repeat the the series of injections. Repeat for additional samples. Calculate the peak height ratios for both duplicate injections preceding and following the sample injections. Average the four values (R'). Calculate the average peak height ratios for the two sample injections (R).

(d) Calculation

$$\text{Diazinon content} = \frac{R \times s \times P}{R' \times w} \, 1\text{g/kg}$$

where:

R = diazinon to HHDN peak height ratio for the sample solution
R' = diazinon to HHDN peak height ratio for the calibration solution
s = mass of diazinon in the calibration solution (mg)
w = mass of diazinon in the sample solution (mg)
P = purity of the diazinon standard (g/kg)

DIAZINON WETTABLE POWDERS

[*] **15**/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 Infrared. Extract the sample with acetone, filter and evaporate the solvent in a stream of clean dry air. Continue as for **15**/TC/M/2.1.
2.2 GLC. As for diazinon technical **15**/TC/M/2.2.

3 Diazinon. As for diazinon technical for **15**/TC/M/3.

4 Suspensibility

REAGENTS AND APPARATUS As for MT 15 and diazinon technical for **15**/TC/M/3 together with:
Aluminium sulphate saturated aqueous solution
Light petroleum boiling range 40 - 60 °C
Ethanol
Sodium chloride saturated aqueous solution
Separating funnels 500 ml

PROCEDURE

(a) Preparation of suspension. MT 15.1 *(i).*

(b) Determination of sedimentation. MT 15.1 *(ii).*

(c) Determination of diazinon in the bottom 25 ml of suspension. After removal of the top 225 ml shake the bottom 25 ml of suspension for a short time and transfer to a separating funnel (500 ml) using light petroleum (100 to 150 ml) and ethanol (20 ml). Add aluminium sulphate solution (10 ml) and shake the mixture. Let the layers separate with occasional horizontal swirling of the funnel. Drain the aqueous layer into a second separating funnel containing light petroleum (100 ml). If a stable emulsion is formed between the two phases, separate off the aqueous layer as far as the emulsion, then add sodium chloride solution (10 ml), shake, and run off the aqueous layer completely. Shake the second separating funnel and then allow the layers to settle. Run off the aqueous layer into a third separating funnel and repeat the extraction with light petroleum (100 ml) as above. All the material soluble in

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[*] AOAC-CIPAC method 1972.

light petroleum should have been extracted and the aqueous layer can be discarded. Distil off the solvent and determine the mass (Q g) of diazinon in the residue by **15**/TC/M/3.

(d) Calculation

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

where:

c = mass of active ingredient in sample taken for the preparation of the suspension (g)
Q = mass of active ingredient in the 25 ml remaining in the cylinder (g)

DIAZINON EMULSIFIABLE CONCENTRATES

[*]**15**/EC/M/-

1 Sampling. Take at least 1 l.

2 Identity test. GLC. As for diazinon technical **15**/TC/M/2.2.

3 Diazinon. As for diazinon technical for **15**/TC/M/3.

DIAZINON SOLUTIONS

[*]**15**/SL/M/-

1 Sampling. Take at least 1 l.

2 Identity test. GLC. As for diazinon technical **15**/TC/M/2.2.

3 Diazinon. As for diazinon technical for **15**/TC/M/3.

DIAZINON GRANULES

[*]**15**/GR/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests

2.1 Infrared. Extract the sample with acetone, filter and evaporate the solvent in a stream of clean dry air. Continue as for **15**/TC/M/2.1.

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[*] AOAC-CIPAC method 1973.

2.2 GLC. As for diazinon technical **15**/TC/M/2.2.
3 Diazinon. As for diazinon technical for **15**/TC/M/3.