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FEB 79 J F SUPROCK, J H VINOPAL, J M HELLER

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PESTICIDE MONITORING STUDY, NO. 17-44-0921-79
EVALUATION OF SILICIC ACID COLUMN PESTICIDE/POLYCHLORINATED BIPHENYL SEPARATION PROCEDURE: RECOVERY AND ELUTION PATTERNS OF 24 PESTICIDES AND PESTICIDE METABOLITES AND TWO POLYCHLORINATED BIPHENYLS.
SEPTEMBER 1977 - FEBRUARY 1979

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J. Howard /Vinopal

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

This report presents silicic acid column recovery and elution characteristics for 24 pesticides and pesticide metabolites and two polychlorinated biphenyls. The average percent recoveries for all except a few compounds studied were essentially quantitative. Overall average recoveries ranged from 72.7 percent for α-BHC to 111.4 percent for o,p'-DDE. The average percent relative standard deviation of the procedure for all but a few compounds studied was within 10 percent. All the compounds studied except for toxaphene, Aroclor 1260, and technical chlordane eluted in only one fraction. An in-depth discussion of the

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Item 20 (Cont). elution characteristics and the quantitation techniques for the above three compounds is presented.

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ABERDEEN PROVING GROUND, MARYLAND 21010

Mr. Suprock/lr/584-3613

HSE-RP-MO/WP

28 MAR 1979

SUBJECT: Pesticide Monitoring Study No. 17-44-0921-79, Evaluation of Silicic Acid Column Pesticide/Polychlorinated Biphenyl Separation Procedure: Recovery and Elution Patterns of 24 Pesticides and Pesticide Metabolites and Two Polychlorinated Biphenyls, September 1977 - February 1979

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A summary of the pertinent findings and conclusions of the inclosed report follows:

This study was conducted to evaluate in detail the silicic acid column recovery and elution characteristics for 24 pesticides/pesticide metabolites and two polychlorinated biphenyls analyzed for in Department of the Army Pesticide Monitoring Program environmental samples. The average percent recoveries for all but a few compounds studied were essentially quantitative. The average recoveries ranged from 72.7 percent for α-BHC to 111.4 percent for o,p'-DDE. The average percent relative standard deviation of the procedure for all but three compounds studied was within 10 percent. Only Aroclor 1254, p,p'-DDT, and heptachlor indicated relative standard deviations greater than 10 percent. All the compounds studied, except for toxaphene, Aroclor 1260, and technical chlordane, eluted in only one fraction. A discussion of column elution characteristics and quantitation techniques for the above three compounds is given.

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PESTICIDE MONITORING STUDY NO. 17-44-0921-79
EVALUATION OF SILICIC ACID COLUMN PESTICIDE/POLYCHLORINATED BIPHENYL SEPARATION PROCEDURE: RECOVERY AND ELUTION PATTERNS OF 24 PESTICIDES AND PESTICIDE METABOLITES AND TWO POLYCHLORINATED BIPHENYLS
SEPTEMBER 1977 - FEBRUARY 1979

1. AUTHORITY.

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

b. AR 200-1, Environmental Protection and Enhancement, 20 January 1978.

2. PURPOSE. To provide necessary data on the recovery and elution patterns of pesticides, pesticide metabolites and polychlorinated biphenyls (PCB) from silicic acid (SilicAR® CC-4) columns currently being used in routine Department of the Army Pesticide Monitoring Program (DAPMP) analytical methodology.

3. GENERAL.

a. Background.

(1) The accurate quantitation and determination of pesticide residues in environmental samples has been hampered by the presence of PCB's since these chemicals were first detected in Sweden in 1966. In 1970, a method was

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reported by Armour and Burke[1] for the separation of PCB's from the more common chlorinated pesticides. Cromartie, et al.[2] later modified this procedure and it was adopted by the DAPMP after a preliminary in-house examination[3].

(2) The need for a more detailed in-house examination of the Cromartie, et al.,[2] procedure was evident due to certain peculiarities and uncertainties in pesticide and PCB elution patterns noted during analyses of routine DAPMP environmental samples.

b. Methodology. Pertinent aspects of the analytical methods and procedures used in this study are available in Appendix A, which is organized as follows:

(1) Part I - Spiking Methodology

(2) Part II - Chromatographic Column Methodology

(3) Part III - Gas Liquid Chromatography (GLC) Techniques

4. RESULTS.

a. Appendix B lists the lower limits of instrumental sensitivity and the analytical limits of detectability in fish and bird samples for the pesticides, pesticide metabolites, and PCB's used in this study.

b. Appendix C lists each compound studied by spiking set and the silicic acid column fraction(s) in which they elute. Appendix C also contains the mean ($\bar{x}$) standard deviation (S), standard deviation of the mean ($S\bar{x}$), and the coefficient of variability (CV) for the six recovery and evaluation experiments (i.e., two replicates at three different spiking levels) performed with each compound.

