



## *Project Summary*

# Determination of Pesticides and PCBs in Industrial and Municipal Wastewaters

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John D. Millar and Richard E. Thomas

Steps in the procedure for the analysis of 25 chlorinated pesticides and polychlorinated biphenyls were studied. Two gas chromatographic columns and two detectors, electron capture and Hall electrolytic conductivity, were evaluated. Extractions were performed with two solvents—dichloromethane and 15% dichloromethane in hexane—at three pHs to determine extraction efficiencies. The effects of storage for seven days, in the presence of residual chlorine, at two temperatures were determined. Florisil and alumina were compared as adsorbents for the clean up of extracts. Recoveries of the substances from clean water and wastewater were measured, and assessments of accuracy and precision were made.

The method is satisfactory for the analysis of clean waters and wastewaters having a relatively low background of interferences. However, it does not work well against medium to high levels of background interferences produced by substances that are electron capture sensitive, especially halogenated ones. Use of the Hall detector is indicated when non-halogenated electron capture sensitive interferences are a problem, even though some loss in sensitivity will

occur. When halogenated interferences are overwhelming, altered gas chromatography conditions and columns, such as temperature programming and columns which produce better resolution than the ones studied in this work, will be required.

*This Project Summary was developed by EPA's Environmental Monitoring and Support Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).*

### Introduction

Under provisions of the Clean Water Act, the Environmental Protection Agency is required to promulgate guidelines establishing test procedures for the analysis of pollutants. The Clean Water Act Amendments of 1977 emphasize the control of toxic pollutants and declare the 65 "priority" pollutants and classes of pollutants to be toxic under Section 307(a) of the Act. This report is one of a series that investigates the analytical behavior of selected priority pollutants and suggests a suitable test procedure for their measurement.

The substances studied in this effort were:

1.  $\alpha$ -BHC
2.  $\beta$ -BHC
3.  $\delta$ -BHC
4. Heptachlor epoxide
5. DDE (p,p')
6. DDD (p,p')
7. DDT (p,p')
8. Endosulfan sulfate
9.  $\gamma$ -BHC
10. Heptachlor
11. Aldrin
12. Endosulfan I
13. Dieldrin
14. Endrin
15. Endosulfan II
16. Endrin aldehyde
17. Chlordane
18. Toxaphene
19. PCB-1016
20. PCB-1221
21. PCB-1232
22. PCB-1242
23. PCB-1248
24. PCB-1254
25. PCB-1260

The study was conducted in two phases. In Phase I, work was conducted with clean water and was intended to provide information that would give direction to Phase II work, conducted on actual wastewaters, and serve as a comparison base for the information developed.

The ultimate objective was to develop a method, having a maximum of applicability, that could be published in the Federal Register and carried out in most analytical laboratories.

Other objectives included the development of accuracy and precision information, procedure variations as required by a particular wastewater, and the maximization of using common sample treatment steps for all the categories of priority pollutants.

## Phase I

### Gas Chromatography

The 25 substances can be gas chromatographed very well. Sensitivity quantities producing a response 10X noise level for the electron capture detector range from 1 to 380 picograms. Sensitivity quantities for the Hall electrolytic conductivity detector are roughly 10 to 100 times greater than this. The 16 single compound pesticides can be fully resolved if chromatographed in two groups of eight each. The PCBs are best chromatographed individually, but several combinations can be ade-

quately chromatographed for some analytical purposes.

The primary gas chromatography column used in this work, 1.5% SP2250 + 1.95% SP 2401 on 100/120 mesh Supelcoport, is preferred on a general use basis over 3% OV-1 on the same solid support. In limited situations, however, it is possible that either the OV-1 column or columns with other liquid phases, such as OV-17, may perform as well or better than the primary column.

A comparison of detectors showed that the Hall electrolytic conductivity detector is more discriminating than the electron capture detector, and it should be used as the primary detector, or used as an auxiliary detector when non-halogenated electron capture sensitive materials present interference problems.

### Extraction Study

The extraction study was initiated to determine the recoveries of the 25 substances of interest from clean water at pH 2, 7 and 10 using 15% dichloromethane (DCM) in hexane and 100% DCM as the extracting solvents.

The water used in the extraction study is a naturally buffered well water obtained from the Southwest Research Institute supply line prior to chlorination. Samples of this water were collected and transported to the laboratory in empty solvent bottles which had previously contained Burdick and Jackson solvents. The pH of the untreated water is close to 8. Adjustment to the required pHs was accomplished by adding strong acid or base.

