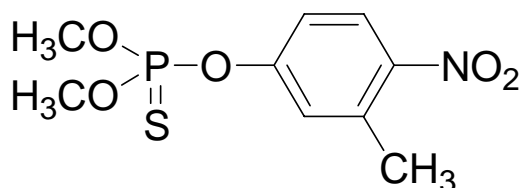


## FENITROTHION

35



<i>ISO common name</i>	Fenitrothion
<i>Chemical name</i>	<i>O,O</i> -Dimethyl <i>O</i> -4-nitro- <i>m</i> -tolyl phosphorothioate (IUPAC) <i>O,O</i> -Dimethyl <i>O</i> -(3-methyl-4-nitrophenyl)-phosphorothioate (CA)
<i>CAS No.</i>	122-14-5
<i>Empirical formula</i>	C <sub>9</sub> H <sub>12</sub> NO <sub>5</sub> PS
<i>RMM</i>	277.2
<i>b.p.</i>	Fenitrothion begins to decompose at around 210°C.
<i>v.p.</i>	700 mPa (6.0 x 10 <sup>-6</sup> Torr) at 20°C
<i>d</i> <sub>4</sub> <sup>20</sup>	1.308
<i>Refractive index n</i> <sub>D</sub> <sup>25</sup>	1.5528
<i>Solubility</i>	Practically insoluble in water, soluble in most organic solvents, e.g. acetone, alcohol, chlorinated hydrocarbons
<i>Description</i>	Brownish yellow liquid
<i>Stability</i>	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<sub>A</sub> and C<sub>B</sub>).

#### 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 $\mu$ l
<i>Detector</i>	flame ionisation
<i>Temperatures</i>	
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
<i>Carrier gas</i>	helium, 35 cm/sec
<i>Retention times</i>	<i>n</i> -butyl benzoate: about 11 min TMPP: about 12.5 min

(b) *Linearity check.* Check the linearity of the detector response by injecting 1  $\mu$ l 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  $\mu$ l portions of the first one until the response factors obtained for two consecutive injections differ by less than 2.0%. Then inject a 1  $\mu$ l 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<sub>A</sub> and S<sub>B</sub>).

(e) *Determination.* Inject in duplicate 1  $\mu$ l portions of each sample solution bracketing them by injections of the calibration solutions as follows; calibration solution C<sub>A</sub>, sample solution S<sub>A</sub>, sample solution S<sub>A</sub>, calibration solution C<sub>B</sub>, sample solution S<sub>B</sub>, sample solution S<sub>B</sub>, calibration solution C<sub>A</sub>, 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  $\mu\text{m}$  filter. Transfer 1 ml of this solution to a vial (10 ml) and add 3 ml of acetone. Mix thoroughly (Solutions S<sub>A</sub> and S<sub>B</sub>).

## 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<sub>A</sub> and S<sub>B</sub>).

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

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

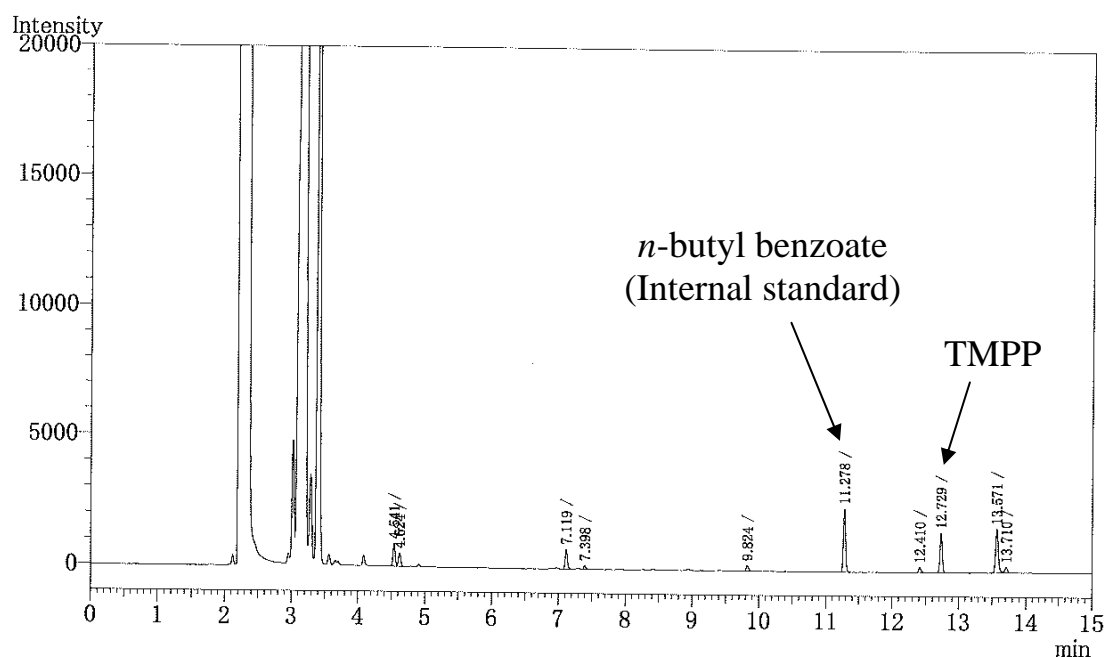


Fig.1 Example of Chromatogram of TMPP