---

[1] Armour, J. A. and J. A. Burke, "Method for Separating PCBs from DDT and its Analogs," J Assoc Offic Anal Chem, 53:761-7 (1970)
[2] Cromartie, E., W. L. Reichel, L. N. Locke, A. A. Belisle, T. E. Kaiser, T. G. Lamont, B. M. Mulhern, R. M. Prouty, and D. M. Swineford, "Residues of Organochlorine Pesticides and Polychlorinated Biphenyls and Autopsy Data for Bald Eagles, 1971-72," Pest Monit J, 9(1):11-14 (1975)
[3] Entomological Special Study No. 44-042-74/75, Extraction and Separation of Polychlorinated Biphenyls from Pesticide Monitoring Samples, 15 April 1975, Natl Tech Inform Serv, AD-A011-242, 10 pp (1975)

5. DISCUSSION.

a. Better than 95 percent of all DAPMP routine biological samples analyzed contain detectable levels of PCB's. These PCB's interfere with the identification and quantitation of those pesticides and their metabolites that elute in the 6-percent ethyl ether/petroleum ether Florisil® cleanup fraction. Therefore, since Calendar Year 1975, all 6-percent Florisil fractions of biological samples have been routinely taken through the silicic acid column fractionation procedure of Cromartie, et al.[2] Hence, the rationale for selecting the particular compounds used in this study is that they all elute in the 6-percent Florisil fraction.

b. Of the 24 pesticides and pesticide metabolites used in this study, only cis- and trans-nonachlor are not presently being routinely analyzed for in DAPMP samples. However, cis- and trans-nonachlor were included in this study because these compounds are analyzed for in certain special request-type DAPMP samples and they are presently being routinely analyzed for by certain other monitoring laboratories. Aroclors® 1254 and 1260 were selected because they are the most commonly occurring PCB's found in biological samples.

c. As evident from the data summarized in Appendix C, the average percent recoveries for all but a few compounds studied were essentially quantitative. Overall, the average recoveries ranged from 72.7 percent for α-BHC to 111.4 percent for o,p'-DDE. The relatively low recovery of α-BHC was probably due to its more volatile nature and, therefore, its susceptibility to losses during concentration and solvent transfer steps.

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[2] Cromartie, E., W. L. Reichel, L. N. Locke, A. A. Belisle, T. E. Kaiser, T. G. Lamont, B. M. Mulhern, R. M. Prouty, and D. M. Swineford, "Residues of Organochlorine Pesticides and Polychlorinated Biphenyls and Autopsy Data for Bald Eagles, 1971-72," Pest Monit J, 9(1):11-14 (1975)

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d. The data in Appendix C also indicate that the average percent relative standard deviation (CV) of the procedure for all but three compounds studied was within 10 percent. Only p,p'-DDT, Aroclor 1254 and heptachlor indicated relative standard deviations greater than 10 percent.

e. All compounds studied except for toxaphene, Aroclor 1260 and technical chlordane eluted in only one silicic acid column fraction at all spiking levels studied. However, in a few instances the elution rate of some columns varied from the standard 5 ml/min before being readjusted. In these instances, small but varying amounts of certain compounds eluted into earlier or later fractions.

(1) At higher spiking levels, many of the components of toxaphene were noted in silicic acid column Fraction II. Although the amount of toxaphene components eluting in Fraction II is a relatively small proportion ($\sim$10 percent) of the total compound applied to the column, significant interference with the identification and quantitation of PCB's (which normally elute in Fraction II) would occur in those samples containing both PCB's and higher residues of toxaphene.

(2) Several components of Aroclor 1260 eluted in silicic acid column Fraction I at all the spiking levels studied.[3] The proportion of the Aroclor 1260 components noted in Fraction I to the total amount of Aroclor 1260 applied to the column remained constant through all the spiking levels studied. One of the Fraction I Aroclor 1260 components had a relative retention time very close to that of mirex (which normally elutes in Fraction I) on the 1.5 percent OV-17 +1.95 percent QF-1 primary column and could possibly be misidentified as mirex. If mirex is suspected of being in a sample containing Aroclor 1260, another column such as 5 percent OV-210 should be used to confirm or negate this suspicion. Both Fraction I and Fraction II should be combined to quantitate the total Aroclor 1260 in the sample.