Extraction of the pesticides from clean water at parts per trillion concentrations when the pH was 2, 7, or 10, was generally 80% or better using 15% DCM in hexane or pure DCM as the extracting solvent. When heptachlor and aldrin were used, however, extraction percentages were generally 50 to 75%. Occasionally, one of the solvents at a particular pH gave a more efficient extraction than did the other solvent-pH combinations, although the improvements were not great enough to demand the particular combination be adhered to exclusively. Under the same extraction conditions, PCB-1242, PCB-1248, PCB-1254, and PCB-1260 at low parts per billion levels were extracted with 90% or better recoveries. At similar levels, PCB-1016, PCB-1221, and PCB-1232 were extracted with adequate efficiency, but the recoveries recorded were occasionally as low as 60%.

No clear advantages have been noted for either solvent studied in this work. In the interest of commonality, either solvent may be chosen without an intolerable loss of efficiency occurring during sample extraction. The pH of the water at the time of extraction is not of substantial concern. However, if the substance being sought is designated beforehand, a slight advantage may be available in some instances by consulting the statistical analysis data for combined pH-solvent effects on the extraction of that particular substance.

### Preservation Study

The preservation study was conducted to determine the effects of a 90-day storage period, at various conditions, on the recovery of the 25 substances of interest from dosed water samples. Each sample consisted of one quart of water dosed with one of the eleven groups, as in the extraction study. Two replicates for each of twelve conditions of pH, temperature and residual chlorine were prepared for each group in the following model (Table 1):

The 2 ppm residual chlorine level was obtained where required, by adding 160 mL of Mallinckrodt sodium hypochlorite analytical reagent (5% minimum available Cl). Storage containers were one-quart, flint glass, round, narrow-mouth bottles closed with aluminum foil-lined caps. A sample was prepared by filling the bottle about two-thirds full with pH adjusted water, adding 160 mL sodium hypochlorite when required, swirling vigorously, adding the 100 mL dosing solution into the vortex, and combining the remainder of the pH adjusted water with the solution in the bottle while it was still swirling. The bottle was capped immediately and stored in a closed cardboard box at 4°C or 24°C. Care was taken not to slosh the bottle contents onto the aluminum lining the cap after closure.

The well water was dosed with a quantity of substance inversely related to the detector response for the substance. Thus,  $\alpha$ -BHC was spiked at the 20 parts per trillion level and toxaphene at the 4 ppb level. Other substances were dosed at levels in between these extremes. Extraction was performed with 15% DCM in hexane and with 100% DCM at pHs of 2, 7 and 10. The most serious problem encountered was the presence of interfering peaks in chromatograms of blanks. The varying magnitude of these peaks made subtraction of a constant blank usually not

possible. Occasionally, work had to be repeated.

At the concentrations used, some conditions of the preservation study produced pronounced effects on the substances. Deleterious effects were found to occur with about half of the 25 substances at some of the 12 combinations of pH, temperature, and residual chlorine levels studied. However, 23 of the substances were stored in the dark for seven days at 4°C, under neutral or acidic pH, and in the presence and absence of two ppb residual chlorine with the only losses suffered being of a tolerable magnitude. Aldrin sustained a 15 to 20% loss at acid or neutral pHs in the presence of chlorine. Endosulfan sulfate losses were 90% or more under all conditions of storage at 230 parts per trillion concentration in clean water. In the storage and recovery tests involving wastewater, however, endosulfan sulfate was dosed at the 15 ppb concentration and no large losses were observed.

Storage of samples before extraction should be at a neutral to an acidic pH and at 4°C. Storage periods of up to seven days' duration do not produce significant decreases in concentrations, except for endosulfan sulfate in concentrations of parts per trillion. For determinations at these levels, samples should be extracted immediately after sampling. If aldrin is to be determined, residual chlorine, if present, should be eliminated before storage of samples.

### Liquid-Solid Column Chromatography

Nearly 100% recoveries were achieved during column chromatography of substances applied to fully activated Florisil PR or alumina mixed with 10% by weight water. Elution patterns of the substances from the Florisil were more desirable than from the alumina. In application studies with wastewaters, the Florisil was slightly superior to the alumina in clean up of extracts.

