

NEIC

COMPLIANCE EVALUATION
AND
WASTEWATER CHARACTERIZATION

UNION CARBIDE COMPANY

National Enforcement Investigations Center, Denver

U.S. Environmental Protection Agency



Office of Enforcement

Environmental Protection Agency
Office of Enforcement
EPA-330/2-79-013

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AND
WASTEWATER CHARACTERIZATION

UNION CARBIDE COMPANY
SOUTH CHARLESTON, WEST VIRGINIA

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March 1979

National Enforcement Investigations Center - Denver
and
Region III - Philadelphia

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I. INTRODUCTION

The Union Carbide Corporation, Chemical and Plastics Division, South Charleston, West Virginia, operates one of the largest petrochemical plants in the world. Union Carbide South Charleston (UCSC) produces about 400 different specialty-type chemicals and mixtures. Most of these products are intermediates used in other processes or sold for use in finished products. The production facilities and mixing and drumming area occupy an area of approximately 93 hectares (230 acres) on Blaine Island and the south bank of the Kanawha River [Appendix A, Figure 2, p. A-12].

The Kanawha Valley contains numerous industrial plants engaged in the production of organic and/or inorganic chemicals. The passage of the Toxic Substance Control and Resources Conservation and Recovery Acts in 1976 focused attention on the need to control the discharges of toxic substances. Large volumes of such wastes are produced and disposed in the Kanawha Valley with a resultant potential for release to the environment.

The Environmental Protection Agency, Region III, requested that the National Enforcement Investigations Center (NEIC) inspect the Union Carbide facility to: a) determine the sources and types of toxic pollutants discharged, b) evaluate pollution abatement practices, and c) determine if NPDES* permit requirements are being met. NEIC conducted a detailed plant inspection and a subsequent field survey.

* NPDES: National Pollutant Discharge Elimination System, Public Law 92-500, Sec. 402 of the Federal Water Pollution Control Act as amended in 1972, and subsequently Sec. 402 of the Clean Water Act as amended in 1977.

The inspection results are summarized in this report, and presented in full context in Appendix A.

The objectives of the April 1978 plant inspection were to:

1. inspect process operations [Appendix A]
2. evaluate pollution sources and abatement practices
3. evaluate self-monitoring procedures
4. analyze a process wastewater sample for toxic pollutants and organic compounds

The objectives of the August 7 to 12, 1978 survey were to:

1. measure flow from each of the cooling water discharges
2. determine if NPDES permit limitations were being met from three selected cooling water discharges
3. collect samples from these three cooling water discharges and the water intake for organic characterization.

In addition to determining the sources and types of toxic pollutants, NEIC evaluated the potential health effects of all organic compounds identified in wastewater and water intake samples.

II. SUMMARY AND CONCLUSIONS

SUMMARY OF INVESTIGATIONS

NEIC conducted an inspection of the UCSC facility in April 1978. During this inspection, each process operation was discussed in detail with Company personnel [Appendix A]. Evaluations were made of air, water and solid waste pollution sources and associated abatement practices. Self-monitoring procedures including sample collection, flow monitoring, sample analysis, bioassay procedures, and discharge monitoring reports (DMRs) were also evaluated. The inspection team collected a sample from the process wastewater for organic analyses in the NEIC Denver laboratory.

From August 7 to 12, 1978, NEIC personnel conducted a survey at the UCSC facility. Cooling water flows were measured using lithium chloride dilution procedures. Twenty-four hour composite samples of cooling water discharges and intake water were collected to determine compliance with NPDES effluent limitations, and to identify organic compounds. Each organic compound was searched in the Registry of Toxic Effects and Chemical Substances and the Toxline data bases to obtain toxic information.

CONCLUSIONS

Inspection

Air pollution control devices used at this facility appear to be adequate. Control devices include scrubbers, electrostatic precipitators, nitrogen blanketing and conservation vents on tanks, and collection

and burning of all combustible wastes. The air emission inventory provided by Company personnel show that pentane, acetone, isopropanol, methylacetate, methanol, diethylamine, butylchloride, propylene oxide and ethanol constitute the majority of the hydrocarbons emitted from this facility.

Solid wastes disposal in the Fillmont Landfill, Goff Mountain Chemical Landfill, and Holz pond appears to be adequate. According to Union Carbide officials, only non-chemical (lumber, paper, scrap polymer, etc.) solid wastes are sent to the Fillmont landfill. Hazardous chemical wastes are trucked to the Goff Mountain Chemical Landfill for disposal. Non-hazardous chemical wastes are pumped to Holz pond.

Sampling techniques were inadequate. The automatic sample containers are not routinely cleaned and some contained algae growth, flaking paint and solid accumulations. Samples are not refrigerated during collection. In addition, the water containers are sometimes full in less than 8 hours precluding the collection of a representative 24-hour sample.

In general, bioassay procedures were adequate. Discrepancies observed include: a) not starting tests within 8 hours as recommended by Standard Methods, b) using city dechlorinated tap water for dilution water instead of Kanawha River Water, c) not running tests in duplicate, d) aerating samples throughout the 96-hour test period. It is advisable, though not required, that the laboratory use a constant temperature water bath to maintain test temperature rather than depending on ambient air temperature.

Discharge monitoring reports, October through December 1977, show that the daily average and/or maximum TOC limitations were exceeded on five outfalls. Based on the sampling technique procedure discrepancies discussed above, these DMR data are questionable, probably low.

The process wastewater discharged to the South Charleston Sewage Treatment Company contains numerous organic chemicals and priority pollutants. A grab sample collected in April showed a total of 39 organic chemicals, including 14 priority pollutants:* benzene, chlorobenzene, 1,2-dichloroethane, chloroform, 1,2-dichlorobenzene, 1,1-dichloroethane, ethylbenzene, methylene chloride, bis (2-ethylhexyl) phthalate, 2,4-dinitrophenol, isophorone, di-N-butyl phthalate, tetrachloroethylene and toluene.

Survey

Total flow discharged by UCSC to the Kanawha River, based on one measurement from each outfall was about 457,000 m³/day. As required by the NPDES permit, Company personnel had previously measured the flows using the same techniques and found flows of approximately 402,000 m³/day.

Effluent data collected during the August survey show that UCSC exceeded permit limitations. Results show daily maximum net TOC concentrations on Outfall 025 ranged from 8 to 20 mg/l with 2 of 3 days exceeding the NPDES permit limitation (12 mg/l). The average net TOC concentration of 15 mg/l was almost 4 times greater than the permit limitation (4 mg/l). Projecting from these data, the daily average limitation would not be expected to be met. Maximum TKN loads were 11 kg/day, approximately 3% of the total plant limitation.

