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Pollutant Analyses
Hooker Chemicals and Plastics Corporation
Waste Disposal Sites
Niagara Falls, New York

(JULY 12 - SEPTEMBER 7, 1979)

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
DENVER, COLORADO

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POLLUTANT ANALYSES
HOOKER CHEMICALS AND PLASTICS CORPORATION
WASTE DISPOSAL SITES
Niagara Falls, New York

[July 12-September 7, 1979]

William J. Stang

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National Enforcement Investigations Center
Denver, Colorado

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I. INTRODUCTION

The EPA and the Department of Justice (DOJ) are investigating the operations at the Hooker Chemicals and Plastics Corporation in regard to its hazardous waste disposal practices at Niagara Falls, New York. The purpose of this investigation is to determine compliance with applicable laws and regulations. Four waste disposal sites are being investigated by EPA: Love Canal, Hyde Park Landfill, S-Area Landfill, and 102nd Street Landfill. Extensive sampling of groundwater, and/or surface waters and sediments have been conducted at these sites by EPA Region II and State agencies. To supplement these data, the National Enforcement Investigations Center (NEIC) was requested to collect additional groundwater and surface water samples from and adjacent to the Hyde Park, S-Area and 102nd Street Landfill sites for mutagenicity and chemical analyses.

Sampling was conducted on July 12, 1979. In addition to the analyses for mutagenic substances,* analyses were performed for organic priority pollutants.** Air sampling was conducted at the Hyde Park Landfill site to determine if airborne pollutants from Hooker operations were present. The potential source of these pollutants is emission of volatile materials from the holding ponds into which the leachate collected from the landfill is pumped. These ponds are presently covered with a 22 mil plastic sheet to prevent emissions.

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* Mutagenicity analyses were by the Salmonella/mammalian microsome mutagenicity procedure (Ames test).

** Priority pollutants are derived from the June 7, 1976 Natural Resources Council (NRDC) vs. Russell Train (USEPA) Settlement Agreement. For a listing of the 129 pollutants see Appendix A.

All samples collected were shipped to the NEIC laboratories Denver, Colorado, for analyses. Document control, Chain-of-Custody, and quality control/quality assurance procedures of the NEIC were followed during this study.

II. SUMMARY OF FINDINGS

To supplement information collected from previous EPA investigations, the NEIC, on July 12, 1979, conducted sampling adjacent to or within three waste disposal sites: Hyde Park Landfill, S-area Landfill, and 102nd Street Landfill of the Hooker Chemicals and Plastics Corporation, Niagara Falls, New York. These samples were analyzed for mutagenic substances and organic pollutants during the period July 16 to September 7. The conclusions and pertinent findings from this investigation are discussed below for each disposal area.

WATER SAMPLE ANALYSES

Hyde Park Landfill

1. Analyses of the sample from the Hyde Park Landfill leachate pond identified 25 organic compounds; 22 of these are priority pollutants.

2. Of the 25 compounds found in the Hyde Park leachate pond, 10 were also identified in a groundwater sample collected near the Hyde Park Landfill. This site contained 18 organic compounds, 10 of which were priority pollutants.

3. Of the 25 compounds found in the Hyde Park leachate pond, 5 were also identified in the surface water sample collected from Bloody Run Creek at University Street. A total of 10 compounds were identified at this station; 6 were priority pollutants.

4. Concentrations of organic compounds identified at or adjacent to the Hyde Park Landfill ranged from low-level detection of less than 1 µg/l to a high of 8,200 µg/l.

5. None of the 129 priority pollutant compounds were detected at the Armagost residential well.

S-Area Landfill

1. Organic characterization of groundwater samples collected from the S-Area Landfill identified compounds from two Hooker monitoring wells (No. 17 & 17a). Twenty-three of the compounds were priority pollutants of which several appeared at high concentrations (range 3 to 15,000 µg/l).

2. Twenty-three organic compounds were also identified in samples collected from two stations (wells CW 2a and 6a) at the Niagara Falls Water Treatment Plant (adjacent to the S-Area Landfill). Twenty of the compounds were priority pollutants. Concentrations ranged from 0.02 to 1,200 µg/l. Two compounds identified in Hooker well samples from the landfill sites were also identified in groundwater collected from the water treatment plant property.

102nd Street Landfill

Groundwater collected from the 102nd Street Landfill contained several priority pollutants. Only 3 compounds were identified at low levels from the well located on Olin Chemical Company property. However, 15 priority pollutants were identified in the groundwater sample collected from Hooker well No. 1. Concentrations ranged from 3 to 1,200 µg/l.

AMBIENT AIR SAMPLE ANALYSES

Air samples were collected using Tenax columns at five locations adjacent to and on the Hyde Park Landfill site. A blank column was carried to the field and returned to Denver for a quality control reference.

The analyses identified benzene, trichloroethylene, hexane, tetrachloroethylene, toluene, and chlorobenzene present in all columns at concentrations greater than the detection limit of 5 $\mu g/m^3$. These substances were also identified in two volatile organics samples collected from the leachate pond. The reference column was later determined to be contaminated and, therefore, failed to meet quality control requirements. No quantitative evaluation was possible. However, the samples collected off-site and on top of the landfill showed no significant amounts of the above substances greater than the blank. The sample collected at the edge of the leachate pond, which was the most likely station to determine high concentrations of these substances, showed that only tetrachloroethylene and toluene were slightly higher than the reference column. No other chemicals were identified in any columns at or above the detection limit of 5 $\mu g/m^3$.

MUTAGEN TESTING

The Ames test for mutagenesis did not demonstrate mutagenic activity in any of the samples collected from stations adjacent to and on the three landfill sites. Mutagenic activity was not apparent in either the concentrated sample extracts or the filtered aliquots of any of the samples.

Inability to detect mutagenic activity in the samples does not necessarily mean that these substances are absent but that the mutagenic effect may be below the detection level of the test system used; additionally, the test system will not detect volatile mutagenic compounds.

TOXICITY EVALUATION

The chemical analyses identified 49 organic compounds. A literature search was done to assess toxicity and health effects of all these compounds. References used were the Registry of Toxic Effects of Chemical Substances (RTECS), the Toxline data base, and other data bases. Health effects and toxicity information was located for 36 of the 49 compounds.

Of the 36 compounds, 18 have demonstrated human health effects including systemic, pulmonary, gastrointestinal, central nervous system, blood and psychotropic effects. Benzene and vinyl chloride are reported to cause cancer in humans. Of the 49 compounds, 5 are reported to produce an irritant effect on the skin, eye, and mucuous membranes.

Of the 36 compounds, 27 have produced animal health effects, including neoplastic, carcinogenic, teratogenic, mutagenic or sensory-organ irritant effect on laboratory animals.

III. SURVEY METHODS

WATER SAMPLING

Ten locations were sampled within or adjacent to the Hyde Park, 102nd Street and S-Area Landfills [Table 1]. All NEIC samples were collected in glass containers of the following volumes:

Analysis	No. of Containers	Sample Volume
Mutagenicity	2	1 gallon[a]
Extractable Organics,	1	1 gallon
PCBs and Pesticides	2	40 ml

a Only 1 gallon was collected at Station 61801, Hyde Park Well, OW-6.

At all locations, duplicate samples were collected for analyses by the Company. The Company formally requested and received a copy of the NEIC procedure for the mutagenicity analysis. This was provided directly to their consultant, Dr. David Brusick of Litton Bionetics.

The NEIC generally followed the same procedure used by EPA Region II during their well sampling surveys conducted in April and June 1979. This required that certain wells, specifically well OW6 (Station 01) and wells W-17, W-17a, CW-6a, and CW-2a (Stations 07-10, respectively), be pumped prior to sampling. The volume pumped was to be ten times the casing volume at static conditions. No pumping was scheduled at Stations 04, 05, and 06. Field conditions caused some variation from the originally planned procedure.

Table 1

STATION LOCATIONS FOR WATER SAMPLES
HOOKER CHEMICALS AND PLASTICS CORPORATION
Niagara Falls, New York
July 12, 1979[a]

Station No.	Time (hr) of Sample Collection	Description
618 01	1020	Monitor Well OW 6 near the Hyde Park Landfill
02	1157	Leachate from ponds on the Hyde Park Landfill
03	1235	Bloody Run Creek at University Street
04	1325	Domestic well at Armagost residence on Penrose Street
05	1455	Olin Well B-2 at 102nd Street Site
06	1530	Hooker Well #1 at 102nd Street Site
07	1745	Monitor Well W-17 at S-Area
08	1745	Monitor Well W-17a at S-Area
09	1645	Monitor Well CW-6a at Niagara Falls Water Treatment Plant
10	1645	Monitor Well CW-2a at Niagara Falls Water Treatment Plant

a Figures 1, 2 and 3 show Station locations.

At Station 01 (OW-6), the pumps became clogged with a black-oily substance within the water column. Company officials were notified that drawdown would have to be done to the extent possible with a 2 cm (3/4 in) I.D. stainless steel bailer* 76 cm (30 in) long. The stated depth in the well prior to bailing was about 2.4 m (8 ft); the casing volume at this depth was approximately 5 liters (1.25 gal). Twenty-three liters (6 gal) were removed from the well in dropping the surface to the minimum level possible [that is, 76 cm (30-in) water depth]. The well recovered to its static head in about 10 minutes, after which sampling commenced. Sample aliquots (ca. 300 ml) were alternately poured into the NEIC and Company containers.

The wells at Stations 07 through 10 were not pumped before sampling. Company officials reported that these wells had been drawn down the previous day to accommodate sample collection by State Health Department personnel and, in their opinion, no additional pumping was necessary. It was mutually agreed that samples could be bailed directly. Samples were also bailed, without prior pumping, from the 102nd Street Landfill wells (Stations 05 and 06).

The Armagost residential well (Station 04) was pumped by the owner for about 10 minutes prior to bailing samples. The static water depth in this 15 cm (6-in) well was 9.5 m (31 ft) [total well depth is 12.5 m (41 ft)]. The actual volume removed during this period was not determined.

To collect leachate pond samples at the Hyde Park Landfill (Station 02), wastewater was pumped into a clean 208 liter (55-gal) drum from which the required sample volumes were taken using a

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* A separate bailer was used for sampling at each well. The bailers had been pre-washed 4 times with methylene chloride, dried and wrapped in aluminum foil before leaving Denver.

stainless steel beaker*. The Bloody Run Creek sample (Station 03) was collected using a stainless steel beaker*.

AMBIENT AIR SAMPLING

Air samples were collected at five stations adjacent to, and on, the Hyde Park Landfill site [Table 2]. Air, at the rate of 260 ml/min was drawn through a 190 mm Tenax column using personnel samplers** (MSA and Bendix-brand names). Two samples were collected at each station. One was provided to the Company, which had requested a split. Information on the type and flow rates of the personnel samplers was also provided. Approximately 2,600 ml of air were drawn through the columns during the 10-minute sampling period. The columns were then recapped, wrapped in tissue paper and, along with a blank Tenax column which was carried to the field and remained capped throughout, were returned to Denver for volatile organics analyses.

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* The beaker had been pre-washed four times with methylene chloride and covered with aluminum foil before leaving Denver. A separate beaker was used for each Station.

** The personnel samplers were calibrated on July 11 at Niagara Falls using a 100 ml bubble meter as the calibration device. The flow rate for both instruments was established at approximately 260 ml/min.

Table 2

AIR SAMPLING LOCATIONS - HYDE PARK LANDFILL AREA
HOOKER CHEMICALS AND PLASTICS CORPORATION
Niagara Falls, N.Y.
July 12. 1979[a]

Station No.	Description	Wind Conditions	Time (hr) Collection
61802	East edge of leachate ponds, Hyde Park Landfill	Slight Breeze W - NW	1228
12	Well OW-3, Northwest of Hyde Park Landfill	Slight Breeze W	1107
13	Located off Hyde Park Site about midway along north property fence, south of Power Authority Road	Slight Breeze Varying W-NW	1130
14	Top of Hyde Park Landfill - midway west to east length	Slight Breeze W-NW	1200
15	Top of Hyde Park Landfill at extreme east end	Slight Breeze W-NW	1217

a Figure 1 shows Station locations.

IV. SURVEY FINDINGS

WATER SAMPLE ANALYSES

Hyde Park Landfill

Characterization of the sample collected from the Hyde Park Landfill leachate pond [Station 02, Figure 1] identified 25 compounds [Tables 3 through 6 and Appendix C]. Twenty-two of these are priority pollutants; the remaining three non-priority pollutants were 2,4-dichlorotoluene and isomers of chlorobenzaldehyde and chlorobenzoic acid. Ten of the 25 compounds identified in the leachate pond were also identified in the groundwater sample at Station 01. The latter sample contained 18 organic compounds; 10 were priority pollutants. Five of the 25 compounds were identified in the surface water sample collected from Bloody Run Creek at University Street (Station 03). A total of 10 compounds were identified at Station 03; 6 were priority pollutants. Station 03 contained 3 compounds (Di-n-butylphthalate, Diethylphthalate and an isomer of tetrachlorobenzene) uncommon to Stations 01 and 02. No priority pollutants were detected at the Armagost residential well on Penrose Street (Station 04). Concentrations of organics identified at Stations 01, 02, and 03 ranged from low-level detection of less than 1 µg/l to a high of 8,200 ug/l. Compounds in concentrations of 1,000 µg/l or greater include two at Station 02 (methylchloride and phenol) and four at Station 01 (carbon tetrachloride, chloroform, 1,2,4-trichlorobenzene and hexachloroethane) at Station 01.

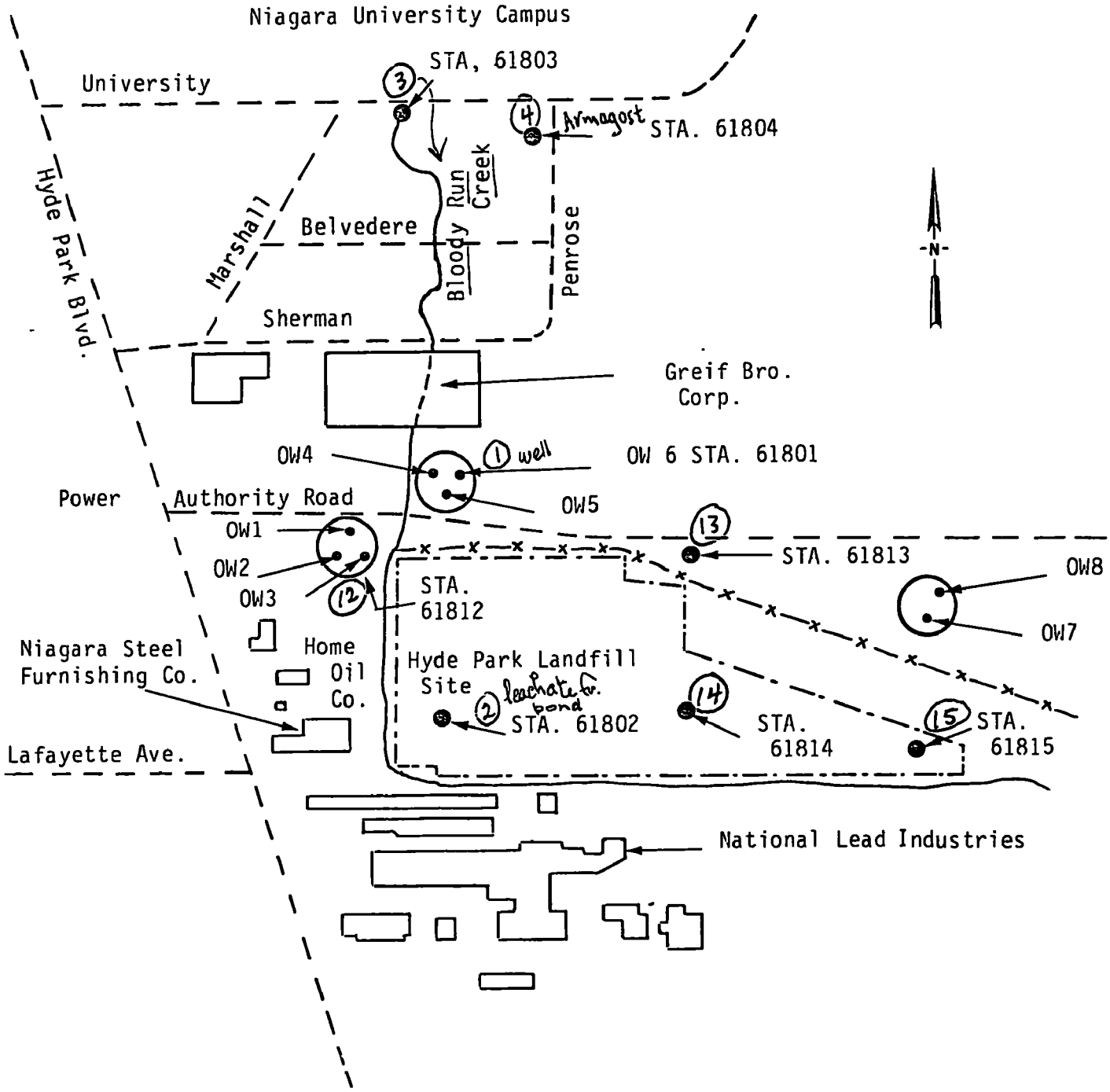

Figure 1

Hyde Park Landfill Area and Bloody Run Creek

Table 3

VOLATILE ORGANICS SAMPLING DATA[a]
HOOKER CHEMICALS AND PLASTICS CORPORATION
WASTE DISPOSAL SITES/NIAGARA FALLS, NEW YORK
July 12-September 7, 1979

Chemical Name	Station No.	Concentration (ppb or µg/l) 01	02	06	08	09	Detection Limit
Acrolein		ND[b]	ND	ND	ND	ND	-[c]
Acrylonitrile		ND	ND	ND	ND	ND	1
Benzene		ND	370	24	590	25	1
Carbon tetrachloride		8,200	270	ND	3,100	ND	1
Chlorobenzene		ND	ND	92	740	510	1
1,2-Dichloroethane		ND	100	ND	ND	ND	1
1,1,1-Trichloroethane		ND	ND	ND	ND	ND	1
1,1-Dichloroethane		ND	ND	ND	ND	ND	1
1,1,2-Trichloroethane		ND	24	ND	75	ND	1
1,1,2,2-Tetrachloroethane		ND	210	ND	ND	ND	1
Chloroethane		ND	ND	ND	ND	ND	1
Chloroform (Trichloromethane)		1,500	940	4	900	ND	1
1,1-Dichloroethylene		ND	ND	ND	7,800	ND	1
1,2-trans-Dichloroethylene		790	340	ND	15,000	41	1
1,2-Dichloropropane		ND	ND	ND	ND	ND	1
1,3-Dichloropropylene (1,3-Dichloropropene)		ND	ND	ND	ND	ND	1
Ethylbenzene		ND	ND	ND	ND	ND	1
Methylene chloride (Dichloromethane)		270	150	ND	52	ND	7[d]
Methyl chloride (Chloromethane)		ND	1,000	ND	ND	ND	10
Methyl bromide (Bromomethane)		ND	ND	ND	ND	ND	10
Bromoform (Tribromomethane)		ND	ND	ND	ND	ND	1
Dichlorobromomethane		ND	ND	ND	ND	ND	1
Trichlorofluoromethane		ND	790	ND	ND	ND	10
Dichlorodifluoromethane		ND	ND	ND	ND	ND	10
Chlorodibromomethane		ND	ND	ND	ND	ND	1
Tetrachloroethylene		ND	690	4	ND	3	1
Toluene		ND	960	14	3	ND	1
Trichloroethylene		ND	550	ND	1,800	3	1
Vinyl chloride		ND	ND	ND	190	ND	10

a Single grab samples, collected July 12, 1979.
b ND means not detected at or above the detection limit.
c Acrolein cannot satisfactorily be determined by the method used.
d Methylene chloride is detected in blank samples at 4 $\pm$ 3 µg/l.

Table 4

BASE-NEUTRAL EXTRACTABLE ORGANICS SAMPLING DATA[a]
HOOKER CHEMICALS AND PLASTICS CORPORATION
WASTE DISPOSAL SITES/NIAGARA FALLS, NEW YORK
July 12-September 7, 1979

Chemical Name	Station No.	Concentration (ppb or µg/l)										
		01	02	03	04	05	06	07	08	09	10	Detection Limit
Isophorone		ND[b]	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Napthalene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Nitrobenzene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
N-Nitrosodimethylamine		NA[c]	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Bis (2-ethylhexyl) phthalate		ND	ND	ND	ND	32	ND	ND	ND	ND	ND	5
Butyl benzyl phthalate		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
Di-n-butyl phthalate		ND	ND	MS[d]	ND	MS	ND	38	ND	ND	ND	5
Di-n-octyl phthalate		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
Diethyl phthalate		ND	ND	MS	ND	ND	ND	ND	ND	ND	ND	5
Dimethyl phthalate		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Benzo (a) anthracene (1,2-Benzarthracene)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
Benzo (a) pyrene (3,4-Benzopyrene)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
3,4-Benzofluoranthene (Benzo(b)fluoranthene)		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo (k)fluoranthene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
Chrysene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
Acenaphthylene		ND	ND	ND	ND	ND	ND	ND	ND	MS	ND	5
Anthracene		ND	ND	ND	ND	ND	ND	ND	ND	MS	ND	5
Benzo(g,h,i)perylene (1,12-Benzoperylene)		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Fluorene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Phenanthrene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Dibenzo(a,h)anthracene		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Indeno (1,2,3-cd)Pyrene		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Pyrene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzidine		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
Acenaphthene		ND	ND	ND	ND	ND	ND	ND	ND	MS	ND	5
1,2,4-Trichlorobenzene		3000	180	MS	ND	ND	40	13	2900	170	1100	5
Hexachlorobenzene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Hexachloroethane		1600	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Bis(chloromethyl) ether		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Bis (2-chloroethyl) ether		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
2-Chloroethylvinyl ether		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
1,2-Dichlorobenzene		210	51	ND	ND	ND	160	ND	440	ND	140	5
1,3-Dichlorobenzene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
1,4-Dichlorobenzene		380	72	ND	ND	ND	710	ND	600	990	190	5
3,3-Dichlorobenzidine		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
2,4-Dinitrotoluene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20
2,6-Dinitrotoluene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
1,2-Diphenylhydrazine		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Fluoranthene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
4-Chlorophenyl phenyl ether		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
Bis (2-chloroisopropyl) ether		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Bis (2-chloroethoxy) methane		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene		700	ND	ND	ND	ND	ND	ND	14	ND	42	5
Hexachlorocyclopentadiene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	20

a Grab samples, collected July 12, 1979.
b ND means not detected at or above the detection limit
c NA means not analyzed for.
d MS means the compound was identified by mass spectrometry but was below the quantitative detection limit.

Table 5

ACID-EXTRACTABLE PHENOLIC COMPOUNDS SAMPLING DATA[a]
HOOKER CHEMICALS AND PLASTICS CORPORATION
WASTE DISPOSAL SITES/NIAGARA FALLS, NEW YORK
July 12-September 7, 1979

Chemical Name	Station No.	Concentration (ppb or µg/l)										
		01	02	03	04	05	06	07	08	09	10	Detection Limit
2,4,6-Trichlorophenol		ND[b]	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
para-Chloro-meta-cresol		ND	ND	ND	ND	ND	15	ND	ND	ND	ND	5
2-Chlorophenol		ND	ND	ND	ND	ND	7	ND	ND	2	ND	5
2,4-Dichlorophenol		ND	240	ND	ND	ND	57	ND	ND	11	ND	5
2,4-Dimethylphenol		ND	ND	ND	ND	ND	3	ND	ND	ND	ND	5
2-Nitrophenol		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
4-Nitrophenol		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5
2,4-Dinitrophenol		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	10
4,6-Dinitro-o-cresol		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	10
Pentachlorophenol		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	10
Phenol		840	3200	54	ND	ND	ND	8	3500	ND	1200	5

a Grab samples, collected July 12, 1979.
b ND means not detected at or above the detection limit.

Table 6

PESTICIDES AND PCB SAMPLING DATA[a]
HOOKER CHEMICALS AND PLASTICS CORPORATION
WASTE DISPOSAL SITES/NIAGARA FALLS, NEW YORK
July 12-September 7, 1979

Chemical Name	Station No.	Concentration (ppb or µg/l)										
		01	02	03	04	05	06	07	08	09	10	Detection Limit
Aldrin		ND[b]	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.1
Dieldrin		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.2
Chlordane		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
4,4' -DDT		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.5
4,4' -DDE(p,p'-DDX)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.1
4,4' -DDD(p,p'-TDE)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.4
α-Endosulfan-Alpha		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.2
β-Endosulfan-Beta		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.2
Endosulfan sulfate		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
Endrin		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.2
Endrin aldehyde		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.4
Heptachlor		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.1
Heptachlor epoxide		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.1
α-BHC-Alpha		ND	90	2.3	ND	0.15	1200	58	180	1.9	14	0.02
β-BHC-Beta		ND	40	ND	ND	ND	8	ND	ND	ND	ND	0.1
γ-BHC(lindane)-Gamma		ND	400	0.17	ND	ND	ND	ND	58	ND	ND	0.02
δ-BHC-Delta		ND	ND	ND	ND	ND	ND	ND	ND	1.3	ND	0.02
PCB-1242 (Arochlor 1242)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
PCB-1254 (Arochlor 1254)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
PCB-1221 (Arochlor 1221)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
PCB-1232 (Arochlor 1232)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
PCB-1248 (Arochlor 1248)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
PCB-1260 (Arochlor 1260)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	2.0
PCB-1016 (Arochlor 1016)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0
Toxaphene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5.0

a Grab samples, collected July 12, 1979.
b ND means not detected at or above the detection limit.

