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PESTICIDE RECOVERY STUDIES FOR EVALUATION OF DEPARTMENT OF THE --ETC(U)
DEC 76 J.H VINOPAL, J F SUPROCK, T M WHITE

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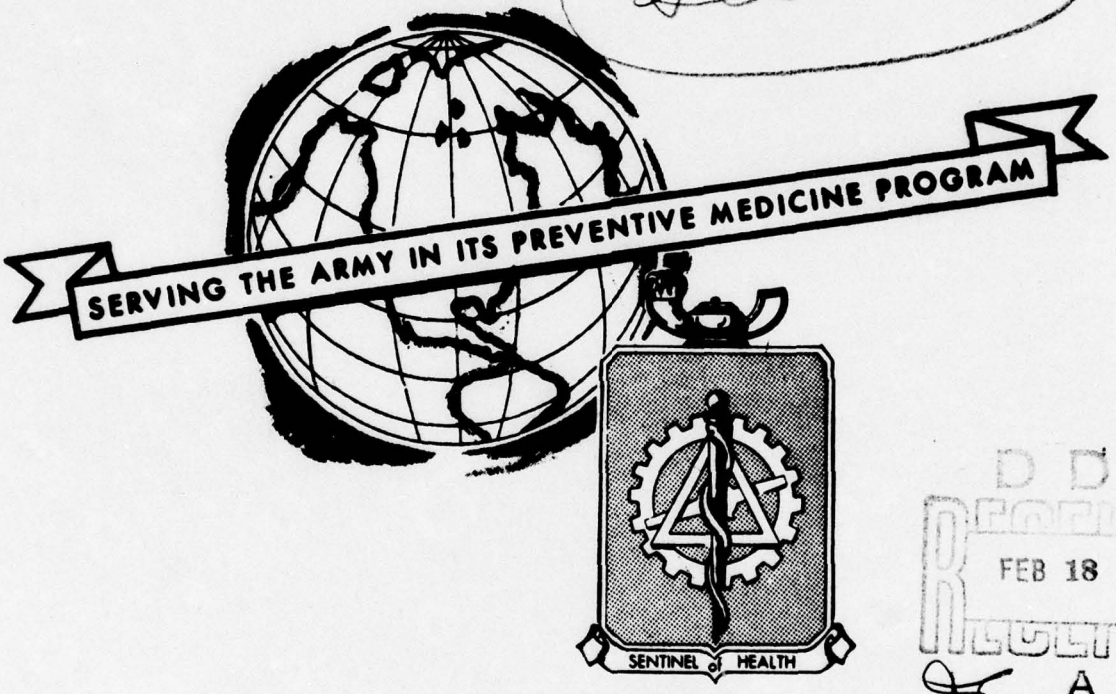
PESTICIDE MONITORING SPECIAL STUDY NO. 44-0131-77
PESTICIDE RECOVERY STUDIES FOR EVALUATION OF DEPARTMENT OF THE ARMY PESTICIDE MONITORING PROGRAM SOIL AND SEDIMENT ANALYSIS METHODOLOGY
PART I. DETERMINATION OF PESTICIDE AND POLYCHLORINATED BIPHENYL RECOVERIES FROM SOIL EXTRACTED IMMEDIATELY FOLLOWING FORTIFICATION
OCTOBER - DECEMBER 1976



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US ARMY
ENVIRONMENTAL HYGIENE AGENCY
ABERDEEN PROVING GROUND, MD 21010

DEPARTMENT OF THE ARMY
U. S. ARMY ENVIRONMENTAL HYGIENE AGENCY
ABERDEEN PROVING GROUND, MARYLAND 21010

HSE-RE/WP

16 FEB 1977

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ABSTRACT

This is a report providing analytical details and analytical results of pesticide and polychlorinated biphenyl (PCB) recovery studies used to evaluate Department of the Army (DA) Pesticide Monitoring Program soil analysis methodology. The present study was specifically involved with the determination of pesticide and PCB recoveries from soil which was extracted immediately following fortification and then carried through routine soil transfer, cleanup, concentration and analysis procedures. The percent recovery values for eight of the nine pesticides studied and for Aroclor® 1260 were essentially quantitative, ranging from 90.0 to 107.6. The recovery data clearly indicated that pesticide losses following immediate extraction of fortified soil samples, and during subsequent transfer, cleanup, concentration and analysis steps were minimal or nonsignificant using routine DA Pesticide Monitoring Program soil analysis methodology. The requirements for further recovery studies assessing the extractability of pesticides and PCB from field or "weathered residue" type soil samples and from sediment samples are discussed.