(3) The silicic acid column fractionation behavior of technical chlordane is discussed below:

(a) Individual components of technical chlordane eluted into both silicic acid column Fraction II and Fraction III. Figures 1 and 2 illustrate the component peaks of technical chlordane contained in each of the fractions as compared to a standard of unfractionated technical chlordane (Figure 3). Technical chlordane is a mixture of five main components (components A, B, C, D and E of Figure 3) on the primarily used 1.5 percent OV-17/1.95 percent QF-1 gas chromatographic column. Due to its silicic acid column fractionation behavior, technical chlordane can present somewhat of a quantitation problem if present in samples carried through the procedure. Fortunately, to date, technical chlordane <u>per se</u> has not been observed in fish and bird samples routinely analyzed under the DAPMP. Only metabolized,

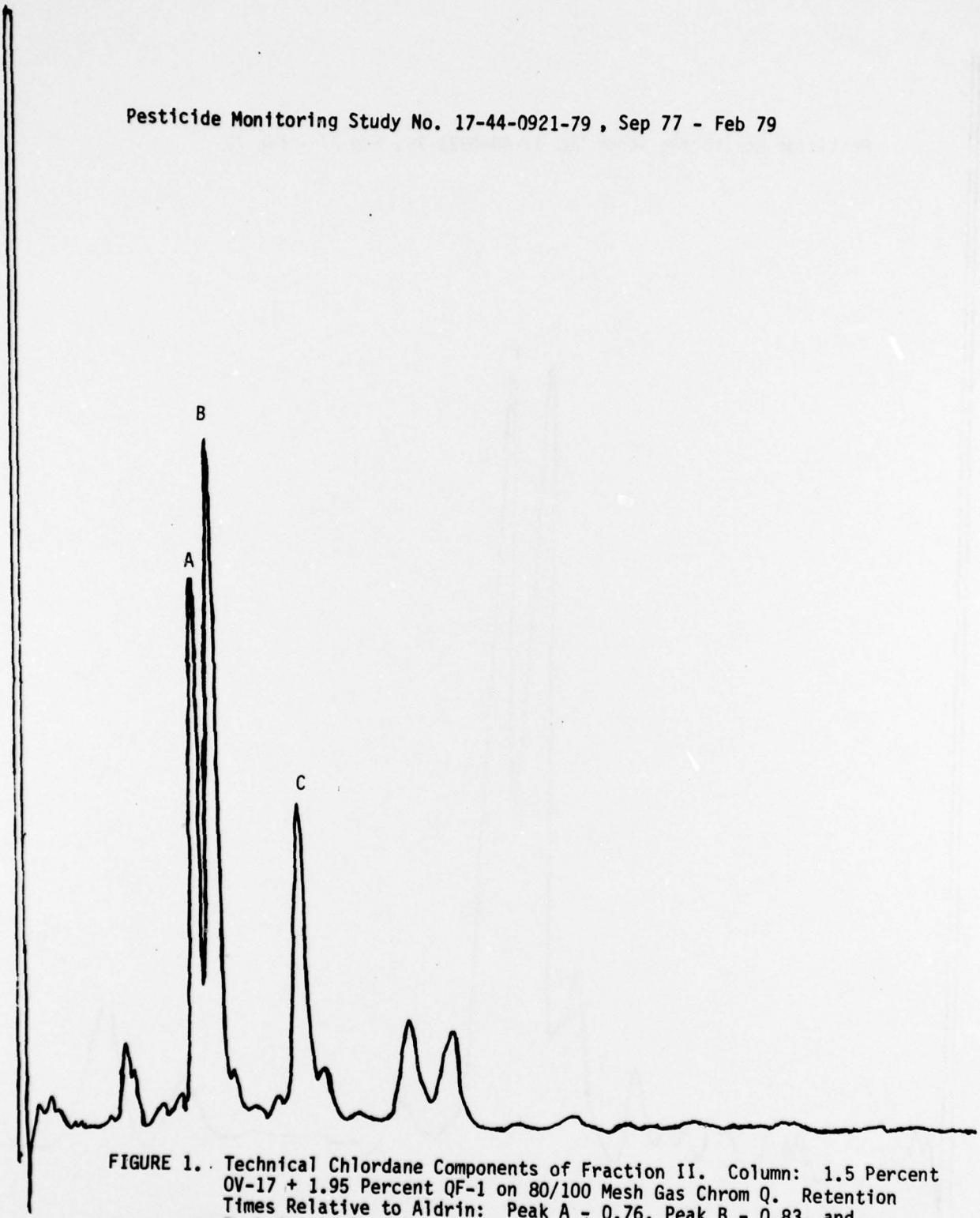

FIGURE 1. Technical Chlordane Components of Fraction II. Column: 1.5 Percent OV-17 + 1.95 Percent QF-1 on 80/100 Mesh Gas Chrom Q. Retention Times Relative to Aldrin: Peak A - 0.76, Peak B - 0.83, and Peak C - 1.15.

