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IX. APPENDIX I

SAMPLING METHOD FOR METHYL PARATHION

The recommended sampling method is based on the methods described by Miles et al, [167] and on that appearing in the NIOSH Manual of Analytical Methods. [82] As stated previously in Chapter IV, the sampling efficiency and the overall precision of the recommended sampling and analytical method are not completely known. In addition, the Environmental Protection Agency has withdrawn the impinger (with ethylene glycol) sampling method from its pesticide manual (RH Hill Jr, written communication, March 1976). However, the recommended method remains the best one presently available for collecting and determining the concentration of methyl parathion in air.

Atmospheric Sampling

When sampling is performed for determination of compliance with the recommended workplace air standard, the sample shall be taken within the breathing zone of the exposed employee to ascertain the employee's actual exposure to airborne methyl parathion. A description of sampling location and conditions, equipment used, time and rate of sampling, and any other pertinent information shall be recorded at the time of the sample collection.

(a) Equipment

The sampling train consists of a midget impinger filled with 15 ml of ethylene glycol, an absorption tube, and an air pump.

(1) Midget impinger: All portions of the impinger which may contact the collection medium or the air stream before collection is effected must be made of glass. The collection medium is ethylene glycol. The ethylene glycol used must be free of substances that will produce interfering peaks upon hexane extraction and subsequent gas-liquid chromatographic (GLC) analysis. Consequently, the only ethylene glycol suitable is that which has been preextracted and found to be free of interfering substances by GLC with a flame photometric detector.

(2) Absorption tube: An absorption tube, loosely packed with a plug of glass wool, is inserted between the exit arm of the impinger and the air pump to protect against splash-over or water condensation.

(3) Air pump: Any air mover capable of drawing the desired flowrate through the impinger may be used, so long as the flowrate does not vary more than ± 5% during the sampling period. The sampling pump must be capable of operating at a pressure drop of 1 inch of mercury while providing the designated flowrate of 2.8 liters/minute. The flowrate of the pump must be calibrated and this calibration checked periodically to ensure that it has not changed.

(b) Calibration

Since the accuracy of an analysis can be no greater than the accuracy of the air volume measurement, the accurate calibration of a sampling pump is essential. How often the calibration must be performed is dependent on the use, care, and handling of the pump. Pumps should also be recalibrated if they have been misused or if they have just been repaired or received from a manufacturer. If the pump receives hard usage, more frequent calibration may be necessary. Regardless of use, maintenance and

calibration should be performed on a regular schedule and records of these kept.

Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, primary standards such as a spirometer or soapbubble meter are recommended, although other standard calibration instruments (such as a wet-test meter or dry-gas meter) can be used. The actual setups will be similar for all instruments.

Instructions for calibration with the soapbubble meter appear below. If another calibration device is selected, equivalent procedures should be used. Since the flowrate given by a pump is dependent on the pressure drop of the sampling device, in this case a midget impinger, the pump must be calibrated while operating with a representative midget impinger in line. The calibration train thus consists of a soapbubble meter, a midget impinger, a pressure gauge capable of measuring 20 inches of water, and an air pump.

(1) The voltage of the pump battery is checked with a voltmeter to ensure adequate voltage for calibration. The battery is charged if necessary.

(2) The pump is turned on. Then the inside of the soapbubble meter is moistened by immersing the buret in the soap solution and drawing bubbles up the inside until they are able to travel the entire buret length without bursting.

(3) The pump rotameter is adjusted to provide the desired flowrate.

(4) A water manometer is checked to ensure that the pressure drop across the sampling train is maintained at approximately 12 inches of water at 2.8 liters/minute.

(5) A soapbubble is started up the buret, and the time it takes the bubble to move from one calibration mark to another is measured with a stopwatch.

(6) The procedure in (5) is repeated at least twice, the results averaged, and the flowrate calculated by dividing the volume between the preselected marks by the time required for the soapbubble to traverse the distance.

(7) Calibration data which are recorded include the volume measured, elapsed time or number of strokes, pressure drop, air temperature, atmospheric pressure, serial number of the pump, date, and name of the person performing the calibration.

(c) Sampling Procedure

Breathing zone samples representative of the individual employee's respiratory exposure are collected with the midget impinger by fastening the impinger to a coat lapel or shirt collar, or by holding the impinger near the face of the employee during the sampling period. The duration of sampling shall be such that a concentration of 10% of the recommended environmental limit specified in Chapter I, Section 1(a), may be detected accurately by the recommended analytical method. An air sample of at least 50 liters must be taken. The temperature and pressure of the atmosphere being sampled are measured and recorded.