S-Area Landfill

Volatile organics were not determined at Station 07 (Monitor Well W-17, Figure 2). A total of five organic compounds were identified from the sample collected at this site; 4 were priority pollutants [Tables 3 through 6]. Concentrations were low, ranging from 8 ug/l to 58 µg/l.

Twenty-two organic compounds were detected from Station 08 (Well W-17a); 19 were priority pollutants, of which several appeared at high concentrations (range 3 ug/l to 15,000 µg/l).

Analyses of groundwater samples collected at Stations 09 and 10 [Wells CW-6a and CW-2a, respectively, Figure 2] identified 15 organic compounds at Station 09; 14 were priority pollutants ranging in concentration from 0.02 ug/l to 990 ug/l. Eight organic compounds were detected at Station 10, 6 were identified as priority pollutants [Tables 3 through 6 and Appendix C]. Concentrations of organic compounds at Station 10 ranged from 14 to 1,200 ug/l. Only two compounds (1,2,4-trichlorobenzene and phenol) were present at concentrations of 1,000 ug/l or greater, both identified at Station 10.

102nd Street Landfill

Groundwater collected from the 102nd Street Landfill [Stations 05 and 06, Figure 3] contained several priority pollutants [Tables 3 through 6]. Only 3 compounds were identified from Station 05. However, 15 priority pollutant compounds were identified from the groundwater sample collected at Station 06 (Hooker Well No. 1). Concentrations ranged from less than 1 to 32 µg/l at Station 05, and from 3 to 1,200 µg/l at Station 06.

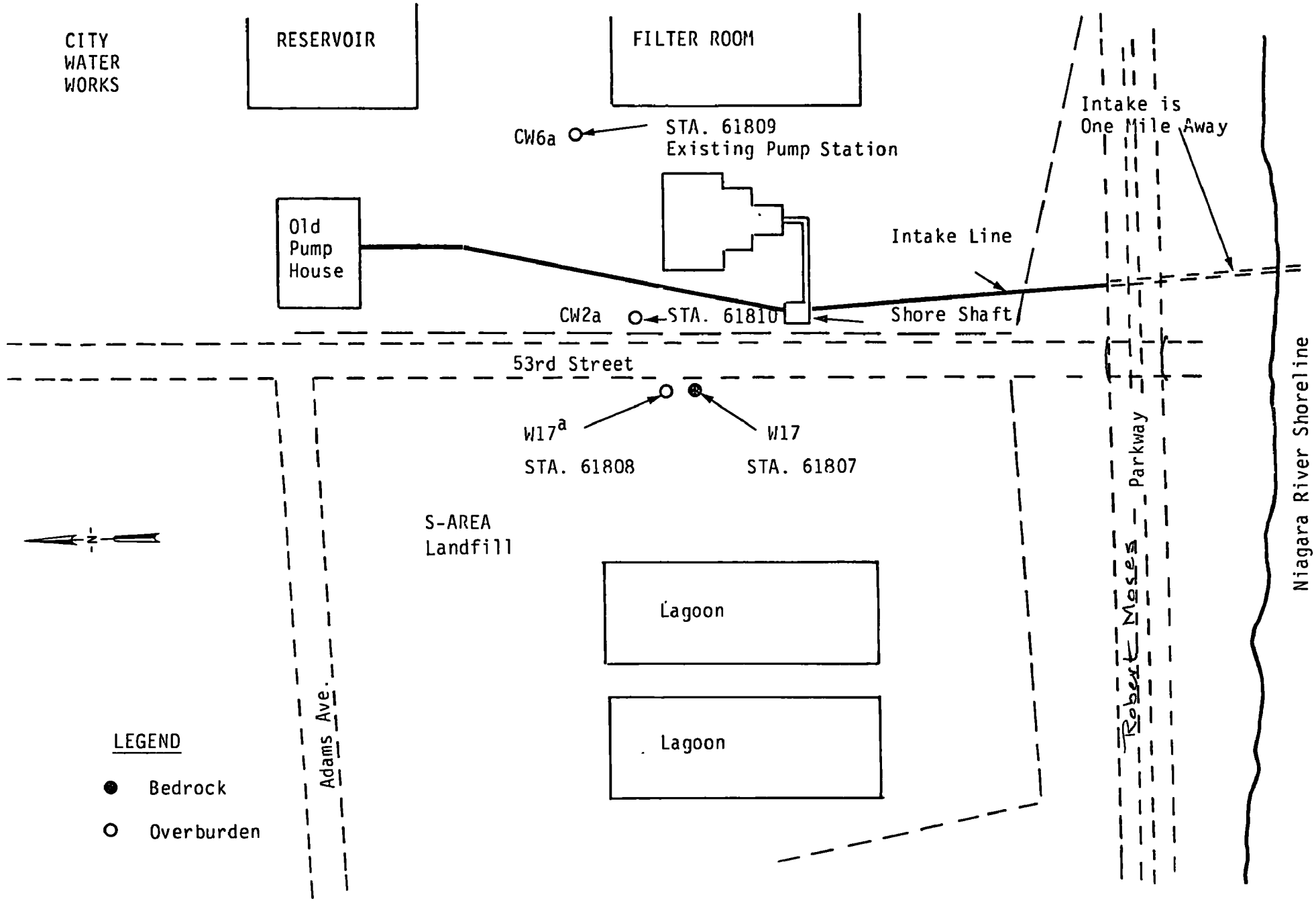

Figure 2

S-Area Landfill and Water Treatment Plant Monitoring Wells Sampled

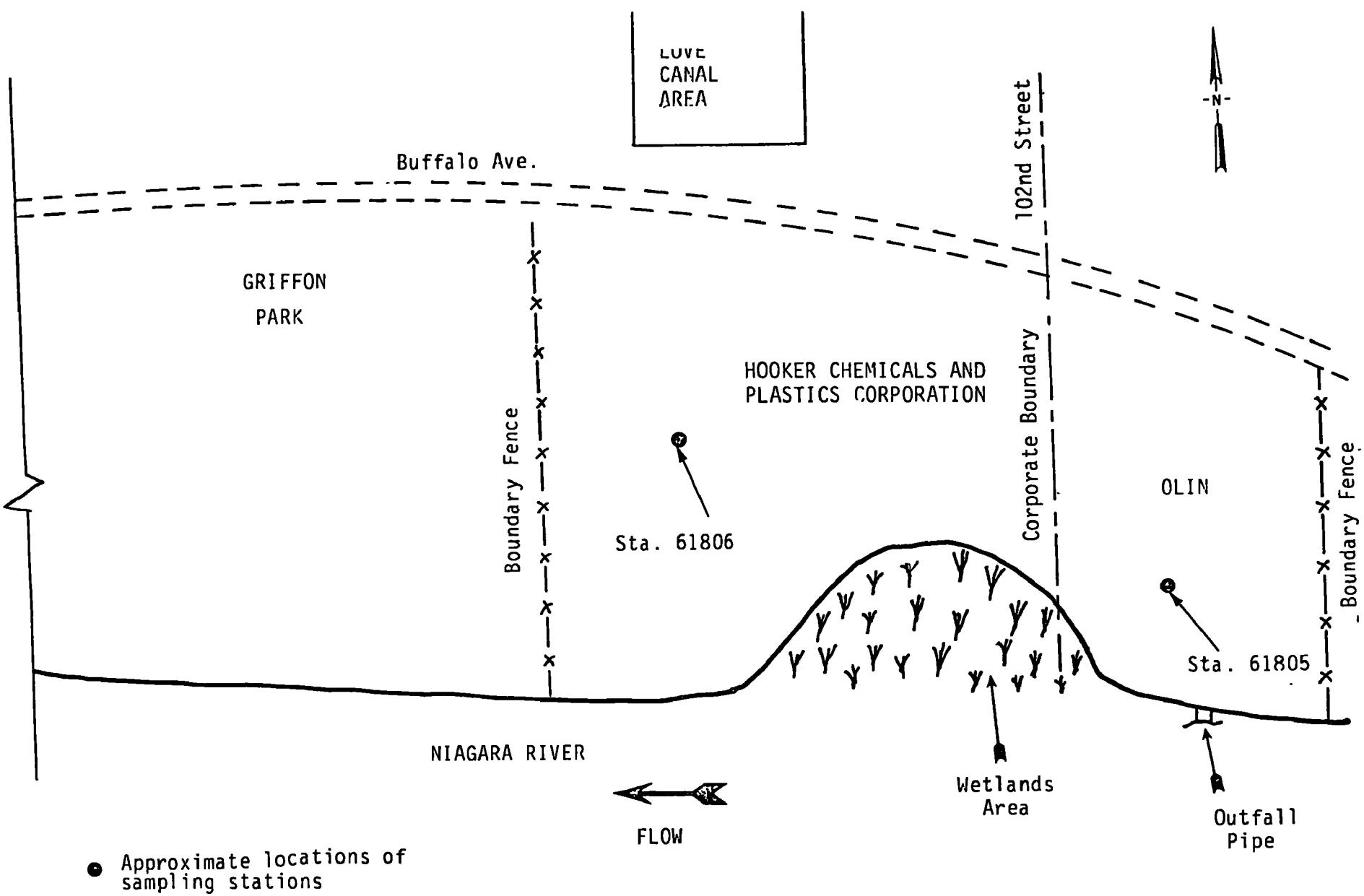

Figure 3

102nd Street Landfill Area

AMBIENT AIR SAMPLE ANALYSES

Analysis of the Tenax columns was performed on a Finnigan 1015 GC/MS.* The chemicals were separated on a 2.4 m x 0.3 cm (8 ft x 1/8 in) stainless steel column packed with 0.2% Carbowax 1500 on 60/80 mesh Carbopack C. The results were checked against Tenax trap blanks and traps loaded from permeation tube standards. The permeation rates were determined by weight loss.

The Tenax column blank and the other columns showed that benzene, trichloroethylene, hexane, tetrachloroethylene, toluene, and chlorobenzene were present at concentrations greater than the detection limit of 5 $\mu g/m^3$. These substances were also identified in the volatile organics samples collected from the leachate pond (Station 02). The reference column was later determined to be contaminated and failed to meet quality control requirements. No quantitative evaluation of the results was possible. However, the Tenax column samples collected at Stations 12, 13, 14, and 15 showed no significant amounts of the above substances greater than the blank. Moreover, significant amounts of benzene, trichloroethylene, hexane, and chlorobenzene were not present in the air sample collected on the edge of the leachate pond (Station 02), which would have been the most likely location for these substances. Tetrachloroethylene and toluene were higher at this station than in the blank. No other chemicals were detected in any of the columns at or above the detection limit of 5 $\mu g/m^3$.

MUTAGEN TESTING

The standard bacterial assay for mutagenicity was performed on liquid sample concentrates using the plate incorporation method, as

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* Gas Chromatograph/Mass Spectrometer.

described by Ames, et al.[1] This test consists of specially developed strains of Salmonella typhimurium that are auxotrophic for the amino acid, histidine (i.e., unable to grow without histidine supplemented to their media). The organisms have been genetically altered so when they are subjected to certain mutagenic and carcinogenic substances they will mutate and regain the natural ability to synthesize histidine. Thus, only mutant colonies can grow on media which does not contain histidine and their growth indicates presence of a mutagenic substance. Mutagenic activity based upon use of bacteria as indicator organisms correlates closely ($\geq$90% probability) with inducement of cancer in laboratory animals by organic compounds.[2,3,4,5,6,7]

Acidic and basic sample extracts and undiluted, filtered samples were prescreened for mutagenic activity using five standard Salmonella tester strains: TA 98, TA 100, TA 1535, TA 1537 and TA 1538. Samples were first tested individually. If they showed negative mutagenicity, they were then subjected to metabolic activation by adding rat liver homogenate (S-9 mix) [Appendix B].

The mutagencity test did not demonstrate mutagenic activity in any of the ten samples.* Concentrated extracts of the sample collected from Station 01 (Monitor Well OW6), adjacent to the Hyde Park Landfill, were toxic to the Salmonella tester strains; therefore, bacterial mutagenicity could not be determined for this material. Mutagenic activity was not apparent in either the concentrated sample extracts or the filtered aliquots of any of the remaining samples.

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* Inability to detect mutagenic activity in the samples does not necessarily mean that these substances are absent but that the mutagenic effect may be below the detection limit of the test system used. The Salmonella test does not detect some of the important chlorinated carcinogens such as chloroform, carbon tetrachloride and hexachlorobenzene. The concentration technique employed eliminates the volatile alkyl halides.

V. TOXICITY EVALUATION

The chemical analyses identified 49 organic compounds. To assess toxicity and health effects, these compounds were searched in the Registry of Toxic Effects of Chemical Substances (RTECS), which is an annual compilation prepared by the National Institute for Occupational Safety and Health. The Registry contains toxicity data for approximately 36,900 substances, but does not presently include all chemicals for which toxic effects have been found. Chemical substances in RTECS have been selected primarily for the toxic effect produced by a single dose, some lethal and some non-lethal. Substances whose principal toxic effects result from exposure over long periods are not included. Toxic information on a chemical substance is determined by examining and evaluating the published medical, biological, engineering, chemical and trade information documents.

The 49 compounds were also searched in the Toxline data base, which is a computerized bibliographic retrieval system for toxicology containing more than 618,000 records taken from material published in primary journals. It is part of the MEDLINE file from the National Library of Medicine and is composed of ten subfiles:

(1) Chemical-Biological Activities 1965 (taken from Chemical Abstracts, Biochemistry Sections)

(2) Toxicity Bibliography 1968 (a subset of Index Medicus)

(3) Abstracts on Health Effects of Environmental Pollutants 1971 (published by Biological Abstracts)

(4) International Pharmaceutical Abstracts 1970 (published by the American Society of Hospital Pharmacists)

(5) Pesticides Abstracts 1967 (compiled by EPA)

(6) Environmental Mutagen Information Center 1969 (Dept. of Energy, Oak Ridge National Lab)

(7) Environmental Teratology Information Center 1950 (Dept. of Energy, Oak Ridge National Lab)

(8) Toxic Materials Information Center (Dept. of Energy, Oak Ridge National Lab)

(9) Teratology file 1971-1974 (a collection of citations on teratology complied by the National Library of Medicine)

(10) The Hayes File on Pesticides (a collection of more than 10,000 citations on the Health aspects of pesticides compiled by Dr. W. J. Hayes, Jr., EPA)

Additional data bases searched to locate or support toxic information on all 49 compounds were: (1) Toxicology Data Bank (TDB), from the National Library of Medicine, which currently contains information on about 2,500 substances; (2) Oil and Hazardous Materials Technical Assistance Data System (OHMTADS), an EPA file, containing toxic data for about 1,000 compounds; (3) Excerpta Medica, a medical file with a section on toxicology and environmental pollution; and (4) Chemical Abstracts.

The RTECS search yielded information on 36 of the 49 compounds. The Toxline search yielded 883 citations to human health effects from the 36 compounds, providing support to the toxic information from RTECS.

Of the 36 compounds, 18 have demonstrated human health effects, including systemic, pulmonary, gastrointestinal, central nervous system, blood and psychotropic effects. Benzene and vinyl chloride are reported to cause cancer in humans. Of the 49 compounds, 5 produce an irritant effect on the skin, eye and mucous membranes [Table 7].

Of the 36 compounds, 27 have produced animal health effects, including neoplastic, carcinogenic, teratogenic, mutagenic or irritation to the skin, eye and mucous membranes of laboratory animals.

The three compounds which were not located in the RTECS were: acenapthene, acenaphthylene, and 2,4-dinitrotoluene. These were searched in Toxline as well. No information was discovered on toxic and health effects to humans. The 11 isomers of non-priority pollutants identified (NEIC Qualitative Data Summary, Appendix C) cannot be searched without more information.

TABLE 7

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Anthracene	$C_{14}H_{10}$	120-12-7[f]		Oral-rat	TDLo:	18 gm/kg	78WI	Carcinogenic	
				Subcutaneous-rat	TDLo:	3,300 mg/kg	33WI	Neoplastic	
Benzene	C_6H_6	71-43-2[f]	TLm 96: 100-10 ppm	Skin-rabbit		15 mg	24H open	Mild Irritation	
				Eye-rabbit		88 mg		Moderate Irritation	
				Oral-human	TDLo:	130 mg/kg		Central Nervous System	TLV (air): Cl 25 ppm
				Oral-human	LDLo:	50 mg/kg			OSHA std (air): TWA 10 ppm;
				Inhalation-human	LCLo:	20,000 ppm	5M		Cl 25 ppm;
				Inhalation-human	TCLo:	210 ppm		Blood	Pk 50ppm/10M/8H
				Inhalation-man	TCLo.	2,100 mg/m^3	4YI	Carcinogenic	
				Oral-rat	LD50:	3,800 mg/kg			NIOSH recm std (air): Cl 1 ppm/60M
				Inhalation-rat	LC50:	10,000 ppm	7H		
				Intraperitoneal-rat	LDLo:	1,150 mg/kg			
				Oral-mouse	LD50:	4,700 mg/kg			
				Oral-mouse	TDLo:	1 mg/kg		Mutagenic	
				Intravenous-rabbit	LDLo:	88 mg/kg			
				Inhalation-mouse	LC50:	9,980 ppm			
				Skin-mouse	TDLo:	1,200 gm/kg	49WI	Neoplastic	
				Intraperitoneal-mouse	LD50:	468 mg/kg			
				Subcutaneous-mouse	TDLo:	2,700 mg/kg	13D (Preg.)	Teratogenic	
				Oral-dog	LDLo:	2,000 mg/kg			
				Inhalation-dog	LCLo:	146,000 mg/m^3			
				Inhalation-cat	LCLo:	170,000 mg/m^3			
				Intraperitoneal-guinea pig	LDLo:	527 mg/kg			
				Subcutaneous-frog	LDLo:	1,400 mg/kg			
				Inhalation-mammal	LCLo:	20,000 ppm	5M		
Benzene, Chloro-	C_6H_5Cl	108-90-7[f]	TLm 96:100-1ppm	Oral-rat	LD50:	2,910 mg/kg			TLV (air): 75 ppm
				Subcutaneous-rat	LDLo:	7,000 mg/kg			
				Oral-rabbit	LD50:	2,830 mg/kg			OSHA std (air): TWA 75 ppm
				Intraperitoneal-rat	LDLo:	7,400 mg/kg			
				Intraperitoneal-guinea pig	LDLo:	4,100 mg/kg			
				Inhalation-mouse	LCLo:	15 gm/m^3			

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Benzene, 1,2-dichloro-	$C_6H_4Cl_2$	95-50-1[f]		Oral-human	LDLo:	500 mg/kg			TLV (air): 50 ppm
				Oral-rat	LD50:	500 mg/kg			
				Inhalation-rat	LCLo:	821 ppm	7H		OSHA std (air): Cl 50 ppm
				Intraperitoneal-rat	LD50:	840 mg/kg			
				Intravenous-mouse	LDLo:	400 mg/kg			
				Oral-rabbit	LD50:	500 mg/kg			
				Intravenous-rabbit	LDLo:	250 mg/kg			
				Oral-guinea pig	LDLo:	2,000 mg/kg			
				Inhalation-guinea pig	LCLo:	800 ppm	24H		
				Eye-rabbit		100 mg	30 sec.	Mild Irritation	
Benzene, 1,4-dichloro-	$C_6H_4Cl_2$	106-46-7[f]		Oral-human	LDLo:	500 mg/kg			TLV (air): 75 ppm
				Oral-human	TDLo:	300 mg/kg		Systemic	
				Eye-human		80 ppm		Irritation	OSHA std. (air): TWA 75 ppm
				Oral-rat	LD50:	500 mg/kg			
				Intraperitoneal-rat	LD50:	2,562 mg/kg			
				Oral-mouse	LD50:	2,950 mg/kg			
				Subcutaneous-mouse	LD50:	5,145 mg/kg			
				Oral-guinea pig	LDLo:	2,800 mg/kg			
Benzene, Ethyl-	C_8H_{10}	100-41-4[f]	TLm 96:100-10 ppm	Inhalation-human	TCLo:	100 ppm	8H	Irritant	TLV (air): 100 ppm
				Oral-rat	LD50:	3,500 mg/kg			
				Inhalation-rat	LCLo:	4,000 ppm	4H		OSHA std (air): TWA 100 ppm (skin)
				Skin-rabbit	LD50:	5,000 mg/kg			
				Inhalation-guinea pig	LCLo:	10,000 ppm			
				Skin-rabbit		15 mg	24H open	Mild Irritation	
				Eye-rabbit		100 mg		Irritation	
Benzene, 1,2,4-trichloro-	$C_6H_3Cl_3$	120-82-1[f]	TLm 96: 10-1 ppm	Oral-rat	LD50:	756 mg/kg			TLV (air): 5 ppm
				Oral-mouse	LD50:	766 mg/kg			
				Intraperitoneal-mouse	LDLo:	500 mg/kg			
1,3-Butadiene, Hexachloro-	C_4Cl_6	87-68-3[f]		Oral-rat	LD50:	90 mg/kg			
				Oral-rat	TDLo:	15 gm/kg	2YC	Carcinogenic	
				Intraperitoneal-rat	LD50:	175 mg/kg			
				Oral-mouse	LD50:	110 mg/kg			
				Inhalation-mouse	LCLo:	235 ppm	4H		
				Intraperitoneal-mouse	LD50:	76 mg/kg			
				Oral-guinea pig	LD50:	90 mg/kg			
				Unreported-mammal	LD50:	200 mg/kg			

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Carbon Tetrachloride	CCl_4	56-23-5[f]	TLM 96: 100-10 ppm	Skin-rabbit		4 mg	Mild Irritation		TLV (air): 10 ppm (skin)
									OSHA std (air): TWA 10 ppm; Cl 25; pk 200/5M/4H
				Eye-rabbit		2,200 ug	30 sec	Mild Irritation	NIOSH recm std (air): Cl 2ppm/60M
				Eye-rabbit		500 mg	24H	Severe Irritation	
				Skin-Guinea pig		800 mg	24H	Moderate Irritation	
				Oral-human	LDLo:	43 mg/kg			
				Oral-woman		1,800 mg/kg		Systemic	
				Inhalation-human	TCLo:	20 ppm		Central Nervous System	
				Oral-woman	TDLO:	1,800 mg/kg		Pulmonary System	
				Oral-man	TDLO:	1,700 mg/kg		Central Nervous System	
				Inhalation-human	LCLo:	1,000 ppm			
				Inhalation-human	TCLO:	317 ppm	30M	Gastrointestinal Tract	
				Oral-rat	LD50:	2,800 mg/kg			
				Inhalation-rat	LCLo:	4,000 ppm	4H		
				Inhalation-rat	TCLO:	300 ppm	6-15D (Preg)	Teratogenic	
				Skin-rat	LD50:	5,070 mg/kg			
				Intraperitoneal-rat	LD50:	1,500 mg/kg			
				Subcutaneous-rat	TDLo:	133 gm/kg	25WI	Neoplastic	
				Oral-mouse	LD50:	12,800 mg/kg			
				Oral-mouse	TDLo:	4,800 mg/kg	88DI	Carcinogenic	
				Inhalation-mouse	LC50:	9,526 ppm	8H		
				Intraperitoneal-mouse	LD50:	4,675 mg/kg			
				Subcutaneous-mouse	LDLo:	12 gm/kg			
				Oral-dog	LDLo:	1,000 mg/kg			
				Intraperitoneal-dog	LD50:	1,500 mg/kg			
				Intravenous-dog	LDLo:	125 mg/kg			
				Inhalation-cat	LCLo:	38,110 ppm	2H		