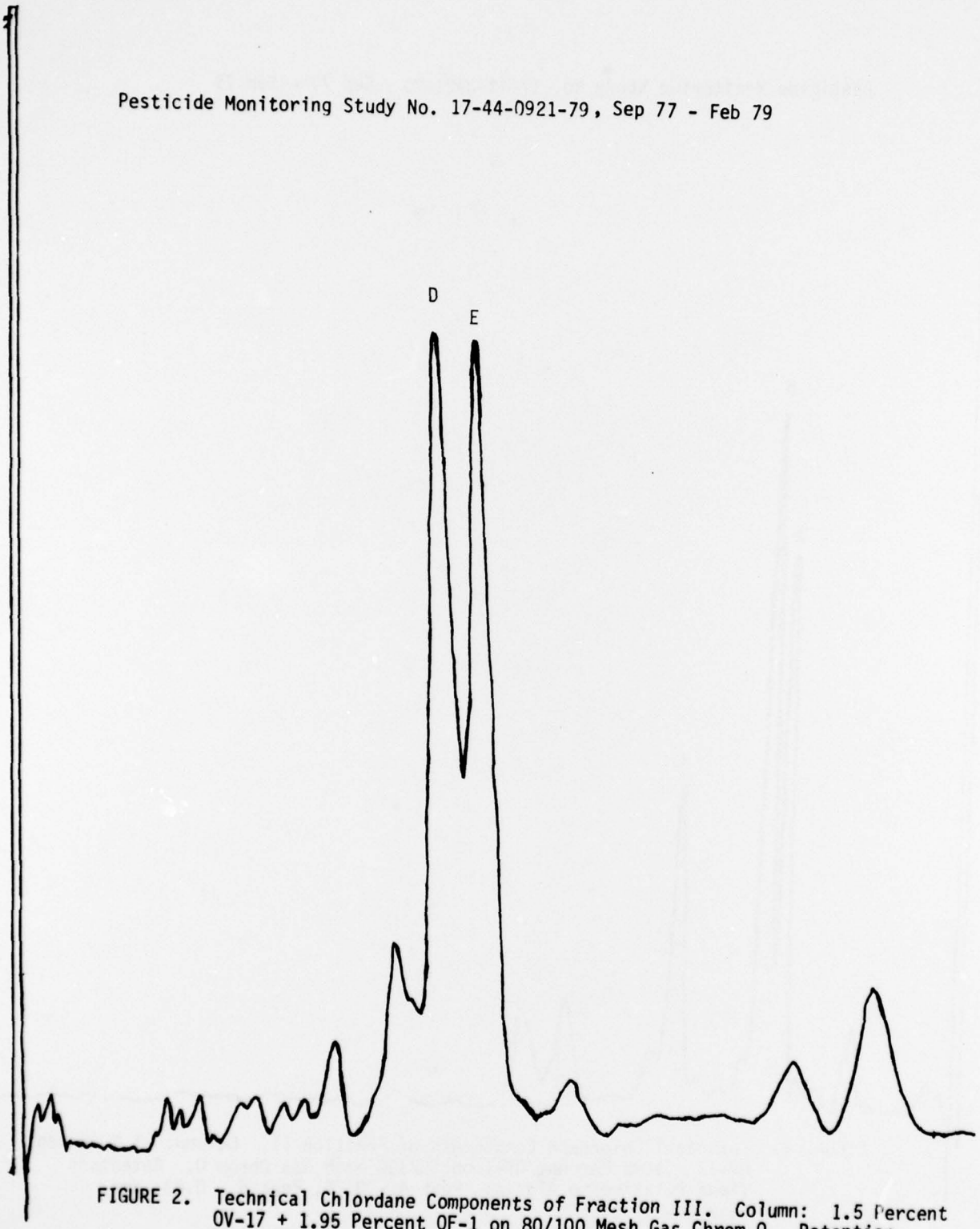

FIGURE 2. Technical Chlordane Components of Fraction III. Column: 1.5 Percent OV-17 + 1.95 Percent QF-1 on 80/100 Mesh Gas Chrom Q. Retention Times Relative to Aldrin: Peak D - 1.67, Peak E - 1.81.