After a sample is taken, the impinger stem is removed and washed with 2-5 ml of ethylene glycol. This wash solution is included in the impinger, and the amount of washing solution recorded. The top of the impinger is sealed tightly with a plastic stopper. The impinger is placed upright in a carrying case, with care taken to prevent losses because of spillage or evaporation. The trapped methyl parathion (and methyl paraoxon, if present) is extracted into hexane and analyzed as described in Appendix II. Other collection methods shown to be equivalent or superior may be used.

X. APPENDIX II

ANALYTICAL METHOD FOR METHYL PARATHION

The method presented in the NIOSH Manual of Analytical Methods [82] for analysis of parathion (ethyl) in air is recommended for the analysis of methyl parathion.

Principle of the Method

Methyl parathion in workplace air is trapped in ethylene glycol contained in a midget impinger. The ethylene glycol solution is diluted with water and extracted with hexane. The resulting solution of methyl parathion in hexane is concentrated and subjected to GLC analysis using a phosphorus-specific flame photometric detector.

Range and Sensitivity

The linear range of the flame photometric detector is 0.5-25 ng for methyl parathion. For a 50-liter air sample, carried through the following procedure to solution in 5 ml of hexane, 2 μl of which is injected into the GLC, the range of workplace air concentrations over which analysis is linear is 25-1,250 μg/cu m. These limits can be lowered or raised by changing (1) the volume of air sampled, (2) the volume of the final hexane solution, or (3) the size of the aliquot injected into the GLC.

Interferences

Phosphorus compounds having retention times close to that of methyl parathion will interfere with the analysis. The equipment used must be scrupulously cleaned to remove any traces of phosphate detergents. Glassware should, in addition, be rinsed with hexane immediately prior to use.

Advantages and Disadvantages

(a) The method is very sensitive, and the detector exhibits high specificity for phosphorus compounds. The analysis is performed directly on the compound of interest. Separation and quantification are accomplished in a reasonable amount of time.

(b) The cost of the equipment and supplies may tax the budget of some laboratories. The sensitivity of the equipment depends on careful adjustment of the operating parameters. Contamination can occur easily through equipment and reagents. If interfering compounds are anticipated, a lengthy cleanup procedure is required.

Apparatus

(a) Forceps.

(b) Glass stirring rods.

(c) Separatory funnels, 125-ml.

(d) Beakers, 100-ml.

(e) Funnels, 65- or 75-mm (diameter at top).

(f) Glass wool, silanized.

(g) Hot water bath.

(h) Kuderna-Danish evaporator concentrator, consisting of a 125-ml Erlenmeyer-type flask, 3-ball Snyder column, and 10-ml receiver graduated in milliliters.

(i) Glass beads, 3-mm.

(j) Volumetric flasks for standards.

(k) Graduated cylinders, 25- or 50-ml.

(l) Syringes, 5- or 10-μl and 100-μl.

(m) Gas-liquid chromatograph, with attendant equipment, including a phosphorus-flame photometric detector.

(n) Gas-liquid chromatography column, 6-ft x 4-mm ID, borosilicate glass (silanized), packed with one of the following:

(1) 10% DC-200 (12,500 cst) on 80-100 mesh Gas Chrom Q.

(2) 7.5% QF-1 (10,000 cst)/ 5% DC-200 (12,500 cst) on 80-100 mesh Gas Chrom Q.

(3) 2% diethylene glycol succinate (C6 stabilized) on 80-100 mesh Gas Chrom Q.

(4) 4% SE-30/6% OV-210 on 80-100 mesh Chromosorb W, HP.

Columns 1 and 2 are conditioned by heating 2-4 days at 240-250 C under nitrogen flowing at 60 ml/minute, then primed by repeated injections of standard methyl parathion solution under the conditions of analysis given below. Column 3 is conditioned by heating 12 hours at 225-230 C under nitrogen flowing at 60 ml/minute. Column 4 is conditioned for at least 3 days at 245 C under nitrogen flowing at 60 ml/minute. A column of 10% Carbowax 20M on 80-100 mesh silanized support (2-in x 4-mm ID glass tubing) is then attached before Column 4, and the assembly is heated at

230-235 C for 17 hours under nitrogen flowing at 20 ml/minute. The 10% Carbowax 20M column is subsequently removed.

Reagents

(a) Ethylene glycol, interference-free.

(b) Hexane, interference-free.

(c) Distilled water, interference-free.

(d) Saturated aqueous sodium chloride, interference-free.

(e) Anhydrous sodium sulfate.

(f) Methyl parathion of known purity.

