Method no.:	PV2105
Target concentration:	0.1 mg/m^3 ACGIH TLV. There is no OSHA PEL for disulfoton.
Procedure:	Samples are collected by drawing known volumes of air through OSHA versatile sampler (OVS-2) tubes, containing a glass fiber filter and two sections of XAD-2 adsorbent. Samples are extracted with toluene and analyzed by gas chromatography (GC) using a flame photometric detector (FPD).
Recommended air volume and sampling rate:	480 minutes a 1.0 L/min (480 Liters)
Detection limit of the overall procedure based on the recommended air volume:	0.0020 mg/m^3
Status of method:	Partially Validated. This method has been only partially evaluated and is presented for information and trial use.

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1 General Discussion

1.1 Background

1.1.1 History of procedure

This evaluation was undertaken to determine the effectiveness of the OVS-2 sampling tube as a sampling device for disulfoton. It follows the procedure developed for several other organophosphorus pesticides. (Ref. 5.1)

It should be noted that in this evaluation for disulfoton several other analytes were also present in the analytical procedure. These other analytes are not mentioned in this evaluation, but can be seen on the sample chromatogram.

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

Organophosphorus pesticides act as irreversible inhibitors of cholinesterase, thereby allowing the accumulation of large amounts of acetylcholine. When a critical level of cholinesterase depletion is reached, usually about 20% of normal, symptoms and signs of acetylcholine accumulation poisoning become manifest. (Ref. 5.2)

These symptoms may include blurred vision, weakness, nausea, headache, abdominal cramps, chest discomfort, and diarrhea. Signs may include miosis, muscle twitching, salivation, sweating, tearing, cyanosis, convulsions, and coma. (Ref. 5.2) Besides being absorbed following inhalation or ingestion, organophosphorus pesticides are readily absorbed through, the intact skin (Ref. 5.2). Disulfoton is a highly toxic chemical with an acute oral LD_{50} for male rats of 6.8 mg/kg and females rates of 2.3 mg/kg. The dermal LD_{50}'s are 25 and 6 mg/kg for male and female rats respectively. (Ref.5.3) Due to these factors, disulfoton has a TLV of 0.1 mg/m3 by the ACGIH. (ref. 5.3)

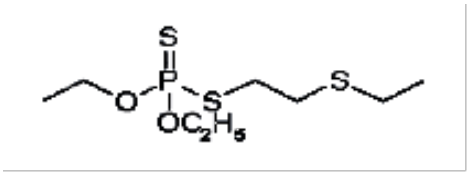
1.1.3 Potential workplace exposure

No estimate of worker exposure to disulfoton was found. Disulfoton is used as a systemic insecticide and acaricide.

1.1.4 Physical properties (Refs. 5.3-5.6)

CAS #:	298-04-4
Molecular weight:	274.38
Molecular formula:	$C_8H_{19}O_2PS_3$
Boiling point:	132-133 °C at 1.5 mmHg
Vapor Pressure:	0.00018 mmHg at 20 °C
Appearance:	colorless oily liquid
Solubility:	insoluble in water, soluble in most organic solvents
Synonyms:	Disyston, dithiodemeton, dithiosystox, dithios
Chemical name:	O,O-Diethyl-S-2-(ethylthio) ethyl phosphorodithioate

Structure:



1.2 Limit defining parameters

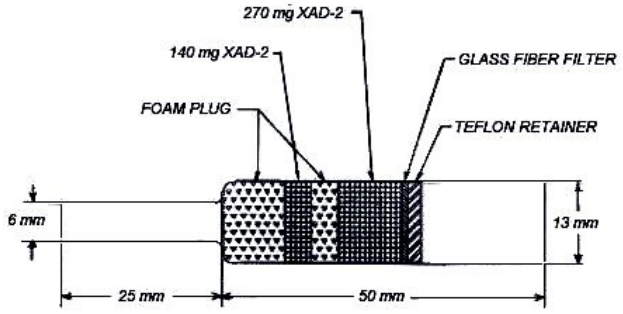
The detection limit of analytical procedure is 0.6 ng per injection. This is the amount of analyte, which will give a peak whose height is approximately five times the baseline noise.

