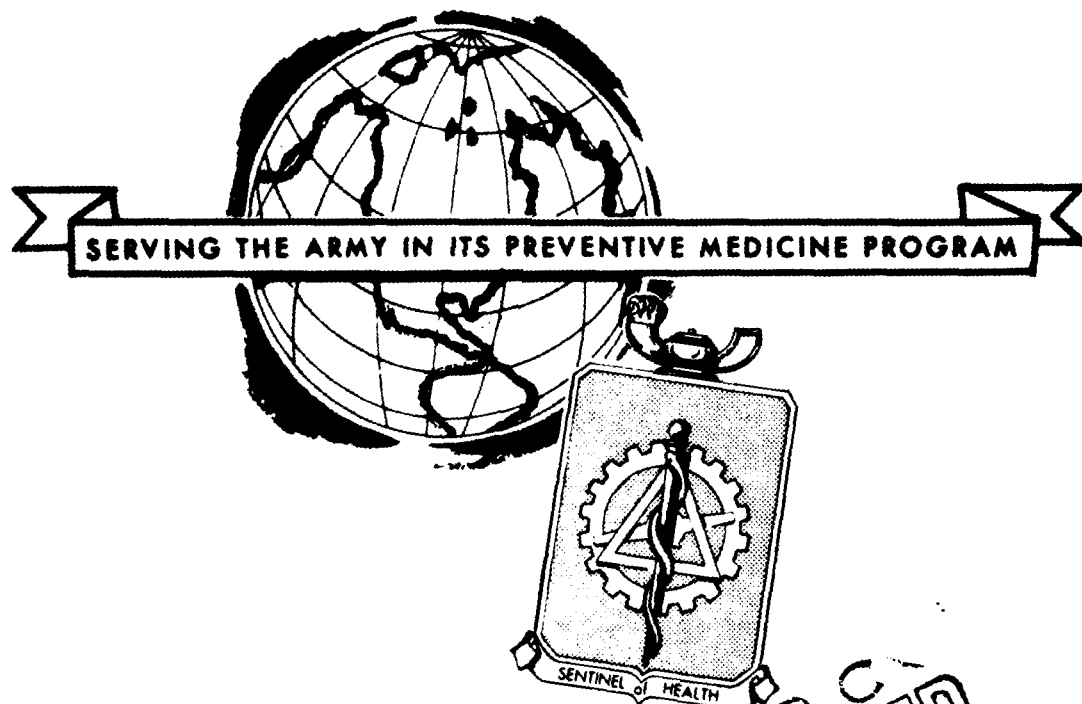


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INTERIM REPORT
ENTOMOLOGICAL SPECIAL STUDY NO. 44-005-75
MONITORING OF PESTICIDE DISPOSAL PRACTICES
IOWA ARMY AMMUNITION PLANT
BURLINGTON, IOWA 52600
SEPTEMBER 1974 - JANUARY 1975



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19. ABSTRACT (Continue on reverse side if necessary and identify by block number) This is an interim report giving the results of analyses of five samples taken near a pesticide disposal pit. The samples were analyzed for routine pesticides and special analyses were done for the phenoxy herbicides and for monuron and diuron. Analyses showed above normal concentrations of silvex ranging from 4.52 to 18.52 ppm. There were also residues of 2,4-D and 2,4,5-T present in the samples. Corrective recommendations will be made upon completion of a follow-up survey by this Agency.		

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15 OCT 1976

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SEPTEMBER 1974 - JANUARY 1975

ABSTRACT

This is an interim report giving the results of analyses of five samples taken near a pesticide disposal pit. The samples were analyzed for routine pesticides and special analyses were done for the phenoxy herbicides and for monuron and diuron.

Analysis showed above normal concentrations of silvex ranging from 4.52 to 18.52 ppm. There were also residues of 2,4-D and 2,4,5-T present in the samples.

Corrective recommendations will be made upon completion of a follow-up survey by this Agency.

SEARCHED	☒
SERIALIZED	☒
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DEPARTMENT OF THE ARMY
U. S. ARMY ENVIRONMENTAL HYGIENE AGENCY
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SEPTEMBER 1974 - JANUARY 1975

1. Reference. Report, ALBO-E, US Army Medical Laboratory, St Louis, 29 July 1974, subject: Report of Entomological Survey, Iowa Army Ammunition Plant, Burlington, Iowa 52600, 3-4 June 1974.