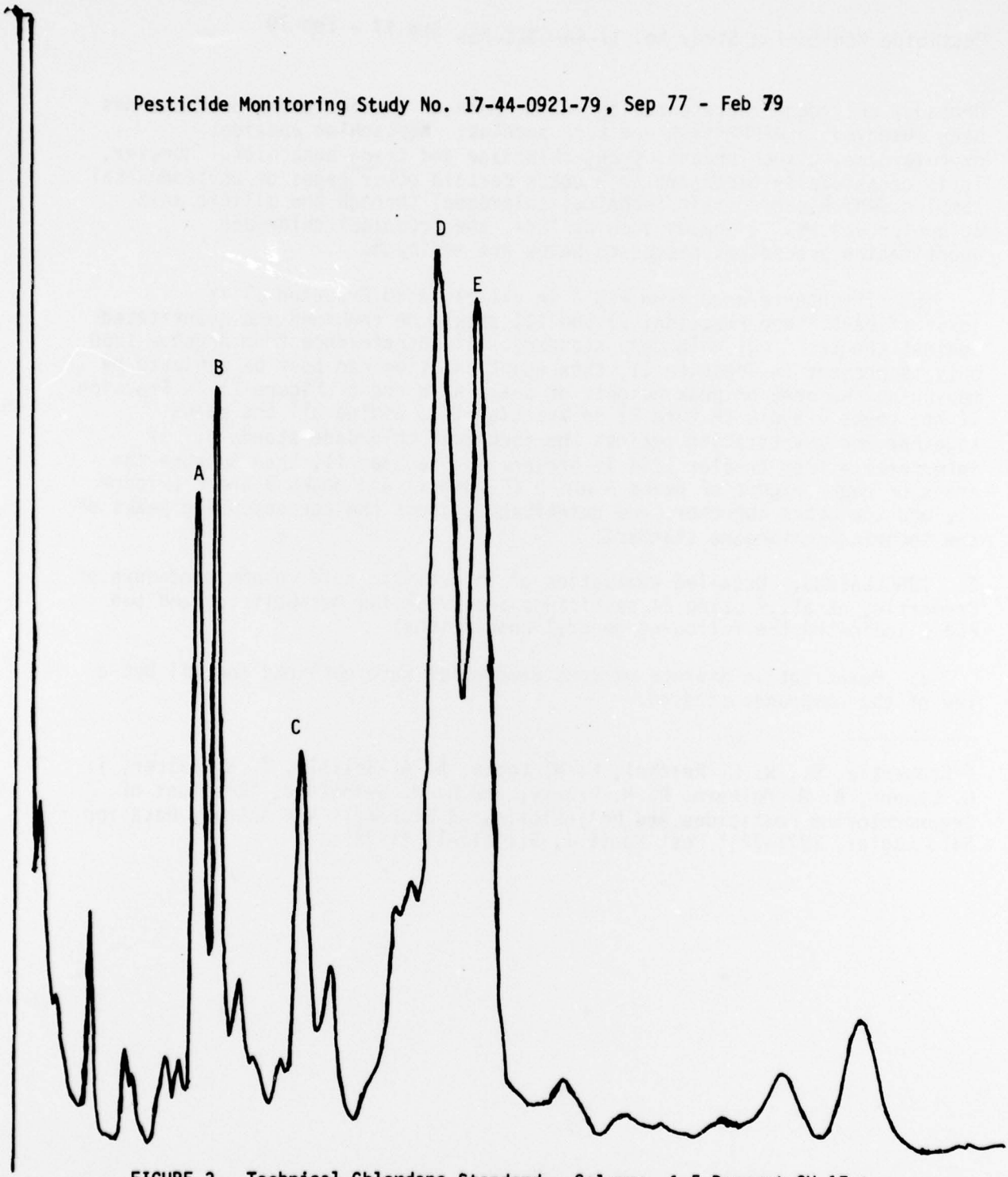

FIGURE 3. Technical Chlordane Standard. Column: 1.5 Percent OV-17 + 1.95 Percent QF-1 on 80/100 Mesh Gas Chrom Q. Retention Times Relative to Aldrin: Peak A - 0.76, Peak B - 0.83, Peak C - 1.15, Peak D - 1.67, and Peak E - 1.81.

degraded chlordane represented by one or more of the following products has been observed in DAPMP fish and bird samples: heptachlor epoxide, oxychlordane, cis-chlordane, trans-chlordane and trans-nonachlor. However, it is occasionally necessary to process certain other types of environmental samples (which can contain technical chlordane) through the silicic acid column procedure. In cases such as this, the technical chlordane quantitation procedures discussed below are employed.

(b) If interference from PCB's in silicic acid Fraction II is insignificant, then Fractions II and III should be combined and quantitated against the technical chlordane standard. If interference from Aroclor 1260 only is present in Fraction II, then quantitization can best be achieved by measuring the area or peak heights of peaks A, B and C (Figure 1) in Fraction II and peaks D and E (Figure 2) in Fraction III, adding all the peaks together and quantitating against the technical chlordane standard. If interference from Aroclor 1254 is present in Fraction II, then measure the areas or peak heights of peaks A and B (Figure 1) and peaks D and E (Figure 2), add the peaks together, and quantitate against the corresponding peaks of the technical chlordane standard.

6. CONCLUSIONS. Detailed evaluation of the silicic acid column procedure of Cromartie, et al.,[2] using 24 pesticides and pesticide metabolities and two PCB's indicated the following general conclusions:

a. Quantitative average percent recoveries were obtained for all but a few of the compounds studied.