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1. AUTHORITY.

a. AR 200-1, Environmental Protection and Enhancement, 7 December 1973.

b. AR 40-5, Health and Environment, 25 September 1974.

c. Public Law 92-516, The Federal Environmental Pesticide Control Act of 1972, as amended by Public Law 94-140.

2. REFERENCES.

a. USAEHA Entomological Special Study No. 44-004-74/75. Revised Department of the Army Pesticide Monitoring Program, 1 April 1975. National Technical Information Service, AD-A004 030, 38 pages, 1975.

b. DA Pesticide Monitoring Program, Evaluation of Data from Environmental Samples Collected Prior to 1 January 1974, Part I, Soil, Sediment, Water, 1 September 1974. National Technical Information Service, AD-A003 228, 11 pages, 1974.

c. USAEHA Entomological Special Study No. 44-011-75/76, Department of the Army Pesticide Monitoring Program, January - December 1974. National Technical Information Service, AD-A017, 14 pages, 1975.

3. PURPOSE. To provide analytical details and analytical results of pesticide and polychlorinated biphenyl (PCB) recovery studies used to evaluate Department of the Army (DA) Pesticide Monitoring Program soil analysis methodology. The present study was specifically involved with the determination of pesticide and PCB recoveries from soil which was extracted immediately following fortification and then carried through routine soil transfer, cleanup, concentration, and analysis procedures.

4. GENERAL.

a. Pertinent aspects of the analytical methods and procedures used in the study are available in Appendix A. Appendix A is organized as follows:

(1) Part 1. Source of soil samples used in pesticide and PCB recovery studies and method used to fortify soil samples with known pesticide and PCB mixtures.

(2) Part 2. Methodology used for extraction and cleanup of fortified and control soil samples.

(3) Part 3. Gas-liquid chromatography (glc) parameters used in analysis of soil sample extracts.

b. The lower limits of instrumental sensitivity, the lower limits of detectability in soil, and Florisil® column elution behavior for those pesticides and PCB currently being analyzed for in the DA Pesticide Monitoring Program are presented in Appendix B.

5. RESULTS. Data including mean ($\bar{x}$), standard deviation (S), standard deviation of the mean ($S\bar{x}$) and coefficient of variability (CV) for the nine recovery experiments (i.e., three replicates at three different spiking levels) performed with each of the nine pesticides and one polychlorinated biphenyl utilized in this study are summarized in Appendix C, Tables 1-10. The fortification, extraction, cleanup, and gas chromatographic procedures detailed in Appendix A were employed in all recovery experiments.

6. DISCUSSION AND CONCLUSIONS.

a. Basis for Selection of DA Pesticide Monitoring Program Soil Methodology.

(1) Most of the aspects concerned with the extraction and analysis of pesticides in soil have been covered in depth by the recent reviews of Chiba[1] and Chesters, et al.[2]

(2) Some important general statements taken from the above cited references concerning the preparation and extraction of pesticides from soil are listed below. These statements were selected because of their direct relevance to currently used DA Pesticide Monitoring Program soil analysis methodology.

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[1] Chiba, M., "Factors Affecting the Extraction of Organochlorine Insecticides," Residue Reviews, 30:63-113 (1969)

[2] Chesters, G., H. B. Pionke and R. C. Daniel, "Extraction and Analytical Technique for Pesticides in Soil, Sediment and Water, In Pesticides in Soil and Water," W. D. Guerrzi, ed., Soil Science Society of America, Inc., Madison, WI, pp 451-550 (1974)

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(a) Techniques of soil sample preservation and preparation which allow samples to completely dry out will likely cause pesticide losses.

(b) Extractability for many pesticides from dry soils can be increased by pre-moistening the soil with water.