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Carbon Tetrachloride (cont'd)				Subcutaneous-cat	LDLo:	300 mg/kg			
				Oral-rabbit	LD50:	6,380 mg/kg			
				Intraperitoneal-rabbit	LDLo:	478 mg/kg			
				Subcutaneous-rabbit	LDLo:	3,000 mg/kg			
				Intravenous-rabbit	LD50:	5,840 mg/kg			
				Inhalation-guinea-pig	LCLo:	20,000 ppm	2H		
				Oral-hamster	TDLo:	3,680 mg/kg	30WI	Carcinogenic	
				Inhalation-frog	LCLo:	58,000 mg/m3			
				Inhalation-mammal	LCLo:	50,000 ppm	5M		
Chloroform (Trichloromethane)	$CHCl_3$	67-66-3[f]	TLm 96:100-10 ppm	Oral-human	LDLo:	140 mg/kg			TLV (air): 25 ppm
				Inhalation-human	TCLo:	1,000 mg/m^3	1Y	Systemic	
				Inhalation-human	TCLo:	5,000 mg/m^3	7M	Central Nervous System	OSHA std (air): TWA 50 ppm
				Oral-rat	LD50:	800 mg/kg			
				Oral-rat	TDLo:	70 gm/kg	78WI	Neoplastic	NIOSH recm std (air): Cl 2 ppm/60M
				Inhalation-rat	LCLo:	8,000 ppm	4H		
				Inhalation-rat	TCLo:	100 ppm	7H/6-15D (Preg)	Teratogenic	
				Oral-mouse	LD50:	1,120 mg/kg			
				Oral-mouse	TDLo:	18 gm/kg	120DI	Carcinogenic	
				Inhalation-mouse	LC50:	28 gm/m^3			
				Intraperitoneal-mouse	LD50:	1,671 mg/kg			
				Subcutaneous-mouse	LD50:	704 mg/kg			
				Oral-dog	LDLo:	1,000 mg/kg			
				Inhalation-dog	LC50:	100 gm/m^3			
				Intraperitoneal-dog	LD50:	1,000 mg/kg			
				Intravenous-dog	LDLo:	75 mg/kg			
				Inhalation-cat	LCLo:	35,000 mg/m^3	4H		
				Oral-rabbit	LDLo:	500 mg/kg			
				Inhalation-rabbit	LC50:	59 gm/m^3			
				Subcutaneous-rabbit	LDLo:	3,000 mg/kg			
				Inhalation-guinea pig	LCLo:	20,000 ppm	2H		
				Inhalation-frog	LCLo:	6,000 mg/m^3			
				Inhalation-mammal	LCLo:	25,000 ppm	5M		
				Skin-rabbit		10 mg	24H open	Mild Irritation	
				Eye-rabbit		148 mg		Irritation	

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
m-Cresol, 4-Chloro	C_7H_7ClO	59-50-7[f]		Oral-rat	LDLo:	500 mg/kg			
				Subcutaneous-rat	LD50:	400 mg/kg			
				Intraperitoneal-mouse	LDLo:	30 mg/kg			
				Subcutaneous-mouse	LDLo:	200 mg/kg			
Cyclohexane, 1,2,3,4,5,6-Hexachloro-, alpha-isomer	$C_6H_6Cl_6$	319-84-6[f]		Oral-rat	LD50:	177 mg/kg			
				Oral-rat	TDLo:	17 gm/kg	48WC	Carcinogenic	
				Oral-mouse	TDLo:	8,350 mg/kg	24WC	Carcinogenic	
				Oral-mouse	TDLo:	10 gm/kg	24WC	Carcinogenic	
Cyclohexane, 1,2,3,4,5,6-Hexachloro-, beta-isomer	$C_6H_6Cl_6$	319-85-7[f]		Oral-rat	LD50:	6,000 mg/kg			
				Oral-mouse	TDLo:	29 gm/kg	2YC	Carcinogenic	
Cyclohexane, 1,2,3,4,5, 6-Hexachloro-, delta-isomer	$C_6H_6Cl_6$	319-86-8[f]		Oral-rat	LD50:	1,000 mg/kg			
Cyclohexane, 1,2,3,4,5,6-Hexachloro-, gamma-isomer (Lindane)	$C_6H_6Cl_6$	58-89-9[f]	TLm 96: under 1 ppm	Oral-child	LDLo:	180 mg/kg			TLV (air): 0.5 mg/m^3
				Oral-child	TDLo:	111 mg/kg		Systemic	
				Oral-rat	LD50:	76 mg/kg			
				Skin-rat	LD50:	500 mg/kg			OSHA std (air): TWA 500 µg/m^3 (skin)
				Intraperitoneal-rat	LDLo:	35 mg/kg			
				Oral-mouse	LD50:	86 mg/kg			
				Oral-mouse	TDLo:	29 gm/kg	52WC	Carcinogenic	
				Intraperitoneal-mouse	LDLo:	75 mg/kg			
				Oral-dog	LD50:	40 mg/kg			
				Intravenous-dog	LDLo:	8 mg/kg			
				Oral-rabbit	LD50:	60 mg/kg			
				Skin-rabbit	LD50:	50 mg/kg			
				Intravenous-rabbit	LDLo:	4,500 ug/kg			
				Oral-guinea pig	LD50:	127 mg/kg			
				Oral-hamster	LD50:	360 mg/kg			
				Oral-bird, wild	LDLo:	100 mg/kg			
				Intramuscular-bird, wild	LDLo:	26 mg/kg			
Ethane, 1,2-Dichloro- (Ethylene Dichloride)	$C_2H_4Cl_2$	107-06-2[f]	TLm 96: 1,000-100 ppm	Inhalation-human	TCLo:	4,000 ppm	H	Central Nervous System	TLV (air): 50 ppm
				Oral-human	TDLo:	428 mg/kg		Gastro-intestinal tract	OSHA std (air): TWA 50 ppm; Cl 100; PK 200/5M/3H
				Oral-man	LDLo:	810 mg/kg			
				Oral-human	LDLo:	500 mg/kg			
				Oral-rat	LD50:	12 µg/kg			

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Ethane, 1,2-Dichloro- (cont'd)				Inhalation-rat	LCLo:	1,000 ppm	4H		
				Intraperitoneal-rat	LD50:	74 µg/kg			
				Subcutaneous-rat	LDLo:	500 mg/kg			
				Oral-mouse	LDLo:	600 mg/kg			NIOSH recm std (air): TWA 1 ppm; Cl 2 ppm/15M
				Inhalation-mouse	LCLo:	5,000 mg/m^3	2H		
				Intraperitoneal-mouse	LD50:	40 µg/kg			
				Subcutaneous-mouse	LDLo:	380 mg/kg			
				Oral-dog	LDLo:	2,000 mg/kg			
				Intravenous-dog	LDLo:	175 mg/kg			
				Oral-rabbit	LD50:	860 mg/kg			
				Inhalation-rabbit	LCLo:	3,000 ppm	7H		
				Subcutaneous-rabbit	LDLo:	1,200 mg/kg			
				Inhalation-pig	LCLo:	3,000 ppm	7H		
				Inhalation-guinea pig	LCLo:	1,500 ppm	7H		
				Intraperitoneal-guinea pig	LDLo:	600 mg/kg			
				Skin-rabbit		625 mg	open	Mild Irritation	
				Eye-rabbit		63 mg		Severe Irritation	
				Oral-rat	TDLo:	26 gm/kg	78WI	Carcinogenic	
				Oral-mouse	TDLo:	81 gm/kg	78WI	Carcinogenic	
Ethane, Hexachloro-	C_2Cl_6	67-72-1[f]		Oral-human	LDLo:	50 mg/kg			TLV (air): 1 ppm (skin)
				Oral-rat	LD50:	6,000 mg/kg			
				Intraperitoneal-mouse	LD50:	4,500 mg/kg			
				Intravenous-dog	LDLo:	325 mg/kg			OSHA std (air): 1 ppm (skin)
				Subcutaneous-rabbit	LDLo:	4,000 mg/kg			
Ethane, 1,1,2,2-tetrachloro-	$C_2H_2Cl_4$	79-34-5[f]		Oral-human	TLDO:	30 mg/kg		Central Nervous System	OSHA std (air): TWA 5 ppm (skin)
				Oral-human	LDLo:	50 mg/kg			
				Inhalation-human	TCLo:	1,000 mg/m^3	30M	Central Nervous System	NIOSH recm std (air): TWA 1 ppm
				Inhalation-rat	LCLo:	1,000 ppm	4H		
				Oral-mouse	TDLo:	58 gm/kg	58WC	Carcinogenic	
				Inhalation-mouse	LCLo:	9,000 mg/m^3	40M		
				Intraperitoneal-mouse	LDLo:	30 mg/kg			
				Oral-dog	LDLo:	300 mg/kg			
				Intravenous-dog	LDLo:	50 mg/kg			
				Inhalation-cat	LCLo:	19,000 mg/m^3	45M		
				Subcutaneous-rabbit	LDLo:	500 mg/kg			

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Ethane, 1,1,2-Trichloro-	$C_2H_3Cl_3$	79-00-5[f]	TLm: 96: 100-10 ppm	Oral-human	LDLo:	50 mg/kg			OSHA std (air): TWA 10 ppm (skin)
				Oral-rat	LD50:	1,140 mg/kg			
				Inhalation-rat	LCLo:	500 ppm	8H		TLV (air): 10 ppm (skin)
				Intraperitoneal-mouse	LD50:	994 mg/kg			
				Subcutaneous-mouse	LD50:	227 mg/kg			
				Oral-dog	LDLo:	500 mg/kg			
				Intraperitoneal-dog	LDLo:	450 mg/kg			
				Intraveneous-dog	LDLo:	95 mg/kg			
				Subcutaneous-rabbit	LDLo:	500 mg/kg			
				Skin-rabbit		500 mg	open	Mild Irritation	
				Skin-guinea pig		1,440 mg	15M	Irritation	
				Inhalation-cat	LCLo:	13,100 mg/m³	4.5H		
Ethene, 1,1-dichloro- (1,1-Dichloro-ethylene)	$C_2H_2Cl_2$	75-35-4[f]	TLm 96: 1,000-100 ppm	Inhalation-human	TCLo:	25 ppm		Systemic	TLV (air): 10 ppm
				Oral-rat	LD50:	200 mg/kg			
				Inhalation-rat	LCLo:	10,000 ppm	24H		NIOSH recm std (air): TWA 1 ppm; Cl 5ppm/15M
				Inhalation-rat	TCLo:	55 ppm	6H/52WI	Neoplastic	
				Inhalation-rat	TCLo:	55 ppm	6H/1YI	Equivocal Tumorigenic Agent	
				Oral-dog	LDLo:	5,750 mg/kg			
				Intravenous-dog	LDLo:	225 mg/kg			
				Subcutaneous-rabbit	LDLo:	3,700 mg/kg			
				Inhalation-mouse	LC50:	98 ppm	22H		
				Inhalation-mouse	TCLo:	55 ppm	6H/1YI	Equivocal Tumorigenic Agent	
Ethylene, Chloro- (Vinyl Chloride)	C_2H_3Cl	75-01-4[f]	TLm 96: over 1,000 ppm	Inhalation-man	TCLo:	500 ppm	4YI	Carcinogenic	TLV (air): 200 ppm
				Oral-rat	LD50:	500 mg/kg			
				Oral-rat	TDLo:	11 gm/kg	136WI	Carcinogenic	OSHA std (air): TWA 1 ppm; Cl 5 ppm/15M
				Inhalation-rat	TCLo:	250 ppm	4H/130WI	Carcinogenic	
				Inhalation-rat	TCLo:	6,000 ppm	4H/12-18D (Preg)	Carcinogenic	
				Inhalation-mouse	TCLo:	250 ppm	35 WI	Carcinogenic	NIOSH recm std (air): TWA 1 ppm; Cl 5 ppm/15M
				Inhalation-hamster	TCLo:	500 ppm	4H/30W-I	Carcinogenic	
				Inhalation-rat	TCLo:	6,000 ppm	4H/12-18D (Preg)	Neoplastic	
				Inhalation-rat	TCLo:	250 ppm	39WI	Carcinogenic	
				Inhalation-mouse	TCLo:	50 ppm	6H/12WI	Carcinogenic	
				Oral-rat	TDLo:	34 gm/kg	136WI	Carcinogenic	

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type[b] of Dose	Dose	Duration[c]	Effects[d]	
Ethylene, 1,2-Dichloro-(E)-	$C_2H_2Cl_2$	156-60-5[f]		Inhalation-human	TCLo:	4,800 mg/m^3	10M	Central Nervous System	
				Inhalation-mouse	LCLo:	75,000 mg/m^3	2H		
				Inhalation-cat	LCLo:	43,000 mg/m^3	6H		
Ethylene, Tetrachloro- (Tetrachloroethene)	C_2Cl_4	127-18-4[f]	TLm 96: 100-10 ppm	Inhalation-human	TCLo:	200 ppm		Systemic	OSHA std (air): TWA 100 ppm;
				Oral-human	LDLo:	500 mg/kg			Cl 200;
				Inhalation-man	TCLo:	280 ppm	2H	Eye	PK 300/5M/3H
				Inhalation-man	TCLo:	600 ppm	10M	Central Nervous System	NIOSH recm std (air):
				Inhalation-rat	LCLo:	4,000 ppm	4H		TWA 50 ppm;
				Oral mouse	LD50:	8,850 mg/kg			Cl 100 ppm/15M
				Inhalation-mouse	LCLo:	23,000 mg/m^3	2H		
				Intraperitoneal-mouse	LD50:	5,671 mg/kg			
				Oral-dog	LDLo:	4,000 mg/kg			
				Intraperitoneal-dog	LD50:	2,100 mg/kg			
				Intravenous-dog	LDLo:	85 mg/kg			
				Oral-cat	LDLo:	4,000 mg/kg			
				Oral-rabbit	LDLo:	5,000 mg/kg			
				Subcutaneous-rabbit	LDLo:	2,200 mg/kg			
				Oral-mouse	TDLo:	86 gm/kg	41WC	Carcinogenic	
Ethylene, Trichloro- (Trichloroethene)	C_2HCl_3	79-01-6[f]	TLm 96: 1,000-100 ppm	Oral-human	LDLo:	50 mg/kg			TLV (air): 100 ppm
				Inhalation-human	TCLo:	6,900 mg/m^3	10M	Central Nervous System	OSHA std (air): TWA 100 ppm;
				Inhalation-human	TCLo:	160 ppm	83M	Central Nervous System	Cl 200; PK 300/5M/2H
				Inhalation-man	TCLo:	110 ppm	8H	Irritant	NIOSH recm std (air): TWA 100 ppm;
				Oral-rat	LD50:	4,920 mg/kg			Cl 150 ppm/10M
				Inhalation-rat	LCLo:	8,000 ppm	4H		
				Oral-mouse	TDLo:	135 gm/kg	27WI	Carcinogenic	
				Inhalation-mouse	LCLo:	3,000 ppm	2H		
				Intravenous-mouse	LD50:	34 mg/kg			
				Oral-dog	LDLo:	5,860 mg/kg			
				Intraperitoneal-dog	LD50:	1,900 mg/kg			
				Intravenous-dog	LDLo:	150 mg/kg			
				Subcutaneous-rabbit	LDLo:	1,800 mg/kg			
				Oral-cat	LDLo:	5,864 mg/kg			
				Inhalation-cat	LCLo:	32,500 mg/m^3	2H		
				Inhalation-guinea pig	LCLo:	37,200 ppm	40M		
				Eye-human		5 ppm		Irritation	

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Ethylene, Trichloro- (Trichloroethene) (cont'd)				Skin-rabbit		500 mg	24H	Severe Irritation	
				Eye-rabbit		20 mg	24H	Severe Irritation	
				Oral-human	LDLo:	7 gm/kg			
				Inhalation-human	TDLo:	812 mg/kg		Systemic	
				Inhalation-man	LCLo:	2,900 ppm			
				Intraperitoneal-mouse	LD50:	3,000 mg/kg			
				Subcutaneous-dog	LDLo:	150 mg/kg			
				Oral-rabbit	LDLo:	7,330 mg/kg			
Methane, Chloro- (Methyl Chloride)	CH_3Cl	74-87-3[f]	TLm 96: over 1,000 ppm	Inhalation-rat	LC50:	152,000 mg/m^3	30M		TLV (air): 100 ppm
				Inhalation-mouse	LC50:	3,146 ppm	7H		
				Inhalation-dog	LCLo:	15,000 ppm	7H		
				Inhalation-cat	LCLo:	128,700 mg/m^3	4H		OSHA std (air): TWA 100 ppm Cl 200; PK 300/ 5M/3H
				Inhalation-guinea pig	LCLo:	20,000 ppm	2H		
Methane, Dichloro- (Methylene Chloride)	CH_2Cl_2	75-09-2[f]	TLm 96: 1,000-100 ppm	Inhalation-human	TCLo:	500 ppm	1YI	Central Nervous System	TLV (air): 200 ppm
				Oral-human	LDLo:	500 mg/kg			OSHA std (air): TWA 500 ppm; Cl
				Inhalation-human	TCLo:	500 ppm	8H	Blood	1,000; PK 2,000/ 5M/2H
				Oral-rat	LD50:	167 mg/kg			
				Inhalation-rat	LC50:	88,000 mg/m^3	30M		
				Inhalation-mouse	LC50:	14,400 ppm	7H		NIOSH recm std (air): TWA 75 ppm; PK 500 ppm/15M
				Intraperitoneal-mouse	LD50:	1,500 mg/kg			
				Subcutaneous-mouse	LD50:	6,460 mg/kg			
				Oral-dog	LDLo:	3,000 mg/kg			
				Inhalation-dog	LCLo:	20,000 ppm	7H		
				Intraperitoneal-dog	LDLo:	950 mg/kg			
				Subcutaneous-dog	LDLo:	2,700 mg/kg			
				Intravenous-dog	LDLo:	200 mg/kg			
				Inhalation-cat	LCLo:	43,400 mg/m^3	4.5H		
				Oral-rabbit	LDLo:	1,900 mg/kg			
				Subcutaneous-rabbit	LDLo:	2,700 mg/kg			
				Inhalation-guinea pig	LCLo:	5,000 ppm	2H		
Methane, Trichlorofluoro-	CCl_3F	75-69-4[f]		Inhalation-rat	LCLo:	10 ppm	20M		TLV (air): 1000 ppm
				Intraperitoneal-mouse	LD50:	1,743 mg/kg			OSHA std (air): TWA 1,000 ppm

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type[b] of Dose	Dose	Duration[c]	Effects[d]	
Phenol	C_6H_6O	108-95-2[f]	TLm 96: 100-10 ppm	Skin-rabbit		500 mg	24H	Severe Irritation	TLV (air): 5 ppm (skin)
				Skin-rabbit		535 mg	open	Severe Irritation	
				Eye-rabbit		5 mg		Severe Irritation	OSHA std (air): TWA 5 ppm (skin)
				Oral-human	LDLo:	140 mg/kg			
				Oral-rat	LD50:	414 mg/kg			
				Skin-rat	LD50:	669 mg/kg			
				Intraperitoneal-rat	LD50:	250 mg/kg			NIOSH recm std (air): TWA 20 mg/m^3; Cl 60 mg/m^3/15M
				Subcutaneous-rat	LDLo:	650 mg/kg			
				Oral-mouse	LD50:	300 mg/kg			
				Skin-mouse	TDLo:	4,000 mg/kg	20WI	Carcinogenic	
				Intraperitoneal-mouse	LD50:	360 mg/kg			
				Subcutaneous-mouse	LD50:	344 mg/kg			
				Intravenous-mouse	LD50:	112 mg/kg			
				Oral-dog	LDLo:	500 mg/kg			
				Parenteral-dog	LDLo:	2,000 mg/kg			
				Oral-cat	LDLo:	80 mg/kg			
				Subcutaneous-cat	LDLo:	80 mg/kg			
				Parenteral-cat	LDLo:	500 mg/kg			
				Oral-rabbit	LDLo:	420 mg/kg			
				Skin-rabbit	LD50:	850 mg/kg			
				Intraperitoneal-rabbit	LDLo:	620 mg/kg			
				Subcutaneous-rabbit	LDLo:	620 mg/kg			
				Intravenous-rabbit	LDLo:	180 mg/kg			
				Parenteral-rabbit	LDLo:	300 mg/kg			
				Intraperitoneal-guinea pig	LDLo:	300 mg/kg			
				Subcutaneous-guinea pig	LDLo:	450 mg/kg			
				Subcutaneous-frog	LDLo:	75 mg/kg			
				Parenteral-frog	LDLo:	290 mg/kg			
				Subcutaneous-frog	LDLo:	290 mg/kg			
Phenol, o-Chloro-	C_6H_5ClO	95-57-8[f]		Oral-rat	LD50:	670 mg/kg			
				Intraperitoneal-rat	LD50:	230 mg/kg			
				Subcutaneous-rat	LD50:	950 mg/kg			
				Oral-mouse	LD50:	670 mg/kg			
				Skin-mouse	TDLo:	4,800 mg/kg	12WI	Neoplastic	
				Subcutaneous-rabbit	LDLo:	950 mg/kg			
				Intravenous-rabbit	LDLo:	120 mg/kg			
				Subcutaneous-guinea pig	LDLo:	800 mg/kg			
				Subcutaneous-frog	LDLo:	400 mg/kg			
				Oral-mammal	LD50:	440 mg/kg			

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[g]
				Route of Entry - Species	Type[b] of Dose	Dose	Duration[c]	Effects[d]	
Phenol, 2-,4-Dichloro-	$C_6H_4Cl_2O$	120-83-2[f]		Oral-rat	LDLo:	580 mg/kg			
				Intraperitoneal-rat	LD50:	430 mg/kg			
				Subcutaneous-rat	LD50:	1,730 mg/kg			
				Oral-mouse	LD50:	1,600 mg/kg			
				Skin-mouse	TDLo:	312 mg/kg	39WI	Carcinogenic	
Phenol, Pentachloro-	C_6HCl_5O	87-86-5[f]		Skin-rabbit		10 mg	24H open	Mild Irritation	TLV (air): 0.5 mg/m^3 (skin)
				Oral-human	LDLo:	29 mg/kg			
				Oral-man	TDLo:	196 mg/kg		Central Nervous System	OSHA std (air): TWA 500 µg/m^3
				Oral-rat	TDLo:	60 mg/kg	9D (Preg)	Teratogenic	
				Oral-rat	LD50:	50 mg/kg			
				Inhalation-rat	LD50:	11,700 ug/kg			
				Skin-rat	LD50:	105 mg/kg			
				Intraperitoneal-rat	LD50:	56 mg/kg			
				Subcutaneous-rat	LD50:	100 mg/kg			
				Subcutaneous-mouse	TDLo:	46 mg/kg		Neoplastic	
				Subcutaneous-dog	LDLo:	135 mg/kg			
				Oral-rabbit	LDLo:	70 mg/kg			
				Skin-rabbit	LDLo:	40 mg/kg			
				Intraperitoneal-rabbit	LDLo:	135 mg/kg			
				Subcutaneous-rabbit	LDLo:	70 mg/kg			
Phthalic Acid, Bis (2-Ethylhexyl) Ester	$C_{24}H_{38}O_4$	117-81-7[f]		Eye-rabbit		500 mg		Irritation	OSHA std (air): TWA 5 mg/m^3
				Oral-man	TDLo:	143 mg/kg		Gastro-intestinal Tract	
				Oral-rat	LD50:	31 gm/kg			
				Intraperitoneal-rat	LD50:	30,700 mg/kg			
				Intraperitoneal-rat	TDLo:	30 gm/kg	5-15D (Preg)	Tetratogenic	
				Intravenous-rat	LDLo:	300 mg/kg			
				Oral-mouse	LD50:	30 gm/kg			
				Oral-mouse	TDLo:	7,500 mg/kg	8D (Preg)	Teratogenic	
				Intraperitoneal-mouse	LD50:	14 gm/kg			
				Oral-rabbit	LD50:	34 gm/kg			
				Skin-rabbit	LD50:	25 gm/kg			
				Skin-guinea pig	LD50:	10 gm/kg			

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES
NIAGARA FALLS, NEW YORK

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data					Exposure Limits[e]
				Route of Entry - Species	Type of Dose[b]	Dose	Duration[c]	Effects[d]	
Phthalic Acid, Dibutyl Ester	$C_{16}H_{22}O_4$	84-74-2[f]	TLM 96: 1000-100 ppm	Oral-human	LDLo:	5,000 mg/kg			TLV (air): 5 mg/m³
				Oral-human	TDLo:	140 mg/kg		Eye	
				Oral-mouse	LD50:	12,000 mg/kg			OSHA std (air): TWA 5 mg/m³
				Intraperitoneal-rat	LD50:	3,050 mg/kg			
				Intraperitoneal-rat	TDLo:	874 mg/kg	5-15D (Preg)	Teratogenic	
Phthalic Acid, Diethyl Ester	$C_{12}H_{14}O_4$	84-66-2[f]		Eye-rabbit		112 mg		Irritation	TLV (air): 5 mg/m³
				Oral-human	LDLo:	500 mg/kg			
				Inhalation-human	TCLo:	1,000 mg/m³		Irritant	
				Intraperitonal-rat	LD50:	5,058 mg/kg			
				Intraperitoneal-rat	TDLo:	1,232 mg/kg	5-15D (Preg)	Teratogenic	
				Intraperitoneal-mouse	LD50:	2,749 mg/kg			
				Oral-rabbit	LDLo:	1,000 mg/kg			
				Intravenous-rabbit	LDLo:	100 mg/kg			
				Subcutaneous-guinea pig	LDLo:	3,000 mg/kg			
Toluene	C_7H_8	108-88-3[f]	TLm 96: 100-10 ppm	Eye-human		300 ppm		Irritation	TLV (air): 100 ppm (skin)
				Oral-human	LDLo:	50 mg/kg			
				Inhalation-human	TCLo:	200 ppm		Central Nervous System	OSHA std (air): TWA 200 ppm
				Inhalation-man	TCLo:	100 ppm		Psychotropic	C1 300; PK 500/10M
				Oral-rat	LD50:	5,000 mg/kg			
				Inhalation-rat	LCLo:	4,000 ppm	4H		NIOSH recm std (air): TWA 100 ppm; C1 200 ppm/10M
				Intraperitoneal-rat	LDLo:	800 mg/kg			
				Inhalation-mouse	LC50:	5,320 ppm	8H		
				Skin-rabbit	LD50:	14 gm/kg			
				Skin-rabbit		435 mg		Mild Irritation	
				Eye-rabbit		870 µg		Mild Irritation	
				Subcutaneous-frog	LDLo:	920 mg/kg			
2,4-Xylenol (2,4-Dimethylphenol)	$C_8H_{10}O$	105-67-9[f]		Oral-rat	LD50:	3,200 mg/kg			
				Skin-rat	LD50:	1,040 mg/kg			
				Oral-mouse	LD50:	809 mg/kg			
				Skin-mouse	TDLo:	5,600 mg/kg	28WI	Carcinogenic	

TABLE 7 (Continued)

TOXICITY OF COMPOUNDS
HOOKER CHEMICALS AND PLASTICS CORPORATION WASTE DISPOSAL SITES

a Aquatic Toxicity: TLm 96: 96-hour static or continuous flow standard protocol, in parts per million (ppm)

b Other Toxicity Data:
- LD50 - lethal dose 50% kill
- LCLo - lowest published lethal concentration
- LC50 - lethal concentration 50% kill
- LDLo - lowest published lethal dose
- TDLo - lowest published toxic dose
- TCLo - lowest published toxic concentration
- TD - toxic dose

c Duration:
- M - minute;
- H - hour
- D - day
- W - week
- Y - year
- C - continuous
- I - intermittent

d Exposure Limits:
- NR - not reported
- NIOSH - National Institute for Occupational Safety and Health
- OSHA - Occupational Safety and Health Act of 1970
- TWA - time-weighted average concentration
- TLV - threshold limit value
- Cl - ceiling
- Pk - peak concentration

e Blood - Blood effects; effect on all blood elements, electrolytes, pH, protein, oxygen carrying or releasing capacity.
Carcinogenic - Carcinogenic effects; producing cancer, a cellular tumor the nature of which is fatal, or is associated with the formation of secondary tumors (metastasis)
Central Nervous System - Includes effects such as headaches, tremor, drowsiness, convulsions, hypnosis, anesthesia.
Eye - Irritation, diplopia, cataracts, eye ground, blindness by affecting the eye or the optic nerve.
Gastrointestinal - diarrhea, constipation, ulceration.
Irritant - Any irritant effect on the skin, eye or mucous membrane.
Mutagenic - Transmissible changes produced in the offspring.
Neoplastic - The production of tumors not clearly defined as carcinogenic.
Psychotropic - Exerting an effect upon the mind.
Pulmonary - Effects on respiration and respiratory pathology.
Systemic - Effects on the metabolic and excretory function of the liver or kidneys.
Teratogenic - Nontransmissible changes produced in the offspring.

f This chemical has been selected for priority attention as point source water effluent discharge toxic pollutant (NRDC vs Train consent decree)

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APPENDIX A

PRIORITY POLLUTANTS LISTING

RECOMMENDED LIST OF PRIORITY POLLUTANTS

Compound Name

1. *acenaphthene
2. *acrolein
3. *acrylonitrile
4. *benzene
5. *benzidine
6. *carbon tetrachloride (tetrachloromethane)

*Chlorinated benezenes (other than dichlorobenzenes)

7. chlorobenezene
8. 1,2,4-trichlorobenzene
9. hexachlorobenzene

*Chlorinated ethanes (including 1,2-dichloroethane, 1,1,1-trichloroethane and hexachloroethane)

10. 1,2-dichloroethane
11. 1,1,1-trichloroethane
12. hexachloroethane
13. 1,1-dichloroethane
14 1,1,2-trichloroethane
15. 1,1,2,2-tetrachloroethane
16 chloroethane

*Chloroalkyl ethers (chloromethyl, chloroethyl and mixed ethers)

17. bis(chloromethyl) ether

*Specific compounds and chemical classes as listed in the consent degree.