Outfalls 023 and 035 samples had net TOC concentrations of 2 mg/l or less which is within permit limitations. Maximum TKN loads were 8 and 11 kg/day respectively, less than 3% of the permit total plant limitation (460 kg/day).

* See explanation in Section III of priority pollutants.

During the NEIC survey, twelve organic compounds: butyl carbitol, pristane, benzene, bromodichloromethane, chlorobenzene chloroform, chlorodibromomethane, 1,2-dichloroethane, 1,1-dichloroethane, 1,2-dichloropropane, ethylbenzene, and methylene chloride were identified and confirmed in the Outfall 023, 025, 035 and water intake samples at low concentrations ranging from 1 to 82 $\mu\text{g/l}$. All except butyl carbitol and pristane are priority pollutants. Of these, benzene is a known carcinogen to man and chloroform is carcinogenic to animals.

III. INSPECTION METHODS AND RESULTS

During the April 4 to 6, 1978, inspection [Appendix A], NEIC personnel obtained information on process operation and associated pollution sources and self-monitoring procedures. Production rates and process schematics are not included because UCSC considers that information confidential.

POLLUTION SOURCES AND DISPOSAL METHODS

The Kanawha River is the source of process, cooling and boiler feed waters. All process and domestic wastewaters are discharged to the South Charleston Sewage Treatment Company (SCSTC) through an open redwood flume. UCSC had installed monitoring stations on their process sewer to measure flow and total carbon [Appendix A, Figure 2]. The pH, temperature and organics are measured at selected stations.

Once-through non-contact cooling water is discharged into the Kanawha River through 22 outfalls.* Two cooling water discharges (Outfalls 023 and 025) are equipped with organic spill detectors calibrated at 50 ppm isopropanol. If the organic concentration of the wastewater should exceed this value, an alarm is sounded. At that time a sample is collected and analyzed in the company laboratory to determine the compounds discharged.

Air pollution emissions are controlled by scrubbers, electrostatic precipitators, nitrogen blanketing and conservation vents on tanks, and collection and burning of all combustible waste gases. Union Carbide

* During the study cooling water was discharged from only 19 of these outfalls.

has installed 11 ambient air monitoring stations in and around the plant to detect leaks and/or equipment malfunctions. The air emission inventory provided by company personnel shows that pentane, acetone, isopropanol, methylacetate, methanol, diethylamine, butylchloride, propylene oxide and ethanol consistute the majority of the hydrocarbons emitted from this facility [Appendix A].

Solid wastes are disposed of in Goff Mountain Chemical landfill, Fillmont landfill, and Holz pond.* Hazardous chemical wastes and toxic substances are hauled to the Goff Mountain landfill. Landfill leachate, which is collected in an under-drainpipe system, is treated in the Union Carbide Institute (UCI) wastewater treatment facility. Non-hazardous chemical wastes, UCI and SCSTC waste-activated sludge and UCSC powerplant fly ash are pumped to Holz pond, an anaerobic lagoon. Overflow from this pond is treated at SCSTC. UCSC non-chemical solid wastes (lumber, paper, scrap polymer, etc.) are disposed of in the Fillmont landfill.

SELF-MONITORING EVALUATION

The evaluation of self-monitoring procedures consisted of interviews with UCSC sampling, analytical and bioassay personnel, and evaluations of sampling, monitoring and analytical equipment [Appendix A]. The findings of this evaluation are discussed below:

Company personnel analyzed standard reference TOC samples provided by NEIC [Appendix A]. TOC reference samples results were 3 to 12% lower than true value, which are acceptable. All chemical analyses are performed according to EPA-approved methods.

* Goff Mountain landfill is operated by Union Carbide Institute personnel. Fillmont landfill and Holz pond are operated by UCSC personnel.

Company personnel conduct bioassay testing quarterly as required by the NPDES permit. Discrepancies noted in their bioassay procedures include a) not starting tests within 8 hours as recommended by Standard Methods, b) using dechlorinated city water as dilution water instead of Kanawha River water, c) not running tests in duplicate, and d) aerating samples throughout the 96-hour test period. It is advisable, though not required, that the laboratory use a constant temperature water bath to maintain test temperature rather than depend on ambient air temperature.

The NPDES permit requires 24-hour flow composite samples. Company personnel have installed automatic samplers designed by their own personnel. Samples are collected starting at approximately 5:00 a.m. daily. Observations made during the initial inspection (April 4 to 6) and the NEIC survey (August 9 to 12) showed that within 7 to 8 hours some of the sample containers were full and others were over 2/3 full indicating an improper sampling rate. The samples collected are not 24-hour composites as required by the NPDES permit nor are they representative.

Other discrepancies noted include the lack of proper sample refrigeration during collection and sampling buckets which contained algae growth, flaking paint and an accumulation of solids. Company officials indicated that they do not have a routine maintenance program for the samplers.

The Discharge Monitoring Reports (DMR) [Appendix A, Tables 3 through 9] show that during the last quarter of 1977 the daily average and/or daily maximum TOC concentration (4 and 8 mg/l respectively) were exceeded on five outfalls.* Based on the discrepancies

* Composite samples from Outfalls 009, 014, 015, 016, 017, 024, 028, 031, 036, 039, 040, 042, 075 and 076 are composited into one sample and the limitation is based on the total discharge from all 16 outfalls. TOC violations are reported for these samples as well as samples from Outfalls 025, 032, 035 and 074.

discussed above, DMR data are considered to be questionable, i.e., probably low.

NEIC SAMPLE ANALYSIS

During the April reconnaissance survey, NEIC personnel collected a grab sample from the UCSC industrial/domestic effluent to SCSTC. This sample was analyzed for toxic pollutants and other organic compounds. A total of 39 organic chemicals, including 14 priority pollutants,* were identified. The priority pollutants were benzene, chlorobenzene, 1,2-dichloroethane, chloroform, 1,2-dichlorobenzene, 1,1-dichloroethylene, ethylbenzene, methylene chloride, 2,4-dinitrophenol, bis-(2-ethylhexyl) phthalate, isophorone, toluene, di-N-butyl phthalate, and tetrachloroethylene.

* Priority Pollutants are derived from the June 7, 1976 Natural Resources Defense Council (NRDC) vs. Russell Train (USEPA) Settlement Agreement.