(c) The choice of extractant is one of the most critical steps in the development of a procedure. The most successfully used solvent systems for the extraction of organochlorine insecticides from soil consist either wholly, or in part, of a polar organic solvent. In the case of organophosphorus insecticides, the solvent most frequently used for soil extractions is acetone either alone or in combination with isopropanol or hexane using shaking or blending procedures.

(3) The procedures currently used for the extraction and cleanup of soil samples collected under the DA Pesticide Monitoring Program were adapted with certain modifications and additions from the method of Wiersma, et al.[3] The main modifications and additions to the method of Wiersma, et al,[3] include:

(a) The substitution of acetone for isopropanol in the extraction mixture which eliminates the need for water washings to remove the isopropanol.

(b) The extraction of 150 g soil samples instead of 300 g samples, although the 1:2 ratio of sample to extracting solvent is not modified.

(c) A Florisil column cleanup of all sample extracts is carried out prior to gas chromatographic analysis. This column cleanup procedure was added for two reasons: the use of cleaner soil extracts increases the lifespan of gas chromatographic columns and detectors, and the fractionation of pesticides among the various Florisil eluates aids in qualitative determinations.

(4) The method of Wiersma, et al,[3] was developed for the analysis of a wide variety of organochlorine and organophosphorus pesticides routinely monitored in the Environmental Protection Agency's National Soils Monitoring Program. Because of similar analytical requirements, this method, with the modifications and additions described above, was adopted for use in the DA Pesticide Monitoring Program. Both the original methodology of Wiersma, et al,[3] and the modified methodology used in the DA Pesticide Monitoring Program embody the important general principles regarding soil preparation and extraction outlined in paragraph (2) above.

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[3] Wiersma, G. B., H. Tai and P. F. Sand, "Pesticide Residue Levels in Soils FY 1969 - National Soils Monitoring Program," Pestic Monit J, 6(3):194-228 (1972)

b. Discussion of Pesticide and Polychlorinated Biphenyl Recovery Data.

(1) As stated earlier, the present study was specifically involved with the determination of pesticide and PCB recoveries from soil which was extracted immediately following fortification and then carried through transfer, cleanup, concentration and analysis steps using routine DA Pesticide Monitoring Program soil analysis methodology. No attempt was made in this study to simulate the pesticide extraction of field or "weathered residue" type soil samples.

(2) The nine pesticides chosen for the present study were selected on the rationale that these pesticides are representative of the entire DA Pesticide Monitoring Program routine monitoring list (see Appendix B). Pesticides from the DDT class, BHC class, cyclodiene class, miscellaneous organochlorine class and organophosphorus class are represented in this study. Aroclor 1260 was selected as a representative polychlorinated biphenyl. PCB analyses on all soil samples received under the DA Pesticide Monitoring Program have recently been initiated commencing with calendar year 1976 soil collections.

(3) From the data summarized in Appendix C, Tables 1-10, it can be seen that percent recovery values for eight of the nine pesticides studied and for Aroclor® 1260 were essentially quantitative, ranging from 90.0 to 107.6. The recovery data for one pesticide (oxychlordane) averaged 78 percent which, although not quantitative, was satisfactory. The recovery data clearly indicate that pesticide losses following immediate extraction of fortified soil samples, and during subsequent transfer, cleanup, concentration and analysis steps were minimal or nonsignificant using routine DA Pesticide Monitoring Program soil analysis methodology. The recovery data from this study are very similar to recovery data reported by Wiersma, et al,[3] using equivalent pesticides and soil fortification techniques.[4]

c. Requirements for Additional Recovery Studies - Soil and Sediment.

(1) The recovery experiments performed in the present study are incomplete since they do not adequately assess the extractability of pesticides from field or "weathered residue" type soil samples. Unfortunately, very little is known about the time and conditions necessary for fortified pesticides to reach conditions equivalent to those of naturally

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[3] Wiersma, G. B., H. Tai and P. F. Sand, "Pesticide Residue Levels in Soils FY 1969 - National Soils Monitoring Program," Pestic Monit J 6(3):194-228 (1972)

[4] Tai, H., Personal Communication, 1976

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treated pesticides in the field.[1] Chesters, et al,[2] indicates that recoveries determined from fortified samples may approximate the extraction of weathered pesticide residues in soil if sufficient equilibration time is allowed with a moistened soil or one subjected to alternate wetting and drying conditions.