---

[2] Cromartie, E., W. L. Reichel, L. N. Locke, A. A. Belisle, T. E. Kaiser, T. G. Lamont, B. M. Mulhern, R. M. Prouty, and D. M. Swineford, "Residues of Organochlorine Pesticides and Polychlorinated Biphenyls and Autopsy Data for Bald Eagles, 1971-72," Pest Monit J, 9(1):11-14 (1975)

b. The average percent relative standard deviations (CV) of the procedure was within 10 percent for all except three of the compounds studied.

c. All of the compounds studied, except toxaphene, Aroclor 1260 and technical chlordane, eluted in only one silicic acid column fraction at all spiking levels studied.

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APPENDIX A

ANALYTICAL METHODS AND PROCEDURES

1. METHODOLOGY USED TO SPIKE SILICIC ACID COLUMNS.

a. Pesticides, pesticide metabolites and PCB's used in this study were made up in Iso-octane from stock solutions and grouped into seven different spiking sets containing from one to six compounds. This was done to prevent compounds with close relative retention times from interfering with the quantitation of one another.

b. Recovery studies for each set of compounds were duplicated at three different spiking levels representing compound concentration ranges from approximately 3 to 50 times above analytical limits of detectability currently used for fish and bird samples. Analytical detection limits for fish and birds are based on a 50-gram wet weight sample and a definitive extract volume of 100 ml for the chlorinated hydrocarbon pesticides and metabolities, and a definitive extract volume of 10 ml for PCB's and organophosphorus pesticides.

c. In a typical spiking procedure, 1 ml of a spiking set solution (containing from one to six compounds) was transferred to a graduated centrifuge tube and diluted to 5 ml with petroleum ether. The solution was mixed and then pipetted directly onto the silicic acid column. In three cases (toxaphene and the two PCB's), 2 to 5 ml of spiking solution had to be added to graduated centrifuge tubes, concentrated to 1 ml, then diluted to 5 ml with petroleum ether.

2. SILICIC ACID COLUMN METHODOLOGY.

a. Apparatus and Materials.

(1) Glassware.

(a) Chromatographic columns - 400 x 22 mm i.d. with 24/40 outer joint, coarse fritted disc, and Teflon® stop cocks (Kontes Glass Co., K420550, C-4)

(b) Separatory funnels - 500 ml with Teflon stopcocks, 24/40 inside joint on stem, and 24/25 outside joint at top (Kontes Glass Co., K633030)

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(c) Kuderna-Danish apparatus - 250 ml and 500 ml flasks, 10 ml concentrator tubes, Snyder columns.

(d) Graduated cylinders - 10 ml, 25 ml, 250 ml and 500 ml

(e) Glass powder funnels

(f) Glass-stoppered reagent bottles - 500 ml

(g) Graduated centrifuge tubes - 15 ml

(h) Beakers - 250 ml

(i) Stainless steel spatulas

(j) Screw-cap culture tubes with Teflon cap liners - 16 x 125 mm

(k) Volumetric pipets - 1, 2 and 5 ml

(2) Reagents, Solvents and Standards.

(a) Petroleum ether - pesticide grade

(b) Hexane - pesticide grade

(c) Methylene chloride - pesticide grade

(d) Acetonitrile - pesticide grade

(e) Iso-octane (2,2,4-trimethylpentane) - pesticide grade

(f) Silicic acid - Mallinckrodt, Silicar CC-4

(g) Pesticide and PCB standards - analytical reference grade

b. Silicic Acid Column Procedures.

(1) Silicic Acid Preparation. Silicic acid was placed in an open porcelain-coated pan and the pan was then covered with aluminum foil. Several small holes were poked in the foil and the pan was then placed in a 130°C oven for 24 hours. After 24 hours, the pan was removed from the oven and 50-g aliquots of silicic acid were quickly weighed into 500-ml glass-stoppered reagent bottles. The bottles were placed in a dessicator and cooled to room temperature. The bottles were then removed from the dessicator and 1.5-ml hexane-washed distilled water was added to each bottle. The bottles were then stoppered and sealed with masking tape, placed on a wrist-action shaker and shaken for 4 hours. After 4 hours, the bottles were

returned to the dessicator and allowed to equilibrate for 15 hours before use. Unusued silicic acid was returned to the dessicator. Desired activity lasts about 5 days.

(2) Elution Mixtures and Volumes.

(a) Nonpolar elution mixture - petroleum ether: 100 ml - Fraction I, 300 ml - Fraction II

(b) Polar elution mixture - 1-percent acetonitrile, 19-percent hexane and 80-percent methylene chloride: 200 ml - Fraction III

(3) Preparation of the Silicic Acid Column. Twenty grams of silicic acid was weighed into a 200-ml beaker and immediately slurried with 80-ml petroleum ether. With the aid of a glass funnel, the slurry was then poured into the column with stopcock open. The beaker was rinsed with a small portion of petroleum ether and then added to the column. The funnel and the inside of the column were then rinsed down with an additional small amount of petroleum ether. As the petroleum ether was draining out, the column was constantly being tapped with a spatula. When the level of petroleum ether was 3 mm above the surface of the silicic acid (never allow column to go dry), the stopcock was closed.