18. bis(2-chloroethyl) ether

19 2-chloroethyl vinyl ether (mixed)

*Chlorinated naphtalene

20. 2-chloronaphthalene

*Chlorinated phenols (other than those listed elsewhere; includes trichlorophenols and chlorinated cresols)

21. 2,4,6-trichlorophenol

22. parachlorometa cresol

23. *chloroform (trichloromethane)

24. *2-chlorophenol

*Dichlorobenzenes

25 1,2-dichlorobenzene

26. 1,3-dichlorobenzene

27. 1,4-dichlorobenzene

*Dichlorobenzidine

28. 3,3'-dichlorobenzidine

*Dichloroethylenes (1,1-dichloroethylene and 1,2-dichloroethylene)

29 1,1-dichloroethylene

30. 1,2-trans-dichloroethylene

31. *2,4-dichlorophenol

*Dichloropropane and dichloropropene

32. 1,2-dichloropropane

33. 1,2-dichloropropylene (1,3-dichloropropene)

34. *2,4-dimethylphenol

*Dinitrotoluene

35. 2,4-dinitrotoluene

36. 2,6-dinitrotoluene

37. *1,2-diphenylhydrazine

38. *ethylbenzene

39. *fluoranthene

*Haloethers (other than those listed elsewhere)

40. 4-chlorophenyl phenyl ether

41. 4-bromophenyl phenyl ether

42. bis(2-chloroisopropyl) ether

43. bis(2-chloroethoxy) methane

*Halomethanes (other than those listed elsewhere)

44. methylene chloride (dichloromethane)

45. methyl chloride (chloromethane)

46. methyl bromide (bromomethane)

47. bromoform (tribromomethane)

48. dichlorobromomethane

49. trichlorofluoromethane

50. dichlorodifluoromethane

51. chlorodibromomethane

52. *hexachlorobutadiene

53. *hexachlorocyclopentadiene

54. *isophorone

55. *naphthalene

56. *nitrobenzene

*Nitrophenols (including 2,4-dinitrophenol and dinitrocresol)

57. 2-nitrophenol

58. 4-nitrophenol

59. *2,4-dinitrophenol

60. 4,6-dinitro-o-cresol

*Nitrosamines

61. N-nitrosodimethylamine

62. N-nitrosodiphenylamine

63. N-nitrosodi-n-propylamine

64. *pentachlorophenol

65. *phenol

*Phthalate esters

66. bis(2-ethylhexyl) phthalate

67. butyl benzyl phthalate

68. di-n-butyl phthalate

69. di-n-octyl phthalate

70. diethyl phthalate

71. dimethyl phthalate

*Polynuclear aromatic hydracrarbons

72. benzo(a)anthracene (1,2-benzanthracene)

73. benzo (a) pyrene (3,4-benzopyrene)

74. 3,4-benzofluoranthene (benzo(b)fluoranthene)

75. benzo(k)fluoranthane (11,12-benzofluoranthene)

76. chrysene

77. acenaphthylene

78. anthracene

79. benzo(ghi)perylene (1,12-benzoperylene)

80. fluroene

81. phenathrene

82. dibenzo (a,h)anthracene (1,2,5,6-dibenzanthracene)

83. indeno (1,2,3-cd)pyrene (2,3-o-phenylenepyrene)

84. pyrene

85. *tetrachloroethylene

86. *toluene

87. *trichloroethylene

88. *vinyl chloride

Pesticides and Metabolites

89. *aldrin

90. *dieldrin

91. *chlordane (technical mixture & metabolites)

*DDT and Metabolites

92. 4,4'-DDT

93. 4,4'-DDE (p,p'-DDX)

94. 4,4'-DDD (p,p'-TDE)

*endosulfan and metabolites

95. a-endosulfan-Alpha

96. b-endosulfan-Beta

97. endosulfan sulfate

*endrin and metabolites

98. endrin

99. endrin aldehyde

*heptachlor and metabolites

100. heptachlor

101. heptachlor epoxide

*hexachlorocyclohexane (all isomers)

102. a-BHC-Alpha

103. b-BHC-Beta

104. r-BHC (lindane)-Gamma

105. g-BHC-Delta

*polychlorinated biphenyls (PCB's)

106. PCB-1242 (Arochlor 1242)

107. PCB-1254 (Arochlor 1254)

108. PCB-1221 (Arochlor 1221)

109. PCB-1232 (Arochlor 1232)

110. PCB-1248 (Arochlor 1248)

111. PCB-1260 (Arochlor 1260)

112. PCB-1016 (Arochlor 1016)

113. *Toxaphene

114. *Antimony (Total)

115. *Arsenic (Total)

116. *Asbestos (Fibrous)

117. *Beryllium (Total)

118. *Cadmium (Total)

119. *Chromium (Total)

120. *Copper (Total)

121. *Cyanide (Total)

122. *Lead (Total)

123. *Mercury (Total)

124. *Nickel (Total)

125. *Selenium (Total)

126. *Silver (Total)

127. *Thallium (Total)

128. *Zinc (Total)

129. **2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD)

*Specific compounds and chemical classes as listed in the consent decree

**This compound was specifically listed in the consent decree. Because of the extreme toxicity (TCDD). We are recommending that laboratories not acquire analytical standard for this compound.

APPENDIX B

METHODS, ANALYTICAL PROCEDURES, AND QUALITY CONTROL

MUTAGEN ASSAY METHODS

Sample Extraction

Prior to extraction, samples were allowed to settle for one hour. The aqueous portion of the samples were then decanted; the sediment was discarded.

For basic-neutral extractions, one-liter portions of decanted sample were adjusted above pH 12 with NaOH. Each one-liter aliquot was extracted three times (5 minutes each) with 35 ml of dichloromethane. The solvent fraction was then separated, mixed with anhydrous sodium sulfate to remove any emulsion and filtered (Whatman No. 1 filter paper) into a one-liter round bottom flask. The aqueous fractions were retained for acidic extraction. These were adjusted below pH 2 and the above procedure repeated.

The combined solvent fractions (approximately 420 ml) were evaporated to dryness at 44° C in a rotoevaporator.* The residue was resuspended into 35 ml** sterile dimethylsulfoxide (DMSO), labeled and refrigerated at 4°C until assayed by the Ames procedure.

An alternate mehtod of preparing samples for the Ames Assay consisted of filtering 50 ml aliquots of unconcentrated sample through a 0.22 micro-meter pore-size membrane filter. Filtered samples were labeled and refrigerated at 4°C until assayed by the Ames procedure.

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* Using this method the estimate of mutagenic activity from complex mixtures is low, because: 1) the volatile alkyl halides are lost in the dichloromethane/DMSO exchange, and 2) the Salmonella test detects only about 90% of carcinogens as mutagens. Some of the important chlorinated hydrocarbons are not detected, i.e., chloroform, hexachlorobenzene, etc.

** Sample No. 01 required 50 ml DMSO for complete solution. This material was later found to be contaminated. The solution was sterilized by filtration through a ultra-fine, fritted-glass filter prior to the Ames Assay.

Bacterial Mutagenicity Assay

The Standard Ames Salmonella/mammalian microsome mutagenicity assay was performed using the agar-plate incorporation procedure as described by Ames, et al.[1] Sample extracts and filtered whole (unconcentrated) aliquots were screened with Salmonella typhimurium tester strains TA 98, TA 100, TA 1535, TA 1537 and TA 1538, first individually and then in the presence of rat liver homogenates (S-9 mix).

Mutagenesis Assay by Preincubation Method

Undiluted extracts of samples 01 and 02 contained large amounts of organic materials. Additionally, Sample No. 01 was toxic to the Salmonella tester strains. To allow the liver homogenate more time to react with the organic mixture, and to possibly reduce the toxicity of Sample No. 01, the sample extracts were preincubated in the presence of S-9 mix and the tester strains at 20°C for 20 minutes prior to the agar-plate assay.

Quality Control

A four-liter volume of sterile distilled water was added to a clean, 1-gallon amber glass bottle and treated as a sample. This served as a quality reference for the sample bottles, distilled water, extracting solvents, emulsion removal, and the concentration process. A DMSO sample was tested to ensure that this material did not interfere with test results. These quality control procedures were repeated five times during the study.

The tester strains TA 1535, TA 1537, TA 1538, TA 98 and TA 100 were exposed to diagnostic mutagens to confirm their natural reversion characteristics. The strains were tested for ampicillin resistance, crystal violet sensitivity, ultra-violet light sensitivity, and histidine requirement. Spontaneous reversion rates were tested with each sample series.

Rat liver homogenate was tested with 2-aminofluorene with strains TA 1538, TA 98 and TA 100 to confirm the metabolic activation process.

Sterility checks were performed on solvents, extracts, liver preparation, and all culture media.

VOLATILE ORGANIC COMPOUNDS BY GC/MS
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

1.0 Scope and Application

1.1 Water and wastewater samples may be analyzed for purgeable organic compounds, typically methylene chloride through ethyl benzene by GC/MS. Both qualitative and quantitative data are generated. This procedure includes data evaluation as defined for screening of industrial wastes for "priority pollutants" as well as data for complete organics characterization of any purgeable components.

2.0 Summary of Method

2.1 Aliquots of aqueous samples are purged with an inert gas. Low molecular weight and slightly soluble components are stripped from the solution and trapped on a porous polymer adsorbent trap. Organic components are then desorbed from the trap by rapid heating onto an analytical gas chromatographic (GC) column. As separated components elute from the GC column, they are detected by a quadrupole mass spectometer. Quantitation of compounds identified from their spectra is effected either by external or internal standard techniques.

3.0 Sample Handling and Preservation

3.1 Samples may be collected as duplicate grab samples. Duplicates are useful for reanalysis of the sample if needed. If data are to be correlated to other 24 hours composite samples, collect multiple grab samples at regular intervals. They may be composited at the lab prior to analysis.

3.2 Preserve the samples by maintaining at or below 4°C during

shipment and storage. Samples containing residual chlorine require the addition of 0.1g $Na_2S_2O_3$ per 100 ml of sample to reduce the remaining chlorine.

4.0 Definitions and Comments

5.0 Interferences

5.1 Samples containing residual chlorine can produce halogenated organics in excess of what was present at the time of collection. Therefore the addition of a reducing agent is necessary if residual chlorine is suspect.

5.2 No head space is allowed in a sample. Samples containing head space may loose volatile species and produce erronous results.

5.3 Samples exposed to vapors of volatile organic compounds may absorb those vapors and produce erronous data. Blanks must be handled and transported concurrently with samples to identify potential contamination.

6.0 Apparatus

6.1 Sample Bottles: 1 oz. glass bottles equipped with teflon-lined silicone septa and screw caps (Pierce #13074 and #12722 or equivalent). Before sampling, wash used bottles with soap (Alconox or equivalent) and tap water, rinse with tap water. New bottles require only washing with tap water. Bake bottles at 200°C and septa at 80°C for 30 minutes. Allow to cool in a desicator with charcoal adsorbant to maintain an organics-free atmosphere. Then cap the bottles and hold for sampling.

6.2 Sample handling syringes: Samples are transferred using 5.0 ml. gas-tight syringes equipped with gas-tight valves and 6" needles. (Tekmar or equivalent)

6.3 Liquid sample concentrator: Tekmar LSC-1 or equivalent with the following modifactions:

6.3.1 Replace existing trap with a thin wall (0.020" stainless steel (SS) trap packed with 15 cm 60/80 mesh Tenax GC (Applied Sciences). Wrap the trap with fiberglass insulated heating wire (Briskheat, 7 ohm per foot Nichrome wire for direct contact with metal or equivalent). Wrap the platinum resistance element between the SS tubing and the heating wire. Attach the heater wire and resistance element to the appropriate terminals.

6.3.2 Add a trap made of 12" of 3/8" copper tubing packed with activated charcoal (190°C for 4 hours) immediately ahead of the purging chamber.

6.3.3 Add a GC flow controller such that flow going to the GC column is regulated. The GC column then becomes completely independent of the existing GC flow systems.

6.4 GC column: Separations are effected using an 8' by 1/8" SS column packed with 0.2% Carbowax 1500 on 60/80 Mesh Carbopack C (available from Supelco).

6.5 Gas chromatograph: A Varian 1400 or equivalent equipped with a linear temperature programmer.

6.6 Detector: Finnigan 1015 mass spectrometer with Systems Industries System 150 data system, or equivalent instrument capable of collecting continuous repetative mass spectra (CRMS) over a range of 33 to 260 amu in 5 seconds or less. The data system must be capable of generating multiple extracted ion current profiles (EIPC).

6.7 Glassware: All glassware is washed as described in section 6.1 and baked at 105°C (up to 200°C) for at least 30 minutes.

6.8 Analytical Balance: Capable of measuring 0.0001g for standards preparation.

7.0 Reagents

7.1 Organic-Free water: Pass tap water through a 2 x 40 cm column of charcoal activated by heating to 190°C for four hours.

7.2.a Concentrated Standards (Liquid components): Stock solutions are prepared at ca. 1 mg/ml in pesticide analysis grade methanol. Due to the high volatility of some compounds, exact concentrations are calculated from the volume of pure compound used and its density. To 10.0 ml of methanol in a 14 ml vial with a teflon-lined screw cap, add 10.0 ul of pure compound, seal, mix and store in a freezer at -20°C. This stock standard may be stable for two months dependent upon the volatility of the component. Calculate the concentration from the volume of pure compound and its density as follows:

$$ng/ul = \frac{10.0 \times 10^{-3} ml}{10.0\ ml} \times (density) \frac{g}{ml} \times \frac{1\ ng}{10^{-9} g} \times \frac{10^{-3}\ ml}{1\ ul}$$

7.2.b Concentrated Standards (Gaseous components): Stock solutions of gaseous components may be prepared similarly to liquid components with the following change. Prepare a vial containing 10.0 ml of methanol, weigh the capped bottle and record this tare weight. Carefully bubble the pure gaseous component into the methanol. When enough gas has been absorbed into the methanol (estimated), reseal the vial and reweigh. The increase in weight represents the amount of pure component added. Calculate

the concentration as follows:

$$ng/ul = (\text{net weight})mg \times \frac{1\ ng}{10^{-6}\ mg} \times \frac{10^{-3}\ ml}{ul}$$

7.3 Working concentrate: Remove the stock standard from the freezer and allow to equilibrate to ambient temperature. With a 250 microliter syringe, prepare a mixed Standard with each component at 20 ng/ul in methanol. Seal the solution in 2 ml crimp seal vials with teflon-lined septa. These working standards may be stable up to one month depending on the volatility of the components.

7.4 Analytical standards for GC/MS: Using a microliter syringe, add 1 to 50 ul of the working concentrate to a 5.0 ml aliquot of organic-free water. Analyze immediately. Each ul of working concentrate when added to 5.0 ml of water is equivalent to 4 ug/l (ppb).

7.5 Internal standards: In the same manner as 7.2 and 7.3, prepare a single working concentrate of bromochloromethane (CH_2BrCl) and 1,4 dichlorobutane ($C_4H_8Cl_2$) at 100 ng/ul each.

8.0 Procedure

8.1 Instrument Preparation

8.1.1 Install the gas chromatographic (GC) column by directly passing through the injection port. Attach the column using teflon ferrules only to allow subsequent dismantling the system. Connect the other end of the tubing to the trap exit of the Tekmar LSC-1. Attach a source of ultra-pure helium to the inlet of the Tekmar. Adjust the column flowrate to 30ml/min. Carefully check the system for leaks.

8.1.2 Periodically, replace the charcoal in the internal filter of the LSC-1.

8.1.3 Set up the GC for 60°C initial and 170°C final temperatures, an 8°C/min. program rate, and hold at the final temperature.

8.2 Mass spectrometer calibration

8.2.1 Adjust and calibrate the mass spectrometer according to the manufacturer's specifications.

8.2.2 Analyze an organics-free-water blank to verify a clean system.

8.2.3 Analyze a standard mix at a concentration near the midpoint of the calibration curve. Check the response of factors calculated for the multipoint calibration curve. Check the response of each compound and verify if it is within the range of response factors calculated for the multi point calibration curve. If not, determine the cause of the problem, make the necessary corrections and reanalyze the standard.

8.3 Sample Analysis

8.3.1 Equilibrate sample bottles to ambient temperature and pour an aliquot directly into a 5.0 ml syringe. Immediately insert the plunger, invert the syringe, expel any air and adjust the volume to 5.0 ml. Composite samples may be prepared by adjusting the volume to the desired amount for the individual aliquot and adding this to a second syringe. Continue preparing the individual

aliquots until the composite is prepared. Dose the sample with 10 ul (1 ug each standard) of the internal standard solution to yeild a concentration of 200 ug/l.

8.3.2 Remove a glass purge device from the oven and cool in the charcoal-filled desicator. Attach to the Tekmar and introduce the sample.

8.3.3 Purge the sample for 12 minutes at 40 ml/min. onto the Tenax trap. At the same time, cool the GC oven to ambient temperature by leaving the oven door open.

8.3.4 Set the trap desorb temperature to 180°C, switch to the desorb mode and start a timer. After 3½ minutes, begin collection of CRMS using the following conditions:

Mass range: 20-27; 33-260

Integration time: 17 ms.

Or scan time up: 4 seconds

And scan time down: 0.1 seconds

After four minutes, switch back to purge mode, close the oven and set the temperature to 60°C.

8.3.5 After eight minutes, begin the GC temperature program.

8.3.6 While the sample is running, remove the purge device and join the purge inlet and outlet line with a short piece of 1/4" tubing. Turn on the trap bake and adjust the temperature to 200°C. Bake out the trap for at least 5 minutes. Wash the purge device with methanol and place in an oven as described in section 6.1.

8.3.7 Collect data until the last components have eluted from the GC column. Typically, 30 minutes.

8.4 Data Evaluation

8.4.1 After each analysis, plot the reconstructed ion chromatogram (RIC) and extracted ion current profiles (EICP) for each internal standard added. Integrate the areas of the selected peaks and compare to the limits calculated in section 9.6. If the base peak areas are outside the acceptable ranges, evaluate the problem and reanalyze the sample. If the data are acceptable, process the data as required for organics characterization or priority pollutants as described below.

8.4.2 Organics Characterization. Select a spectrum and subtract the background for each peak of interest. Generate a plot of the spectrum for analysis. In addition, perform a search of the current NBS spectra library and print out the results. (ref. 2)

8.4.3 Priority Pollutant Evaluation. Using the protocal procedures (ref. 3), generate and evaluate each compound's EICP for the selected ions. Compounds that are present may be quantitated as described in the protocal and summarized in section 8.4.4.

8.4.4 Quantitation. Compounds identified are quantitated by the internal standard techniques. An ion of the compound is selected and integrated over the GC peak. The area of an internal standard (typically 1,4-dichlorobutane, m/e 55) ion is also determined. The concentration of the component is then determined based on the amount of internal standard (200 ppb here) and the relative response

factor determined in section 8.4.5 by the following equation:

$$C_c = \frac{Ac}{As} \times \frac{Cs}{Rf}$$

Where: Cc = concentration of component (ppb)
Ac = area of component ion
As = area of internal standard ion
Cs = concentration of internal standard (ppb)
Rf = relative response facton (unitless)

8.4.5 Determination of Response factors: Prior to the analysis of samples, response factors for the compourds of interest relative to the internal standard must be determined and verified over a concentration range. Analyze 200 ppb (typical for VOA's) for each compound. Mixed standards are acceptable. Measure the areas of the ions of interest of the internal standard and the components in the standards. Calculate the response factors as follows:

$$Rf = \frac{Ac}{As} \times \frac{Cs}{Cc}$$

Where: Rf = relative response factor
As = area of internal standard ion
Ac = area of component ion
Cs = concentration of internal standard (ppb)
Cc = concentration of component. (ppb)

9.0 Quality Control

9.1 Standard Curve - Prior to the determination of any sample components by GC/MS using internal standards, linearity for each standard component must be established over a typical working range of 20 to 200 ppb. This requires analysis of at least four concentration levels: 0, 20, 100 and 200 ppb. Calculate the response factors relative to one internal standard

and determine the mean and percent relative standard deviation (%RSD). Acceptable data are indicated by a %RSD of less than 20. Values outside this range indicate problems with response linearity and the linear range must be carefully evaluated. Table I shows typical data for 22 of the priority pollutants.

Daily, one standard mix at the midpoint of the linear range must be analyzed and the response factors should fall within the range indicated above. The %RSD range should be updated as more data are generated to reflect changes in the method's performance.

9.2 Precision - To determine the percision of the method a regular program of analyses of replicate aliquots of environmental samples must be carried out. The precision criterion should be developed from 15 sets of replicate results accumulated over a period of time during the routine analysis program. At least two replicate aliquots of a well mixed sample must be analyzed with each set of 20 samples or less analyzed at a given time. These replicate data must be obtained for each parameter of interest.

Initially, samples selected for replicate analysis should be those that are most representative of the interference potential of the sample type. As the program progresses, samples representing the entire range of concentrations and interference potential should be designed into the replicate analysis program.

After 15 replicate results have been obtained, calculate the range (R_i) of these results as follows:

$$R_i = X_{i1} - X_{i2}$$

where R_i is the difference between the results of the pair (X_{i1} and X_{i2}) from sample i-1 through n. The concentration of each sample is represented by the mean:

$$\bar{X}_i = \frac{(X_{i1} + X_{i2})}{2}$$

where $\bar{X}$ is the average of the results of the replicate pair. A preliminary estimate of the critical difference (R_c) between replicate analysis for any specific concentration level (C) can be calculated as:

$$R_c = 3.27 \ (C\Sigma R_i)/(\Sigma \bar{X}_i) \quad \text{(sums from } i=1 \text{ to } n\text{)}$$

From these data develop a table of such R_c values for various C values that span the concentration range of interest.