IV. SURVEY METHODS AND RESULTS

During August 1978 NEIC personnel measured the cooling water discharge flow, determined compliance with NPDES permit [No. WV0000078] effluent limitations from three selected cooling water outfalls and characterized the wastewater discharged from these three outfalls and the water intake. Permit compliance was based on the following effluent limitations which have been in effect since May 1, 1977:

Parameter	Limitation	
	Daily Average	Daily Maximum
TOC mg/l (net)	4	12
Temp °C (°F)	NA	43.3 (110)
pH range		6-9

The permit also established net load limitations for the following parameters based on the total plant discharge:

Parameter	Limitation in kg/day (lb/day)	
	Daily Average	Daily Maximum
Chlorides	5,774 (12,718)	8,820 (19,427)
Phenolics	NA	NA
Dissolved Solids	NA	NA
Kjeldahl Nitrogen	233 (492)	460 (1,014)
Organic Nitrogen	198 (436)	396 (872)

Instantaneous flow is to be measured on each outfall once/year by the dye tracer technique. These measurements are then used to

determine flows for the next 12 months based on intake meter readings. In addition, the company is required to monitor Outfalls 023 and 025 twice/year for vinyl chloride monomers. Toxicity is to be monitored by bioassays conducted quarterly.

FLOW MEASUREMENT

The NPDES permit requires that flow be measured once/year on each outfall by the dye tracer technique. These measurements are then used for the next 12 months to determine flows from each outfall based on intake meter readings. During the period August 7 to 11, NEIC personnel measured the flow once from each cooling water discharge using lithium chloride as a tracer* [Appendix B].

NEIC results show that the discharge flow was approximately 456,700 m³/day [Table 1]. Company data collected one year prior to the NEIC data indicate the flow was about 10% less.

A comparison between intake meter readings and NEIC lithium flow results cannot be made as some intake flow meters were not operating during the study. Company personnel, however, estimated the total cooling water being discharged based on rated pump capacities and consumptive uses as follows:

9 pumps at 9500 gpm - 123 mgd
less water treated - 3.2 mgd
less water supplied to technical center - 1.7 mgd
total cooling water - 118.2 mgd or 447,000 m³/day

* Company personnel also use lithium chloride to measure flows from these outfalls.

Table 1
COOLING WATER FLOWS^a
UNION CARBIDE SOUTH CHARLESTON

Outfall No.	NEIC		Company	
	m ³ /day	mgd	m ³ /day	mgd
009	3,670	0.97	727	0.192
014	25,000	6.6	30,848	8.15
015	2,120	0.56	318	0.084
016	2,230	0.59	2,225	0.588
017	17,800	4.7	18,054	4.77
023	41,600	11	40,500	10.70
024	24,200	6.4	11,620	3.07
025	106,000	28	81,378	21.50
027	7,570	2.0	2,271	0.600
028	--	No Discharge	--	--
031	32,600	8.6	16,805	4.44
032	19,700	5.2	18,054	4.77
035	53,000	14	22,029	5.82
036	4,160	1.1	3,085	0.815
039	9,080	2.4	10,484	2.77
040	26,500	7.0	33,914	8.96
042	29,900	7.9	38,985	10.30
072	--	No Discharge	--	--
074	23,500	6.2	46,555	12.30
075	110	0.029	636	0.168
076	28,000	7.4	23,202	6.13
TOTAL	456,700	120.65	401,700	106.13

a Flows were measured using lithium chloride. Company data are approximately one year prior to NEIC measurements.

SAMPLING

Three cooling water discharges (Outfalls 023, 025 and 035) and the water intake were sampled August 9 to 12, 1978. The three cooling water discharges were selected by NEIC personnel based on volume discharged and past self-monitoring data which showed that discharges from Outfalls 025 and 035 were in violation of NPDES TOC limitations. Aliquots were manually collected every two hours and continually composited on an equal volume basis for total organic carbon, phenolic compounds (i.e., phenols), NH_3 , TKN and organics analyses. Grab samples were randomly collected three times per day for volatile organic analysis* (VOA). Temperature and pH were measured each time a sample was collected. Details on sampling and flow measurement procedures, Chain-of-Custody procedures, and analytical and quality control procedures are contained in Appendices B, C and D respectively.

Sampling results are summarized in Tables 2 and 3 and discussed by individual outfalls.

Outfall 023

The maximum net TOC concentrations in the cooling water discharged through Outfall 023 was 2 mg/l, 17% of the NPDES permit limitation (i.e., daily maximum of 12 mg/l). These data are somewhat lower than those reported in UCSC's self-monitoring data [Appendix A, Table 4], which show monthly maximum values ranging from 3 to 7 mg/l. The net TKN and NH_3 loads for the survey were each 8 kg (17 lb)/day.

Outfall 023 neutral extractable organics results showed that this discharge contained a few organic compounds with concentrations estimated at less than 2 ppb. Of these compounds only pristane could

* GC/MS analysis was requested only on an equal volume composite of those grab samples collected August 11, 1978.

Table 2
SUMMARY OF FIELD MEASUREMENTS AND ANALYTICAL DATA
UNION CARBIDE SOUTH CHARLESTON
August 9-11, 1978

Station Description	Date	Flow		pH range	Temperature Range °C	TOC		Phenol µg/l ^a	Chlorides			NH ₃ -N			TKN		
		m ³ /day	mgd			mg/l Gross	Net		mg/l Gross	kg/day Net	lb/day Net	mg/l Gross	kg/day Net	lb/day Net	mg/l Gross	kg/day Net	lb/day Net
Water Intake	8/09			6.9-7.4	23-25	8		<15	6			<0.2			0.5		
	8/10			7.2-7.6	23-26	7		<10	6			<0.2			0.6		
	8/11			7.2-7.9	24-25	8		<10	5			<0.2			0.5		
Outfall 023	8/09	41,600	11	6.7-7.7	28-31	8	<2	<10	6			0.2	8	18	0.7	8	18
	8/10	41,600	11	7.1-7.6	28-31	7	<2	<10	5			<0.2			0.5		
	8/11	41,600	11	7.1-7.7	27-30	10	2	<10	7	83	180	<0.2			0.7	8	18
Outfall 025	8/09	106,000	28	7.0-10.3	27-30	16	8	<15	29	2400	5400	<0.2			0.6	11	23
	8/10	106,000	28	6.6-7.4	27-30	25	18	<10	21	1600	3500	<0.2			0.4		
	8/11	106,000	28	7.0-7.5	28-30	28	20	<10	22	1800	4000	<0.2			0.6	11	23
Outfall 035	8/09	53,000	14	6.8-7.7	24-41	8	<2	<15	7	53	120	<0.2			0.7	11	23
	8/10	53,000	14	7.2-7.9	23-27	7	<2	<10	7	53	120	<0.2			0.7	5	12
	8/11	53,000	14	6.8-7.8	24-26	9	<2	<10	6	53	120	<0.2			0.4		

a Values reflect change in detection limit due to the small volume of samples collected.