Procedure

(a) The sample in 17-20 ml of ethylene glycol is transferred to a 125-ml separatory funnel. (The reagent quantities below apply to a sample in 20 ml of ethylene glycol and must be scaled for different volumes of collection media). Wash the sample container with a measured amount of water and add the washings to the separatory funnel. Dilute the ethylene glycol with a total of 70 ml of water.

(b) Extract the aqueous solution three times with 12 ml of hexane.

(c) Dry the hexane solution by passing it through 2.6 g of anhydrous sodium sulfate contained in a funnel with a glass-wool retaining plug at the top of the stem. Collect the eluate in a 125-ml Kuderna-Danish flask which has been fitted with a 10-ml receiving tube containing one 3-mm glass bead. When the extract has eluted, rinse the separatory funnel with three consecutive 2-ml portions of hexane, washing down the walls of the

funnel. Allow each rinse to elute before adding the next. Finally, rinse the funnel and the sodium sulfate with two more 2-ml portions of hexane.

(d) Place the Kuderna-Danish assembly in a boiling water bath and concentrate the extract to a volume of approximately 5 ml. Remove the assembly from the bath and, after it is cool, disconnect the receiving tube from the flask, rinsing the joint with a little hexane. Place the tube under a nitrogen stream at room temperature and further concentrate the extract to approximately 0.5 ml. Rinse down the wall of the tube with hexane delivered from a 100-μl syringe, dilute to exactly 1.0 ml, and stir.

(e) Inject an aliquot of the hexane solution into the GLC and obtain a chromatogram. The chromatographic conditions are:

Column temperature	220 C for Columns 1 and 2 210 C for Column 3 200 C for Column 4
Injection port temperature	225 C
Detector temperature	200 C
Transfer line temperature	235 C
Switching valve temperature	235 C
Carrier gas (nitrogen) flow	60 ml/minute

The retention times (relative to methyl parathion) at these conditions for methyl parathion and some interfering organophosphorus insecticides are shown in Table X-1.

TABLE X-1

RELATIVE RETENTION TIMES OF METHYL PARATHION AND OTHER ORGANOPHOSPHORUS (OP) INSECTICIDES

OP Compound	Column 1	Column 2	Column 3	Column 4
Methyl parathion	1.00 (3.2 min)	1.00 (6.2 min)	1.00 (4.0 min)	1.00
Methyl paraoxon	0.77	1.13	1.20	1.11
Ethyl parathion	1.37	1.28	0.85	1.33
Ethyl paraoxon	1.05	1.45	1.04	1.47
Amino parathion	1.42	1.00		
Dursban	1.37			
Fenthion	1.33			
Ruelene		1.29		
Phosphamidon				1.36

Adapted from reference 82

The solvent-flush sample injection technique is recommended. Duplicate injections should be made. The hexane which precedes the methyl parathion and methyl paraoxon should be vented so that the detector flame is not extinguished. The conditions of the run should be such that no methyl parathion is lost during the venting process.

(f) By comparison to standard curves for methyl parathion, the average of the area under the methyl parathion peak is converted to the amount (ng) of methyl parathion seen by the detector.

Calibration and Standards

(a) Prepare at least three standard solutions in the concentration range 100-10,000 ng/ml from a stock solution of methyl parathion in hexane.

(b) Make duplicate injections of aliquots of each methyl parathion standard solution and determine the peak areas.

(c) Plot the amount (ng) of methyl parathion seen by the detector vs the peak area. A straight line passing through the origin should result. If these conditions are not observed, either the linear range of the detector has been exceeded or a system malfunction has occurred.

(d) Injections of standards should be interspersed among sample injections in order to monitor detector sensitivity.

Calculations

(a) Determine the total weight (ng) of methyl parathion present in the sample:

$$\text{Sample weight of methyl parathion (ng)} = \text{ng(0)} \times \frac{\text{Soln vol}}{\text{Inj vol}}$$

where:

ng(0)	=	nanograms of methyl parathion determined from calibration curve based on peak area responses
Soln vol	=	volume in μl of the final hexane solution
Inj vol	=	volume in μl of the aliquot of the final hexane solution injected into the gas chromatograph

(b) Convert the volume of air sampled to standard conditions (25 C, 760 mmHg):

$$V(s) = V \times \frac{P}{760} \times \frac{298}{(T + 273)}$$

where:

V(s) = volume of air in liters at 25 C, 760 mmHg

V = volume of air in liters as measured

P = barometric pressure in millimeters of mercury

T = temperature of air in degrees centigrade

(c) The concentration of methyl parathion (or methyl paraoxon) can be expressed in ng/liter or μg/cu m:

$$\mu g/cu\ m = ng/liter$$

or

$$\mu g/cu\ m = \frac{\text{total ng}}{V(s)}$$