These preliminary critical difference values may be used to judge the acceptability of the succeeding replicate results. To do this, calculate the mean ($\bar{X}$) and difference (R) between the replicate results. Referring to the table of critical range values developed above, find the C nearest to $\bar{X}$ and use its R_c to evaluate the acceptability of R. If the R is greater than R_c, the system precision is out of control and the source of this unusual variability should be identified and resolved before continuing with routine analysis and periodically (after 25 to 30 additional pairs of replicate results are obtained) revise, update, and improve the table of critical range values.

9.3 Recovery - Determine the recovery of the method for the analysis

of environmental samples by adding a spike (T_i, true value) sufficient to approximately double the background concentration level ($\bar{X}_i$) of the sample selected earlier for replicate analysis (Section A1). If the original concentration is higher than the midpoint of the standard curve (range of the method), then the concentration of the spike should be approximately one-half the original concentration. If the concentration of the original sample was not detectable, the concentration of the spike should be five to fifteen times the lower limit of detection. The volume of standard added in aqueous solution should not dilute the sample by more than ten percent. The volume of standard added in an organic solvent solution should be kept small (100 ul/l or less), so that the solubility of the standard in the water will not be affected.

Analyze the sample, calculate the observed value (0_i), and then calculate the recovery for the spike as follows:

$$P = 100(0_i - \bar{X}_i)/T_i$$

where P_i is the percent recovery. If the sample was diluted due to the addition of the spike, adjust $\bar{X}_i$ accordingly.

After determining P_i for at least 15 spike results, calculate the mean percent recovery ($\bar{P}$) and standard deviation (S_p) of the recovery as follows:

$$\bar{P} = \left(\sum_{i-1}^{n} P_i^2 - \left(\sum_{i=2}^{n} P_i\right)^2/n\right)$$

$$S_p = \frac{1}{n-1}\left(\sum_{i-1}^{n} P_i^2 - \left(\sum_{i=2}^{n} P_i\right)^2/n\right)$$

where n = the number of percent recovery values available.

If the percent recovery of the spike is not within the interval of $\bar{P} \pm 3\ S_p$, the system accuracy is out of control and the source of this systematic error should be identified and resolved before continuing with routine analysis.

At least one spiked sample must be analyzed along with each set of 20 samples or less that is analyzed at a given time. This spiked data must be obtained for each parameter of interest. Record the recovery data of all spiked analyses and periodically (every 25 to 30 data points) revise, update, and improve the accuracy criteria.

9.4 System Blank - An organics-free-water blank must be analyzed daily showing no contamination of the analytical system. If EICP methods are being used to located pollutants, the blank must also be subjected to the same analysis procedure. Data collected from blanks may also be used to determine detection limits based upon the responses of any components present. Calculate detection limits for each component as twice the noise measured. Typical detection limits are 1 to 2 ppb.

9.5 Field Blanks - A field blank must be analyzed with each set of samples from a given source. This is particularly important since volatile organics samples can potentially be contaminated due to exposure of organic solvents. The blanks must be analyzed in the same manner as the sample. Field blanks for purgeables are sent from the laboratory to the sampling site and returned as a check on possible contamination of the sample by permeation of volatiles through the septum seal.

When interferences occur, the analytical results must be discarded unless sufficient data from these blanks is available to permit correction of the results.

9.6 Internal Standards - Measure the areas of the quantitation ions selected for the internal standards. Record the measured values in the GC logbook. Since instrument variations are usually small within an operating day, let the internal standard response from the calibration standard be X and reference any variation to X. Check each subsequent measurement and if it is outside the range of X ± 15%, consider the analysis out of control. Resolve the problem and reanalyze the sample. As more data are collected, update the limits periodically.

10.0 Calculations

10.1 If the concentration of standard solutions and internal standards in aqueous solutions are reported in ppb (parts per billion), no further calculations are necessary. Dilutions, when necessary, may be calculated assuming a 10% solution is one part sample diluted to 10 parts with organic-free water by:

$$\text{true conc.} = \text{measured conc.} \times \frac{100}{\%\text{sol.}}$$

11.0 Precision and Accuracy - This section summarizes the quality control for precision, accuracy, recoveries and detection limits. These data show that for the 16 pollutants evaluated:

a. The within-day precision is ca. ± 10% (compound dependent).

b. The day-to-day precision is ca. ± 26% (based on the second internal standard response).

c. The mean average recovery below 50 ppb is 110%.

d. All 16 compounds are detectable at 1 ppb.

11.1 Precision - Insufficient data have been collected to determine ranges described in section 9.2. However, the data from 2 Replicate Analyses are reported here:

Name	Avg.	Diff.	Avg.	Diff.
benzene	1	2	5.6	0
carbon tetrachloride	ND	--	ND	--
chlorobenzene	ND	--	1.85	0.1
1,2-dichloroethane	ND	--	1.45	0.1
1,1,1-trichloroethane	ND	--	4.65	0.7
1,1,2-trichloroethane	2.4	0.2	333	14
1,1,2,2-tetrachloroethane	ND	--	ND	--
chloroform	ND	--	20	2
1,2-trans-dichloroethene	ND	--	ND	--
1,2-dichloropropane	ND	--	ND	--
ethyl benzene	ND	--	ND	--
methylene chloride	(a)	--	13.8	1.5
bromoform	ND	--	ND	--
bromo dichloropropane	ND	--	ND	--
toluene	ND	--	8.75	0.1
trichloroethene	ND	--	1.45	0.1

(a) Replicate Analysis contaminated with methylene chloride

These data show the method to be reproducible to ca. 10% (compound dependent) for analysis performed on the same day. Another measure of precision is the analysis of samples collected

in duplicate at the sampling site. One such sample was analyzed and the results shown below:

Name	Avg.	Diff.
benzene	1.4	0
carbontetrachloride	ND	--
chlorobenzene	3.75	0.3
1,2-dichlorcethane	7.6	4.4
1,1,1-trichloroethane	ND	--
1,1,2-trichloroethane	981	29
1,1,2,2-tetrachloroethane	ND	--
chloroform	1.35	2.7
1,2-trans-dichloroethene	ND	--
1,2-dichloropropane	ND	--
ethylbenzene	ND	--
methylenechloride	3.87	6.06
bromoform	ND	--
bromochloromethane	ND	--
toluene	1.25	2.5
trichloroethene	1.65	0.5

11.2 Accuracy - The accuracy of the method may be estimated from the recovery data in section 11.3. Another measure of the overall method accuracy may be obtained from evaluation of the measured concentrations for the second internal standard (bromochloromethane). Since this standard is added to every sample at a constant concentration, it provides a measure of the accuracy of the results in each sample. Overall, for 90

determinations, the measured concentration was 199, ± 51 ppb (199 ± 26%) for bromochloromethane at 200 ppb added concentration.

11.3 Recovery - The accuracy of the method may be estimated based on the recoveries obtained from spiking real samples with known amounts of pollutants. For 5 samples spiked below 50 ppb, the average recoveries and standard deviations are shown below:

Name	%Recovery
benzene	136 ± 37
carbontetrachloride	110 ± 20
chlorobenzene	124 ± 32
1,2-dichloroethane	102 ± 15
1,1,1-trichloroethane	115 ± 19
1,1,2-trichloroethane	93 ± 28
1,1,2,2-tetrachloroethane	112 ± 26
chloroform	113 ± 24
1,2-trans-dichloroethene	110 ± 26
1,2-dichloropropane	104 ± 9.5
ethylbenzene	103 ± 14
methylene chloride	96 ± 33
bromoform	91 ± 22
bromodichloromethane	113 ± 24
toluene	142 ± 31
trichloroethene	106 ± 19

One sample spiked at 200 ppb yielded the following recoveries:

Name	%Recovery
benzene[a]	29
carbontetrachloride	76
chlorobenzene	107
1,2-dichloroethane[a]	75
1,1,1-trichloroethane	78
1,1,2-trichloroethane[a]	42
1,1,2,2-tetrachloroethane	96
chloroform[a]	11
1,2-trans-dichloroethene	79
1,2-dichloropropane	10
ethyl benzene	144
methylene chloride	24
bromoform	80
bromodichloromethane	91
toluene	83
trichloroethene	86

Compounds noted "a" were present in the sample at high concentrations and the addition of 200 ppb exceeded the linear response range.

11.4 Detection Limit - When using automatic data processing procedures, the detection limit is difficult to define. Since the first step in data processing is identification of the spectrum, the detection limit has been defined here as: The minimum amount producing an identifiable mass spectrum. Once the compound is identified, the amount present is measured.

A reagent water blank was spiked at 1 ppb, analyzed and the data automatically processed. The results are listed below:

Name	%Recovery
benzene	139
carbontetrachloride	97
chlorobenzene	148
1,2-dichloroethane	129
1,1,1-trichloroethane	133
1,1,2-trichloroethane	120
1,1,2,2-tetrachloroethane	106
trichloroethane	119
1,2-trans-dichloroethene	132
1,2-dichloropropane	94
ethyl benzene	105
methylenechloride	176
bromoform	75
bromodichloromethane	171
toluene	114
trichloroethane	111

These data show detection of all the compounds spiked at 1 ppb. During the reduction of sample data, many compounds can be identified at concentrations as low as 0.2 ppb. In these cases, the concentrations are reported as "MS" indicating a mass spectral identification but the concentration is below the verified limit of 1 ppb. Compounds not detected are reported as "ND".

Methylene chloride generally shows large variability in

quantitative results near the detection limit. This is due to 2 factors, first is its volatility and second is the potential contamination of samples from the laboratory air. Therefore the detection limit is defined as 3 times the standard deviation of blank determinations (16 ppb). The mean background (2.9 ppb) is subtracted from each value followed by application of the detection limit.

Toluene elutes coincident with the internal standard 1,4-dichlorobutane. The carbon isotope peak at m/e 91 therefore yields a constant toluene background (2.8 ± 1.2 ppb). The detection limit is then defined as 3 standard deviations of the background (3.6 ppb). Due to the consistancy of the background, 2.8 is subtracted from each value measured before applying the 3.6 ppb detection limit.

12.0 References

(1) Memo from James Eichelberger and William Budde to EPA GC/MS users titled "Perfluorobromobenzene Reference Compound for use with Typical Purge and Trap Columns that do not Transmit DFTPP Readily," March 10, 1978.

(2) National Bureau of Standards, EPA-NIH-MSDC Mass Spectral Library.

(3) "Samples and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants," U.S. EPA, Environmental Monitoring and Support Laboratory - Cincinnati, Ohio, March, 1977 revised April, 1977.

(4) "The Determination of Volatile Organic Compounds at the ug/l Level in Water by Gas Chromatography." Thomas A. Bellar and James J. Lichtenberg, Jour. Am. Water. Works Assoc., 66 (12), 739, (1974).

Compound	Mean R_f [c]	% RSD
TRICHLOROFLUOROMETHANE	0.180	35
1,1-DICHLOROETHYLENE	1.20	24
BROMOCHLOROMETHANE [a]	0.783	14
1,1-DICHLOROETHANE	1.03	15
TRANS-1,2-DICHLOROETHYLENE	0.762	16
CHLOROFORM	0.957	16
1,2-DICHLOROETHANE	0.734	15
1,1,1-TRICHLOROETHANE	0.544	21
CARBON TETRACHLORIDE	0.593	18
BROMODICHLOROMETHANE	0.992	3.5
1,2-DICHLOROPROPANE	0.735	13
TRANS-1,3-DICHLOROPROPENE	0.314	15
TRICHLOROETHYLENE	0.559	15
DIBROMOCHLOROMETHANE	0.464	36
CIS-1,3-DICHLOROPROPENE	0.240	10
1,1,2-TRICHLOROETHANE	0.429	6.3
BENZENE	1.40	13
BROMOFORM	0.200	11
TETRACHLOROETHYLENE	0.441	11
1,4-DICHLOROBUTANE [a,b]	1.0	NA
1,1,2,2-TETRACHLOROETHANE	0.725	5.7
TOLUENE	1.38	16
CHLOROBENZENE	0.866	7.5

[a] Internal standards always at 200 ppb.

[b] Used as relative response of 1.0.

[c] Mean of 4 determinations at 20, 50, 100, and 200 ppb.

Quality Control Data
Volatile Organics Analysis (Purgeables)

One sample, Station 6, was analyzed in replicate and also spiked. In the replicate analyses, four components were detected each time (benzene, chlorobenzene, tetrachloroethene and toluene) with an average deviation of 8%, with a range of relative percent deviation from 2 to 14 percent. Chloroform was detected at 8 ug/l in one analysis and was ND in the second.

In the spiked sample, all 25 components were detected, with an average 81% recovery.

Table 1
Purgeables-QC Results

Spiked Sample	Component	Conc. ug/l	Recovered	Percent
	Benzene	64	61	95
	Carbon tetrachloride	100	73	73
	Chlorobenzene	192	180	94
	1,2-Dichloroethane	40	28	70
	1,1,1-Trichloroethane	40	15	38
	1,1-Dichloroethane	100	69	69
	1,1,2-Trichloroethane	100	94	94
	1,1,2,2-Tetrachloroethane	40	50	125
	Chloroethane	300	310	103
	Chloroform	100	64	64
	1,2-Dichloropropane	100	69	69
	1,1-Dichloroethene	100	110	110
	cis-1,3-Dichloropropene	40	26	65
	Ethylbenzene	40	2	5
	Methylene chloride	100	64	64
	Methyl chloride	300	64	21
	Methyl bromide	300	220	73
	Bromoform	40	44	110
	Bromodichloroethane	40	30	75
	Trichlorofluoromethane	40	100	250
	Dibromochloromethane	100	97	97
	Tetrachloroethene	100	84	84
	Toluene	40	28	70
	Trichloroethane	100	75	75
	Vinyl chloride	300	140	47
	Average	81%		

Table 1 (Cont.)

Replicate	Component	Analysis 1	Analysis 2
	Benzene	23 ug/l	24
	Chlorobenzene	100	84
	Chloroform	8	ND
	Tetrachloroethene	4	3
	Toluene	13	15

BASE/NEUTRAL PRIORITY POLLUTANT ANALYSIS BY GLASS CAPILLARY GAS CHROMATOGRAPHY/MASS SPECTROMETRY NATIONAL ENFORCEMENT INVESTIGATIONS CENTER-JUNE 1979

1.0 Scope and Application

1.1 This method is applicable to the extractable base/neutral priority pollutant organics. The majority of the base/neutrals can be analyzed with this technique.

1.2 The limit of detection for this method is from 5 to 20 ug/l (ppb) depending on the type of compound.

1.3 The nominal concentration range is from 5 to 100 ug/l (ppb). Higher concentrations may be handled by dilution prior to analysis.

2.0 Summary of Method

2.1 Concentrated solvent extracts of aqueous, sediment, or solid samples are injected into a glass capillary column gas chromatograph directly coupled to a quadrupole electron-impact mass spectrometer via a small diameter heated glass lined stainless steel tube. A splitless injection technique is used. The resultant mass spectra are collected and stored by a computer controlled data system. The identifications are made by automatic computer matching of the sample spectra and relative retention times with those of standard spectra from a special stored library of the base/neutral priority pollutants. Quantitative results are obtained for each compound using a response factor for each standard relative to an internal standard.

3.0 Interferences

3.1 Concentrated solvent extracts can contribute interferences. Common solvent interferences are: diacetone alcohol (4-methyl-4-hydroxy-2-pentanone) from acetone and cyclohexene from dichloromethane.

3.2 Common interferences from sodium sulfate are the phthalates.

4.0 Comments

4.1 Several of the base/neutrals are difficult to identify by this method. Two-Chloroethylvinyl ether, bis(chloromethyl)ether, and 3,3-dichlorobenzidine have never been identified using this column in our laboratories.

4.2 Isophorone and hexachlorocyclopentadiene chromatograph fairly well, but cannot be identified by the computer search on most occasions at 40 ug/l (ppb) and obviously higher concentrations are required.

4.3 Butylbenzylphthalate is often misidentified as dibutylphthalate and phenanthrene and anthracene are always identified as anthracene. Daily updating of the quantitation parameters and manual data auditing readily solves this problem.

4.4 Several of the PAH's are more difficult to identify at lower levels, but with higher concentrations above 50 ug/l, they should be readily identified.

5.0 Apparatus

5.1 Finnigan Model 9500 gas chromatograph equipped with a glass capillary column.

5.1.1 Grob type glass lined injector for splitless injection.

5.1.2 Capillary glass column, 25 meters X 0.25 mm ID, OV-101.

5.2 Finnigan Model 3200 electron impact mass spectrometer.

5.2.1 Glass lined stainless steel tubing direct coupling to GC.

5.3 Finnigan INCOS data system (1).

5.3.1 MSDS software 3.1, 7/1/78, Revision B

6.0 Procedure

6.1 Gas Chromatography

6.1.1 Inject 1 to 2 ul of sample into the gas chromatograph with purge valve turned off for 1 min. after injection. At precisely 1 min. open purge valve (Purge flow 50 ml/min)

6.1.2 The initial column oven temperature is equilibrated at 60°C and held for 1 min. after injection, then a temperature program is initiated at 4°C/min to 22°C. The final temperature is held until 70 minutes have elapsed. The column flow is adjusted to give a nominal flow of 1.5 ml/min at 100°C. The injector temperature is 250°C.

6.2 Mass Spectrometry.

6.2.1 The following MS instrumental parameters are used:

Electron multiplier voltage	-	1800 volts
Lens voltage	-	50 volts
Collecter voltage	-	35 volts
Extractor energy voltage	-	6 volts
Ion Energy voltage	-	10 volts
Electron energy voltage	-	70 volts
Emission current	-	0.5 ma

6.2.2 The following data acquisition parameters are used:

Scan time	-	2 seconds
Mass range	-	35 - 350 AMU
Sensitivity	-	10^{-7} amp.

6.2.3 The data acquisition is initiated immediately upon sample injection in a suspended mode. At 4 minutes the ionizer is turned on and at 5 minutes the data collection is begun. The data acquisition contunues for a total of 70 minutes from injection then stops. This data handling is automatically controlled by an in house procedure. (See Appendix I)

6.2.4 The quantitation and presentation of the scan number of an added d_{10} anthracene internal standard are hardcopied for monitoring the integrity of the GC/MS system. Again this is done utilizing an in house procedure. (See Appendix II)

6.2.5 The data is then processed using an automatic conputerized search and quantitation procedure. The quantitation is obtained based on the response factors relative to an internal standard. This procedure is also an in house procedure

(See Appendix III), utilizing standard operating methods. (1)

7.0 Precision and Accuracy

7.1 Data not available

8.0 Calculations

8.1 The quantitation is done using the nanograms/microliter obtained from the response factors derived from analysis of a standard mix of priority pollutants with an internal standard added. Response factors are calculated based upon the integrated areas of selected ions for each component in a standard mix. Appendix IV lists the ions selected to date. Response factors are calculated as follows:

Resp. Fact = Areas * REF. AMNT/(REF. AREA * AMNT)
Area = area of component response
Ref. Amnt = amount of internal reference standard
Ref. Area = area of internal reference standard response
Amnt. = amount of component analyzed in standard.

To determine the concentration of an identified component in a sample, rearrange the equation and solve for the amount. Usually, concentrations are in ug/ul.

Amnt = Area * REF. AMNT/(REF. AREA * Resp. Fact)

8.2 The concentration of the sample component is calculated in ug/l as follows:

$$\text{ug/l} = \frac{\text{ng}}{\text{ul}} \times \frac{\text{100\% conc. vol in ml}}{\text{extract vol in liters}} \times \frac{\text{100\%}}{\text{\% soln.}}$$

9.0 Quality Control

9.1 The mass spectrometer is tuned and calibrated daily using a perfluorotributylamine (FC-43) calibration compound.

9.2 A standard mix containing eight compounds is analyzed on the GC/MS. These compounds give a representative cross section of types of compounds. The compounds are:

1 1,2-Dichlorobenzene
2 N-Methylaniline
3 2,6-Dimethylphenol
4 Napthalene
5 p-Nitrotoluene
6 1,2,4,5-Tetrachlorobenzene
7 Biphenyl
8 Tetradecane

10.0 References

(1) "INCOS Data System, MSDS Operators Manual, Revision 3," Finnigan Instruments, March 1978.

Attachment I

```
TRACE OF PROCEDURE BNPPAO
  * ERASE;C            PRIORITY POLLUTANT BASE-NEUTRALS DATA ACQUISITION SEUP]
  * [TIME (SEC)  PROMPT              ACTION                              ]
  * [  0         NONE                TURN DIVERTER OFF                   ]
  * [ 30         BEEP;BEEP;BEEP;     INJECT SAMPLE                       ]
  * [ 90         BEEP;BEEP           TURN DIVERTER ON; START GC PROGRAM]
  * [250         BEEP                TURN ON IONIZER                     ]
  * [300         NONE                ACQUISTION STARTED                  ]
  * ;SCPP;[TO START RUN PRESS CARRIAGE PETURN.]
  * [AFTER PRESSING CARRIAGE PETURN YOU WILL HAVE]
  * [30 SECONDS BEFORE YOU INJECT]PAUSE;
  * ACOU (I;MI00;T2;S;G:55:00;E)::ERASE;
  * [INJECT SAMPLE IMMEDIATELY AFTER THIRD BEEP]
  * WAIT=15;BEEP;BEEP;BEEP;ERASE;
  * [SAMPLE SHOULD HAVE BEEN INJECTED AT THIS TIME]
  * [WHEN THE TERMINAL BEEPS AGAIN.]
  * [TURN DIVERTER ON AND START GC PROGRAM]
  * WAIT =45;BEEP;BEEP;ERASE;
  * WAIT=47
  * [DIVERTER SHOULD BE ON AND THE GC PROGRAMMING AT THIS TIME]
  * [TURN ON IONIZER AT THE SOUND OF THE FANFARE];
  * WAIT=120;
  * SONG(OFF);EPASE;
  * WAIT=125;
  * [IONIZER SHOULD BE ON AT THIS TIME];
  * WAIT=150;ACOU (S;E);MAP/C (I;V100000;D150,1650,500;E)
  *
  EPASE
  SCPP
     *  SCAN(MASS RANGE LOW 35;HIGH 350;UP 1.95;DOWN 0.00;HOLD TIME TOP 0.00;BOTTOM 0.05)
     *
     SCAN  (MASS RANGE LOW 35;HIGH 350;UP 1.95;DOWN 0.00;HOLD TIME TOP 0.00;BOTTOM 0.05)
  PAUSE
  ACOU  (I;MI00;T2;S;G:55:00;E)
  EPASE
  WAIT =15
  BEEP
  BEEP
  BEEP
  EPASE
  WAIT =45
  BEEP
  BEEP
  ERASE
  WAIT =47
  WAIT =120
  SONG  (OFF)
  EPASE
  WAIT =125
  WAIT =150
  ACOU  (S;E)
  MAP  (I,V100000;D150,1650,500;E)/C
```

Attachment II

```
TRACE OF PROCEDURE GNDONE
   * PARA(I;H;E);MAP(I;V200000;H1,2000,700;E)
   * ;CHRO (I;R;$PP,121;&PP,121;%PP,121;N1,2;A5,3;G-15,15;H-15,15;E);FEED;BEEP
   *
   PARA  (I;H;E)
   MAP   (I;V200000;H1,2009,700;E)
   CHRO  (I;R;$PP,121;&PP,121;%PP,121;N1,2;A5,3;G-15,15;H-15,15;E)
   FEED
   BEEP
```