Table 3
VOLATILE ORGANICS DATA^a
UNION CARBIDE SOUTH CHARLESTON COOLING WATER DISCHARGES

Station Description Compound	→ Outfall 023	Outfall 025	Outfall 035	Water Intake
	Concentration (µg/l)			
Acrolein	ND ^b	ND	ND	ND
Benzene	1	ND	1	ND
Bromodichloromethane	2	2	6	ND
Bromoform	ND	ND	ND	ND
Carbon tetrachloride	ND	ND	ND	ND
Chlorobenzene	ND	2	ND	ND
2-Chloroethylvinyl ether	ND	ND	ND	ND
Chloroform	8	7	25	2
Chlorodibromomethane	ND	1	2	ND
1,2-Dichloroethane	3	2	ND	5
1,1-Dichloroethene	ND	8	ND	ND
trans-1,2-Dichloroethene	ND	ND	ND	ND
1,2-Dichloropropane	ND	2	ND	ND
Ethylbenzene	ND	ND	ND	4
Methylene chloride	ND	ND	82	14
1,1,2,2-Tetrachloroethane	ND	ND	ND	ND
Tetrachloroethene	ND	ND	ND	ND
Toluene	ND	ND	ND	ND
1,1,1-Trichloroethane	ND	ND	ND	ND
1,1,2-Trichloroethane	ND	ND	ND	ND
Trichloroethene	ND	ND	ND	ND
Vinyl Chloride	ND	ND	ND	ND

a Equal volume composite of three grab samples collected August 11, 1978.

b ND - none detected. Detection limit 1 µg/l for all components except acrolein, which has a detection limit of 50 µg/l.

be identified. The concentration of pristane, however, was less than the detection limit (1 $\mu\text{g/l}$).

VOA data [Table 3] show the cooling water contained 4 organic compounds (benzene, bromodichloromethane, chloroform and 1,2-dichloroethane) at low concentrations ranging from 1 to 8 $\mu\text{g/l}$. The intake water contained 1,2-dichloroethane at a higher concentration (5 $\mu\text{g/l}$) than the discharge (3 $\mu\text{g/l}$). These volatile compounds are priority pollutants.*

Outfall 025

The daily maximum TOC limitation on Outfall 025 was exceeded. The net TOC concentrations ranged from 8 to 20 mg/l [Table 1] with two out of the three days exceeding the NPDES limitation (i.e., 12 mg/l) by 50 and 67% DMR data for the last quarter of 1977 show that this outfall exceeded the maximum limitation by 2.3 times in November.

The average net TOC concentration for the three-day survey was 15 mg/l , almost 4 times greater than allowed by the NPDES permit (i.e., daily average of 4 mg/l). The NPDES permit defines the daily average limitation as the arithmetic average of all the daily determinations made during a calendar month. Because samples were only collected for 3 days, compliance with the average cannot be determined. Survey results, however, indicate that the daily average TOC limitation would not be met. DMR data show that the monthly average concentration for December was 5 mg/l , or 25% greater than the permit limitation (4 mg/l).

Numerous organic compounds were observed in Outfall 025 samples. Concentrations, however, were estimated to be below 5 ppb except for

* Listed in Consent Agreement, Natural Resources Defense Council vs. Russell E. Trains, June 1976.

two compounds which were estimated at less than 10 ppb. Some of the compounds were halogenated oxygenated aliphatics but could not be specifically identified. Two compounds, butyl carbitol and pristane, were identified but, due either to interferences or difficulties in correlation to the flame ionization chromatogram, could not be quantified.

The volatile organic analyses showed that this discharge contained bromodichloromethane, chlorobenzene, chloroform, chlorodibromomethane, 1,2-dichloroethane, 1,1-dichloroethene and 1,2-dichloropropane at low concentrations ranging from 1 to 8 µg/l [Table 3]. All of these compounds are priority pollutants.

Outfall 035

During the survey, the gross TOC, NH₃ and TKN concentrations at this outfall ranged from 7 to 9 mg/l, <0.2* mg/l and 0.4 to 0.7 mg/l, respectively. These concentrations are similar to those found in the intake water (7 to 8 mg/l TOC, <0.2* mg/l NH₃ and 0.5 to 0.6 mg/l TKN). DMR data for October, November, and December 1977 show maximum TOC concentrations of 21, 10 and 39 mg/l, respectively [Appendix A]. The NPDES limitation (12 mg/l) was exceeded during October and December.

Samples collected from this outfall contained several neutral extractable organics compounds with concentrations less than 2 ppb. None of these compounds, however, could be identified by GC/MS. Volatile organic samples contained benzene, bromodichloromethane, chloroform, chlorodibromomethane and methyl chloride at concentrations of 1, 6, 25, 2 and 82 µg/l, respectively. These compounds are also on the priority pollutant list.

* All values were the same.

Water Intake

As previously noted, the water intake contained small concentrations of TOC (7 to 8 mg/l), chlorides (5 to 6 mg/l), NH_3 (<0.2 mg/l) and TKN (0.5 to 0.6 mg/l). Self-monitoring data for the last quarter of 1977 are similar to those obtained during the survey.

At least eight isomers of bis (C_6) phthalic acid esters were identified but could not be verified in the intake water composite samples. Concentrations were estimated as less than 10 ppb for all isomers. Pristane was also identified in the intake samples but, due to interfering compounds and/or difficulty in correlation to the flame ionization chromatogram, could not be quantified. Volatile organic analysis showed that the intake water contained small amounts of chloroform, 1,2-dichloroethane, ethylbenzene, and methylene chloride (2, 5, 4 and 14 $\mu\text{g/l}$, respectively), all of which are priority pollutants.

TOXICITY EVALUATION

The twelve organic compounds identified in the cooling water and water intake samples were searched in the Registry of Toxic Effects of Chemical Substances (RTECS)* and in the Toxline** database to obtain health effects data [Appendix E].

The RTECS search yielded information on 10 to 12 compounds [Table 4]. The Toxline search yielded 205 references to human health effects from the 10 compounds, providing support to the toxic data from RTECS.

* This Registry is compiled annually by the National Institute for Occupational Safety and Health.

** Toxline is a computerized bibliographic retrieval system for toxicology.

No information on toxic and health effects of dichlorobromomethane and chlorodibromomethane was available in either RTECS or Toxline. However, it should be noted that bromodichloromethane was included in the National Cancer Institute's Carcinogenesis Bioassay program as of February 1978.