2. BACKGROUND.

a. Five samples were received from Iowa Army Ammunition Plant 13 September 1974. The samples were analyzed for routine pesticides and special analyses were done for the phenoxy herbicides and for monuron and diuron. This is an interim report giving the results of the analysis performed on these samples. Sample locations and results are listed below. Recommendations will be withheld pending the results of a follow-up survey to be conducted by this Agency.

<u>Sample Location</u>	<u>Analysis Results</u>	
	<u>Pesticide</u>	<u>Concentration (ppm)</u>
SP-172 4-foot depth	p,p'-DDD	Trace
	p,p'-DDE	Trace
	o,p'-DDT	Trace
	p,p'-DDT	0.06
	chlordane	0.06
	silvex	17.09
	2,4-D	0.015
SP-170 6-foot depth	o,p'-DDT	Trace
	p,p'-DDT	0.03
	2,4-D	0.021
	2,4,5-T	0.021
	silvex	18.52

Use of trademarked names does not imply endorsement by the US Army.

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Sample Location

Analysis Results

	<u>Pesticide</u>	<u>Concentration (ppm)</u>
SP-173	Routine pesticides	
8-foot depth	Sample broken	
	2,4-D	0.047
	2,4,5-T	0.018
silvex	5.24	
SP-169	p,p'-DDE	Trace
10-foot depth	p,p'-DDT	Trace
	2,4-D	0.053
	2,4,5-T	0.033
	silvex	10.40
SP-171	p,p'-DDD	0.07
12-foot depth	p,p'-DDE	0.072
Sediment Sample	p,p'-DDT	0.046
	p,p'-DDT	0.39
	2,4-D	0.149
	2,4,5-T	0.009
	silvex	4.523

b. The methods of analysis for the routine pesticides are listed in Appendix A. Appendix B lists the methods of analysis for the chlorophenoxy herbicides and Appendix C lists the methods of analysis for the substituted urea herbicides.

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APPENDIX A

ROUTINE PESTICIDE EXTRACTION AND CLEAN-UP FOR SOIL AND SEDIMENT

1. SCOPE AND APPLICATION.

a. This method covers the determination of various organochlorine and organophosphate pesticides. The pesticides analyzed for and lower limits of detectability are shown in the Table. Such compounds are composed of carbon, hydrogen, chlorine in the organochlorines and carbon, hydrogen and phosphorus in the organophosphates. Both these groups may contain oxygen, sulfur, nitrogen or other halogens.

b. This method may also extract polychlorinated biphenyls (PCB) from the sample. The method of removing the PCB from the pesticides is indicated in Appendix D.

2. APPARATUS AND MATERIALS.

a. Gas Liquid Chromatographic (GLC) Material.

(1) Gas Chromatograph: Equipped with glass lined injection port (Tracor MT 222 or equivalent).

(2) Detector Capability - Electron Capture - Radioactive (Ni 63), Electrolytic Conductivity. Flame Photometric.

(3) Recorder: Potentiometric strip chart (10 in 1 mv) compatible with the detector.

(4) Gas Chromatographic Columns:

(a) Solid Support - Supelcon (100-120 mesh), Chromasorb W (100-120 mesh).

(b) Liquid Phases - Expressed as weight-percent coated on solid support.

3% OV-1

1.5% OV-17 + 1.95% QF-1

4% SE-30 + 6% QF-1

(c) Tubing - Pyrex (6' x 1/4" o.d. U shaped)

TABLE
LIMITS OF DETECTABILITY OF PRIMARY PESTICIDES IN WATER, SOIL AND SEDIMENT

Pesticide	Amounts for 10% full-scale deflection (EC detector). (pg)	Limit of Detectability (ppm) (Soil and Sediment)
α BHC	2.4	.0016
β BHC	5.1	.0034
aldrin	2.4	.0016
chlordane	78.0	.052
o,p'-DDE	15.8	.013
p,p'-DDD	19.8	.013
o,p'-DDE	11.3	.0095
p,p'-DDE	10.2	.0085
o,p'-DDT	19.8	.013
p,p'-DDT	28.9	.017
dieldrin	10.2	.0065
endrin	30.0	.025
heptachlor	3.8	.0025
heptachlor epoxide	7.5	.005
lindane	3.8	.0025
methoxychlor	120.0	.08
mirex	60.0	.04
toxaphene	1200.0	.80
chlorpyrifos	12.0	.008
diazinon	48.0	.032
malathion	75.0	.05
malathion (FPD)	300.0	.02
methyl parathion	22.5	.015
parathion	26.3	.018
ronnel	7.5	.005

(5) Routine Analysis Parameters for GLC.