Attachment III

```
TRACE OF PROCEDURE PRIPOL
   * ;CPRIORITY POLLUTANT EVALUATION PROGRAM.  SEE PRIPOL.DS FOP EXPLAINATON]
   * ;CWRITTEN APRIL 24,1979 BY O.J.LOGSDON II  US EPA NEIC 303-234-4661]
   * ;CREVISED APRIL 24,1979 BY O.J.LOGSDON II  US EPA NEIC 303-234-4661]
   * ;SETL $1;SETS $2
   * ;EDSL YES (-;1;W;E);EDSL NO (-;W;E)
   * ;SETN PRIPOL
   * ;PRIP00
   * ;FEED
   * ;BEEP;BEEP;BEEP
   *
   SETL $1
   SETS $2
   EDSL YES (-;1;W;E)
   EDSL NO (-;W;E)
   SETN PRIPOL
   PRIP00
      * GETN;PRIP01;LOOP
      *
      GETN
      PRIP01
         * ;SET0 $1;EDOL (-;W;E);SETL #0;SET1 #1
         * ;FILE (K PRIN.99/N;E)
         * ;PARA (I;H;E);CHP0 (I;H1,2000;400;E)
         * ;PRIP06;SETL #0;SET10 '14;PRIP02
         * ;EDLL (B'1;E);PRIN (0P1);FILE (C PRIN.99,M;/N;E)
         * ;FEED
         * ;QUAN (I;H;E)
         * ;FEED;BEEP
         *
         SET0 $1
         EDOL  (-;W;E)
         SETL
         SET1 #1
         FILE  (K PRIN.99/N;E)
         PARA  (I;H;E)
         CHR0  (I;H1,2000;400;E)
         PRIP06
            * SET14 #1;GETL #1
            * ;SEAR/V (I;S,&;V200000;N2.10.500;D-25.25;E)
            * ;PRIP07
            *
            SET14 #1
            GETL #1
            SEAR  (I;S;&;V200000;N2.10.500;D-25.25;E)/V
            PRIP07
               * IF PRIP07 #1.'14
               * ;PPIN (0P2)
               * ;BEEP.BEEP.BEEP;BEEP;BEEP;BEEP;BEEP;BEEP
               * ;RETU PRIP01
               *
               IF PRIP07>1.'14
               PRIN  (0P2)
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               RETU PRIP01
         SETL
         SET10 '14
         PRIP02
            * SET1 '10
            * ;SET14 #0
            * ;GETL
            * ;SEAR/V (I;S, &;V200000;N1.10.10;D-20.20;E)
            * ;FI[illegible] ('4,',14,6,'15,6,'16,6,C,E)
```

```
        * ;PRIP03
        * ;LOOP
        *
        SET1 !10
        SET14
        GETL
        SEAR  (I;S;*;V200030-N1,10,10;D-20,20;E)/V
        PRIN  ('4,2;'14,G,'15,G;'16,6;C;E)/KX
        PRIP03
           * PRIP04
           * ;EDOL (-;N;*;A;E)
           *
           PRIP04
              * IF PRIP04 '16,PRIP04 *500
              * ;SET1 '14
              * ;CHRO (I;R;S;*;N1,2;A)5,3;G-4,4;D-5,5;E)
              * ;PRIP05
              * ;RETU PRIP03
              *
              IF PRIP04'16,PRIP04*500
              SET1 !14
              CHRO  (I;R;S;*;N1,2;A)5,3;G-4,4;D-5,5;E)
              PRIP05
                 * IF PPIP05 '27,PRIP05
                 * ;LIBR (!;C;DS;HS;E)
                 *
                 IF PRIP05'27,PRIP05
                 LIBP  (I;C;DS;HS;E)
              RETU PRIP03
           EDOL  (-;N;*,A,E)
        LOOP
     EDLL  (B'I;E)
     PRIN  (@P1)
     FILE  (C PRIN.99,M:/N;E)
     FEED
     QUAN  (I;H;E)
     FEED
     BEEP
  LOOP
FEED
BEEP
BEEP
BEEP
```

Attachment IV

	Name	Quantitation Ion
	D10-ANTHRACENE (INTERNAL STANDARD)	188
01	ACENAPHTHENE	154
08	1,2,4-TRICHLOROBENZENE	74
09	HEXACHLOROBENZENE	284
12	HEXACHLOROETHANE	117
18	BIS(2-CHLOROETHYL)ETHER	93
20	2-CHLORONAPHTHALENE	162
25	1,2-DICHLOROBENZENE	146
26	1,3-DICHLOROBENZENE	146
27	1,4-DICHLOROBENZENE	146
28	3,3-DICHLOROBENEZIDINE	252
35	2,4-DINITROTOLUENE	165
36	2,6-DINITROTOLUENE	165
37	1,2-DIPPHENYLHYDRAZINE (MEAS. AS ASOB)	77
39	FLUORANTHENE	202
40	4-CHLOROPHENYL PHENYL ETHER	204
41	4-BROMOPHENYL PHENYL ETHER	248
42	BIS(2-CHLOROISOPROPYL)ETHER	45
43	BIS(2-CHLOROETHOXY)METHANE	93
52	HEXACHLOROBUTADIENE	225
53	HEXACHLOROCYCLOPENTADIENE	237
54	ISOPHORONE	82
55	NAPHTHALENE	128
56	NITROBENZENE	77
62	N-NITROSODIPHENYLAMINE (MEAS AS DIPH)	109
63	N-NITROSODIPROPYLAMINE	130
66	DI-(2-ETHYLHEXYL)PHTHALATE	149
67	BUTYL BENZYL PHTHALATE	149
68	DI-N-BUTYLPHTHALATE	149
69	DI-OCTYLPHTHALATE	149
70	DIETHYLPHTHALATE	149
71	DIMETHYLPHTHALATE	163
72	BENZO(A)ANTHRACENE	228
74	3,4-BENZOFLUORANTHENE	252
75	BENZO/K/FLUORANTHENE	252
76	CHRYSENE	228
77	ACENAPHTHYLENE	152
78	ANTHRACENE	178
80	FLUORENE	166
81	PHENANTHRENE	178
84	PYRENE	202

Quality Control Data
Base-neutral Extractables

Two samples were spiked with nine priority pollutants at 133 ug/l, extracted and analyzed. The same samples were analyzed in duplicate as well. The average recovery was 78 percent. The low recovery reflects the problems of water-solvent emulsions which required centrifugation to separate.

Duplicates data were minimal, with only five compounds showing results above the detection limits in the original samples.

Table 1
Base-neutral Extractables-QC Results

Spiked Samples

Component	Sample 06 Conc. ug/l	Sample 06 Recovered	Sample 06 Percent	Sample 07 Conc.	Sample 07 Recovery	Sample 07 Percent
p-Dichlorobenzene	840	180	21%	133	140	105%
Isophorone	133	210	160	133	100	75
1,2,4-Trichlorobenzene	173	160	92	146	84	58
2-Chloronaphthalene	133	99	74	133	97	73
Acenaphthalene	133	98	73	133	102	77
Dinitroluene	133	140	105	133	103	77
Anthracene	133	79	59	133	100	75
Di-n-butyl phthalate	133	67	50	133	95	72
Pyrene	133	125	94	133	83	62
Averages		81%			75%	

Duplicate Samples

Sample 06

Component	Analysis 1	Analysis 2
p-Dichlorobenzene	710 ug/l	1000
1,2,4-Trichlorobenzene	40	160
o-Dichlorobenzene	160	590

Sample 07

1,2,4-Trichlorobenzene	13	2
Di-n-butyl phthalate	38	5

ADJUSTED pH EXTRACTION TECHNIQUE FOR ORGANICS ANALYSIS NATIONAL ENFORCEMENT INVESTIGATIONS CENTER-JANUARY 1979

1.0 Scope and Application

1.1 This procedure is applicable to the analysis of water and wastewater samples for a broad spectrum of organic pollutants. The primary use is to extract Priority Pollutants (1) for analysis by GC-MS.

2.0 Summary of Method

2.1 Water and wastewater samples are extracted with CH_2Cl_2 (dichloromethane) at a basic pH to extract neutrals and bases and then at an acidic pH to extract phenols. The extracts are dried and filtered by passing over anhydrous Na_2SO_4 and concentrated to 5-10 ml in a Kuderna-Danish (KD) apparatus, then finally concentrated to 1.0 ml in a graduated centrifuge tube under a gentle stream of purified air.

2.2 The concentrated extracts are sealed in 1 ml serum vials and stored in a refrigerator until analysis.

3.0 Sample Handling and Preservation

3.1 Prior to extraction, samples are refrigerated and extracted as soon as possible, generally within 48 hours. Samples may be held 5 days or more if necessary.

4.0 Interferences and Detection Limits

4.1 The detection limits must be 10 ug/l or less. (2) Concentration of a sample containing 10 ug/l of a component to 1.0 ml yields an extract concentration of 10 ng/ul.

4.2 In some samples, industrial wastes, in particular, the concentration of some components may be so great that dilution is necessary for analysis on glass capillary GC.

In most cases, however, the extreme sensitivity of glass capillary GC will allow dilution by a factor of 10 without lowering the detection limit below 10 ug/l.

5.0 Apparatus

5.1 Separatory funnels: 2 l glass with teflon or glass stoppers and stopcocks. No stopcock grease is used.

5.2 Drying column: All glass 3 cm diameter by 50 cm with attached 250 ml reservoir.

5.3 Concentrator: 250 ml Kuderna-Danish (KD) evaporative concentrator equipped with a 5 or 10 ml receiver amphule and a 3 ball Snyder column.

5.4 Centrifuge tubes: 12 ml glass tubes graduated in 0.1 ml marks.

5.5 Graduate: 1 l glass graduated cylinder.

5.6 Vials: 1 ml with teflon-coated septum sealing caps.

6.0 Reagents

6.1 Extraction solvent: Pesticide analysis grade CH_2Cl_2 (dichloromethane). Burdick and Jackson, distilled in glass, or equivalent.

6.2 Dilution solvent: Pesticide analysis grade acetone, Burdick and Jackson, distilled in glass or equivalent.

6.3 Drying agent: Analytical reagent grade granular anhydrous Na_2SO_4 (sodium sulfate), rinsed with CH_2Cl_2 immediately before use.

6.4 Glass wool that has been extracted with CH_2Cl_2.

6.5 6N NaOH for pH adjustment.

6.6 6N HCl for pH adjustment.

6.7 pH paper for pH measurement.

6.8 Purified air: Compressed air filtered through activiated charcoal.

7.0 Procedure

7.1 Thoroughly mix the sample and measure 1 liter of sample with a graduate. Transfer the sample to a 2 l separatory funnel.

7.2 Measure and record the initial sample pH.

7.3 Base-Neutral Fraction

7.3.1 Adjust the pH with 6N NaOH to 11 or greater and record the value.

7.3.2 Serially extract with 3 successive portions of 100, 50 and 50 ml of CH_2Cl_2. Shake each extract at least 2 minutes.

7.3.3 If emulsions form, use a wire or stirring rod to break it up, pass the emulsion through glass wool, or centrifuge if necessary.

7.3.4 Measure the volume of solvent recovered (graduations on a beaker are adequate) and record. More than 85 percent recovery constitutes a satisfactory extraction.

7.3.5 Place a glass wool plug in a drying column and add ca. 10 cm of Na_2SO_4 with at least 50 ml of CH_2Cl_2. Pour the combined extract through the column. Follow with 100 ml of acetone. Collect the CH_2Cl_2 and acetone and transfer to a KD assembly.

7.3.6 Concentrate on a hot water bath at 80-90°C until the extract almost stops boiling. Quantitatively transfer the receiving tube contents to a graduated centrifuge tube. Concentrate the extract to 1.0 ml by blowing a gentle stream of purified air over the surface of the solvent. Transfer the concentrate to a 1 ml vial and cap. Mark the liquid level and label with the sample number, fraction identifier (B for base neutrals and A for acids), your initials and the date.

7.4 Acids Fraction

7.4.1 Adjust the pH of the aqueous layer with 6N HCl to 2 or less and record the result.

7.4.2 Proceed with the extraction as in 7.3.2.

7.5 Analysis Preparation

7.5.1 If the sample is being analyzed by capillary GC, typically dilute an aliquot 1:5 (20% solution) in acetone.

7.5.2 If CH_2Cl_2 solvent is a problem during the analysis, exchange the extract into acetone. Add 2 ml of acetone to the extract in a centrifuge tube and concentrate to 1.0 ml with a gentle stream of purified air.

8.0 Quality Control

8.1 With each batch of samples, the following quality control checks must be performed. Two of each type check is to be done for the first 20 samples with one of each check done on each additional 20 samples.

8.1.1 Reagent Blank: Extract 1 l of organics free water using the same procedure as for samples. These should be done randomly with samples to check for contamination of various reagents, etc.

8.1.2 Duplicate Extraction: Select a sample, split it and extract both aliquots. Carry each extract through the entire analytical scheme. Determine the relative percent differences for each component.

8.1.3 Spike: If a number of pollutants are suspected, prepare a spike by splitting the sample and adding known amounts of the pollutants to one aliquot and extract both aliquots. Carry each extract through the entire analytical scheme and determine the percent recoveries for each compound added. If no specific pollutants are suspected, spike with the standard mix described in Reference 3.

8.2 If reference samples (external audit samples) are available that are applicable to the project, analyze one sample during the project.

9.0 Calculations

9.1 Solvent recovery:

$$\% \text{ Recovery} = \frac{\text{volume recovered (ml)} * 100}{\text{volume added (ml)}}$$

9.2 Pollutant recovery (spiked samples):

$$\% \text{ recovery} = \frac{(\text{concentration measured} - \text{initial concentration}) * 100}{\text{concentration added}}$$

9.3 Relative percent difference (RPD):

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} * 100$$

Where D_1 = first sample value
D_2 = second sample value (duplicate)

Table I. Recovery Data Obtained from Analysis of Spiked Tap Water.

	Mean[a]	Std. Dev.	% Std.[a]
Phenol	56.7[b]	25.2[b]	44.4[b]
Hexachloroethane-Nitrobenzene	43.0	2.4	5.7
Isophorone	45	9.9	21.9
1,2,4-Trichlorobenzene	58.8	4.6	7.8
Naphthalene	55.0	13.3	24
Hexachlorobutadiene	46.5	3.4	7.3
Hexachlorocyclopentadiene	31.7[b]	1.2[b]	3.6[b]
2-Chloronaphthalene	63.8	4.8	7.5
Acenaphthalene	76	3.2	4.2
Dimethyl Phthalate	55	11.2	20.4
Acenaphthene	76	4.7	6.2
2.4-Dinitrotoluene	61.8	5.7	9.2
Fluorene	77.8	1.7	2.2
Diethyl Phthalate	76.2	1.2	1.6
n-Nitrosodiphenylamine (Diphenylamine)	75.8	3.3	4.4
4-Bromodiphenyl ether	76.6	4.6	6.0
Hexachlorobenzene	74.8	6.2	8.3
Phenanthrene	75.5	4.0	5.4
Anthracene	67.3	4.0	6.0
Di-N-Butyl Phthalate	77.3	3.3	4.3
Fluoranthene	75.0	9.0	12.0
Pyrene	70.5	2.1	3.0
Butylbenzyl Phthalate	82.0	1.4	1.7
Average	65.1		9.4

[a] Based on 4 samples

[b] Based on 3 samples

10.0 Precision and Accuracy

10.1 Precision and accuracy vary with the pollutants measured. Table I shows data obtained from the analysis of 4 tap water samples spiked with the listed pollutants at 100 ug/l.

11.0 References

(1) NRDC v. Train, 8, E.R.C. 2120 (1976).

(2) "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S. EPA, EMSL-Cincinnati, March, 1977, revised April, 1977.

(3) Organics Analytical Quality Control Manual, EPA-NEIC, February, 1979.

Quality Control Data
Base-neutral Extractables

Two samples were spiked with nine priority pollutants at 133 ug/l, extracted and analyzed. The same samples were analyzed in duplicate as well. The average recovery was 78 percent. The low recovery reflects the problems of water-solvent emulsions which required centrifugation to separate.

Duplicates data were minimal, with only five compounds showing results above the detection limits in the original samples.

Table 1
Base-neutral Extractables-QC Results

Spiked Samples

Component	Sample 06 Conc. ug/l	Sample 06 Recovered	Sample 06 Percent	Sample 07 Conc.	Sample 07 Recovery	Sample 07 Percent
p-Dichlorobenzene	840	180	21%	133	140	105%
Isophorone	133	210	160	133	100	75
1,2,4-Trichlorobenzene	173	160	92	146	84	58
2-Chloronaphthalene	133	99	74	133	97	73
Acenaphthalene	133	98	73	133	102	77
Dinitroluene	133	140	105	133	103	77
Anthracene	133	79	59	133	100	75
Di-n-butyl phthalate	133	67	50	133	95	72
Pyrene	133	125	94	133	83	62
Averages		81%			75%	

Duplicate Samples

Sample 06

Component	Analysis 1	Analysis 2
p-Dichlorobenzene	710 ug/l	1000
1,2,4-Trichlorobenzene	40	160
o-Dichlorobenzene	160	590

Sample 07

1,2,4-Trichlorobenzene	13	2
Di-n-butyl phthalate	38	5

Quality Control Data
Acid Extractables

The same two samples spiked for base-neutrals were also spiked and analyzed in duplicate for phenolics. The spike concentrations were between 21 and 79 ug/l for the eleven components. The average recovery was 73%.

Five components were detected in sample 06, and one component in sample 07. The average deviation was 25%.

Table 2
Acid Extractables-QC Results

Spiked Samples

Component	Sample 06 Conc. ug/l	Recovered	Percent	Sample 07 Conc.	Recovered	Percent
2,4,6-Trichlorophenol	54	31	57%	54	57	105
4-Chloro-3-methylphenol	71	19	27	56	51	91
2-Chlorophenol	69	29	47	62	57	92
2,4-Dichlorophenol	115	5	4	58	50	86
2,4-Dimethylphenol	43	13	30	40	26	65
2-Nitrophenol	70	26	37	70	51	73
4-Nitrophenol	21	15	72	21	28	133
2,4-Dinitrophenol	55	43	78	55	86	156
4,6-Dinitro-o-cresol	48	30	63	48	79	164
Pentachlorophenol	63	17	27	46	51	111
Phenol	71	22	31	79	46	58
Averages		43%			103%	

Duplicate Samples

Sample 06

Component	Analysis 1	Analysis 2
4-Chloro-3-methylphenol	14 ug/l	16
2-Chlorophenol	6	7
2,4-Dichlorophenol	49	65
2,4-Dimethylphenol	2	3
Pentachlorophenol	28	5

Sample 07

Phenol	10	5

COMPUTER ASSISTED EVALUATION OF ORGANIC PRIORITY POLLUTANT GC/MS DATA NATIONAL ENFORCEMENT INVESTIGATIONS CENTER-JUNE 1979

1.0 Introduction

1.1 This procedure is applicable to GC/MS data collected under constant analytical conditions for the organic priority pollutant defined in "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants". (1) By developing appropriate libraries, data for any groups of selected organic pollutants can be evaluated.

2.0 Summary of Method

2.1 GC/MS data files are processed by location of an internal standard that is used for response and retention time reference. Components of interest are then located by reverse searching from library spectra. If a compound is located and the match is sufficient, it is quantitated and its spectrum optionally printed. The concentrations are then calculated from each component found using a relative response quantitation technique. Printed reports of both quantitative and qualitative results are available.

3.0 Definitions and Comments

3.1 Unlike the 3 ion and retention time compound identification technique described for priority pollutant analysis in reference 1, this procedure allows the user to audit each identification where the spectra are printed. Thus, each identification is unambiquous and marginal data may be eliminated.

4.0 Interferences

4.1 In some cases, a spectrum may match the library reference sufficently to be passed. During quantitation, however, the ion of interest may be too weak to locate and no entry will be made in the quantitation list. In such a case, no entry at all (e.g. no "not found" entry) will appear in the quantitation report. The name and match results will, however, appear in the qualitative data report.

4.2 Occasionally, multiple peaks will be detected during quantitation due to background interferences and multiple entries will be made in the quantitation list. Generally, the entry having the same label as the correct spectrum is used for quantitation and the others are disregarded. In some instances, however, the correct selection is not obvious and manual evaluation of the quantitation results must be done.

4.3 When isomers of a chemical elute too close to one another, the system may misassign them. Manual evaluation then is usually required to properly identify the isomers.

5.0 Apparatus

5.1 Finnigan INCOS data system software, Revision 3.1 or later. To initially set up this procedure, the user must understand and be proficient in the use of MSDS. (2)

6.0 Procedure

6.1 Procedure Set Up

6.1.1 Load the procedures listed in Appendix I into the system disc or create the procedures from the trace of PRIPOL in Appendix II.

6.2 Library Set Up

6.2.1 Build a user library containing spectra of interest. Each entry should have relative retention time (RRT) data, response factor (RF) data and a reference peak for RRT and RF references. Appendix III is a typical library. The library should include the internal standard (S).

6.2.2 Create library lists on the system disc with entries that reference the desired library entries. The first entry of the library list must be an internal standard. Appendix IV shows three library lists used for selected priority pollutants

6.2.3.1 If the RRT and RF data initially entered in the library was about correct, evaluate data from a standard mix. Edit the resulting quantitation list and manually add any entries not identified.

6.2.3.2 If no RRT and RF data were initially available, manually locate and quantitate the components of interest including the internal standard. Write each quantitation result into a quanlist and edit the list to include the peak references.

6.2.4 Using "QUAN", update the response factors (R), retention times (T) and relative retention times (S). Using the "QUAN" commands F3 and H, print out the updated list and response factors, retention times and relative retention times.

6.3 Routine Use

6.3.1 Analyze samples, standards and quality control samples using the same instrument conditions used to set up the libraries.

6.3.2 Using the namelist editor, create a name list "PRIPOL" containing the names of the data files to be processed.

6.3.3 Execute the procedure as follows:

PRIPOL library list, yes (no)

Where: Library list is the appropriate user library list name.

Yes (no) selects printout of the spectra at a peak that was identified by the procedure.

6.3.4 Appendix V is an example of PRIPOL output using a library list containing one internal standard and one component. The "yes" option was selected.

7.0 Quality Control

7.1 Each identification can be manually audited if the "yes" option was selected. Inaccurate qualitative results may then be checked and manually corrected.

7.2 Quantitation data accuracy is monitored by use of standard quality control techniques such as daily standardization, replicate analysis and spikes. (3) Daily calibration of the method can be accommodated by analyzing the standard data first, updating the relative response factors, obtaining hard copy of the new factors and then analyzing sample data.

8.0 Precision and Accuracy

8.1 The overall precision and accuracy is limited to the quality of the raw data being processed.

9.0 References

(1) "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1977, Revised April 1977.

(2) "INCOS Data System - MSDS Operators Manual - Revision 3", Finnigan Instruments, March 1978.

(3) "Quality Assurance Program for the Analyses of Chemical Constituents in Environmental Samples", U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1978.

(4) "Organic Pollutant Analysis Quality Assurance and Document Control Procedures", U.S. EPA, NEIC, Denver, Colorado, Revision 1, April 1979.

