



## Temephos

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Method number:	PV2056
Target Concentration:	10 mg/m <sup>3</sup> total dust 5 mg/m <sup>3</sup> respirable dust
Tested Concentration:	5mg/m <sup>3</sup> with a sampling rate of 1.0 L/min
Procedure:	Samples are collected by drawing known volumes of air through glass fiber filters. The filters are extracted with acetonitrile and analyzed by high performance liquid chromatography (HPLC) using an ultraviolet detector (UV).
Recommended air Volume and sampling rate:	60 minutes at 1.0 L/min (60 L)
Detection limit of the overall procedure	0.14 mg/m <sup>3</sup> (based on the recommended air volume and the analytical detection limit):
Status of method:	Partially Validated method. This method has been partially evaluated and is presented for information and trial use only.

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## 1 General Discussion

### 1.1 Background

#### 1.1.1 History of procedure

This evaluation was undertaken because OSHA recently adopted the temephos TLV as a PEL. Glass fiber filters were tested as an effective sampling device for temephos.

#### 1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.)

The oral LD<sub>50</sub> in male and female rats is 8600 and 13000 mg/kg respectively. (Ref. 5.1) All animal species tested tolerated 10 mg/kg without clinical effect, and 1 mg/kg without effect on cholinesterase activity (Ref. 5.2). Humans have tolerated an oral dose of 256 mg/man/day for 5 days or 64 mg/man/day for four weeks without detected effects on plasma cholinesterase or red blood cells. (Ref. 5.2)

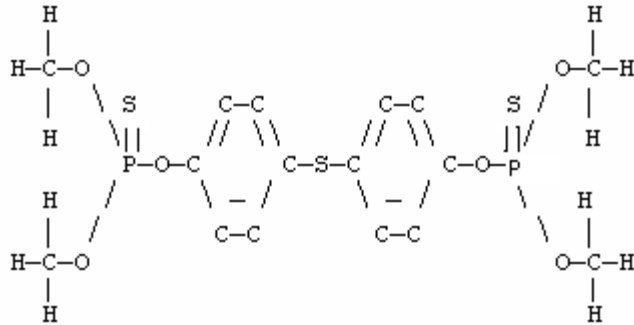
#### 1.1.3 Potential workplace exposure

Temephos is a cholinesterase inhibiting insecticide. No information was available on the number of workers that may be exposed to temephos. There was an estimated  $1.5 \times 10^8$  grams produced in 1972 by the U.S.

#### 1.1.4 Physical properties (Ref. 5.1 to 5.4)

CAS number:	3383-96-8
IMIS number:	T195
Molecular weight:	466.46
Molecular formula:	C <sub>16</sub> H <sub>20</sub> O <sub>6</sub> P <sub>2</sub> S <sub>3</sub>
Melting point:	30.0 - 30.5 °C
Solubility:	Soluble in acetonitrile, carbon tetrachloride, ether, dichloroethane, and toluene; almost insoluble in water and hexane; insoluble in methylcyclohexane
Chemical name:	O,O'-(thiodi-4, 1-phenylene) bis (O,O-dimethyl phosphorothioate)
Synonyms:	O,O'-(thiodi-4, 1-phenylene) phosphorothioic acid O,O,O,O'-tetramethyl ester; Abat; Abate; Abathion; AC 52160; American Cyanamid CL-52160; American Cyanamid E.I. 52,160; Biothion; Bithion; CL 52160; Difenthos; O,O-dimethyl phosphorothioate O,O-diester with 4,4'-thiodiphenol; Ecopro; EI 52160; ENT 27,165; Experimental Insecticide 52160; Nimitex; Nimitox; phenol, 4,4'-thiodi-, O,O-diester with O,O-dimethyl phosphorothioate; phosphorothioic acid, O,O'-(thiodi-4,1-phenylene) O,O,O',O'-tetramethyl ester; Swebate; Temefos; Temephos (ACGIH, OSHA); Temophos; tetramethyl-o,o'-thiodi-p-phenylene phosphorothioate; O,O,O',O'-tetramethyl O,O'-thiodi-p-phenylene phosphorothioate; O,O'-(thiodi-4,1-phenylene)bis(O,O-dimethyl phosphorothioate); O,O'-(thiodi-p-phenylene) O,O,O',O'-tetramethyl bis(phosphorothioate)
Description:	white crystalline solid
UV Scan:	Figure 1.

Structure:



## 1.2 Limit defining parameters

The detection limit of the analytical procedure is 2.0 ng per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise. (Figure 2.)