### Florisil

The Florisil clean up column was prepared by gently packing Florisil PR, which was taken directly from storage at 130°C for a minimum of 24 hours, to a height of 10 cm (about 21 grams) in a 400mm x 25mm (OD) glass column containing a coarse fritted disc and fitted with a Teflon stopcock. A layer of sodium sulfate 1-2 cm in height was added. The column was prewetted with

Table 1. Experimental Model for Preservation Study

	4°C		24°C	
	0 ppm Cl	2 ppm Cl	0 ppm Cl	2 ppm Cl
pH 2	2	2	2	2
pH 7	2	2	2	2
pH 10	2	2	2	2

60 mL of hexane and drained to the top of the sodium sulfate. Next, a 5 mL volume of hexane containing a dose of one of the groups was put on the column, which was again drained to the top of the sodium sulfate. Elution was then performed using 200 mL volumes of 6, 15 and 50% ethyl ether in hexane, with each 200 mL elution fraction received in a Kuderna-Danish flask. Each fraction was concentrated to a 10 mL final volume and an appropriate volume was injected into the gas chromatograph.

### Alumina

Add 10 mL water to 90 grams alumina (Woelm N-Super I), as purchased in a jar. Cap the jar, shake until the mixture is flowing smoothly, then let stand 24 hours before using. Slurry 22 grams of the prepared alumina in enough hexane so it can be poured into a 400mm x 25mm OD glass column with a coarse fritted disc and a Teflon stopcock. Settle the alumina with gentle tapping on the glass column and add a layer of sodium sulfate 1-2 cm in height. Wash the column with 20 mL hexane and add the dosed hexane which should be about 5 mL in volume. Successively elute with 40 mL hexane, 110 mL hexane, 100 mL 50% ethyl ether containing 2% ethyl alcohol in hexane, and receive each fraction in a Kuderna-Danish flask. Concentrate the fractions to final volumes of 10 mL and inject appropriate quantities into the gas chromatograph.

### Evaluation of Results

Recoveries from both columns are excellent. Elution times with the alumina column are shorter than those with the Florisil column. However, the single compound pesticides, chlordane, and toxaphene, are more frequently found in two fractions when using a alumina column than when using a Florisil column. Thus, processing and analyzing additional fractions, as would be the case with alumina, would likely be more time consuming than would be the longer elution times with Florisil. Since more components are recovered near the 100% level in the first fraction

(Florisil), this advantage appears to more than offset the less significant advantages of the alumina. Furthermore, Florisil may be stored as received in a 130°C oven for indefinite periods of time and still be usable. The alumina as received must be cautiously deactivated to Activity IV (10% water by weight) and then protected with care until used. How long Activity IV alumina may be held before use is not known. However, it appears to be a reasonably long period, perhaps two weeks or longer.

### Phase II

#### Wastewater Application

With the assistance and approval of the project officer, five wastewaters were procured and analyzed. The sources of these wastewaters and the results of the analyses are given below. All samples were put into one-gallon glass bottles which had previously contained Burdick and Jackson "distilled in glass" solvents, suitable for pesticide residue analysis. Sample sizes ranged from 20 to 24 gallons. All samples were shipped by air, unrefrigerated. Time in shipment was five days for Wastewater 1 and about two days for the others. Upon receipt at Southwest Research Institute, the samples were pooled in a 25-gallon stainless steel vessel, where the pH was adjusted to 3-4, as required, with concentrated sulfuric acid. The samples were then returned to the bottles in which they were shipped and stored at 4°C until used.

#### Analysis of Wastewaters

In order to develop improvements upon the method and to provide base data for the dosing and recovery experiments, as well as the accuracy and precision evaluations to follow, each wastewater was analyzed in triplicate for each substance of interest in this program. As an arbitrary working device and to allow for small errors attendant to injection, flow control, temperature control, and so on, the coincidence of retention times was considered for identification purposes to be anything within 0.03 minutes.

## Dosing of Wastewaters and Recovery Analyses

In order to accumulate data for accuracy and precision evaluations, each group was put into six, one-liter portions of each wastewater, one group at a time. Three of these dosed samples were immediately analyzed while three were stored in the dark at 4°C for seven days in sealed, glass containers. The dosed quantities were keyed to the specific response of the substance to electron capture detection, ranging from 1 µg/L for the substances giving the highest response, to 200 µg/L for toxaphene, except where interfering background required increase of the quantity. Except where noted to the contrary, extraction, clean up and GC analysis of the extracts were as given in earlier sections of this report.