2 Sampling procedure

2.1 Apparatus

2.1.1 Samples are collected using a personal sampling pump that can be calibrated to within ±5% of the recommended flow rate with the sampling device in line.

2.1.2 Samples are collected with OVS-2 tubes, which are specially made 13-mm o.d. glass tubes that are tapered to 6-mm o.d., packed with a 140-mg backup section and a 270-mg sampling section of cleaned XAD-2 and a 13-mm diameter glass fiber filter. The backup section is retained by two foam plugs and the sampling section is between one foam plug and the glass fiber filter. The glass fiber filter is held next to the sampling section by a polytetrafluoroethylene (PTFE) retainer.



2.2 Reagents

No sampling reagents are required.

2.3 Sampling technique

2.3.1 Attach the small tubing of the sampling tube to the sampling pump with flexible, plastic tubing such that the large, front section of the sampling tube is exposed directly to the atmosphere. Do not place any tubing in front of the sampler. The sampler should be attached vertically (large end down) in the worker's breathing zone in such a manner that it does not impede work performance.

2.3.2 After sampling for the appropriate time, remove the sampling device and seal the tube with plastic end caps.

2.3.3 Wrap each sample end-to-end with a Form OSHA-21 seal.

2.3.4 With each set of samples, submit at least one blank. The blank should be handled the same as the other samples except that no air is drawn through it.

2.3.5 Bulk samples should be submitted for analysis in a separate container. Do not ship with the air samples.

2.4 Extraction efficiency

Two 13-mm glass fiber filters were each spiked with 48.06 µg of disulfoton. The two filters, along with a blank filter, were each extracted with 2 mL of toluene in separate 4-mL vials which also contained 270 mg of XAD-2 adsorbent. The average extraction efficiency for these two filters (with the XAD-2 adsorbent present, also) was 89%.

2.5 Retention efficiency

Two OVS-2 tubes were each spiked with 48.06 µg of disulfoton by spiking the 13-mm glass fiber filter in the tube with the analyte of interest. 350 liters of humid air was drawn through each filter. The two filters were then extracted as above. The average retention efficiency for these two filters was 8O%.

2.6 Sample storage

Two OVS-2 tubes were each spiked with 48.06 µg of disulfoton as above. 470 liters of humid air was drawn through each filter. These two tubes were stored for ten days at ambient temperature in a drawer. They were then extracted as above. The average recovery after ten days of storage was 77%.

2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 480 L.

2.7.2 The recommended flow rate is 1.0 L/min.

2.8 Interferences

It is not known if any compounds will interfere with the collection of disulfoton. Suspected interferences should be reported to the laboratory with submitted samples.

2.9 Safety precautions

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 GC equipped with an FPD detector operating in the phosphorus mode. A Hewlett-Packard 5730A GC fitted with an FPD was used in this evaluation. Injections were performed using a Hewlett-Packard 7671A automatic sampler.

3.1.2 A GC column capable of resolving disulfoton from any interference. A 30-m × 0.53-mm i.d. (1.0 µm d_f DB-210) Megabore capillary column, was used in this evaluation and is available from J&W Scientific, Inc., Rancho Cordova, CA.

3.1.3 An electronic integrator or other suitable means of measuring detector response. A Hewlett-Packard 3357 data system was used in this evaluation.

3.1.4 Vials, 2-mL and 4-mL glass with PTFE-lined septa.

3.1.5 Volumetric flasks, pipettes, and syringes for preparing standards, making dilutions, and performing injections.

3.2 Reagents

3.2.1 Hydrogen, air, oxygen, and nitrogen, GC grade.

3.2.2 Toluene. Pesticide grade.

3.2.3 Disulfoton, 98.1% pure (Chem Services Inc.).

3.3 Standard preparation

Stock standard solutions are prepared by adding toluene to preweighed amounts of disulfoton. Working range standard solutions are prepared by diluting stock solutions with toluene. Stock and dilute standards are stored in a freezer.

3.4 Sample preparation

3.4.1 Transfer the 13-mm glass fiber filter and the 270-mg section of the sampling tube to a 4-mL vial. Place the first foam plug and the 140-mg section in a separate vial. A small glass funnel can be used to facilitate the transfer of the adsorbent. Discard the rear foam plug. Do not discard the glass sampling tube; it can be reused after it has been cleaned with surfactant or suitable solvent.