As previously noted, the concentrations of these compounds ranged from 1 to 82 $\mu\text{g/l}$ [Table 3]. Seven of the 12 compounds identified have demonstrated human effects associated with them [Table 4]. The hazards of ingesting minute quantities of these organic pollutants in drinking water over long periods of time are difficult to evaluate. From the standpoint of adverse health effects, three of the compounds are known carcinogens. Benzene is carcinogenic to man and chloroform to animals.

APPENDIX A

UNION CARBIDE SOUTH CHARLESTON
(INSPECTION)
April 1978

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ATTACHMENTS

- A - Company Organic Chemical Monitoring
- B - Organic Chemicals Screening Analyses
- C - Bioassay Procedures Evaluation
- D - Chemical Laboratory Procedures Evaluation

I. INTRODUCTION

Union Carbide Corporation, Chemicals and Plastics Division (NPDES Permit No. WV0000078), operates a chemical manufacturing facility at South Charleston, West Virginia producing approximately 400 different chemicals and mixtures. The plant operates 24 hr/day, year around and employs 1,700 people.

The Environmental Protection Agency, Region III, requested that the National Enforcement Investigations Center (NEIC) inspect the Union Carbide facility to: a) determine the sources and types of toxic pollutants* discharged, b) evaluate pollution abatement practices, and c) determine if NPDES permit requirements were being met.

On April 4, 5 and 6, 1978, Dr. Wayne C. Smith, Mr. James L. Hatheway, Mr. Bruce A. Binkley and Mr. D. David Vietti of NEIC visited the plant to inspect process operations, probable pollution sources, waste disposal practices and pollution abatement practices and to evaluate self-monitoring procedures which included sampling, flow measurement, analytical and bioassay procedures used. The Company, represented by Mr. J.L. Worstell, provided information and assistance.

* Toxic Pollutant List published January 31, 1978 in Federal Register Vol. 43, No. 21.

II. PROCESS OPERATIONS AND POLLUTION SOURCES

Union Carbide produces approximately 400 different chemicals and mixtures that include about fifty major products. These 50 products and about 700 other chemicals are used to formulate the balance of the products. Production rates and most process schematics are considered confidential. Figure 1 shows the major production units, products and raw materials for this plant.

Kanawha River water, about 340,000 m³/day (90 mgd), is used for non-contact cooling and process water. The non-contact cooling water is discharged, untreated, to the river through 22 NPDES-permitted outfalls. The Company has no NPDES limits for the process wastewater. The two major cooling water outfalls (023 and 025) are continuously monitored for specific organics. If the organics concentration exceeds 50 ppm (calibrated as isopropanol), a grab sample of this cooling water is collected and analyzed to determine what process area is causing the problem. NPDES limitations [Table 1] for these discharges were effective May 1, 1977. Approximately 18,000 m³/day (5 mgd) process wastewater, domestic waste and floor washings, are collected and discharged to the South Charleston Sewage Treatment Company (SCSTC) for treatment. Union Carbide has an extensive total carbon monitoring system (21 analyzers) on the process sewers [Table 2 and Figure 2]. These total carbon analyzers are used to detect process upsets and for treatment billing costs to the individual processes.

Solid waste is disposed in one of two landfills or in an on-site lagoon. Non-chemical (lumber, paper, scrap polymer, etc.) solid wastes are disposed in the Company owned and operated Fillmont landfill (State approved). Chemical solid waste is sent to the Goff Mountain landfill

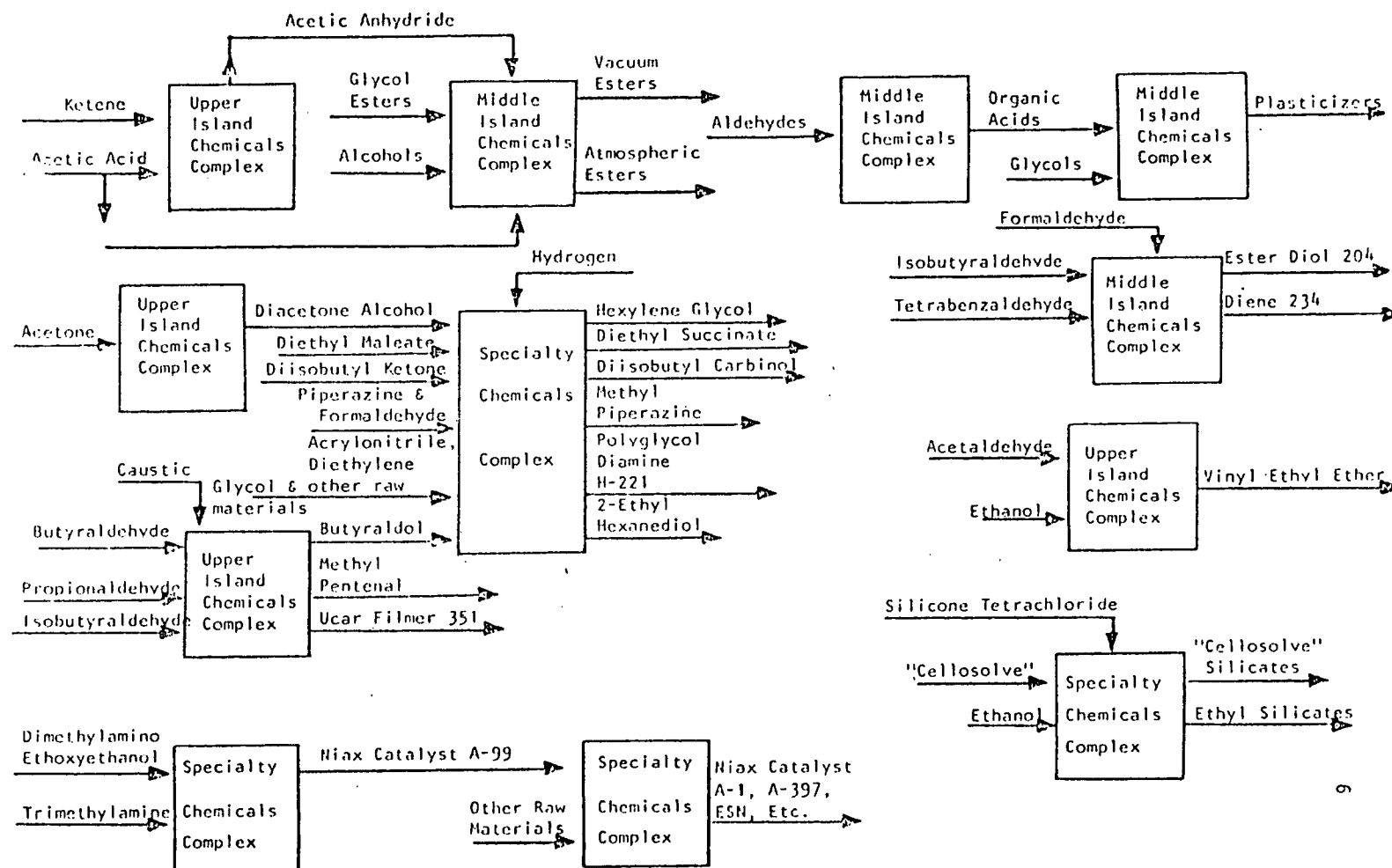


Figure 1. Major Production Units, Products and Raw Materials
South Charleston Plant