### Wastewater 1

This wastewater was sampled by personnel of the Surveillance and Analysis Division, USEPA, Athens, Georgia and was identified there as HI-003. The pH as received was between 7.5 and 8. The wastewater was light yellow to light orange in color, free from solids and had a mild odor, reminiscent of pine oil with a sweet overtone. The plant producing the sample manufactures toxaphene and other organic materials, and it was understood that this effluent was derived from all the plant operations, and not from the toxaphene unit. This was due to the toxaphene plant's effluent treating unit being "down" on the day of sampling, and as a result no effluent was being discharged from it at that time.

As a preliminary step to the analysis of Wastewater 1, extracts were cleaned up with Florisil and alumina. Of the three fractions taken from each column, alumina produced the cleaner initial two fractions but a much dirtier third fraction. Since six single compound pesticides were determined in the third fraction from the alumina, but only two in the third fraction were determined from the Florisil, Florisil was the better overall choice and was subsequently used in all work with Wastewater 1. A comparison with standards rules out all of the pollutants of interest in this program, except heptachlor and endosulfan II and PCB-1242. The presence of PCB-1242 is more questionable than the other two substances, since only six of the nine peaks being followed in the analysis of this substance were present, and the amount present, as indicated by

Table 2. Ranges of Recoveries, Wastewater 1

	Ranges of Recoveries, %, avg.	
	Zero Time	7 Days' Storage
<i>Single compound pesticides</i>	90 - 110	90 - 99
<i>Chlordane (6 peaks)</i>	88 - 106	93 - 109
<i>Toxaphene (8 peaks)</i>	99 - 105	96 - 106
<i>PCB-1254 (7 peaks)</i>	93 - 98	102 - 105
<i>PCB-1016 (8 peaks)</i>	86 - 97	98 - 106
<i>PCB-1260 (11 peaks)</i>	91 - 102	97 - 112
<i>PCB-1221 (7 peaks)</i>	98 - 117	97 - 101
<i>PCB-1232 (10 peaks)</i>	89 - 134	92 - 96
<i>PCB-1242 (9 peaks)</i>	92 - 101	93 - 97
<i>PCB-1248 (7 peaks)</i>	98 - 101	100 - 103

each peak, was not a constant value. This is strong, but not conclusive evidence that PCB-1242 is not present. However, if the substance present is PCB-1242, the amount indicated is less than two ppb, and the other two substances are at 20 and 40 parts per trillion, respectively.

The dosing and recovery experiments for Wastewater 1 went smoothly. It is estimated that the dosing quantities could have been reduced to 25% of the quantities used with analytical results remaining satisfactory in most instances. The greatest difficulties occurred with interferences for one or two early eluting peaks of PCB-1221 and PCB-1232. If these peaks had been eliminated from the analyses, adequate peaks would have remained for analytical purposes, and the ranges given for these substances would have been narrowed. Recovery ranges were as follows in Table 2.

### Wastewater 2

Wastewater 2 was sampled by personnel of the Eastern District Office, USEPA, Westlake, Ohio. No information was supplied as to the source or character of this sample. The sample as received was amber in color, free from solids, had a urinelike odor, and had a pH of 4.

Despite the absence of substances of interest, the substances which were present provided many interferences in the spiking and recovery experiments. Due to the interference problems, especially from the substances eluting from the GC column during the first four minutes, a considerable effort was made to identify the source of these interferences, with the hope that such identification would suggest approaches for their removal. The presence of elemental sulfur was strongly suspected. Treatment of first fractions of extracts with elemental mercury began,

but the results were erratic. Careful drying of the extracts, addition of a mercuric salt, addition of acid-washed copper, and prolonging the agitation period were without consistently beneficial effects.

During concentration of the extracts in the Kuderna-Danish apparatus, a deposit of yellowish needle crystals and a gummy residue were noted. Infrared spectroscopy examination suggested that pentachlorophenol might have been present.

GC/MS analysis of the concentrated extract indicated the presence of naphthalene, methyl naphthalene, two dichlorinated compounds, 2-methylthiobenzothiazole, phthalates, dioctyl adipate, anthracene, and two hydrocarbons. No pesticides were found.

The difficulties with this wastewater were so great that dosing and recovery experiments were not completed for several of the pesticides and PCBs. An erratic response to clean up, especially with mercury, produced inconsistent background values in many instances. The greatest difficulties occurred within the first four minutes after injection. It was not unusual to experience no reduction in the interferences in undosed wastewater replicates treated with mercury, and yet, find substantial reductions in interferences in the dosed replicates, or vice versa. An impasse was reached and work with this wastewater was set aside so attention could be given to the other wastewaters.