(2) Additional recovery studies designed to more adequately assess the extractability of pesticides under simulated field conditions are underway. In these studies, soil samples will be fortified with several representative pesticides and with Aroclor 1260, and then allowed to equilibrate for 14-21 days at room temperature under alternate wetting and drying conditions prior to extraction and processing using routine DA Pesticide Monitoring Program soil analysis methodology.

(3) Sediment samples are also collected routinely under the DA Pesticide Monitoring Program. Methodology used for the extraction, cleanup and analysis of sediment samples is essentially similar to that described in Appendix A, Parts II and III for soil. According to Chesters, et al,[2] soil monitoring procedures should not be applied to sediment systems without first validating the procedure. In addition, pesticide recoveries performed on fortified sediment samples indicate generally lower recoveries than those obtained by application of similar techniques to soil systems.

(4) Appropriate pesticide recovery studies designed to assess the applicability and efficiency of DA Pesticide Monitoring Program soil analysis methodology to sediment analysis are underway at the present time.

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[1] Chiba, M., "Factors Affecting the Extraction of Organochlorine Insecticides," Residue Reviews, 30:63-113 (1969)

[2] Chesters, G., H. B. Pionke and R. C. Daniel, "Extraction and Analytical Technique for Pesticides in Soil, Sediment and Water, In Pesticides in Soil and Water," W. D. Guerrzi, ed., Soil Science Society of America, Inc., Madison, WI, pp 451-550 (1974)

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APPENDIX A

ANALYTICAL METHODS AND PROCEDURES

PART 1. SOURCE OF SOIL SAMPLES USED IN PESTICIDE AND PCB RECOVERY STUDIES AND METHOD USED TO FORTIFY SOIL SAMPLES WITH KNOWN PESTICIDE AND PCB MIXTURES.

a. Soil samples used in pesticide and PCB recovery studies were obtained by compositing a number of soil samples routinely received under the DA Pesticide Monitoring Program during CY 1975. These soil samples were collected from various geographic locations within CONUS and represented several soil types. Only soil samples which had been previously analyzed and found to be negative for all routine pesticides were composited for use in this study.

b. Soil samples (150 g) were fortified by the addition of 50 ml of acetone solution containing known concentrations of pesticides or PCB (Aroclor 1260). In a typical recovery experiment, 50 ml of acetone solution containing known concentrations of four to five different pesticides or Aroclor 1260 was added to triplicate 150 g soil samples. Fortification of each soil sample was carried out in 1-qt, wide-mouth glass jars. After the addition of the pesticide or PCB-acetone spiking solution to the soil sample, the jar was swirled to ensure that the soil was completely and uniformly wetted with the spiking solution. The acetone was then evaporated from the soil samples under a gentle nitrogen stream with periodic stirring of the soil. After complete evaporation of the acetone, which usually required about 2 hours, the soil sample was then immediately extracted using the procedures described in Appendix A, Part IIc. Reagent blanks were run with every recovery experiment and, additionally, control soil samples containing no added pesticides or PCB were run several times during the course of the study. Recovery studies for each pesticide and Aroclor 1260 were carried out at three different fortification levels representing a 50- to 100-fold concentration range. Only nanograde or equivalent grade acetone was used in the above described procedures.

PART 2. METHODOLOGY USED FOR EXTRACTION AND CLEANUP OF FORTIFIED AND CONTROL SOIL SAMPLES.

a. Apparatus and Materials.

(1) Glassware, Balances, Shakers.

(a) 1-qt mason jars with Teflon®-lined lids

(b) Chromatographic columns - 22 mm x 300 mm

(c) Graduated cylinders - 100 ml

(d) Kuderna-Danish aparatus - 250 ml flasks, 10 ml concentrator tubes, Snyder columns

(e) Tared Beakers - 250 ml

(f) Volumetric flasks - 200 ml

(g) Screw cap, 15-ml culture tubes with Teflon cap liners

(h) 0.5, 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10 ml volumetric pipets

(i) Graduated centrifuge tubes - 15 ml

(j) Glass stoppered, screw lid reagent bottles - 100 ml

(k) Analytical laboratory balance

(l) General laboratory top loading balance

(m) Variable speed mechanical shaker capable of accommodating up to 12 1-qt jars

(2) Reagents, Solvents, Standards.