Attachment I

PROCEDURES NEEDED TO RUN PRIPOL

PRIPOL
PRIP00
PRIP01
PRIP02
PRIP03
PRIP04
PRIP05
PRIP06
PRIP07

METHODS NEEDED TO RUN PRIPOL

PRINP1
PRINP2

Attachment IIa

```
TRACE OF PROCEDURE PRIPOL
   * ;[PRIORITY POLLUTANT EVALUATION PROGRAM.          ]
   * ;[WRITTEN APRIL 24,1979 BY D.J.LOGSDON II  US EPA NEIC 303-234-4661]
   * ;[REVISED JULY 12,1979 BY D.J.LOGSDON II  US EPA NEIC 303-234-4661]
   * ;SETL $1;SETS $2;SETQ TEMP
   * ;EDSL YES (-;1;W;E);EDSL NO (-;W;E)
   * ;SETN PRIPOL
   * ;PRIP00
   * ;FEED
   * ;BEEP;BEEP;BEEP
   *
   SETL $1
   SETS $2
   SETQ TEMP
   EDSL YES (-;1;W;E)
   EDSL NO (-;W,E)
   SETN PRIPOL
   PRIP00
      * GETN;PRIP01;LOOP
      *
      GETN
      PRIP01
         * ;EDOL $1 (-;W;E);SETL #0;SET1 #1
         * ;FILE (K PRIN.99/N;E)
         * ;PARA (I;H;E);CHRO (I;H1,2000,400;E)
         * ;PRIP06;SETL #0;SET!O !14;PRIP02
         * ;EDLL (B!1;E);PRIN (@P1);FILE (C PRIN.99,M:/N;E)
         * ;FEED
         * ;QUAN $1 (I;F2;H;E)
         * ;FEED,BEEP
         *
         EDOL $1 (-;W;E)
         SETL
         SET1 #1
         FILE  (K PRIN.99/N;E)
         PARA  (I;H;E)
         CHRO  (I;H1,2000,400;E)
         PRIP06
            * SET14 #1;GETL #1
            * ;SEAR/V (I;$;&;V200000;N2,10,500;D-25,25;E)
            * ;PRIP07
            *
            SET14 #1
            GETL #1
            SEAR  (I;$;&;V200000,N2,10,500;D-25,25;E)/V
            PRIP07
               * IF PRIP07 #1,!14
               * ;PRIN (@P2)
               * ;BELP,BEEP;BEEP;BEEP;BEEP,BEEP;BEEP
               * ;RETU PRIP01
               *
               IF PRIP07#1,!14
               PRIN  (@P2)
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               BEEP
               RETU PRIP01
            SETL
            SET!O !14
            PRIP02
               * SET1 !0
               * ;SET11 #0
               * ;GETL
               * ;SEAR/V (I;$;&;V200000,N1,10,10;D-10,10;E)
               * ;PRIN/V (I4,',',I3.3  15,6;!16.6,C E)
```

Attachment IIb

```
            * ;PRIP03
            * ;LOOP
            *
            SET1 !10
            SET14
            GETL
            SEAR  (I;S;%;V200000;N1,10,10;D-10,10;E)/V
            PRIN  ('4,2;!14,6;!15,6;!16,6;C;E)/KX
            PRIP03
               * PRIP04
               * ;EDOL S1 (-;N;*;A;E)
               *
               PRIP04
                  * IF PRIP04 !16,PRIP04 *500
                  * ;SET1 !14
                  * ;EDOL (-;W,E)
                  * ;CHRO (I;R,S;*;N1,2;A>5,3;G-4,4;D-5,5;E)
                  * ;EDOL TEMP,S1 (U*20,100;A,E)
                  * ;PRIP05
                  * ;RETU PPIP03
                  *
                  IF PRIP04!16,PRIP04*500
                  SET1 !14
                  EDOL  (-;W;E)
                  CHRO  (I;R;S;*;N1,2:A>5,3;G-4,4;D-5,5;E)
                  EDOL TEMP,S1 (U*20,100;A;E)
                  PRIP05
                     * IF PRIP05 !27,PRIP05
                     * ;LIBR (I;C;DS;HS;E)
                     *
                     IF PRIP05!27,PRIP05
                     LIBR  (I;C;DS;HS;E)
                  RETU PRIP03
               EDOL S1 (-;N;*;A;E)
            LOOP
         EDLL  (B'1;E)
         PRIN  (QP1)
         FILE  (C PRIN.99,M:/N:E)
         FEED
         QUAN S1 (I;F2;H;E)
         FEED
         BEEP
      LOOP
FEED
BEEP
BEEP
BEEP
```

Attachment IIc

```
PRINP2.ME = C20;T:                                    PRIORITY POLLUTANT EVALUATION:
C;T:                    NO INTERNAL STANDARD WAS FOUND IN SAMPLE :S1:
C;T:                                    ;D          ... .. ..
PRINP1.ME = C2;D;T:  IDENTIFICATION REPORT          FILE:
;S1;C2;T;NO  SCAN  PURITY  FIT
;C;E
```

Attachment III

NAM NUM: NAME WT FORMULA		RET TIME	BASE	AREA	U.P.#1	U.P.#2
PP 1: 01 ACENAPHTHENE 154 C12.H10	0.706 154.000 40.00	21:26 PP	154 121	0. :S	0.000 0.812	0.000
PP 2: 02 ACROLEIN 56 C3.H4.O	0.000 0.000100.00	0:00	56 0	0.	0.000 1.000	0.000
PP 3: 03 ACRYLONITRILE 53 C3.H3.N	0.000 53.000100.00	0:00 PP	53 122	0. VS	0.000 0.125	0.000
PP 4: 04 BENZENE 78 C6.H6	0.650 78.000100.00	2:12 PP	78 122	0. VS	0.000 4.023	0.000
PP 5: 05 BENZIDINE 184 C12.H12.N2	1.345 184.000 20.00	5:00 PP	184 121	0. :S	0.000 0.047	0.000
PP 6: 06 CARBONTETRACHLORIDE 152 C.CL4	0.493 117.000100.00	1:40 PP	117 122	0. VS	0.000 2.979	0.000
PP 7: 07 CHLOROBENZENE 112 C6.H5.CL	1.103 112.000100.00	3:45 PP	112 122	0. VS	0.000 2.499	0.000
PP 8: 08 1,2,4-TRICHLOROBENZENE 180 C6.H3.CL3	0.385 74.000 40.00	11:42 PP	180 121	0. :S	0.000 0.084	0.000
PP 9: 09 HEXACHLOROBENZENE 282 C6.CL6	0.924 284.000 40.00	28:04 PP	284 121	0. :S	0.000 0.338	0.000
PP 10: 10 1,2-DICHLOROETHANE 98 C2.H4.CL2	0.414 62.000100.00	1:24 PP	62 122	0. VS	0.000 1.234	0.000
PP 11: 11 1,1,1-TRICHLOROETHANE 132 C2.H3 CL3	0.473 97.000100.00	1:36 PP	97 122	0. VS	0.000 3.100	0.000
PP 12: 12 HEXACHLOROETHANE 234 C2.CL6	0.283 117.000 40.00	8:36 PP	201 121	0. :S	0.000 0.113	0.000
PP 13: 13 1,1-DICHLOROETHENE 98 C2.H4.CL2	0.300 63.000100.00	1:01 PP	63 122	0. VS	0.000 2.792	0.000
PP 14 14 1,1,2-TRICHLOROETHANE 132 C2.H3.CL3	0.704 97.000100.00	2:23 PP	97 122	0. VS	0.000 0.695	0.000
PP 15 15 1,1,2,2-TETRACHLOROETHANE 166 C2.H2.CL4	0.900 83.000100.00	3:19 PP	83 122	0. VS	0.000 0.731	0.000
PP 16: 16 CHLOROETHANE 64 C2.H5 CL	0.064 64.000500.00	0:12 PP	64 122	0. VS	0.000 1.036	0.000
PP 17: 17 BIS(CHLOROMETHYL) ETHER 114 C2 H4.0 CL2		0:00	79	0.	0.000	0.000

Attachment IV

NAM	NUM:	WT	FORMULA	NAME
PP	121:	188		D10-ANTHRACENE (INTERNAL STANDARD)
PP	1:	154	C12.H10	01 ACENAPHTHENE
PP	8:	180	C6.H3.CL3	08 1,2,4-TRICHLOROBENZENE
PP	9:	282	C6.CL6	09 HEXACHLOROBENZENE
PP	12:	234	C2.CL6	12 HEXACHLOROETHANE
PP	18:	142	C4.H8.O.CL2	18 BIS(2-CHLOROETHYL)ETHER
PP	20:	162	C10.H7.CL	20 2-CHLORONAPHTHALENE
PP	25:	146	C6.H4 CL2	25 1,2-DICHLOROBENZENE
PP	26:	146	C6.H4.CL2	26 1,3-DICHLOROBENZENE
PP	27:	146	C6.H4.CL2	27 1,4-DICHLOROBENZENE
PP	28:	252	C12.H10.N2.CL2	28 3,3'-DICHLOROBNEZIDINE
PP	36:	182	C7.H6.O4.N2	35 2,4-DINITROTOLUENE
PP	37:	182	C7.H6.O4.N2	36 2,6-DINITROTOLUENE
PP	38:	182	C12.H10.N2	37 1,2-DIPHENYLHYDRAZINE (MEAS. AS AZOB
PP	40:	202	C16.H10	39 FLUORANTHENE
PP	41:	204	C12.H9.O.CL	40 4-CHLOROPHENYL PHENYL ETHER
PP	42:	248	C12.H9.O.BR	41 4-BROMOPHENYL PHENYL ETHER
PP	43:	170	C5.H12.O.CL2	42 BIS(2-CHLOROISOPROPYL)ETHER
PP	44:	172	C5.H10.O2.CL2	43 BIS(2-CHLOROETHOXY)METHANE
PP	53:	258	C4.CL6	52 HEXACHLOROBUTADIENE
PP	54:	270	C5.CL6	53 HEXACHLOROCYCLOPENTADIENE
PP	55:	138	C9.H14.O	54 ISOPHORONE
PP	56:	128	C10.H8	55 NAPHTHALENE
PP	57:	123	C6.H5.O2.N	56 NITROBENZENE
PP	63:	169	C12.H11.N	62 N-NITROSODIPHENYLAMINE (MEAS AS DIPH
PP	64:	130	C6.H14.O.N2	63 N-NITROSODIPROPYLAMINE
PP	67:	390	C24.H38.O4	66 DI-(2-ETHYLHEXYL)PHTHALATE
PP	68:	312	C18.H20.O4	67 BUTYL BENZYL PHTHALATE
PP	69:	278	C16.H22.O4	68 DI-N-BUTYLPHTHALATE
PP	70:	390	C24.H38.O4	69 DI-OCTYLPHTHALATE
PP	71:	222	C12.H14.O4	70 DIETHYLPHTHALATE
PP	72:	194	C10.H10.O4	71 DIMETHYLPHTHALATE
PP	73:	228	C18.H12	72 BENZO(A)ANTHRACENE
PP	75:	252	C20.H12	74 3,4-BENZOFLUORANTHENE
PP	76:	252	C20.H12	75 BENZO\K\FLUORANTHENE
PP	77:	228	C18.H12	76 CHRYSENE
PP	78:	152	C12.H8	77 ACENAPHTHYLENE
PP	79:	178	C14.H10	78 ANTHRACENE
PP	81:	166	C13.H10	80 FLUORENE
PP	82:	178	C14.H10	81 PHENANTHRENE
PP	85:	202	C16.H10	84 PYRENE

Attachment Va

QUANTITATION REPORT FILE: VSM13S14

DATA: VSM13S14.MI
0:00:00
SAMPLE: VOA STD MIX R 13 MAY 31, 1979
CONDS.:
FORMULA:
SUBMITTED BY:

INSTRUMENT: SYSIND
ANALYST:

WEIGHT: 0.000
ACCT. NO.:

AMOUNT=AREA * REF.AMNT/(REF.AREA* RESP.FACT)

NO	NAME
1	1,4-DICHLOROBUTANE (INTERNAL STANDARD)
2	BROMOCHLOROMETHANE (INTERNAL STANDARD)
3	04 BENZENE
4	06 CARBONTETRACHLORIDE
5	07 CHLOROBENZENE
6	10 1,2-DICHLOROETHANE
7	11 1,1,1-TRICHLOROETHANE
8	13 1,1-DICHLOROETHANE
9	14 1,1,2-TRICHLOROETHANE
10	15 1,1,2,2-TETRACHLOROETHANE
11	16 CHLOROETHANE
12	23 CHLOROFORM
13	29 1,1-DICHLOROETHENE
14	30 1,2-TRANS-DICHLOROETHYLENE
15	32 1,2-DICHLOROPROPANE
16	33A CIS-1,3-DICHLOROPROPENE
17	38 ETHYLBENZENE
18	44 METHYLENECHLORIDE
19	45 METHYL CHLORIDE
20	46 METHYL BROMIDE
21	47 BROMOFORM
22	48 BROMODICHLOROMETHANE
23	49 TRICHLOROFLUOROMETHANE
24	51 DIBROMOCHLOROMETHANE
25	85 TETRACHLOROETHENE
26	86 TOLUENE
27	87 TRICHLOROETHENE
28	88 VINYLCHLORIDE

NO	M/E	SCAN	TIME	REF	RPT	METH	AREA	AMOUNT	%TOT
1	55	203	3:23	1	1.000	A BB	166494.	200.000 UG/L	4.35
2	49	55	0:55	1	0.271	A BB	280909.	200.000 UG/L	4.35
3	78	132	2:12	1	0.659	A BB	334940.	100.000 UG/L	2.17
4	117	100	1:40	1	0.484	A BB	247990.	100.000 UG/L	2.17
5	112	225	3:45	1	1.109	A BB	208056.	100.000 UG/L	2.17
6	62	84	1:24	1	0.411	A BB	102756.	100.000 UG/L	2.17
7	97	96	1:36	1	0.473	A BB	258064.	100.000 UG/L	2.17
8	63	61	1.01	1	0.300	A BB	232452.	100.000 UG/L	2.17
9	97	143	2.33	1	0.704	A BB	57813	100.000 UG/L	2.17
10	83	190	3 19	1	0.931	A BB	68890.	100.000 UG/L	2.17
11	64	13	0:13	1	0.064	A BB	431236.	500.000 UG/L	10.87
12	83	70	1:10	1	0.344	A BB	425633.	100.000 UG/L	2.17
13	96	67	1.07	1	0.333	A BB	72096.	100.000 UG/L	2.17
14	61	67	1:07	1	0.330	A BB	165562.	100.000 UG/L	2.17
15	65	123	2 03	1	0.605	A BB	159055	100.000 UG/L	2.17
16	75	142	2.22	1	0.702	A BB	73630.	100.000 UG/L	2.17
17	91	259	4 19	1	1.273	A BB	373312.	100.000 UG/L	2.17

Attachment Vb

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	%TOT
18	84	25	0:25	1	0.123	A 88	221038.	100.000 UG/L	2.17
19	50	5	0:05	1	0.025	A 88	288968.	500.000 UG/L	10.87
20	94	7	0:07	1	0.034	A 88	587988.	500.000 UG/L	10.87
21	173	173	2:53	1	0.852	A 88	57085.	100.000 UG/L	2.17
22	83	111	1:51	1	0.547	A 88	223082.	100.000 UG/L	2.17
23	101	39	0:39	1	0.192	A 88	151888.	100.000 UG/L	2.17
24	129	143	2:23	1	0.704	A 88	120233.	100.000 UG/L	2.17
25	129	195	3:15	1	0.961	A 88	122326.	100.000 UG/L	2.17
26	91	203	3:23	1	1.000	A 88	316701.	100.000 UG/L	2.17
27	130	132	2:12	1	0.650	A 88	124234.	100.000 UG/L	2.17
28	62	9	0:09	1	0.044	A 88	564238.	500.000 UG/L	10.87

NO	RET(L)	RATIO	RPT(L)	RATIO	AMNT	AMNT(L)	R.FAC	R.FAC(L)	RATIO
1	3:23	1.00	1.000	1.00	200.00	200.00	1.020	1.000	1.00
2	0:55	1.00	0.271	1.00	200.00	200.00	1.687	1.687	1.00
3	2:12	1.00	0.650	1.00	100.00	100.00	4.023	4.023	1.00
4	1:40	1.00	0.493	1.00	100.00	100.00	2.979	2.979	1.00
5	3:45	1.00	1.108	1.00	100.00	100.00	2.499	2.499	1.00
6	1:24	1.00	0.414	1.00	100.00	100.00	1.234	1.234	1.00
7	1:36	1.00	0.473	1.00	100.00	100.00	3.100	3.100	1.00
8	1:01	1.00	0.300	1.00	100.00	100.00	2.792	2.792	1.00
9	2:23	1.00	0.704	1.00	100.00	100.00	0.695	0.695	1.00
10	3:19	1.00	0.980	1.00	100.00	100.00	0.731	0.731	1.00
11	0:13	1.00	0.064	1.00	500.00	500.00	1.036	1.036	1.00
12	1:18	1.00	0.384	1.00	100.00	100.00	5.089	5.089	1.00
13	1:07	1.00	0.330	1.00	100.00	100.00	0.938	0.938	1.00
14	1:07	1.00	0.330	1.00	100.00	100.00	1.962	1.962	1.00
15	2:03	1.00	0.606	1.00	100.00	100.00	1.899	1.899	1.00
16	2:22	1.00	0.700	1.00	100.00	100.00	0.885	0.885	1.00
17	4:19	1.00	1.276	1.00	100.00	100.00	4.484	4.484	1.00
18	0:25	1.00	0.123	1.00	100.00	100.00	2.655	2.655	1.00
19	0:05	1.00	0.025	1.00	500.00	500.00	0.694	0.694	1.00
20	0:07	1.00	0.034	1.00	500.00	500.00	1.413	1.413	1.00
21	2:53	1.00	0.852	1.00	100.00	100.00	0.686	0.686	1.00
22	1:51	1.00	0.547	1.00	100.00	100.00	2.680	2.680	1.00
23	0:39	1.00	0.192	1.00	100.00	100.00	1.823	1.823	1.00
24	2:23	1.00	0.704	1.00	100.00	100.00	1.444	1.444	1.00
25	3:15	1.00	0.961	1.00	100.00	100.00	1.468	1.468	1.00
26	3:23	1.00	1.000	1.00	100.00	100.00	3.804	3.804	1.00
27	2:12	1.00	0.650	1.00	100.00	100.00	1.492	1.492	1.00
28	0:09	1.00	0.044	1.00	500.00	500.00	1.356	1.356	1.00

Attachment Vc

NAM	NUM:	WT	FORMULA
PP	122:	126	C4.H8.CL2
PP	123:	128	C.H2.CL.BR
PP	4:	78	C6.H6
PP	6:	152	C.CL4
PP	7:	112	C6.H5.CL
PP	10:	98	C2.H4.CL2
PP	11:	132	C2.H3.CL3
PP	13:	98	C2.H4.CL2
PP	14:	132	C2.H3.CL3
PP	15:	166	C2.H2.CL4
PP	16:	64	C2.H5.CL
PP	23:	118	C.H.CL3
PP	29:	96	C2.H2.CL2
PP	30:	96	C2.H2.CL2
PP	32:	112	C3.H6.CL2
PP	33:	110	C3.H4.CL2
PP	39:	106	C8.H10
PP	45:	84	C.H2.CL2
PP	46:	50	C.H3.CL
PP	47:	94	C.H3.BR
PP	48:	250	C.H.BR3
PP	49:	162	C.H.CL2.BR
PP	50:	136	C.CL3.F
PP	52:	206	C.H.CL.BR2
PP	85:	164	C2.CL4
PP	87:	92	C7.H8
PP	88:	130	C2.H.CL3
PP	89:	62	C2.H3.CL

NAME	
	1,4-DICHLOPOBUTANE (INTERNAL STANDAR
	BROMOCHLOROMETHANE (INTERNAL STANDAR
04	BENZENE
06	CARBONTETRACHLORIDE
07	CHLOROBENZENE
10	1,2-DICHLOROETHANE
11	1,1,1-TRICHLOROETHANE
13	1,1-DICHLOPCETHANE
14	1,1,2-TRICHLOROETHANE
15	1,1,2,2-TETRACHLOROETHANE
16	CHLORGETHANE
23	CHLOROFORM
29	1,1-DICHLOROETHENE
30	1,2-TRANS-DICHLOROETHYLENE
32	1,2-DICHLOROPROPANE
33A	CIS-1,3-DICHLOROPROPENE
38	ETHYLBENZENE
44	METHYLENECHLORIDE
45	METHYL CHLORIDE
46	METHYL BROMIDE
47	BROMOFORM
48	BROMODICHLOROMETHANE
49	TRICHLOROFLUOROMETHANE
51	DIBROMOCHLOROMETHANE
85	TETRACHLOROETHENE
86	TOLUENE
87	TRICHLOROETHENE
88	VINYLCHLORIDE

0/00/00 0:00:00 IDENTIFICATION PEPORT FILE: D:VSM13314.MI

NO	SCAN	PURITY	FIT
*1	203	353	870
*1	55	835	952
4	132	429	954
6	100	921	993
7	225	653	922
10	84	630	981
11	96	611	901
13	61	264	957
14	143	291	935
15	199	322	959
16	13	509	930
23	78	870	941
29	67	764	975
30	67	740	956
32	123	651	935
33	142	360	874
39	259	654	981
45	25	777	969
46	5	398	976
47	7	503	987
48	173	693	940
49	111	896	995
50	39	454	968
52	143	354	969
86	195	770	945
87	203	573	972
88	132	425	924
89	9	593	963

METHOD FOR ORGANOCHLORINE PESTICIDES IN ENVIROMENTAL WATER SAMPLES NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

1. SCOPE AND APPLICATION

1.1 This method is an adaptation of that described in ref. 1 and covers the determination of various organochlorine pesticides, including some pesticidal degradation products and related compounds in industrial effluents. Such compounds are composed of carbon, hydrogen, and chlorine, but may also contain oxygen, sulfur, phosphorus, nitrogen or other halogens.

1.2 The following compounds may be determined individually by this method with a sensitivity of at least 1 µg/liter: BHC, lindane, heptachlor, aldrin, heptachlor epoxide, dieldrin, endrin, DDE, DDD, DDT, methoxychlor, endosulfan, mirex, trifluralin, endrin aldehyde, and endosulfan sulfate. Under favorable circumstances, Strobane, toxaphene, chlordane (tech) and others may also be determined. The usefulness of the method for other specific pesticides must be demonstrated by the analyst before any attempt is made to apply it to sample analysis.

1.3 When organochlorine pesticides exist as complex mixtures, the individual compounds may be difficult to distinguish. High, low, or otherwise unreliable results may be obtained through misidentification and/or one compound obscuring another of lesser concentration. Provisions incorporated in this method are intended to minimize the occurrence of such interferences.

2. SUMMARY

2.1 The method offers several analytical alternatives, dependent on the analyst's assessment of the nature and extent of interferences and/or the complexity of the pesticide mixtures found. Specifically, the procedure describes the use of an effective co-solvent for efficient sample extraction; provides, through use of column chromatography and liquid-liquid partition, methods for elimination of non-pesticide interferences and the pre-separation of pesticide mixtures. Identification is made by selective gas chromatographic separations and may be corroborated through the use of two or more unlike columns. Detection and measurement is accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography. Results are reported in micrograms per liter.

2.2 This method is recommended for use only by experienced pesticide analysts or under the close supervision of such qualified persons.

3. INTERFERENCES

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.2 The interferences in industrial effluents are high and varied and often pose great difficulty in obtaining accurate and precise measurements of organochlorine pesticides. Sample clean-up procedures are generally required and may result

in the loss of certain organochlorine pesticides. Therefore, great care should be exercised in the selection and use of methods for eliminating or minimizing interferences. It is not possible to describe procedures for overcoming all of the interferences that may be encountered in industrial effluents.

3.3 Polychlorinated Biphenyls (PCB's) - Special attention is called to industrial plasticizers and hydraulic fluids such as the PCB's which are a potential source of interference in pesticide analysis. The presence of PCB's is indicated by a large number of partially resolved or unresolved peaks which may occur throughout the entire chromatogram. Particularly severe PCB interference will require special separation procedures (2,3).

3.4 Phthalate Esters - These compounds, widely used a plasticizers, respond to the electron capture detector and are a source of interference in the determination of organochlorine pesticides using this detector. Water leaches these materials from plastics, such as polyethylene bottles and tygon tubing. The presence of phthalate esters is implicated in samples that respond to electron capture but not to the microcoulometric or electrolytic conductivity halogen detectors or to the flame photometric detector.

3.5 Organophosphorus Pesticides - A number of organophosphorus pesticides, such as those containing a nitro group, e.g., parathion, also respond to the electron capture detector and may intefere with the determination of the organochlorine pesticides. Such compounds can be identified by their response to the alkali flame ionization or flame photometric detectors.

3.6 Anaerobic extracts may contain gross interference due to the presence of sulfur compounds. This interference can be removed by reacting the extract with a small amount of metal-

lic mercury to precipitate the sulfur compounds. After alumina column cleanup, the sulfur interferences are confined to the first fraction, and only this fraction need be reacted with metallic mercury (4).

4. APPARATUS AND MATERIALS

4.1 Gas Chromatograph - Equipped with glass lined injection port.

4.2 Detector Options:

- 4.2.1 Electron Capture - Radioactive (tritium or nickel 63)
- 4.2.2 Microcoulometric Titration
- 4.2.3 Electrolytic Conductivity

4.3 Recorder - Potentiometric strip chart (10 in) compatible with the detector.

4.4 Gas Chromatographic Column Materials:

- 4.4.1 Tubing - Pyrex (180 cm long x 4 mm ID)
- 4.4.2 Glass Wool - Silanized
- 4.4.3 Solid Support - Gas-Chrom Q (60-80 mesh)
- 4.4.4 Liquid Phases - Expressed as Weight percent coated on solid support.
  - 4.4.4.1 OV-101, 3%
  - 4.4.4.2 OV-210, 5%
  - 4.4.4.3 OV-17, 3% or any column yielding equivalent separation

4.5 Kuderna-Danish (K-D) Glassware (Kontes)

- 4.5.1. Snyder Column - three ball (macro)
- 4.5.2 Evaporative Flasks - 500 ml
- 4.5.3 Receiver Ampuls - 10 ml, graduated

4.6 Chromatographic Column - pyrex (approximately 340 mm long x 20 mm ID) with coarse fritted place on bottom (Kontes

K422000) modified to include a reservoir for 50 ml of solvent and fitted with a ball joint.

4.7 Micro Syringes - 10, 25, 50 and 100 µl

4.8 Separatory Funnels - 125 ml, 1000 ml and 2000 ml with Teflon stopcock.

4.9 Graduated cylinders - 100, 250 and 1000 ml.

4.10 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.

4.11 Alumina, Basic, Brockman Activity I; 80-200 mesh. The amount of water needed for proper deactivation is determined by the elution pattern for a technical chlordane standard. A 1.75% deactivation is usually sufficient to yield the correct elution pattern (see Table IV).

5. REAGENTS, SOLVENTS, AND STANDARDS

5.1 Ferrous Sulfate - (ACS) 30% solution in distilled water.

5.2 Potassium Iodide - (ACS) 10% solution in distilled water.

5.3 Sodium Chloride - (ACS) Saturated solution in distilled water (pre-rinse NaCl with hexane).

5.4 Sodium Hydroxide - (ACS) 10 N in distilled water.

5.5 Sodium Sulfate - (ACS) Granular, anhydrous (conditioned at 300 °C for 4 hours).

5.6 Sulfuric Acid - (ACS) Mix equal volumes of conc. H_2SO_4 with distilled water.

5.7 Diethyl Ether - Nanograde, redistilled in glass, if necessary.

5.7.1 Must contain 2% alcohol and be free of peroxides by following test: To 10 ml of ether in glass-stoppered cylinder previously rinsed with ether, add one ml of freshly prepared 10% KI solution. Shake

and let stand one minute. No yellow color should be observed in either layer. Alternately the peroxide test may be done with EM Quant™ Ether Peroxide - Test stacks. The peroxide level must be less than 1.5 ppm.

5.7.2 Decompose either peroxides by adding 40 g of 30% ferrous sulfate solution to each liter of solvent. CAUTION: Reaction may be vigorous if the solvent contains a high concentration of peroxides.

5.7.3 Distill deperoxidized ether in glass and add 2% ethanol.

5.8 Acetonitrile, Hexane, Methylene Chloride, Petroleum Ether (boiling range 30-60°C) - nanograde,redistill in glass if necessary.