### Wastewater 3

Wastewater 3 was procured by Southwest Research Institute-Houston personnel, and as received, had a mildly bad odor, a cloudy appearance, and a pH near 7. It was the final chlorinated effluent from the Northside Treatment plant in Houston, which treats both residential sewage and industrial plant effluents.

The analyses of three, one-liter replicates suggests the presence of DDE at 22 parts per trillion, dieldrin at 38 parts per trillion and chlordane at about 600 parts per trillion. Although only two peaks are shown as matching with chlordane, they are the major peaks for the substance. The lesser peaks might not be detectable in this matrix at this level. Five of the ten peaks matched the standard for PCB-1232, but the amount present, as indicated by each peak, was quite variable. This is strong evidence against its presence, and therefore, PCB-1232 was not considered to be present. The difficulties with this wastewater included emulsion formation and erratic response to clean up with mercury. The latter difficulty, although not as pronounced as with Wastewater 2, persisted throughout the work with this sample. As with Wastewater 2, the undosed replicates were more erratic as a rule than the dosed ones. As a consequence, the background values were from replicates not exposed to mercury, and the dosed replicates all received clean up with mercury. In some instances, this resulted in recovery percentages which were perhaps too low by a few percentage points Aldrin, DDE, DDT, and toxaphene suffered the highest losses during the seven-day storage period, with losses ranging from 12 to 17%. Recovery ranges were as follows in Table 3.

#### Wastewater 4

Wastewater 4 was procured by Southwest Research Institute, and was from one of the influent streams in a highly industrialized area of the city, flowing to the Northside Treatment Plant in Houston. As received, this sample had a rank sewage odor, some solid material, a cloudy appearance, and a pH near 7.

Florisil was used to clean up the extracts, and was followed by agitation of the first fractions with mercury. Emulsions formed as with Wastewater 3 and were treated as reported for that wastewater.

Treatment with mercury produced somewhat erratic reductions of interferences. Six replicates were treated. The last two were subjected to an increased amount of violent agitation with the vortex tube agitator as compared to the first four. This resulted in more uniform and lower analytical values in the data derived from first fractions. Comparison of retention

**Table 3. Ranges of Recoveries, Wastewater 3**

	Ranges of Recoveries, %, avg	
	Zero Time	7 days' Storage
<i>Single compound pesticides</i>	79-102	83-106
<i>Chlordane (6 peaks)</i>	78-89	98-105
<i>Toxaphene (8 peaks)</i>	89-99	83-88
<i>PCB-1254 (7 peaks)</i>	89-94	91-95
<i>PCB-1016 (8 peaks)</i>	92-99	92-98
<i>PCB-1260 (11 peaks)</i>	95-146	88-92
<i>PCB-1221 (7 peaks)</i>	86-97	91-93
<i>PCB-1232 (10 peaks)</i>	81-171	85-108
<i>PCB-1242 (9 peaks)</i>	79-87	109-114
<i>PCB-1248 (7 peaks)</i>	83-91	91-96

times with standards indicates the presence of DDE at 31 parts per trillion, heptachlor at 119 parts per trillion, dieldrin at 32 parts per trillion, and chlordane at 1.4 ppb.

As mentioned earlier, Wastewater 4 was quite similar to Wastewater 3, so for this and other reasons, only certain substances were dosed and recovered. This was performed at zero time, only. There were no unexpected problems met, and mercury clean up was less erratic than with Wastewater 3 and with earlier undosed replicates of this wastewater. Treatment of an extract with BioBeads SX-2 was without beneficial effects.

#### Wastewater 5

This sample was taken by Southern Research Institute personnel, through arrangements made by the EPA, from the final effluent stream from a pesticide manufacturing plant in Memphis, Tennessee. As received, this effluent was clear, had a chemical odor, and a pH around 3.

Emulsification was not a problem. Column clean up was carried out with Florisil after a preliminary comparison with alumina showed no advantage resulting from the use of alumina. When this wastewater was analyzed, it was found that Florisil afforded a slightly better, although inadequate, clean up than did alumina. In another experiment, the first fraction from Florisil of a dosed sample extract was exchanged into DCM and applied to a 22mm x 31cm column of BioBeads SX-2. During elution of this column with DCM, several fractions were collected and analyzed. There was no improvement in the separation of the interfering materials from the dosed pesticides.

