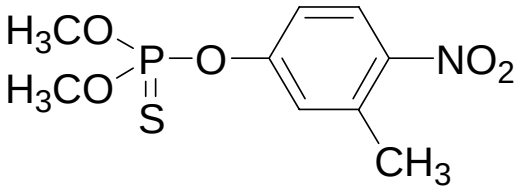
FENITROTHION

35



ISO common name	Fenitrothion
Chemical name	*O,O*-Dimethyl *O*-4-nitro-*m*-tolyl phosphorothioate (IUPAC) *O,O*-Dimethyl *O*-(3-methyl-4-nitrophenyl)-phosphorothioate (CA)
CAS No.	122-14-5
Empirical formula	$C_9H_{12}NO_5PS$
RMM	277.2
b.p.	Fenitrothion begins to decompose at around 210°C.
v.p.	700 mPa (6.0 x 10^{-6} Torr) at 20°C
d_4^{20}	1.308
Reflactive index n_D^{25}	1.5528
Solubility	Practically insoluble in water, soluble in most organic solvents, e.g. acetone, alcohol, chlorinated hydrocarbons
Description	Brownish yellow liquid
Stability	Hydrolyzed by alkali. Do not store at temperatures above 40°C because the material will isomerize

FENITROTHION
35/TC/m3/-

5 TETRAMETHYL PYROPHOSPHOROTHIOATE (TMPP).

OUTLINE OF METHOD The content of TMPP in the test sample is determined by capillary GC using flame ionisation detection and internal standardisation.

REAGENTS

Acetone

n-Heptane

TMPP analytical standard analytical standard of known purity. Store refrigerated.

n-Butyl benzoate internal standard. Must not show any peaks at the same retention time of TMPP.

Internal standard solution. Dissolve *n*-butyl benzoate (100 mg) in *n*-heptane (100 ml). Transfer 10 ml of this solution into a volumetric flask (100 ml) and make up to volume with *n*-heptane. Ensure that a sufficient quantity of this solution is prepared for all samples and calibration standards to be analysed.

Calibration solution Prepare calibration solutions in duplicate. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) approximately 30 mg (*s* mg) of TMPP analytical standard. Make up to volume with acetone. Mix thoroughly. Transfer by pipette 2.0 ml of this solution into a vial (30 ml) and add by pipette 2.0 ml of internal standard solution. Add 16 ml of acetone. Mix thoroughly. (Solutions C_A and C_B).

APPARATUS

Gas chromatograph equipped with a split/splitless injection and a flame ionisation detector.

Capillary column fused silica, 30 m x 0.25 (i.d.) mm, film thickness: 1 μm, coated with crosslinked dimethyl polysiloxane (DB-1 or equivalent)

Electric integrator or data system

PROCEDURE

(a) Gas chromatographic conditions (typical):

Column	fused silica, 30 m x 0.25 (i.d.) mm, film thickness: 1 μm, coated with crosslinked dimethyl polysiloxane (DB-1 or equivalent)
Injection system	
Injector	split injection
Split flow	approximately 20 ml/min

Injection volume	1 µl	
Detector	flame ionisation	
Temperatures		
Column oven	100°C (0 min), ramp at 10°C/min to 300°C, then hold at 300°C for 20 min	
Injection port	200°C	
Detector	310°C	
Carrier gas	helium, 35 cm/sec	
Retention times	*n*-butyl benzoate:	about 11 min
	TMPP:	about 12.5 min

(b) Linearity check. Check the linearity of the detector response by injecting 1 µl of solutions with TMPP concentrations 0.5, 1 and 2 times that of the calibration solution before conducting analysis.

(c) System equilibration. Prepare two calibration solutions. Inject 1 µl portions of the first one until the response factors obtained for two consecutive injections differ by less than 2.0%. Then inject a 1 µl portion of the second solution. The response factor for this solution should not deviate by more than 2.0% from that for the first calibration solution, otherwise prepare new calibration solutions.

(d) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) into a vial (10 ml) sufficient sample to contain about 200 mg (*w* mg) of fenitrothion. Add by pipette 2.0 ml of internal standard solution. Add 3 ml of acetone. Mix thoroughly. Transfer 1 ml of this solution to a vial (10 ml) and add 3 ml of acetone. Mix thoroughly (Solutions S_A and S_B).

(e) Determination. Inject in duplicate 1 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows; calibration solution C_A, sample solution S_A, sample solution S_A, calibration solution C_B, sample solution S_B, sample solution S_B, calibration solution C_A, and so on. Measure the relevant peak areas.