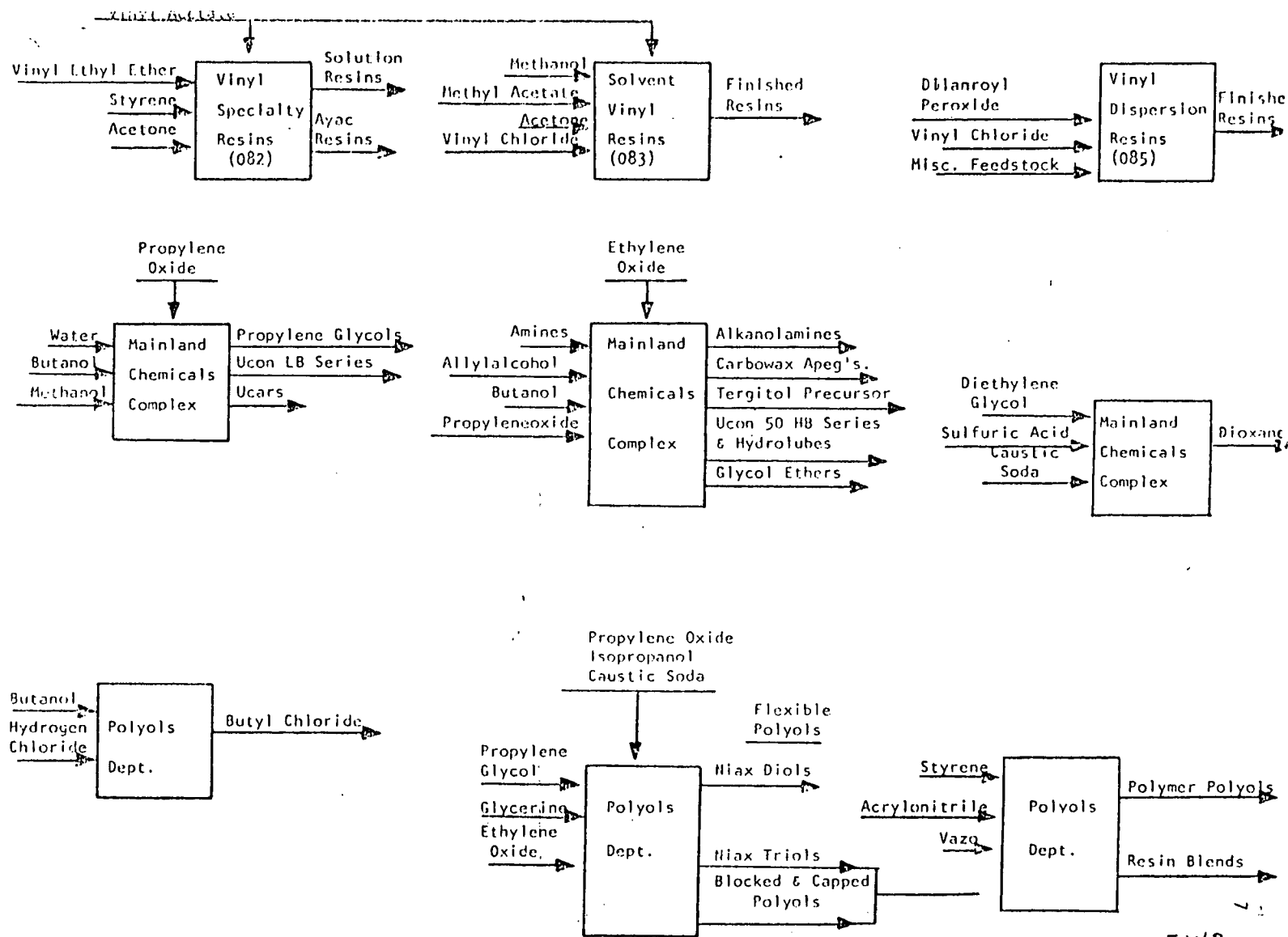


Figure 1. (Cont.)
Major Production Units, Products and Raw Materials
South Charleston Plant

Table 1
NPDES PERMIT LIMITATIONS
UNION CARBIDE
SOUTH CHARLESTON, WEST VIRGINIA

Parameter	Net Discharge Limitations		Monitoring Requirements	
	Daily Avg.	Daily Max.	Measurement Frequency	Sample Type
	kg/day (lb/day)			
Flow m ³ /day (mgd)		NA	a	
TOC	4 mg/l	12 mg/l	5/week	24 hr. com. ^b
Temperature	N/A	43.3 (110)	1/week	Instantaneous
Vinyl Chloride Monomer		N/A	2/year	24 hr. com.
pH (range)		6.0-9.0	1/week	Grab
Other	There shall be no discharge of floating solids or visible foam in other than trace amounts.			

a Outfalls 009, 014, 015, 016, 017, 023, 024, 025, 027, 028, 031, 035, 036, 039, 040, 042, 072, 074, 075, 076. Flow measurements shall be made once a year by the dye tracer test method on each of the twenty-two outfalls. These measurements will then be used to determine the relative flows of the twenty-two outfalls for the next twelve months. Each month, the flow for any of the individual outfalls shall be determined from (a) the plant cooling water intake for that month (which is metered) and (b) relative outfall flowrates as determined from the last dye tracer test measurements.

From May 1, 1977, until the expiration date, TOC analyses shall be made using 24-hour composite samples for 4 days of the week, and a 72-hour composite sample for the remaining 3 days of the week.

Beginning on the effective date of the permit and continuing through December 31, 1977, the Environmental Protection Agency and the permittee will jointly investigate the qualitative and quantitative presence of organic chemicals in the outfalls using analytical techniques and instruments appropriate to the probable constituents and their expected concentration ranges in the discharges. Beginning on January 1, 1978, the permittee shall initiate monitoring and reporting for such specific organic chemicals at such frequency as jointly determined to be appropriate in the prior testing period.