5.9 Pesticide Standards - Reference grade: sources

5.9.1 Quality Assurance Section, Environmental Toxicology Division, EPA, HERL, Research Traingle Park, N.C. 27711, MD-69

5.9.2 Pesticides Reference Standards Section, Bldg 048 Range 3 and 3rd Street, BARC, West, Beltsville, MD 20705

5.9.3 Nanogens, P.O. Box 1025, Watsonville, CA 95076

6. CALIBRATION

6.1 Gas chromatographic operating conditions are considered acceptable if a Standard Mix B elutes from the GC with correct retention times and sensitivity. Standard Mix B consists of 0.025 µg/ml lindone, 0.050 µg/ml heptachlor, 0.075 µg/ml aldrin, 0.100 µg/ml γ chlordane, 0.125 µg/ml dieldrin, 0.250 µg/ml o, p'-DDT and 0.250 µg/ml p,p'-DDT

in hexane. The chromatographic conditions chosen should yield at least 30% full-scale deflection for all of the components of Std. Mix B (see Figures 1 through 3). For all quantitative measurements, the detector must be operated within its linear response range and the detector noise level should be less than 2% of full-scale.

6.2 Standards are injected frequently as a check on the stability of operating conditions. Gas chromatograms of several standard pesticides are shown in Figures 1, 2 and 3 and provide reference operating conditions for recommended columns.

6.3 The elution order and retention ratios of various organochlorine pesticides are provided in Table I, as a guide. The sensitivity of these compounds is given in Table II.

7. QUALITY CONTROL

7.1 Replicate and spiked sample analyses are recommended as quality control checks. At a minimum, one replicate and one spiked analysis should be included per 20 sample analyses. If less than 20 sample analyses are required, one duplicate and one spiked analysis should still be included. Data for recovery of specific organochlorine pesticides from water is given in Table III.

7.2 In addition, one method blank is required per 20 sample analyses. If less than 20 sample analyses are required, one method blank should still be included.

7.3 One sample should be injected in replicate into the gas chromatograph per 20 samples analyzed. If less than 20 sample analyses are required, a replicate GC injection should still be made.

8. SAMPLE PREPARATION

8.1 Shake the sample if suspended matter is present and adjust pH to near neutral (pH 6.5-7.5) with 50% sulfuric acid or 10 N sodium hydroxide.

8.2 Quantitatively transfer 1 liter of sample into a two-liter separatory funnel. Less sample may be analyzed if necessary, with the realization that detection limits will be affected.

9. EXTRACTION

9.1 Add 60 ml of 15% methylene chloride in hexane (v:v) to the sample in the separatory funnel and shake vigorously for two minutes.

9.2 Allow the mixed solvent to separate from the sample, then draw the water into a one-liter beaker. Pour the organic layer into a 250 ml beaker. Return the water phase to the separatory funnel. Rinse the one-liter beaker with a second 60 ml volume of solvent; add the solvent to the separatory funnel and complete the extraction procedure a second time. Perform a third extraction in the same manner.

9.3 Transfer the combined solvent extract to a 500 ml Kuderna-Danish evaporative concentrator by passing it through a funnel plugged with glass wool and filled with sodium sulfate which has been prewashed with hexane.

9.4 Concentrate the extract to 10 ml in the K-D evaporator on a hot water bath.

9.5 Analyze by gas chromatography unless a need for cleanup is indicated (see Section 10).

10. CLEAN-UP AND SEPARATION PROCEDURES

10.1 Interferences in the form of distinct peaks and/or high background in the initial gas chromatographic analysis, as well as the physical characteristics of the extract (color, cloudiness, viscosity) and background knowledge of the sample will indicate whether clean-up is required. When these interfere with measurement of the pesticides, or affect column life or detector sensitivity, proceed as directed below.

10.2 Acetonitrile Partition - This procedure is used to isolate fats and oils from the sample extracts. It should be noted that not all pesticides are quantitatively recovered by this procedure. The analyst must be aware of this and demonstrate the efficiency of the partitioning for specific pesticides.

10.2.1 Quantitatively transfer the previously concentrated extract to a 125 ml separatory funnel with enough hexane to bring the final volume to 15 ml. Extract the sample four times by shaking vigorously for one minute with 30 ml portions of hexane-saturated acetonitrile.

10.2.2 Combine and transfer the acetonitrile phases to a one-liter separatory funnel and add 650 ml of distilled water and 40 ml of saturated sodium chloride solution. Mix throughly for 30-45 seconds. Extract with two 100 ml portions of hexane by vigorously shaking about 15 seconds.

10.2.3 Combine the hexane extracts in a one-liter separatory funnel and wash with two 100 ml portions of distilled water. Discard the water layer and pour the hexane layer into a 500 ml K-D flask

through a funnel plugged with glass wool and filled with sodium sulfate which has been pre-washed with hexane. Rinse the separatory funnel and column with three 10 ml portion of hexane.

10.2.4 Concentrate the extracts to 10 ml in the K-D evaporator in a hot water bath.

10.2.5 Analyze by gas chromatography unless a need for further clean-up is indicated.

10.3 Florisil Column Adsorption Chromatography

10.3.1 Adjust the sample extract volume to 10 ml with hexane.

10.3.2 Prepare a 20 mm I.D. column that contains 4 inches (after settling) of activated Florisil topped with 0.5 inch anhydrous sodium sulfate.

10.3.3 Pre-elute the column with 50-60 ml of petroleum ether. Just prior to exposure of the sulfate layer to air, quantitatively transfer the sample extract onto the column. Just prior to exposure of the sodium sulfate layer to air, add the first eluting solvent, 200 ml of 6% ethyl ether in petroleum ether. Collect the eluate in a 250 ml beaker. Perform the second elution with 200 ml of 15% ethyl ether in petroleum ether, the third elution with 200 ml of 50% ethyl ether-petroleum ether, and the fourth elution with 200 ml fo 100% ethyl ether. (See Eluate Composition 10.3.6).

10.3.4 Concentrate the eluates to 10 ml in a K-D in a hot water bath. Fifty mls of petroleum ether must be added to the fourth fraction prior to concentration to eliminate the ethyl ether from the concentrated extract.

10.3.5 Analyze by gas chromatography.

10.3.6 Eluate Composition - The composition of the eluate should be checked for each new batch of Florisil with a standard mix consisting of gamma-BHC (lindane) heptachlor, endosulfan A and B. If the composition of the eluate varies from that given below, the amount of Florisil used in the column should be altered i.e., an increase in the amount of Florisil will increase the amount of solvent needed to elute compounds from the column. The majority of the compound should elute in the fraction listed below.

6% Eluate

Aldrin	DDT
BHC	Heptachlor
Chlordane	Heptachlor Epoxide
DDD	Lindane
Endosulfan A	Mirex
Toxaphene	PCB's
DDE	Methoxychlor

15% Eluate

Endrin
Dieldrin
Phthalate esters

50% Eluate

Endosulfan B

Certain thiophosphate pesticides will occur in each of the above fractions as well as the 100% fraction. For additional information regarding eluate composition, refer to the FDA Pesticide Analytical Manual (5).

10.4 Alumina Column Adsorption Chromatography (6).

10.4.1 Adjust the sample extract volume to 10 ml with hexane.

10.4.2 Prepare a 15 cm (after settling) x 2 cm column of properly deactivated alumina (see 4.11). The alumina should be settled by tapping the column.

10.4.3 Pre-eluate the column with 40-50 ml of hexane. Adjust the flow of the solvent through the column to 5 ml/min with air. Just prior to exposure of the alumina surface to air, quantitatively transfer the sample extract to the column using several hexane washes. This transfer should be done without disturbing the surface of the alumina.

10.4.4 Just prior to the exposure of the alumina surface to air, add 50 ml of a 10% ethyl ether in hexane solution. Collect the eluate in a 50 ml beaker. Ten 50 ml fractions are collected in like manner and each fraction is concentrated to 10 ml on a hot plate under a gentle stream of air.

10.4.5 Analyze by gas chromatography.

10.4.6 Eluate Composition. The composition of the eluate should be checked for each new batch of alumina with a technical chlordane standard. If the composition of the eluate varies from that given in Table IV, the amount of water added to the alumina should be altered, i.e., an increase in the amount of water will decrease the amount of solvent needed to elute compounds from the column.

11. CALCULATION OF RESULTS

11.1 Determine the pesticide concentration by using the absolute calibration procedure described below:

(1) $$\text{Micrograms/liter} = \frac{(A)\ (B)\ (V_t)}{(V_i)\ \ (V_s)}$$

$$A = \frac{\text{ng standard}}{\text{Standard area}}$$

B = Sample aliquot area

V_i = Volume of extract injected (µl)

V_t = Volume of total extract (µl)

V_s = Volume of water extracted (ml)

12. REPORTING RESULTS

12.1 Report results in micrograms per liter without correction for recovery data. When duplicate and spiked samples are analyzed, all data obtained should be reported.

REFERENCES

1. "Method for Organochlorine Pesticides in Industrial Effluents", Natinal Pollutant Discharge Elimination System, Appendix A, Federal Register, 38, No. 75, Pt. II.

2. Monsanto Methodology for Arochlors - Analysis of Environmental Materials for Biphenyls, Analytical Chemistry Method 71-35, Monsanto Company, St. Louis, Missouri, 63166, 1970.

3. "Method for Polychlorinated Biphenyls in Industrial Effluents," Environmental Protection Agency, National Environmental Research Center, Cincinnati, Ohio, 45268, 1973. (Also NPDES, Appendix A, Fed. Reg., 38, No. 75, Pt. II.)

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Table I

RETENTION TIMES OF ORGANOCHLORINE PESTICIDES RELATIVE TO ALDRIN

Liquid Phase Solid Support	3% OV-101 2 mm x 6' glass on 60/80 GCQ	3% OV-17 4 mm x 6' glass on 60/80 GCQ	5% OV-210 2 mm x 6' glass
Column Temperature	180°C	200°C	200°C
Flow rate[a] (ml/min)	25	80	37
Pesticide	**RRT**	**RRT**	**RRT**
α-BHC	0.40	0.45	0.68
β-BHC	0.44	0.49	0.96
γ-BHC (lindane)	0.48	0.53	0.83
σ-BHC	0.50	0.54	1.54
heptachlor	0.80	0.82	0.88
aldrin	1.00	1.00	1.00
heptachlor epoxide	1.26	1.19	1.71
γ-chlordane	1.44	1.38	1.64
Endosulfan A	1.60	1.47	2.16
α-chlordane	1.62	1.50	1.64
dieldrin	1.88	1.73	2.55
p,p' DDE	1.96	1.68	1.78
endrin	2.11	1.89	2.97
Endosulfan B	2.20	1.93	3.72
o,p' DDT	~~2.37~~ 2.64	2.25	2.22
DDD	2.52	2.10	2.94
Endrin aldehyde	2.52	2.10	5.76
endosulfan sulfate	2.99	2.46	8.42
p,p' DDT	3.37	2.77	3.18
methoxychlor	5.31	4.01	4.60
aldrin (min absolute)	3.80	2.28	1.74

a Argon 10% methane, RRT for other columns are given in ref. 1, Table I.

Table II

SENSITIVITY OF ORGANOCHLORINE PESTICIDES USING ELECTRON CAPTURE (EC) DETECTOR

Instrument	Tracor MT-220
Liquid Phase	3% OV-17
Solid Support	4 mm x 6' glass 60/80 GCQ
Column Temperature	200°C
Flow Rate	81.6 ml/min
Injection Size	2 µl

Pesticide	conc. (µg/ml)	att.	peak height (mm)
Lindane	0.025	8	79
Heptachlor	0.05	8	113
Aldrin	0.075	8	140
γ-Chlordane	0.10	8	132
Dieldrin	0.125	8	136
o,p' DDT	0.250	8	95
p,p' DDT	0.250	8	92
α-BHC	0.05	8	256
endosulfan A	0.10	8	77
p,p' DDE	0.10	8	104
endosulfan B	0.10	8	60
DDD	0.10	8	46
endosulfan sulfate	0.50	8	215
β-BHC	0.050	8	79
Heptachlor Epoxide	0.100	8	134
Endrin	0.100	8	58
Endrin Aldehyde	0.100	8	31

Table III

RECOVERY DATA FOR SELECTED ORGANOCHLORINE PESTICIDES
(EXTRACTION FROM WATER ONLY)

Compound	Spiking Level (µg)	Number of Determinations	Average % Recovery	Standard Deviation
lindane	0.25	12	110	8.3
heptachlor	0.50	12	89	7.6
aldrin	0.75	12	91	12.4
γ-chlordane	1.00	12	97	2.5
dieldrin	1.25	12	100	3.8
o,p' DDT	2.50	12	98	6.4
p,p' DDT	2.50	11	109	5.5
DDD	1.00	11	100	14.8
Endosulfan A	1.00	12	99	4.3
Endosulfan B	1.00	12	95	6.0
α-BHC	0.50	9	102	3.8
p,p' DDE	1.00	12	98	4.1
Endosulfan sulfate	5.00	11	107	11.6
β-BHC	0.50	8	103	5.2
heptachlor epoxide	1.00	9	99	6.2
endrin	1.00	7	115	12.2
endrin aldehyde	1.00	8	89	7.2

Table IV

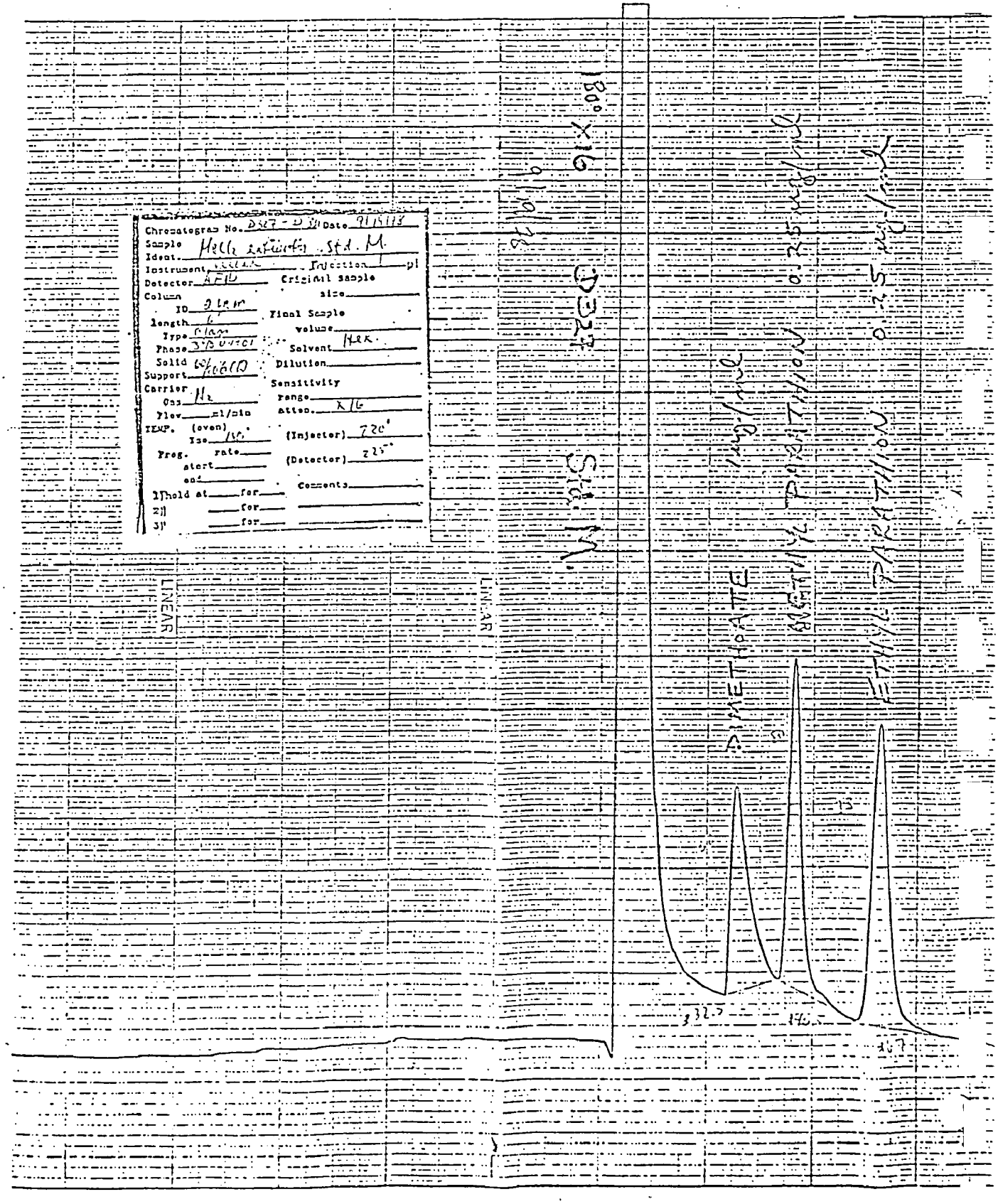
ORDER OF ELUTION OF CHLORINATED INSECTICIDES FROM ALUMINA ADSORPTION COLUMN[a] (6)

Insecticide	50 ml Eluate Fractions - % of Total Recovered										% Recovery
	1	2	3	4	5	6	7	8	9	10	
DDE	95	5									94
Aldrin	93	7									97
Heptachlor	75	25									96
Tech. Chlordane	30	30	35	5							99
Toxaphene	15	55	30	Trace							93
DDT	5	95									94
γ-Chlordane		2	80	18							99
α-Chlordane			95	5							97
DDD			60	40							93
Lindane			35	65							40
Endrin				45	55						95
Heptachlor Epoxide				35	50	15					95
Dieldrin						20	40	20	15	5	96
Methoxyclor						5	30	50	10	5	96
Aroclor 1242	100										100
Aroclor 1248	98	2									100
Aroclor 1254	95	5									100
Aroclor 1260	95	5									100
Lindane	(Acid Alumina)				25	60	15				100
Lindane	(Neutral Alumina)			3	75	20	2				91

a 9/1 Hexane/Ethyl Ether Eluting Solvent.

FIGURE 1. EC/GC, 3% OV-17

FIGURE 2. AFID, 3% OV-101

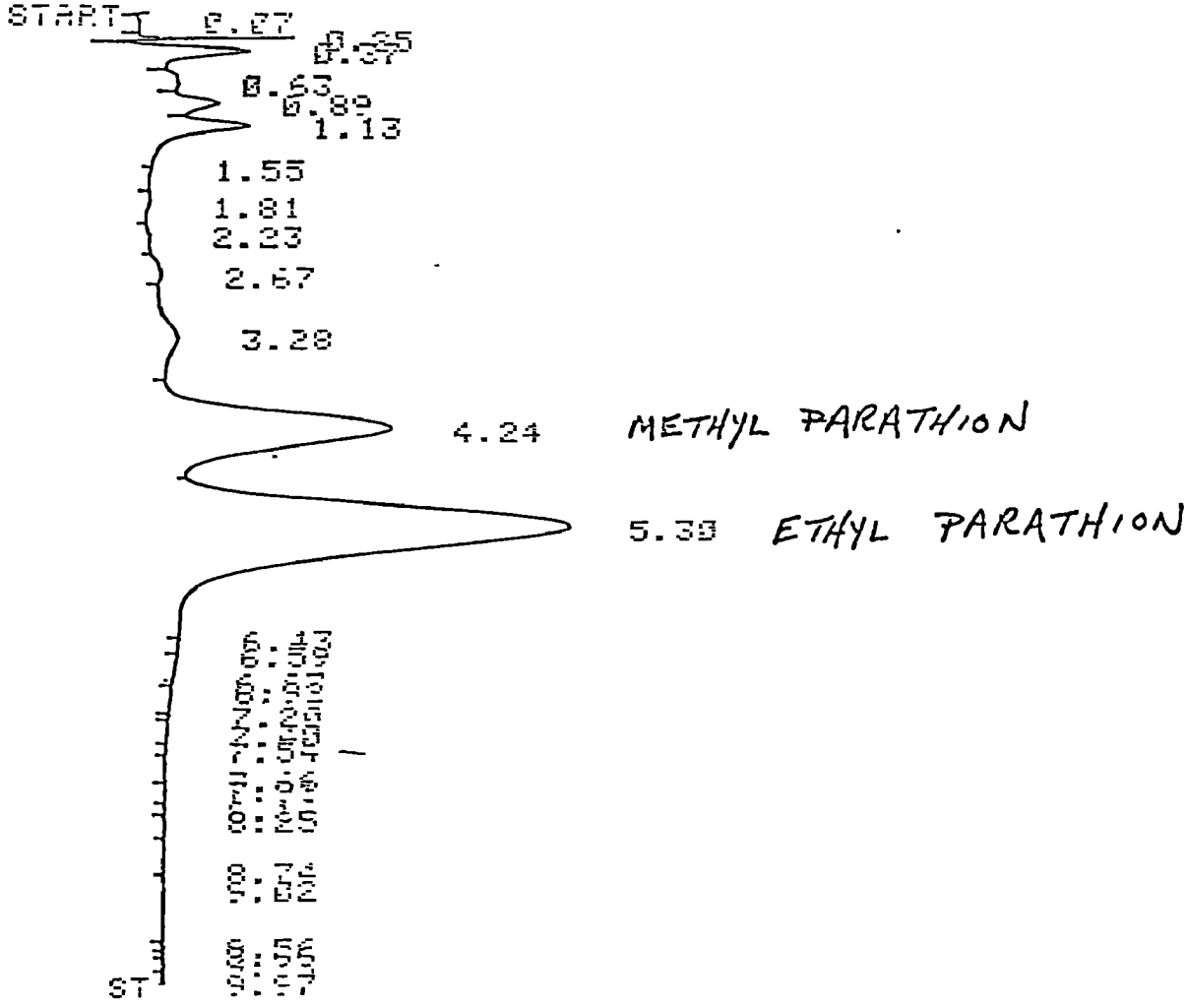


```
TIME        0.00
INJ TEMP  400   335   275
AUX TEMP  400   300   300

CHT SPD     1.27
ZERO       10.0
ATTN 2^     10
AUX SGNL    +8
SLP SENS    0.10
AREA REJ        10000
FLOW  A     8.5     8.5
FLOW  B    34.7    34.2

18.00 STOP
```

FIGURE 3.
EC/GC, 5% OV-210



HP RUN # 25 JAN/17/78 TIME 21:41:47
BOTTLE 30
AREA %

RT	AREA	AREA %
0.25	46320	0.743
0.31	147305	2.365
0.63	104955	1.684
0.89	172500	2.771
1.13	286700	4.603
1.55	65500	1.052
1.81	93100	1.495
2.23	103700	1.665
2.67	150600	2.418
3.28	447300	7.179

Quality Control Data
Pesticides and PCB

Sample 05 was analyzed after spiking with seven pesticide components, with average recovery of 91%.

Sample 08 was analyzed in replicate. Two components, alpha-BHC and gamma-BHC were detected, with an average deviation of 5 percent.

Table 1
Pesticides-PCB's-QC Results

Spiked Sample: 05

Component	Recovery, percent
Aldrin	90%
Gamma-Chlordane	82
o,p'-DDt	85
p,p'DDT	93
Dieldrin	92
Heptachlor	94
Lindane	99

Duplicate Sample: 08

Component	Analysis 1	Analysis 2
Alpha-BHC	170 ug/l	188
Gamma-BHC	61	55

APPENDIX C

NON-PRIORITY POLLUTANTS
QUALITATIVE DATA SUMMARY

APPENDIX C

NON-PRIORITY POLLUTANTS (QUALITATIVE DATA SUMMARY)[a]
HOOKER CHEMICALS AND PLASTICS CORPORATION
WASTE DISPOSAL SITES/NIAGARA FALLS, NEW YORK
July 12-September 7, 1979

Chemical Name	Station No.	Relative Values									
		01	02	03	04	05	06	07	08	09	10
Aminobenzotrifluoride isomer		ND[b]	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chlorobenzaldehyde isomer		ND	3	ND	ND	ND	ND	ND	ND	ND	ND
2,4-dichlorotoluene		39	1	1	ND	ND	ND	ND	ND	ND	ND
Dichlorotoluene isomer (other than 2,4)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichlorobenzene isomer (other than 1,2,4)		ND	ND	ND	ND	ND	ND	ND	MS[c]	ND	ND
Chlorobenzoic acid, methyl ester isomer - #1		3	6	ND	ND	ND	ND	ND	ND	ND	ND
Chlorobenzoic acid, methyl ester isomer - #2		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dichloro-alpha-chlorotoluene isomer - #1		8	ND	1	ND	ND	ND	ND	ND	ND	ND
Dichloro-alpha-chlorotoluene isomer - #2		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachlorobenzene isomer - #1		ND	ND	MS	ND	ND	ND	ND	ND	ND	8
Tetrachlorobenzene isomer - #2		9	ND	MS	ND	ND	ND	3	82	MS	36
Pentachlorobenzene isomer		MS	ND	ND	ND	ND	ND	ND	MS	ND	ND
Chlorobenzoic acid isomer		9	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachlorotoluene isomer - #1		1	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachlorotoluene isomer - #2		2	ND	ND	ND	ND	ND	ND	ND	ND	ND

a This information includes the results of the NEIC Qualitative Evaluation of samples collected July 12, 1979 for other non-priority pollutants. The data format is the same as previously reported data. The results are shown as relative quantities. Because the same respone factors were used as for the previous data, these data may be directly compared.

b ND means not detected.

c MS means the compounds was identified by mass spectrometry but was below the quantitation detection limit.