- (a) Oven temp 200°C
- (b) Injector temp 250°C
- (c) Outlet temp 250°C
- (d) Detector temp 290°C
- (e) Carrier gas flow 60 ml/min
- (f) Sensitivity - 1.7×10^{-9} amp full scale (Input 10^2 Output 16)
- (g) Recorder Speed - 2 in/min

b. Glassware.

- (1) 1 quart wide mouth jar fitted with Teflon[®] cap-liner or 2 layers of foil.
- (2) Graduated cylinders - 250 ml.
- (3) Kuderna-Danish (K-D) flasks - 250 ml.
- (4) Snyder Column - three ball
- (5) Receiver Ampuls - 10 ml, graduated.
- (6) Chromatographic Column - Chromaflex (400 mm long x 19 mm ID) with Teflon stopcock.
- (7) 1 ounce screw cap bottles with foil cap liners.

c. Reagents, Solvents, and Standards.

- (1) Hexane - nanograde.
- (2) Acetone - nanograde.
- (3) Petroleum ether - nanograde.
- (4) Ethyl ether - nanograde.
- (5) No. 43 filter paper - pre-extracted Whatman.
- (6) Sodium Sulfate - anhydrous - Hexane washed.

[®]Teflon is a registered trademark of E. I. du Pont de Nemours & Co., Inc., Wilmington 98, Delaware.

(7) Glass wool - Silanized - Hexane washed.

(8) Florisil® - PR Grade (60-100 mesh); purchase activated at 1250°F and store in dark in glass containers with glass stoppers or foil-lined screw caps. Before use, activate each batch overnight at 130°C in foil-covered glass container.

(9) Pesticide Standards - Reference grade.

3. EXTRACTION AND CLEAN-UP.

a. Extraction.

(1) Weigh out 150 g soil or sediment (air dried) on balance. Remove large rocks, stones, debris from sample before weighing.

(2) Place sample into clean 1 quart jar (fitted with Teflon cap-liner or 2 layers of foil).

(3) Add 300 ml of hexane:acetone (3:1) to jar. Shake jar vigorously for 10 minutes to thoroughly wet all soil particles. Allow jar to set overnight. In the morning, shake jar 10 minutes more, allow soil to settle to bottom (1 hour).

(4) After settling is complete, pour 100 ml of extract into 250 ml graduated cylinder.

(5) Slowly filter 100 ml of extract through pre-extracted No. 43 filter paper, glass wool and enough sodium sulfate to cover glass wool into another 250 ml graduated cylinder.

(6) After filtration, rinse funnel with enough hexane to bring volume in graduated cylinder to 125 ml.

(7) Concentrate extract to 10 ml in K-D flask.

b. Clean-up.

(1) Prepare Florisil column that contains 4 inches activated Florisil topped with ½" anhydrous Na₂SO₄. Prewet column with 40-50 ml hexane. Place collection container under column.

(2) Transfer extract to column letting it pass through at about 5 ml/min. Rinse container with two, about 5 ml portions hexane, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions hexane. Elute column at about 5 ml/min with 200 ml 6% ethyl ether/petroleum ether eluant. Change receivers and elute at about 5 ml/min

®Florasil is a registered trademark of Floridin Company, P.O. Box 989, Tallahassee, Florida.

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with 200 ml 15% ethyl ether/petroleum ether eluant. For soil samples continue on with 50% elution.

(3) Pour out an aliquot (10-15 ml) from each elution mixture (200 ml definitive volume) into 1 oz screw cap bottle with foil cap liners.

(4) Proceed to GLC analysis.

4. GLC ANALYSIS.

a. Eluate Composition - using the Florisil column the pesticides will be separated into the eluates indicated below.