## 2 Sampling Procedure

### 2.1 Apparatus

- 2.1.1 A personal sampling pump that can be calibrated to within  $\pm 5\%$  of the recommended flow rate with the sampling device in line.
- 2.1.2 Glass fiber filters, 37-mm diameter, Gelman Type A or equivalent.
- 2.1.3 Cassette filter holders for 37-mm filters, Millipore M000037A0, or equivalent.

### 2.2 Reagents

No sampling reagents are required.

### 2.3 Sampling technique

- 2.3.1 Immediately before sampling, remove the plastic plugs from the cassette.
- 2.3.2 Attach the cassette to the sampling pump with flexible tubing.
- 2.3.3 Attach the cassette vertically in the employee's breathing zone in such a manner that it does not impede work performance.
- 2.3.4 After sampling for the appropriate time, remove the cassette and seal with plastic plugs.
- 2.3.5 Wrap each sample end-to-end with a Form OSHA-21 seal.
- 2.3.6 Record the air volume for each sample, and list any possible interference.
- 2.3.7 Submit at least one blank for each set of samples. Handle the blank in the same manner as the samples, except no air is drawn through it.
- 2.3.8 Submit bulk samples for analysis in a separate container. Do not ship with air samples.

## 2.4 Extraction efficiency

Twenty-four glass fiber filters were each liquid spiked with 12  $\mu\text{L}$  of a 25.47 mg/mL solution of temephos. These samples were allowed to equilibrate overnight in a drawer at ambient temperature. The next day six samples were each desorbed with 4.0 mL of acetonitrile, shaken for 30 min and analyzed as in Section 3. The results are listed in Table 2.4.

Table 2.4  
Extraction Efficiency

sample #	$\mu\text{g}$ spiked	$\mu\text{g}$ found	% recovered
1	305.6	292.7	95.8
2	305.6	288.9	94.5
3	305.6	286.9	93.9
4	305.6	294.8	96.5
5	305.6	303.4	99.3
6	305.6	312.4	102.2

average = 97.0%

## 2.5 Retention efficiency

The remaining eighteen-spiked glass fiber filters from Section 2.4. were placed on a humid air generator and 60 L of humid air (~78% relative humidity) were drawn through each filter at 1 L/min. Six of the filters were each desorbed with 4.0 mL of acetonitrile, shaken for 30 min and then analyzed as in Section 3. The results are listed in Table 2.5. The remaining samples were stored, six in a drawer at ambient temperature and six in a freezer, for use in the storage study below in Section 2.6.

Table 2.5  
Retention Efficiency

sample #	$\mu\text{g}$ spiked	$\mu\text{g}$ found	% recovered
1	305.6	294.2	96.3
2	305.6	290.5	95.1
3	305.6	294.3	96.3
4	305.6	310.0	101.4
5	305.6	309.2	101.2
6	305.6	294.8	96.5

average = 97.8%

## 2.6 Sample storage

After four days of storage, six samples were each desorbed with 4.0 mL of acetonitrile, shaken for 30 min and then analyzed as in Section 3. Three of the samples were from ambient storage and the other three were from the freezer storage samples. The remaining samples were analyzed after seven days of storage. The results are given in Tables 2.6.1 and 2.6.2.

Table 2.6.1  
Ambient Storage

sample #	µg spiked	µg found	% recovered
4	305.6	342.4	112.0
4	305.6	296.0	96.9
4	305.6	305.6	100.0
7	305.6	298.7	97.7
7	305.6	311.2	101.8
7	305.6	293.9	96.2

average of 4 days = 103%

average of 7 days = 98.6%

Table 2.6.2  
Freezer Storage

sample #	µg spiked	µg found	% recovered
4	305.6	304.0	99.5
4	305.6	297.5	97.3
4	305.6	290.6	95.1
7	305.6	306.8	100.4
7	305.6	284.1	93.0
7	305.6	302.4	99.0

average of 4 days = 97.3%

average of 7 days = 97.5%

## 2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 60 L.

2.7.2 The recommended flow rate is 1.0 L/min.

## 2.8 Interferences (sampling)

It is not known if any compounds will interfere with the collection of temephos. Any suspected interferences should be reported to the laboratory.

## 2.9 Safety precautions (sampling)

2.9.1 Attach the sampling equipment in such a manner that it will not interfere with work performance or employee safety.

2.9.2 Follow all safety practices that apply to the work area being sampled.

## 3 Analytical Procedure

### 3.1 Apparatus

3.1.1 A balance capable of weighing to the nearest tenth of a milligram. A Mettler HL52 balance was used in this evaluation.