(4) Elution of the Compounds from the Columns. One-hundred ml graduated cylinders were placed under the columns for collection of the Fraction I eluate. The spiking set solutions (adjusted to 5 ml with petroleum ether) were then pipetted onto each column. This was done slowly and carefully, touching the tip of the pipet to the side of the columns so as not to disturb the top of the silicic acid. The stopcock was then opened until the solvent level was 3 mm above the surface of the silicic acid. Six ml of petroleum ether was then used to rinse down the walls of each column by pipetting in the same manner as above. The stopcock was opened and the columns allowed again to drain to 3 mm above the surface of the silicic acid. An additional 10 ml of petroleum ether was then added to the columns with the same care as before but not allowed to drain. Separatory funnels containing 400-ml petroleum ether were then placed on top of the columns, the stopcocks were opened, and an elution rate of 5 ml/min was commenced. When the Fraction I eluate volume reached 100 ml, the graduated cylinders were removed quickly and replaced by 500-ml graduated cylinders without closing the stopcocks. When the Fraction II eluate volume reached 300 ml, the graduated cylinders were quickly removed and replaced by 500-ml Kuderna-Danish apparatus and the elution continued until the petroleum ether in the columns was 3 mm above the surface of the silicic acid. The stopcocks were then closed and 200 ml of polar eluting mixture was added to each separatory funnel reservoir. Ten ml of the 200-ml polar eluting mixture was then pipetted onto each column with the same care used earlier; the stopcocks were opened and, again, an elution rate of 5 ml/min was obtained. The elution was continued until all Fraction

III eluant passed through the columns. All three eluant fractions were then concentrated to about 10 ml in Kuderna-Danish apparatus in a water bath. Fraction I and II eluates were transfered to 10-ml graduated cylinders and concentrated (under nitrogen) or diluted (using iso-octane) as required to achieve exactly 10 ml. The eluate fractions were mixed and then transferred to Teflon-lined, screw-cap, culture tubes and stored in a freezer until analysis. Each Fraction III eluate was transferred to 10-ml graduated cylinders and further concentrated to approximately 2 ml. Each eluate was then brought up to 10-ml volume with petroleum ether and reconcentrated to 2 ml again. The 2-ml eluate concentrates were finally adjusted to exactly 10 ml using Iso-octane and mixed. The eluate fractions were transferred to culture tubes and stored in a freezer until analysis. The extra concentration steps carried out with Fraction III eluates are required for removal of methylene chloride prior to analysis.

3. GAS-LIQUID CHROMATOGRAPHY (GLC) ANALYSIS PARAMETERS AND TECHNIQUES.

a. Apparatus and Materials.

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

(2) Detectors: Tracor high-temperature Ni^{63} electron-capture (EC) and Melpar dual flame photometric (FPD).

(3) Recorder: Honeywell Electronic potentiometric strip chart (10 in, 1 mV).

(4) Gas Chromatographic Columns: 1.5 percent OV-17/1.95 percent QF-1 on 80/100 mesh Gas Chrom Q (EC), 3 percent OV-1 on 100/120 mesh Gas Chrom Q (FPD).

(5) Routine Analysis Parameters for GLC:

(a) Oven temperature - 200°C

(b) Injection port temperature - 230°C

(c) EC detector temperature - 305°C

(d) FPD detector temperature - 215°C

(e) Carrier gas flow EC (5 percent methane in argon) - 52 ml/min

(f) Carrier gas flow, FPD (nitrogen) - 60 ml/min

(g) FPD detector gas: Hydrogen - 50 ml/min, Air - 90 ml/min

(h) Electrometer sensitivity - electron-capture: 0.8×10^{-9} amps full scale (input 10^2; output 8)

(i) Electrometer sensitivity - FPD: 4.0×10^{-9} amps full scale (input 10^3, output 4)

(j) Recorder speed: 0.5 in/min

b. GLC Quantitation Techniques.

(1) Automatic Integration Technique. All but five compounds were quantitated using the Spectra Physics - SP4000 Computing Integrator (Spectra Physics, Mountain View, CA).

(2) Manual (Peak Height) Technique. In this technique, heights of chromatographic peaks were measured from their baselines to their apex. Organophosphorus compounds and toxaphene were quantified using this method.

(3) Manual (Peak Area) Technique. In this technique, the area of chromatographic peaks were measured by the formula: Area = peak height x width at one-half the peak height. The PCB's were quantitated by this method using the total areas of six of the most prominent symmetrical peaks.