	<u>6% Eluate</u>	<u>15% Eluate</u>	<u>50% Eluate</u>
γ BHC	chlordane	dieldrin	malathion
β BHC	DDT	endrin	
aldrin	DDD	diazinon	
heptachlor	DDE	methyl parathion	
heptachlor epoxide	chlorpyrifos	parathion	
lindane	ronnel		
methoxychlor	mirex		

b. If there are a large number of partially resolved or unresolved peaks which occur throughout the entire chromatogram in the 6% fraction proceed to the PCB clean-up.

APPENDIX B

CHLOROPHENOXY HERBICIDE EXTRACTION AND CLEAN-UP FOR SOIL AND SEDIMENT

1. SCOPE AND APPLICATION. This method covers the determination of four chlorophenoxy herbicides including 2,4-D, 2,4,5-T; silvex and picloram. These compounds are analyzed for and reported at the butyl esters.

2. APPARATUS AND MATERIALS.

a. GLC Material.

(1) Gas Chromatograph: Equipped with glass lined injection port (Tracor MT-220 or equivalent).

(2) Detector Options: Electron Capture - Radioactive (Ni 63), Electrolytic Conductivity.

(3) Recorder: Potentiometric strip chart (10 in, mv) compatible with detector.

(4) Gas Chromatographic Columns:

(a) Solid Support - Supelcon (100-120-mesh), Chromasorb W (100-120 mesh)

(b) Liquid Phases - Expressed as weight-percent coated on solid support.

3% OV-1

1.5% OV-17 + 1.95% QF-1

4% SE-30 + 6% QF-1

(c) Tubing - Pyrex (6' x 1/4" o.d. U shaped)

(5) Routine Analysis Parameters for GLC:

(a) Oven temperature 200°C.

(b) Injector temperature 250°C.

(c) Outlet temperature 250°C.

(d) Detector temperature 290°C.

(e) Carrier gas flow 60 ml/min.

(f) Sensitivity 1.7×10^{-9} amps full scale (Input 10^2 , Output 16)

(g) Recorder speed 2 in/min.

b. Glassware.

(1) 1 quart wide mouth jar fitted with Teflon cap-liner or 2 layers of foil.

(2) Graduated cylinders - 50 ml, 125 ml, 150 ml.

(3) Separatory funnel - 125 ml.

(4) Volumetric pipettes - 1 ml, 2 ml.

(5) Erlenmeyer flask - 125 ml.

(6) Centrifuge tube - 35 ml graduated.

(7) 1 oz screw cap bottles with foil cap-liners.

c. Reagents, Solvents and Standards.

(1) Ethyl Ether - nanograde.

(2) Sulfuric Acid - (1:1/v/v).

(3) Sodium Sulfate - anhydrous, hexane washed.

(4) Celite[®] 545 - Must be free of electron capturing substances.

(5) NaOH - 7N.

(6) n-butanol - ACS grade.

(7) Sulfuric Acid - concentrated.

(8) Deionized H₂O.

(9) 2,2,4-trimethylpentane - nanograde.

3. EXTRACTION AND DERIVATIZATION.

a. Extraction.

(1) Weigh out 75 g soil or sediment (air dried) on balance. Remove large rocks, stones, debris from sample before weighing.

[®]Celite is a registered trademark of Johns-Manville, 22 E. 40 St., New York 16, New York.

(2) Place sample in clean quart jar (fitted with Teflon cap-liner or 2 layers of foil).

(3) Add 30 ml distilled H_2O and 150 ml ethyl ether.

(4) Add H_2SO_4 (1:1/v/v) until pH is about 2.

(5) Shake 4 hours and filter through Na_2SO_4 /Celite into a 1 quart sample bottle.

(6) Aliquot 50 ml ethyl ether extract into 125 ml separatory funnel. Add 2 ml 7 N NaOH slowly for 5 min. Swirl while adding. Add 50 ml distilled H_2O . Test pH - if not greater than 10 add more base. Shake funnel for 2 min and drain the aqueous layer into another 125 ml funnel.

(7) Adjust pH of aqueous layer to about 2 with 1:1 H_2SO_4 . Add 50 ml ethyl ether and shake for 2 min then discard aqueous layer.