Quarterly the permittee shall determine the 96-hour median tolerance limit applicable to the fathead minnow (*Pimephales promelas*) using the latest EPA approved static bioassay procedures and 24-hour composite samples from the outfalls as indicated below. The results of the bioassay tests shall be reported quarterly to the Environmental Protection Agency, Region III, and to the State of West Virginia, Department of Natural Resources, Division of Water Resources. Separate tests shall be conducted for 24-hour composite samples from outfalls 023, 025, 035, 072 and 074, and a single test shall be conducted on a flow-weighted aggregate prepared from 24-hour composite samples from outfalls 009, 014, 015, 016, 017, 024, 027, 028, 031, 036, 039, 040, 042, 075, and 076.

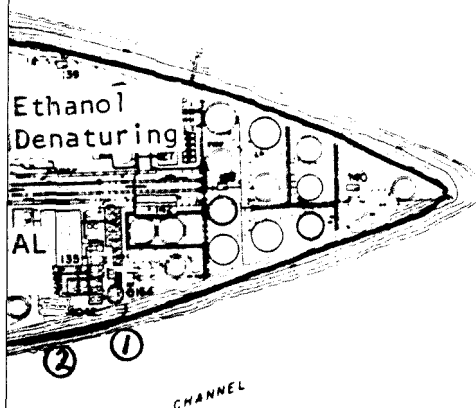
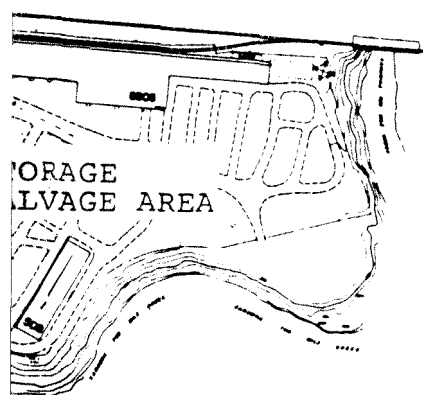
b Composite

Table 2
 WASTEWATER MONITORING STATIONS
 UNION CARBIDE CORPORATION
 SOUTH CHARLESTON PLANT
 South Charleston, West Virginia

Station No.	Area Monitored	Parameters Monitored
1	UICC	Flow, TC ^{a,b}
2	UICC	Flow, TC ^{a,b}
3	Distribution	Flow, TC ^b
4	UICC	Flow, TC ^{a,b}
5	EPD (Main Flume)	Flow, TC ^{a,b}
6	MICC	pH Flow, TC ^{a,b}
7	MICC	pH Flow, TC ^{a,b}
8	EPD	Flow, TC ^b
9	EPD (Main Flume)	Flow, TC ^{a,b}
10	EPD (Main Flume)	pH Flow, TC ^{a,b}
11	Polyols	pH Flow, TC ^b
12	Chemical Mixing	Flow, TC ^b
13	Solvent Vinyl Resins	Flow, TC ^b
14	EPD (Main Flume)	Flow, TC ^{a,b} Specific Organics Analyzer (SOA), pH, Temperature
15	Distribution	Flow, TC ^b
16	Specialty Chemicals LPH	Flow, TC ^b
17	Specialty Chemicals Moderinzation	Flow, TC ^b
18	Specialty Chemicals	Flow, TC ^{a,b}
19	Polyols	Flow, TC ^{a,b}
20	Mainland Chemicals	Flow, TC ^{a,b} pH
21	EPD (Holz Return)	Flow
22	Tech Center	Flow
23	SCWTW	Flow, TC ^{a,b} pH, Biomonitor, Temperature

a Continuous Total Carbon Analysis

b Daily Composite Total Carbon Analysis



MONITORING STATIONS			
#	AREA MONITORED	#	AREA MONITORED
1	UPPER ISLAND CHEMICAL COMPLEX	13	SOLVENT VINYL RESINS
2	U I C C	14	EPD (MAIN FLUME)
3	DISTRIBUTION	15	DISTRIBUTION
4	U I C C	16	SPECIALTY CHEMICALS, LPH
5	EPD (MAIN FLUME)	17	SPECIALTY CHEMICALS MODERNIZATION
6	MIDDLE ISLAND CHEMICAL COMPLEX	18	SPECIALTY CHEMICALS
7	M I C C	19	POLYOLS (NEW)
8	EPD	20	MAINLAND CHEMICALS
9	EPD (MAIN FLUME)	21	EPD (HOLD RETURN)
10	EPD (MAIN FLUME)	22	TECH. CENTER
11	POLYOLS	23	SCW T W
12	CHEMICAL MIXING		