- 3.1.2 A mechanical shaker.
- 3.1.3 An HPLC equipped with a UV detector. A Hewlett Packard (HP) 1090M equipped with an autosampler and diode array detector was used in this evaluation.
- 3.1.4 An HPLC column capable of separating temephos from any interference. A 100-mm × 2.1-mm i.d. Hypersil ODS liquid chromatography column was used in this evaluation.
- 3.1.5 An electronic integrator or some other suitable means for measuring detector response. The Hewlett-Packard 1090M Data System was used in this evaluation.
- 3.1.6 Volumetric flasks and pipettes.
- 3.1.7 Vials, 2-mL and 20-mL.

### 3.2 Reagents

- 3.2.1 Acetonitrile, HPLC grade. This was obtained from Burdick and Jackson for this evaluation.
- 3.2.2 Temephos, reagent grade. A standard obtained from EPA (EPA # 0020, 98.34% purity) was used in this evaluation.
- 3.2.3 Water, HPLC grade, Milli-Q filtered water, Millipore Inc.

### 3.3 Standard preparation

Prepare temephos stock standards by weighing 10 to 15 mg of temephos. Transfer the temephos to separate 10-mL volumetric flasks, and add acetonitrile to the mark. Make working range standards of 2.0 to 185 µg/mL by diluting the stock standards with acetonitrile. Store stock and diluted standards in a freezer.

### 3.4 Sample preparation

- 3.4.1 Transfer the glass fiber filter to a 20-mL vial.
- 3.4.2 Add 4.0 mL of acetonitrile to each vial and seal with a PTFE-lined cap.
- 3.4.3 Shake the vials for 30 minutes on a mechanical shaker.
- 3.4.4 If necessary, transfer the samples to 2-mL vials for use on an HP autosampler.

### 3.5 Analysis

#### 3.5.1 Instrument conditions

Column:	100-mm × 2.1-mm Hypersil ods
Mobile phase:	60% acetonitrile/40% water
Flow rate:	0.3 mL/min
Wavelength:	200 nm
Retention time:	6.1 min
Injection volume:	1.0 µL

#### 3.5.2 Chromatogram (Figure 3.)

### 3.6 Interferences (analytical)

- 3.6.1 Any collected compound having a similar retention time to that of the analyte is a potential interference.
- 3.6.2 HPLC conditions may generally be varied to circumvent interferences.
- 3.6.3 Retention time on a single column is not proof of chemical identity. Analysis on an alternate HPLC column and confirmation by mass spectrometry are additional means of identification.

### 3.7 Calculations

- 3.7.1 Construct a calibration curve (Figure 4.) by plotting detector response versus concentration ( $\mu\text{g/mL}$ ) of temephos.
- 3.7.2 Determine the  $\mu\text{g/mL}$  of temephos in each sample and blank from the calibration curve.
- 3.7.3 Blank correct each sample by subtracting the  $\mu\text{g/mL}$  found in the blank from the  $\mu\text{g/mL}$  found in the sample.
- 3.7.4 Determine the air concentration by using the following formula.

$$\text{mg} / \text{m}^3 = \frac{(\mu\text{g} / \text{mL, blank corrected})(\text{desorption volume, mL})}{(\text{air volume, L})(\text{desorption efficiency, decimal})}$$

### 3.8 Safety precautions (analytical)

- 3.8.1 Avoid skin contact and air exposure to temephos.
- 3.8.2 Avoid skin contact with all solvents.
- 3.8.3 Wear safety glasses at all times.

## 4 Recommendation for Further Study

This method should be fully validated.