This wastewater had a very high background of substances which were electron capture sensitive compared to

the other four wastewaters. However, most of the substances producing peaks are not substances of interest on this program. Comparison of retention times with standards indicates the presence of heptachlor at 6.6 ppb. GC/MS examination of the first and second fractions of an extract indicated that nearly all of the substances are chlorinated but only the presence of heptachlor was confirmed.

This wastewater represented a serious challenge to this program. It was an effluent from a pesticide manufacturing plant, and it contained numerous non-priority, chlorinated pollutants which interfered with the analyses for many of the priority pollutants which are the subject of this program.

The ability of concentrated sulfuric acid and fuming sulfuric acid (7.5%) to extract interfering materials from a hexane solution of the first fraction from Florisil was tested. Concentrated sulfuric acid removed a small, but insignificant, amount of these materials, and the fuming acid was no better.

By operating the GC on a temperature program, advantages were realized with some of the substances, for example, single compound pesticides and chlordane. This technique was applied to analyze recovered doses of  $\gamma$ -BHC, heptachlor, aldrin, endosulfan I, dieldrin, endrin, endosulfan II, endrin aldehyde, and chlordane from this wastewater at zero time. The temperature was programmed from 160° to 200°C at 4° per minute. Only dieldrin defied determination completely. Recoveries for the other components ranged from 62 to 107%. Endrin determination gave more reasonable values under isothermal conditions that programmed conditions. Peak height measurements produced the more reasonable recovery values. Recoveries ranged from 57 to 109%. It is noted that the 57% value for the

heptachlor component of chlordane checks fairly well with the 62% recovery obtained for heptachlor.

Dosing and recovery were carried out at zero time only. PCB-1242 and PCB-1254 were spiked into the same samples and measured using the procedure in ASTM Method D3304-77. In this method, alkaline hydrolysis followed by extraction with concentrated sulfuric acid is performed to reduce non-PCB interferences. Some interfering peaks remained in the treated fraction, especially in the early part of the chromatogram. Three such replicates were dosed, extracted, treated, and analyzed. The data show that peaks 1 and 5 in PCB-1242 were obscured by interferences to the extent they could not be read. Recoveries of other components ranged from 22 to 85%. The poor recoveries indicated by the other PCB-1242 peaks, except for peak 6, are probably the result of poor quantitation, as a consequence of interferences, rather than poor extraction. This view is supported by the fact that the indicated recoveries for PCB-1254 are much higher and uniform in values.

The other PCBs should give comparable results. Those which elute early (PCB-1016, PCB-1221, PCB-1232, PCB-1248) will be analyzed with greater difficulty and less precision and accuracy, probably, than the other late eluter, PCB-1260, in Wastewater 5.

### **Discussion**

In Phase I, endosulfan sulfate was found to disappear almost entirely from dosed clean water replicates stored for seven days at 4° and 24°C. In Phase II, no similar inordinate disappearance of endosulfan sulfate occurred in the replicates of Wastewaters 1, 2 and 3 during storage at 4°C.

Only a few of the 25 substances were found in the five wastewaters analyzed. Endrin, which was tentatively identified in Wastewater 5, was the only one of the substances present above the 10 parts per billion level. Its concentration was estimated at 32 ppb.

Recovery of the substances dosed into the five wastewaters at the 1 to 20 ppb level was satisfactory to very good with respect to accuracy and precision for Wastewaters 1, 3 and 4. Gas chromatographic analysis to determine the recoveries from the Wastewaters 2 and 5 could not be performed in some instances because of interferences that were not sufficiently removed by clean-

up columns, agitation with mercury, alkaline hydrolysis and/or extraction with sulfuric acid. It is very probable that Wastewater 2 would have been analyzed with greater success with the Hall detector than with the electron capture detector. Since Wastewater 5 was from a pesticide plant, the limited analytical success with this wastewater is indicative of the limitations imposed on the method by high concentrations of chlorinated substances that are not among the 25 substances of interest in this program but that have extraction and chromatography characteristics similar to them. Therefore, neither the Hall detector nor the electron capture detector is completely satisfactory in this situation. Better gas chromatography resolution or separation of such interferences before gas chromatography is required.

*John D. Millar and Richard E. Thomas are with Southwest Research Institute, San Antonio, TX 78229.*

*James E. Longbottom is the EPA Project Officer (see below).*

*The complete report, entitled "Determination of Pesticides and PCBs in Industrial and Municipal Wastewaters," (Order No. PB 82-214 222; Cost: \$9.00, subject to change) will be available only from:*

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