(8) Place extract in 125 ml erlenmeyer flask and evaporate slowly to 5 ml on H_2O bath ($50^\circ C$).

(9) Transfer extract to 35 ml centrifuge tube and evaporate to dryness in warm H_2O under stream of dry Nitrogen.

b. Derivatization.

(1) Add 1 ml n-butanol and 3 drops concentrated H_2SO_4 . Stopper tube and place in boiling water bath for 30 minutes. Release pressure after 2-3 minutes.

(2) Add 20 ml deionized H_2O + 5 ml 2,2,4-trimethylpentane and shake for 2 minutes.

(3) Allow water and solvent to separate and analyze iso-octane fraction by GLC.

Lower Limits of Detectability of
Chlorophenoxy Herbicides in Soil and Sediment

<u>Pesticide</u>	<u>Lower Limit of Detectability (ppm)</u>
2,4-D	0.002
2,4,5-T	0.0004
silvex	*
picloram	*

*Lower limits of detectability not determined at this time.

APPENDIX C

SUBSTITUTED UREA HERBICIDE EXTRACTION AND CLEAN-UP FOR SOIL AND SEDIMENT

1. SCOPE AND APPLICATION. This method covers the determination of monuron and diuron and is also suitable for benzomarc, chlorbromuron, diuron, flometuron, metobromuron metoxymarc and neburon in soils and sediment.

2. APPARATUS AND MATERIALS.

a. GLC Material.

(1) Gas Chromatograph: Equipped with glass lined injection port (Tracor MT-222 or equivalent).

(2) Detector: Electron Capture - Radioactive (Ni 63).

(3) Recorder: Potentiometric strip chart (10 in, 1 mv) compatible with detector.

(4) Gas Chromatographic Columns.

(a) Solid Support - Supelcon (100-120 mesh) or Chromasorb W (100-120 mesh).

(b) Liquid Phases - Expressed as weight-percent coated on solid support.

3% OV-1.

1.5% OV-17 + 1.95% QF-1.

4% SE-30 + 6% QF-1.

(c) Tubing - Pyrex (6' x 1/4" o.d. U shaped).

(5) Analysis Conditions for GLC.

(a) Oven temp - 150°C.

(b) Injector temp ~ 250°C.

(c) Outlet temp - 250°C.

(d) Detector temp ~ 290°C.

(e) Carrier gas flow - 60 ml/min.

(f) Sensitivity - 1.7×10^{-9} amp full scale (Input 10^2 Output 16).

(g) Recorder Speed - 2 in/min.

b. Glassware and Reagents.

(1) Conical flask - 100 ml, 250 ml.

(2) Centrifuge tube - 15 ml graduated.

(3) Filter Paper - Whatman No. 1. Hexane washed.

(4) Sodium Sulfate - Anhydrous, Hexane Washed.

(5) Methanol - Nanograde.

(6) 2,4,5-trimethylpentane - Nanograde.

3. EXTRACTION.

a. Weigh out 25 g soil or sediment (air dried) on balance. Remove large rocks, stones, debris from sample before weighing.

b. Place soil into stoppered 250 ml conical flask.

c. Add 50 ml methanol and shake for 1 hour, allow to settle.

d. Filter through Whatman No. 1 filter paper into stoppered tube. Collect 10 to 15 ml.

e. Transfer 5 ml aliquot to 100 ml stoppered conical flask. Add glass bead and concentrate to 0.5 ml on water bath.

f. Remove remaining methanol under nitrogen.

g. Dissolve residue in 5 ml iso-octane. Add 0.5 g Sodium sulfate. Stopper flask and shake vigorously for 1 min.

h. Proceed to GLC analysis.

Entomological Sp Study No. 44-005-75, IAAP, Burlington, IA, Sep 74 - Jan 75

Method tested only for Monuron at US Army Environmental Hygiene Agency.

Procedure modified from:

J. Chromatography

V.44:60-66 1969

C.E. McKone

The Determination of Some Substituted Urea Herbicide Residues
in Soil By Electron-Capture Gas Chromatography

Lower Limits of Detectability of
Substituted Urea Herbicides in Soil and Sediment

<u>Pesticide</u>	<u>Lower Limit of Detectability (ppm)</u>
monuron	*
benzomarc	*
chlorbromuron	*
diuron	*
flometuron	*
metobromuron	*

*Lower limits of detectability not determined at this time.

