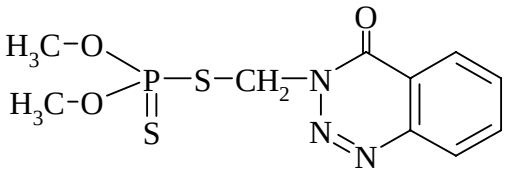
AZINPHOS-METHYL
37(37.a)



ISO common name	Azinphos-methyl
Chemical name	*S*-[3,4-dihydro-4-oxobenzo[d]-(1,2,3)-triazin-3-yl-methyl] O,O-dimethyl phosphorodithioate (IUPAC); O,O-dimethyl S-[(4-oxo-1,2,3-benzo-triazin-3(4*H*)-yl-methyl] phosphorodithioate (CA; *86-50-0*)
Empirical formula	$C_{10}H_{12}N_3O_3PS_2$
RMM	317.3
m.p.	73-74 °C
v.p.	Less than 10^{-3} Pa at 20 °C
d_4^{20}	1.44
Solubility	In water 33 mg/l at 20 °C. Readily soluble in common organic solvents
Description	White crystals
Stability	Rapidly hydrolysed in alkaline media, hydrolysed more slowly in acidic media
Formulations	Emulsifiable concentrates, wettable powders, dustable powders and suspension concentrates

AZINPHOS-METHYL TECHNICAL
*37 (37.a)/TC/(M3)/-

HIGH PRESSURE LIQUID CHROMATOGHRAPHY METHOD

1 Sampling. Take at least 100 g.

2 Identity test. Use the HPLC method below. The relative retention time of azinphos-methyl with respect to the internal standard for the sample solution should not deviate by more than 1% from that for the calibration solution.

3 Azinphos-methyl

OUTLINE OF METHOD The sample is dissolved in acetonitrile and the azinphos-methyl content is determined by reversed phase high performance liquid chromatography, using n-butyrophenone as internal standard.

REAGENTS
Acetonitrile HPLC grade or distilled in glass
Water HPLC grade or distilled in glass
Mobile phase acetonitrile-water, 65 + 35 (v/v)
n-Butyrophenone purity better than 995 g/kg
Internal standard solution. Weigh into a volumetric flask (100 ml) n-butyrophenone (10 g). Dissolve in acetonitrile and dilute to volume.
Azinphos-methyl of known purity, better than 995 g/kg. Store in a refrigerator at -4 to -8 °C.
Calibration solution. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) about 220 mg (*s* mg) pure azinphos-methyl. Pipette internal standard solution (10.0 ml) into the flask, dilute to volume with acetonitrile, and mix thoroughly. Filter a portion of this solution and hold it for HPLC analysis.

APPARATUS
Liquid chromatograph able to generate more than 7 MPa of pressure, and equipped with a spectrophotometric detector measuring at 285 nm, and an integrator or recorder
Chromatographic column stainless steel 250 × 4.6 (i.d.) mm packed with C18 bounded silicagel with a particle size of less than 10 µm (DuPont Zorbax ODS, or equivalent)
Filter 0.45 µm porosity (Gelman Acrodisc-CR, or equivalent)

* Provisional AOAC-CIPAC method 1989.

PROCEDURE

(a) Operating conditions (typical):

Flow rate of mobile phase	1.5 ml (at about 7 MPa)
Column temperature	ambient
Injection volume	10 µl
Detector wavelength	285 nm
Detector sensitivity	0.16 absorbence unit full scale (AUFS)
Chart speed	0.5 cm/min
Column equilibration	Pump mobile phase through the column until the system is equilibrated i.e. until a flat base line is obtained.
Retention times	azinphos-methyl: about 4.0 min internal standard: about 4.5 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) enough sample to contain about 220 mg (*w* mg) of pure azinphos-methyl. Pipette internal standard solution (10.0 ml) into the flask, dilute to volume with acetonitrile, and shake for 1 min. Filter a portion of this solution and hold it for the analysis.