(f) Calculation of TMPP content. Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating TMPP content of the bracketed sample injections.

$$f_i = \frac{I_r \times s \times P}{50 \times H_s}$$

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

where:

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

FENITROTHION WETTABLE POWDER

35/WP/m3/-

5. TMPP. As for **35**/TC/m3/5 except:
add 'APPARATUS' as follows:
Ultrasonic bath

change 'PROCEDURE *(d) Preparation of sample solution.*' as follows:
(d) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) into a vial (10 ml) sufficient sample to contain about 200 mg (*w* mg) of fenitrothion. Add by pipette 2.0 ml of internal standard solution. Add 3 ml of acetone. Place the vials in an ultrasonic bath for 10 min and filter a portion of each sample through a 0.45 μm filter. Transfer 1 ml of this solution to a vial (10 ml) and add 3 ml of acetone. Mix thoroughly (Solutions S_A and S_B).

FENITROTHION EMULSIFIABLE CONCENTRATE

35/EC/m3/-

5. TMPP. As for **35**/TC/m3/5 except:
add 'APPARATUS' as follows:
Ultrasonic bath
Column for solid phase extraction Varian Megabond Elut SI 1g/6mL, or equivalent.

change 'PROCEDURE *(d) Preparation of sample solution.*' as follows:
(d) Preparation of sample solution. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) into a vial (10 ml) sufficient sample to contain about 200 mg (*w* mg) of fenitrothion. Add by pipette 2.0 ml of internal standard solution. Add 3 ml of *n*-heptane. Place the vials in an ultrasonic bath for 10 min and filter a portion of each sample through a 0.45 μm filter. Charge 1 ml of this solution onto Megabond Elut SI column (1 g/6 ml) which is pre-conditioned by eluting 5 ml of acetone and 10 ml of *n*-heptane in this order. Remove formulants in the sample solution by eluting 10 ml of *n*-heptane. Then charge 4 ml of acetone and collect the fraction (Solutions S_A and S_B).

FENITROTHION ULTRA-LOW VOLUME LIQUID
35/UL/m/-

5 TMPP. As for **35**/TC/m3/5

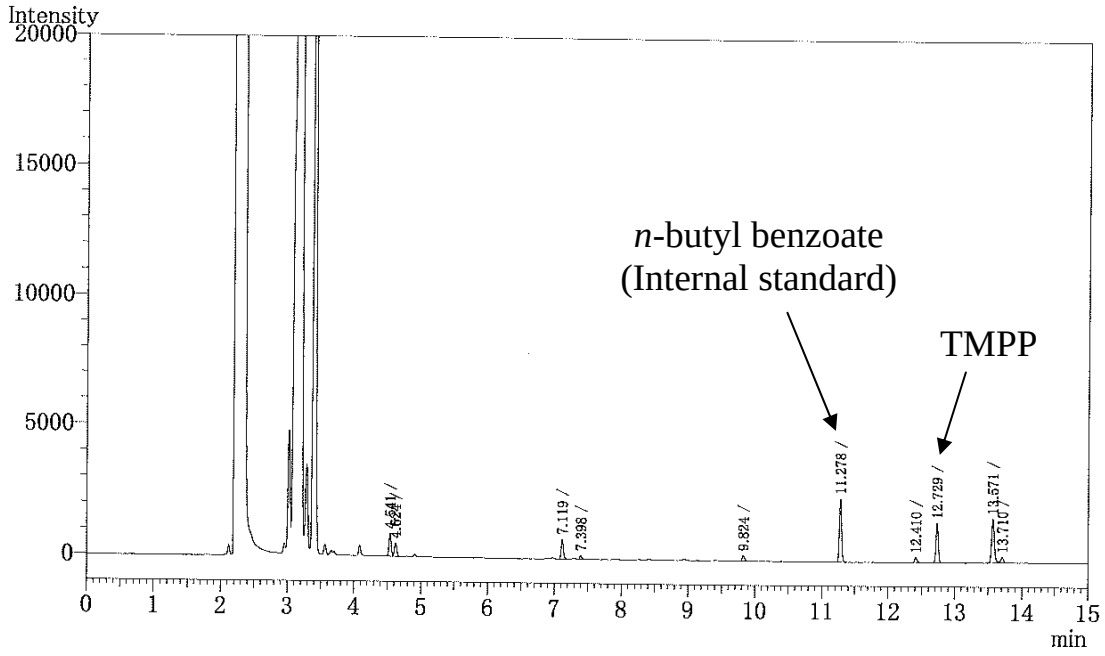


Fig.1 Example of Chromatogram of TMPP