(a) Hexane - Nanograde

(b) Acetone - Nanograde

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(c) Petroleum ether - Nanograde

(d) Ethyl ether - Nanograde

(e) Ethyl alcohol - absolute

(f) Sodium sulfate - anhydrous - hexane washed

(g) Florisil - Pesticide grade (60/100 mesh)

(h) Glass wool - silinized - hexane washed

(i) Pesticide standards & PCB standards - reference grade

b. Extraction and Cleanup Procedures.

(1) Preparation of Soil Prior to Extraction.

(a) All soil samples received under the DA Pesticide Monitoring Program are received in 1-qt glass jars with Telfon-lined lids. Upon receipt the samples are placed in refrigerator storage at ~ 4° C until extraction.

(b) At the time of extraction the entire soil sample is dumped out onto a piece of aluminum foil and mixed. After mixing, a 50 g subsample is removed for the determination of soil moisture content. (Soil moisture content is determined by placing the 50 g subsample in a foil weighing boat and allowing it to stand at room temperature for 1 week. After 1 week, the subsample is reweighed and the percent moisture calculated.) A 150 g subsample is then weighed into a 1-qt mason jar and carried through the extraction procedure described in paragraph 2 below.

(c) The soil moisture content of most soil samples received for analysis range from 10 to 30 percent. These samples are extracted "as is" after mixing and subsampling as described above. Certain soil samples, i.e. from desert locations, are obviously very dry upon receipt. In the case of this type of sample, 30 ml of distilled water is added to the 150 g subsample prior to extraction.

(d) The composited soil used in the present recovery study had a moisture content of 12 percent.

(2) Extraction.

(a) One hundred and fifty gram subsamples of the composited soil prepared as described in Part Ia were weighted into 1-qt mason jars and appropriate fortifications were carried out as described in Part Ib.

(b) The samples were then extracted with 300 ml of 3:1 n-hexane:acetone for 2 hours in a mechanical shaker. After shaking, the samples were allowed to stand for 1 hour to allow settling of particulate matter.

(c) Using a graduated cylinder, 100 ml aliquots of the sample extracts were measured. The aliquots were then passed through chromatographic columns containing approximately 6 inches of sodium sulfate. Following elution of the sample extracts, the columns were rinsed with 25-30 ml of hexane. The extracts and rinses were collected in 250 ml Kuderna-Danish apparatus. The extracts were concentrated in a water bath to 10 ml. The extracts were transferred to 15 ml screw-cap culture tubes with Teflon cap liners and placed in a freezer until cleanup.

(3) Cleanup.

(a) Florisil columns were prepared as follows: To a chromatographic column (22 mm x 300 mm) containing a glass wool plug was added 40 ml (measured in a small Tared beaker) of Florisil - pesticide grade (60/100 mesh). After settling of the Florisil by gentle tapping, the column was topped with a one-half-inch layer of sodium sulfate. The Florisil column was activated by placing it in an oven at 80°-100° C for a minimum of 16 hours.

(b) Florisil columns, prepared and activated as described above, were allowed to cool and then were pre-wet with 40-50 ml of hexane. Sample extracts from extraction step (2)(c) were further concentrated to 2-3 ml under a nitrogen stream and carefully transferred using Pasteur pipets onto the Florisil columns.

(c) Graduated Erlenmeyer beakers (250 ml) were placed under the columns and the columns were eluted with 200 ml of 6 percent ethyl ether/petroleum ether mixture. The beakers were changed and the columns eluted next with 200 ml of 15 percent ethyl ether/petroleum ether mixture. The beakers were again changed and the columns eluted finally with 200 ml of 50 percent ethyl ether/petroleum ether mixture. The elution rate for each of the three fractions was maintained at approximately 5 ml/min. NOTE: Ethyl ether should be free of peroxides and must contain 2 percent v/v of absolute ethanol.