(c) Determination. Adjust the operating parameters so that azinphos-methyl elutes in 3.8 to 4.2 min. Adjust the injection volume and attenuation to give the largest possible on-scale peaks. If the peaks cannot be brought on scale at 0.32 AUFS settings with a 10 µl injection, further dilute the calibration and sample solutions by pipetting 10 ml of each into volumetric flasks (100 ml), diluting to volume with acetonitrile, and mixing thoroughly. Re-adjust the injection volume and the attenuation to give the largest on-scale peaks. After each injection allow 10 min after the internal standard for the elution of formulation excipients.
Using the same injection volume for the sample and the calibration solutions, make repetitive injections of the calibration solution and calculate the response ratios by dividing the peak height of azinphos-methyl by that of internal standard peak (area measurements are not acceptable). The response ratios must agree within 1% before continuing.

Inject in duplicate aliquots of each sample solution (no more than 2 samples [4 injections] between calibration injections). The response ratios of the sample injections must agree within ± 1%. If not, repeat the determination, starting with the injections of the calibration solutions. Re-inject the calibration solution twice. Average the response ratios of the calibration solutions immediately preceding and following the injections of the sample solutions. These must agree within ± 1%. If not, repeat the determination.

(d) Calculation

$$\text{Azinphos-methyl content} = \frac{R \times s \times P}{R' \times w} \quad \text{g/kg}$$

where:

R	=	average peak height ratio of azinphos-methyl to internal standard for the sample solution
R'	=	average peak height ratio of azinphos-methyl to internal standard for the calibration solution
s	=	mass of azinphos-methyl in the calibration solution (mg)
w	=	mass of sample taken (mg)
P	=	purity of standard azinphos-methyl (g/kg)

AZINPHOS-METHYL WETTABLE POWDERS
*37 (37.a)/WP/(M3)/-

1 Sampling. Take at least 500 g.

2 Identity test. As for azinphos-methyl technical **37 (37.a)/**TC/(M3)/2.

3 Azinphos-methyl. As for azinphos-methyl technical **37 (37.a)/**TC/(M3)/3.

Repeatability r = 5.3 g/kg at 477 g/kg active ingredient content

Reproducibility R = 14.9 g/kg at 477 g/kg active ingredient content

AZINPHOS-METHYL DUSTABLE POWDERS
*37 (37.a)/DP/(M3)/-

1 Sampling. Take at least 1 kg.

2 Identity test. As for azinphos-methyl technical **37 (37.a)**/TC/(M3)/2.

3 Azinphos-methyl. As for azinphos-methyl wettable powders, **37 (37.a)**/WP/(M3)/3.

* Provisional AOAC-CIPAC method 1989.

AZINPHOS-METHYL EMULSIFIABLE CONCENTRATES
*37 (37.a)/EC/(M3)/-

1 Sampling. Take at least 500 ml.

2 Identity test. As for azinphos-methyl technical **37 (37.a)**/TC/(M3)/2.

3 Azinphos-methyl. As for azinphos-methyl technical, **37 (37.a)/**TC/(M3)/2.

Repeatability r = 4.8 to 11.5 g/kg at 220 to 230 g/kg active ingredient content

Reproducibility R = 7.0 to 15.7 g/kg at 220 to 230 g/kg active ingredient content

AZINPHOS-METHYL SUSPENSION CONCENTRATES
*37 (37.a)/SC/(M3)/-

1 Sampling. Take at least 1 l.

2 Identity test. As for azinphos-methyl technical **37 (37.a)/**TC/(M3)/2.

3 Azinphos-methyl. As for azinphos-methyl technical, **37 (37.a)/**TC/(M3)/2.

Repeatability r = 15.4 g/kg at 284 g/kg active ingredient content

Reproducibility R = 50 g/kg at 284 g/kg active ingredient content

* Provisional AOAC-CIPAC method 1989.