APPENDIX D
SILICIC ACID COLUMN PROCEDURE

1. REFERENCE. Patuxent National Wildlife Research Center.

2. SUMMARY. The pesticides and PCB's are separated on the silicic acid column into three fractions. The first fraction contains mirex, hexachlorobenzene and some late eluting (on a GC column) PCB's. The second fraction contains PCB's and DDE. The third or polar fraction contains most of the common pesticides including toxaphene and 5442.

3. REAGENTS.

a. Silicic acid - Mallinckrodt, Silicar CC-4 special for column chromatography. When using Silicar CC-4 air pressure is not needed in order to obtain the desired flow rate.

b. Place approximately 800 gms of silicic acid in an open pan. (Cover pan with aluminum foil and poke some holes in it.) Heat for 24 hours or longer in a 130°C oven. After heating, put in jar, place jar in a desiccator and let cool to room temperature. Quickly weigh silicic acid into glass-stoppered flask and add approximately 3 percent water. (100 gms of Silicar plus 3 ml hexane washed distilled water.) The amount of added water must be adjusted by using standards. (Silicar is more stable and need not be adjusted everytime.) All of DDE should be in the second fraction. Stopper flask tightly and seal with parafilm tape. Shake well on shaker 3 hours and place sealed container in desiccator and allow to equilibrate 15 hours. Return container to desiccator immediately after use. Desired activity remains for about 5 days.

4. NONPOLAR ELUTING MIXTURE. Petroleum ether.

5. POLAR ELUTING MIXTURE. 1% acetonitrile, 19% hexane, and 80% methylene chloride. Pipet 10 ml acetonitrile into 1 liter volumetric flask; add 190 ml hexane and make to volume with methylene chloride.

6. APPARATUS. Chromatographic column - 400 x 22 mm i.d. with 24/40 outer joint, coarse fritted plate, and Teflon stopcock (Kontes Glass Co. K420550, C-4). Separatory funnel - 500 ml with Teflon stopcock, 24/40 inside joint on stem, and 24/25 outside joint at top (Kontes Glass Co. K633030).

7. PROCEDURE.

a. Weigh 20 gms of silicic acid and immediately slurry with 80 ml petroleum ether, mixing well. Pour slurry into column with stopcock open, rinsing side of column with small portion of petroleum ether. Tap the column with a spatula while allowing the petroleum ether to drain out. When the

petroleum ether level is about 3 mm above surface of the silicic acid (never allow column to go dry) close the stopcock. Discard solvent.

b. Place 100 ml volumetric receiver under column to collect eluate. Pipet an aliquot of not more than 5 ml cleaned-up sample in hexane or petroleum ether and place on column. Add the sample slowly and carefully; touch the tip of the pipet to the side of the column so as not to disturb the top of the silicic acid. Open stopcock until solvent level is 3 mm above the silicic acid. Pipet 3,2 ml portions of petroleum ether onto column allowing the ether to flow slowly down inside the column and draining each portion to 3mm above surface of silicic acid, close stopcock. Pipet 10 ml of petroleum ether onto the column with the same care as before but do not open stopcock. Place separatory funnel containing 400 ml of petroleum ether on top of the column, open stopcock and obtain an elution rate of approximately 5 ml/min. When eluate volume is exactly 100 ml, remove 100 ml volumetric receiver and without stopping, place 300 ml volumetric receiver under column. When the second eluate volume is exactly 300 ml, place a 500 ml Kuderna-Danish dish with a 10 ml concentrator tube under the column and continue elution until petroleum ether eluate is approximately 3 mm above silicic acid, close stopcock. Add 200 ml of polar eluting mixture to reservoir. Pipet 10 ml of this eluting mixture slowly down the sides of the column so as not to disturb the surface of the silicic acid. Open stopcock and continue elution until all eluant passes through column. Concentrate the three eluant fractions and transfer to a 10 ml centrifuge tube.

8. FRACTION I. 100 ml petroleum ether containing mirex and HCB.
9. FRACTION II. 300 ml petroleum ether containing DDE and PCB's.
10. FRACTION III. Rest of pesticides.