(d) The beakers containing the 6 percent, 15 percent and 50 percent eluate fractions were tared to exactly 200 ml. Aliquots (10 ml for the 6 percent and 15 percent fractions and 12 ml for the 50 percent fraction) were transferred to 15 ml graduated centrifuge tubes and concentrated to obtain appropriate definitive volumes for gas chromatographic analysis. Routine definitive volumes used were 160 ml for the 6 percent fraction, 160 ml for the 15 percent fraction and 16.7 ml for the 50 percent fraction (based on 200 ml total volume for each fraction).

(e) After appropriate concentration, the 6 percent, 15 percent, and 50 percent extract fractions were transferred to 15 ml screw cap culture tubes and stored in a freezer until gas chromatographic analysis.

PART 3. GAS LIQUID CHROMATOGRAPHY PARAMETERS USED IN ANALYSIS OF SOIL SAMPLE EXTRACTS.

a. Instrumental Parameters.

(1) Gas chromatograph. Tracor MT-220, equipped with glass-lined injection ports.

(2) Detectors.

(a) Tracor high-temperature Ni^{63} electron-capture detector (EC) - used for organochlorine pesticides and PCB.

(b) Tracor flame photometric detector operating in phosphorus mode (FPD) - used for organophosphorus pesticides.

(3) Gas chromatographic columns.

(a) 1.5 percent OV-17/1.95 percent QF-1 on 80/100 Gas chrom Q

(b) 3 percent OV-1 on 80/100 Gas chrom Q

(4) Recorder. Honeywell Electronic potentio-metric strip chart (1 mV)

(5) Routine Analysis Parameters for GLC.

(a) Oven temperature - 200° C

(b) Injection port temperature - 225° C

(c) Outlet temperature - 240° C

(d) Detector temperatures (1) EC - 305° C
(2) FPD - 210° C

(e) Carrier gas flows (1) EC column (95% Argon - 5% methane) - 65 ml/min
(2) FPD column (nitrogen) - 60 ml/min

(f) Detector gas flows - FPD (1) Hydrogen - 50 ml/min
(2) Zero air - 90 ml/min

(g) Electrometer Settings.

(1) EC - 1.6×10^{-9} amps full scale sensitivity (Input 10^2; output 16)
(2) FPD (Input 10^3; output 8)

(h) Recorder Speed - 0.5 in/min

b. GLC Quantitation Methods.

(1) Automatic Integration Method - An Auto Lab System IV Computing Integrator (Spectra-Physics, Mountain View, CA) was used for quantitation of all organochlorine pesticide and PCB peaks.

(2) Manual (Peak Height) Method - This method was used for the quantitation of organophosphorus pesticide peaks.

(3) With both methods of quantitation, sample and reference standard peaks of approximately equal (i.e., within 25 percent) peak area or peak height were used to calculate results.

APPENDIX B

SUMMARY OF LOWER LIMITS OF INSTRUMENTAL SENSITIVITY, LOWER LIMITS OF DETECTABILITY IN SOIL, AND FLORISIL COLUMN ELUTION BEHAVIOR FOR PESTICIDES AND PCB ON DA PESTICIDE MONITORING PROGRAM ROUTINE MONITORING LIST (AS OF 1 JANUARY 1977)

Pesticide or PCB	Lower Limits of Instrumental Sensitivity - Picograms Required for 10% Full Scale Recorder Deflection using EC Detection* (Based on 5 µl injection volume)	Lower Limits of Detectability in Soil† (ppm)	Florisil Column Elution Behavior‡ (EE/PE eluate fraction)
α-BHC	3.1	0.003	6%
β-BHC	12.5	0.010	6%
aldrin	10.0	0.008	6%
chlordane (tech.)	75.0	0.060	6%
cis-chlordane	10.0	0.008	6%
trans-chlordane	10.0	0.008	6%
oxychlordane	10.0	0.008	6%
o,p'-DDD	25.0	0.020	6%
p,p'-DDD	20.0	0.016	6%
o,p'-DDE	25.0	0.020	6%
p,p'-DDE	20.0	0.016	6%
o,p'-DDT	25.0	0.020	6%
p,p'-DDT	37.5	0.030	6%
dieldrin	15.0	0.012	15%
endrin	26.5	0.021	15%
heptachlor	4.0	0.003	6%
heptachlor epoxide	10.0	0.008	6%
lindane	5.0	0.004	6%
methoxychlor	100.0	0.080	6%
mirex	25.0	0.020	6%
toxaphene	1000.0	0.800	6%
chlorpyrifos	15.0	0.012	6%
	200.0 (FPD - 10 µl)		
diazinon	65.0	0.052	15%
	160.0 (FPD - 10 µl)		
malathion	100.0	0.010	50%
	250.0 (FPD - 10 µl)		
methylparathion	37.5	0.030	15%
	150.0 (FPD - 10 µl)		
parathion	25.0	0.020	15%
	175.0 (FPD - 10 µl)		
PCB (*includes Aroclors* 1242, 1248, 1254, 1260)	500.0	0.400	6%