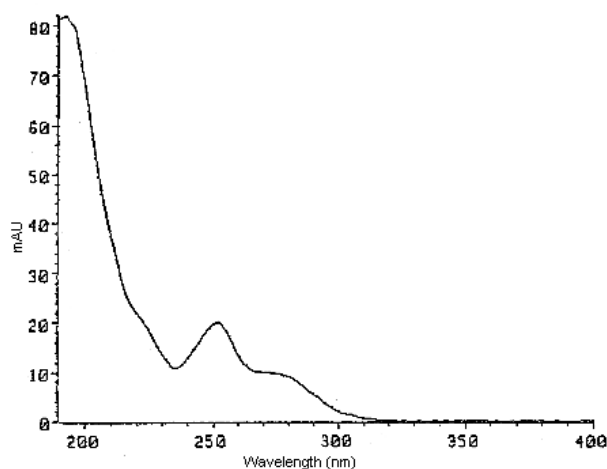


Figure 1.  
UV Scan of Temephos in Mobile Phase

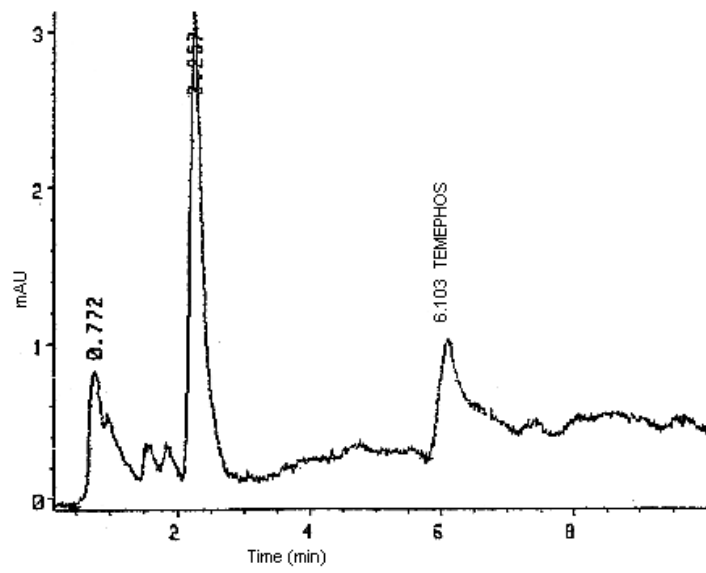


Figure 2.  
Detection Limit Chromatogram of Temephos

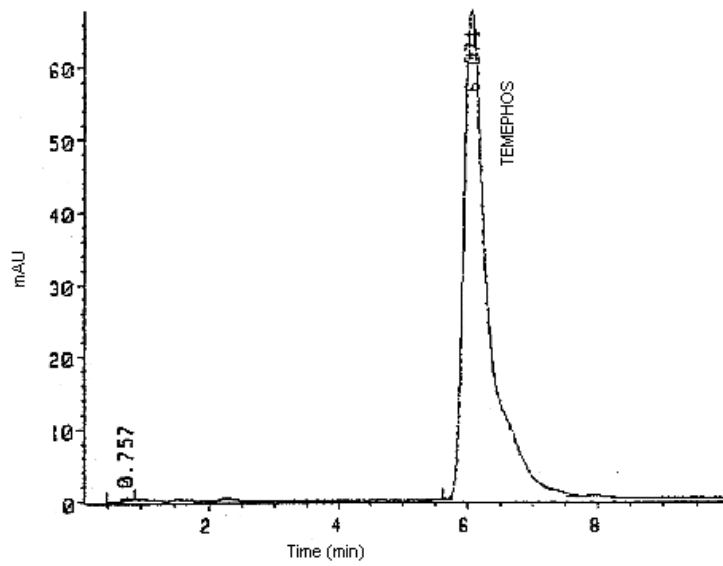


Figure 3.  
Chromatogram of Temephos



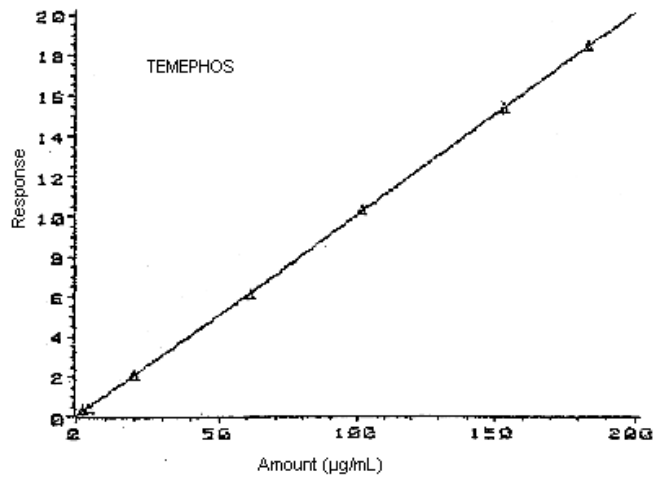


Figure 4.  
Calibration Curve

## 5 References

- 5.1 Merck Index, 10th ed.; Windholz, Martha Ed.; Merck: Rahway, NJ, 1983; pp 1308-1309.
- 5.2 Documentation of Threshold Limit Values and Biological Exposure Indices; American Conference of Governmental Industrial Hygienists Inc., Fifth Edition, 1986, p 557.
- 5.3 Registry of Toxic Effects of Chemical Substances 1985-86 Edition; DHHS (NIOSH) Publication No. 87-114, U.S. Department of Health and Human Services: Cincinnati, OH, 1987; p 3433.
- 5.4 Farm Chemicals Handbook; Berg, Gordon L. Ed.; Meister: Willoughby, OH, 1989; p C5.