3.4.2 Add 2.0 mL of toluene to each vial.

3.4.3 Seal the vials with PTFE-lined septa and allow them to extract for one hour. Shake the vials by hand with vigorous force periodically during the one-hour extraction time.

3.5 Analysis

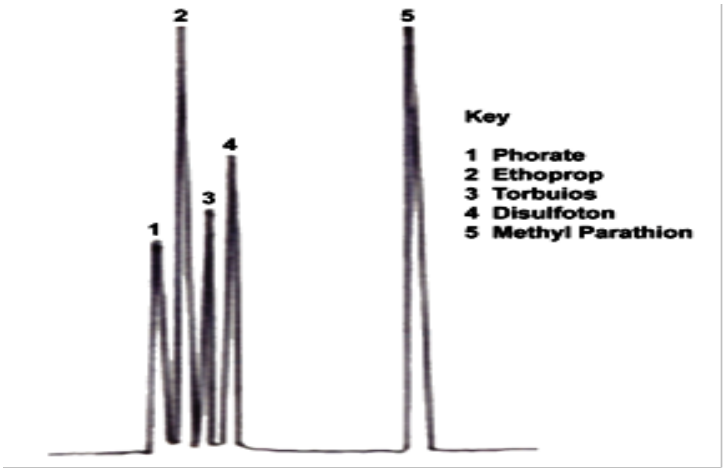
3.5.1 GC conditions

GC column:	30-m × 0.53-mm i.d., (1.0-µm d_f DB-21) Megabore capillary
Injector temperature:	200 °C
Detector temperature:	300 °C
Column temperature: (initial)	150 °C
Initial hold time:	0 min
Temperature program rate:	8 °C/min
Column temperature: (final)	200 °C
Final hold time:	4 min

FPD conditions

Nitrogen flow rate:	5 mL/min
Hydrogen flow rate	200 mL/min
Oxygen flow rate:	60 mL/min
Airflow rate:	30 mL/min
Injection volume:	1.3 µL
Retention time:	6.0 min

3.5.2 Chromatogram



3.6 Interferences

3.6.1 Any compound having a similar retention time to the analyte is a potential interference. Generally, chromatographic conditions can be altered to separate interference from the analyte.

3.6.2 Retention time on a single column is not proof of chemical identity. Analyses by an alternate GC column, detection by an FPD in the sulfur mode for the sulfur containing pesticides, and confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

3.7.1 A calibration curve is constructed by plotting detector response versus standard concentration.

3.7.2 The concentration of disulfoton in a sample is determined from the calibration curve. If disulfoton is found on the backup section, it is added to the amount found on the front section. Blank corrections for each section should be performed before adding the results together.

3.7.3 The air concentration is then determined by the following formula.

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

3.8 Safety precautions

3.8.1 Avoid exposure to all standards.

3.8.2 Avoid exposure to all solvents.

3.8.3 Wear safety glasses at all times.

4 Recommendations for further study

There appears to be some loss of disulfoton with increased sampling time and/or storage time at ambient temperature. More statistically valid retention and storage studies should be done to clarify this loss of analyte. This method should be fully validated.

5 References

5.1 Burright, D., Method #62, "Clorphyrifos, DDVP, Diazinon, Malathion, and Parathion," OSHA Analytical Laboratory, unpublished, 1986.

5.2 "OCCUPATIONAL DISEASES, A Guide to their Recognition", U.S. Department of Health, Education, and Welfare; Public Health Service, Public Heath Service Publication No. 1097, U.S. Government Printing Office, Washington, D.C., 1965.

5.3 "Documentation of the Threshold Limit Values and Biological Exposure Indices," American Conference of Governmental Industrial Hygienists Inc., fifth edition, 1986.

5.4 Farm Chemicals Handbook, Meister Publishing Co., 1985.

5.5 Windholz, M., Ed. "Merck Index," 10th ed.; Merck and Co., Rahway, NJ, 1983.

5.6 "Chemical Information File", U.S. Department of Labor, Occupational Safety and Health Administration, Directorate of Science, Technology and Medicine, June 14, 1985.