* Lower limits of instrumental sensitivity for organophosphorus pesticides listed in this Appendix using flame photometric detection (FPD) are also given as indicated.

† Based on the procedures described in Appendix A, Parts 2 and 3 and using EC detection, except where noted.

‡ Based on the Florisil column cleanup procedure described in Appendix A, Part 2b.

APPENDIX C

SUMMARY OF RECOVERY DATA OBTAINED FOR NINE PESTICIDES AND ONE POLYCHLORINATED BIPHENYL USING FORTIFICATION, EXTRACTION, CLEANUP, AND GAS CHROMATOGRAPHIC PROCEDURES DETAILED IN APPENDIX B

TABLE 1. CHLORDANE

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.12-6.00 ppm	9	$\bar{x}$ = 94.3 S = 11.6 $S\bar{x}$ = 3.9 CV = 12.3

TABLE 2. OXYCHLORDANE

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.016-0.80 ppm	9	$\bar{x}$ = 78.1 S = 8.9 $S\bar{x}$ = 3.0 CV = 11.4

TABLE 3. p,p'-DDE

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.032-1.60 ppm	9	$\bar{x}$ = 99.6 S = 7.8 $S\bar{x}$ = 2.6 CV = 7.9

TABLE 4. MALATHION

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.020-1.00 ppm	9	$\bar{x}$ = 102.1 S = 10.0 $S\bar{x}$ = 3.3 CV = 9.8

TABLE 5. β-BHC

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.020-1.00 ppm	9	$\bar{x}$ = 101.2 S = 14.3 $S\bar{x}$ = 4.8 CV = 14.1

TABLE 6. CHLORPYRIFOS

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.024-1.20 ppm	9	$\bar{x}$ = 102.9 S = 14.7 $S\bar{x}$ = 4.7 CV = 14.3

TABLE 7. p,p'-DDT

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.060-3.00 ppm	9	$\bar{x}$ = 105.9 S = 11.2 $S\bar{x}$ = 3.7 CV = 10.6

TABLE 8. MIREX

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.040-2.00 ppm	9	$\bar{x}$ = 107.6 S = 11.1 $S\bar{x}$ = 3.7 CV = 10.4

TABLE 9. DIELDRIN

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.024-1.20 ppm	9	$\bar{x}$ = 101.4 S = 11.5 $S\bar{x}$ = 3.9 CV = 11.4

TABLE 10. AROCLOR 1260

Spiking Level (Range)	No. Recovery Experiments	Recovery Data (Percent)
0.10-10.00 ppm	9	$\bar{x}$ = 90.0 S = 6.6 $S\bar{x}$ = 2.2 CV = 7.4

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

(9) REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This is a report providing analytical details and analytical results of pesticide and polychlorinated biphenyl (PCB) recovery studies used to evaluate Department of the Army (DA) Pesticide Monitoring Program soil analysis methodology. The present study was specifically involved with the determination of pesticide and PCB recoveries from soil which was extracted immediately following fortification and then carried through routine soil transfer, cleanup, concentration and analysis procedures. The percent

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recovery values for eight of the nine pesticides studied and for Aroclor® 1260 were essentially quantitative, ranging from 90.0 to 107.6. The recovery data clearly indicated that pesticide losses following immediate extraction of fortified soil samples, and during subsequent transfer, cleanup, concentration and analysis steps were minimal or nonsignificant using routine DA Pesticide Monitoring Program soil analysis methodology. The requirements for further recovery studies assessing the extractability of pesticides and PCB from field or "weathered residue" type soil samples and from sediment samples are discussed.