APPENDIX B

LOWER LIMITS OF INSTRUMENTAL SENSITITIVITY AND ANALYTICAL LIMITS OF DETECTABILITY IN FISH AND BIRDS FOR PESTICIDES, PESTICIDE METABOLITES AND PCB's STUDIED

Compound	Lower Limits of Instrumental Sensitivity - Picograms Required for 10% Full Scale Recorder Deflection Using EC Detection* (Based on 5-ml Injection Volume)	Lower Limit of Detectability in Fish and Birds (ppm)
α-BHC	3.1	0.002
β-BHC	12.5	0.005
aldrin	10.0	0.004
chlordane (tech)	75.0	0.030
cis-chlordane	10.0	0.004
trans-chlordane	10.0	0.004
o,p'-DDD	25.0	0.010
p,p'-DDD	20.0	0.008
o,p'-DDE	25.0	0.010
p,p'-DDE	20.0	0.008
o,p'-DDT	25.0	0.010
p,p'-DDT	37.5	0.015
heptachlor	4.0	0.002
heptachlor epoxide	10.0	0.004
lindane	5.0	0.002
methoxychlor	100.0	0.040
mirex	25.0	0.010
oxychlordane	10.0	0.004
toxaphene	1000.0	0.400
cis-nonachlor	25.0	0.010
trans-nonachlor	10.0	0.004
chlorypyrifos	200 pg (FPD-10 µl)	0.004 (FPD)
ronnel	200 pg (FPD-10 µl)	0.004 (FPD)
hexachlorobenzene	4.0	0.002
Aroclor 1254	500.0	0.020
Aroclor 1260	500.0	0.020

* For chlorypyrifos and ronnel, lower limits of instrumental sensitivity and analytical limits of detectability in fish and birds using flame photometric detection (FPD) are given.

APPENDIX C

SUMMARY OF SILICIC ACID COLUMN RECOVERY DATA AND
ELUTION PATTERNS FOR PESTICIDES, PESTICIDE METABOLITES AND PCB's STUDIED

Compound (Grouped by Spiking Set)	Silicic Acid Column Elution Fraction	Spiking Range Studied (µg)*	Total No. Recovery Experiments	Recovery Data (percent) $\bar{x}$	S	$S\bar{x}$	CV
Aroclor 1260	I + II	3-50	6	104.0	4.31	1.76	4.14
Aroclor 1254	II	3-50	6	98.8	14.3	5.84	14.48
toxaphene	II + III	60-1000	6	104.2	5.47	2.23	5.25
oxychlordane	III	0.6-10.0	6	99.6	4.80	1.96	4.82
o,p'-DDE	II	1.5-25.0	6	111.4	1.38	0.56	1.24
ronnel	III	0.6-10.0	6	95.6	7.22	2.95	7.55
lindane	III	0.3-5.0	6	96.0	7.20	2.94	7.50
p,p'-DDE	II	1.2-20.0	6	108.9	1.97	0.81	1.81
hexachlorobenze	I	0.3-5.0	6	106.6	5.26	2.15	4.94
technical chlordane	II + III	4.5-75.0	6	94.0	7.0	2.86	7.44
o,p'-DDD	III	1.5-25.0	6	99.1	9.6	3.92	9.69
p,p'-DDT	III	2.25-37.5	6	96.3	10.48	4.28	10.88
methoxychlor	III	6.0-100.0	6	107.6	8.46	3.45	7.86
mirex	I	1.5-25.0	6	102.0	4.84	1.98	4.74
heptachlor	II	0.3-5.0	6	95.1	15.06	6.15	15.83
α-BHC	III	0.3-5.0	6	72.7	6.39	2.61	8.79
chlorpyrifos	III	0.6-10.0	6	90.5	5.73	2.34	6.33
cis-chlordane	III	0.6-10.0	6	93.6	5.53	2.26	5.90
trans-chlordane	III	0.6-10.0	6	93.5	8.02	3.27	8.58
cis-nonachlor	III	1.5-25.0	6	95.5	3.30	1.35	3.45
aldrin	II	0.6-10.0	6	105.0	4.59	1.87	4.37
trans-nonachlor	III	0.6-10.0	6	89.2	7.18	2.93	8.05
β-BHC	III	0.75-12.5	6	87.7	2.47	1.01	2.81
heptachlor epoxide	III	0.6-10.0	6	88.2	3.61	1.47	4.09
o,p'-DDT	III	1.5-25.0	6	96.8	2.25	0.92	2.32
p,p'-DDD	III	1.2-20.0	6	102.1	3.15	1.28	3.08

* Spiking ranges studied for each compound represent concentration ranges from appropriately 3X to 50X above analytical detection limits for fish and bird samples.

APPENDIX D

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