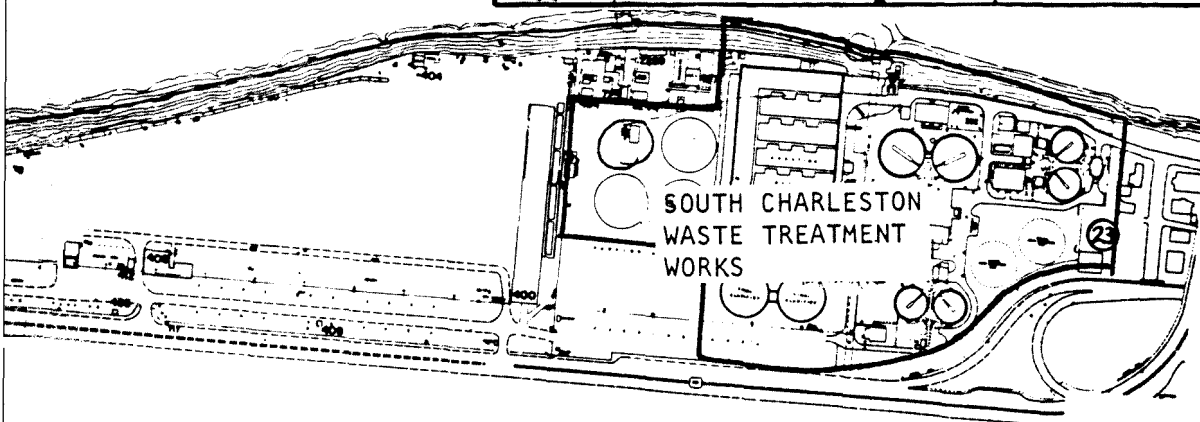
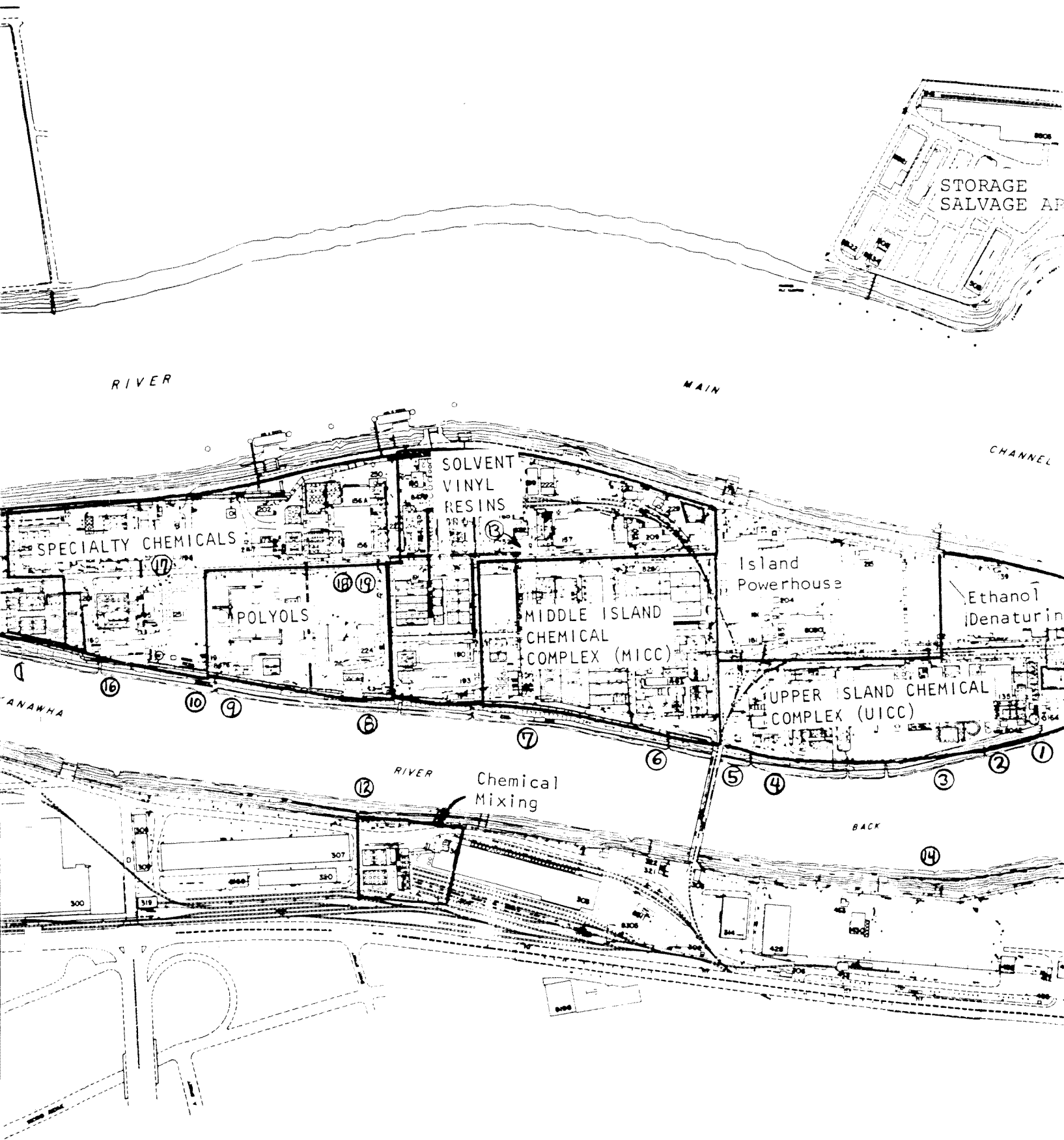
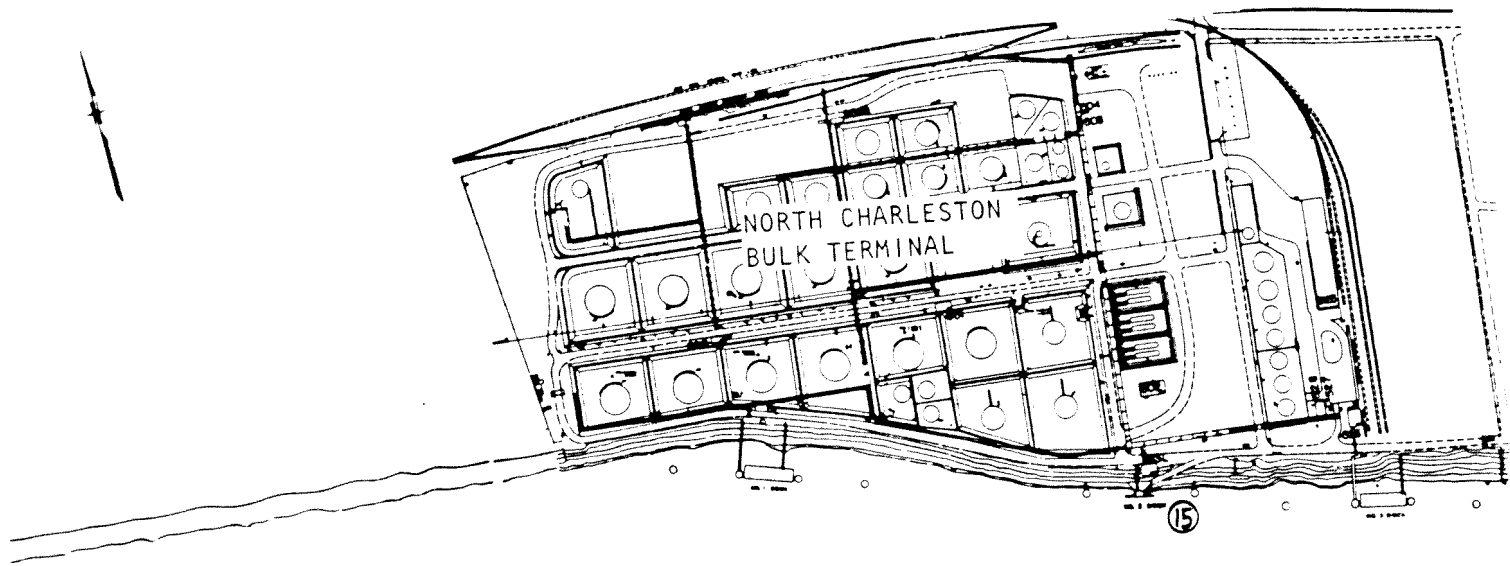
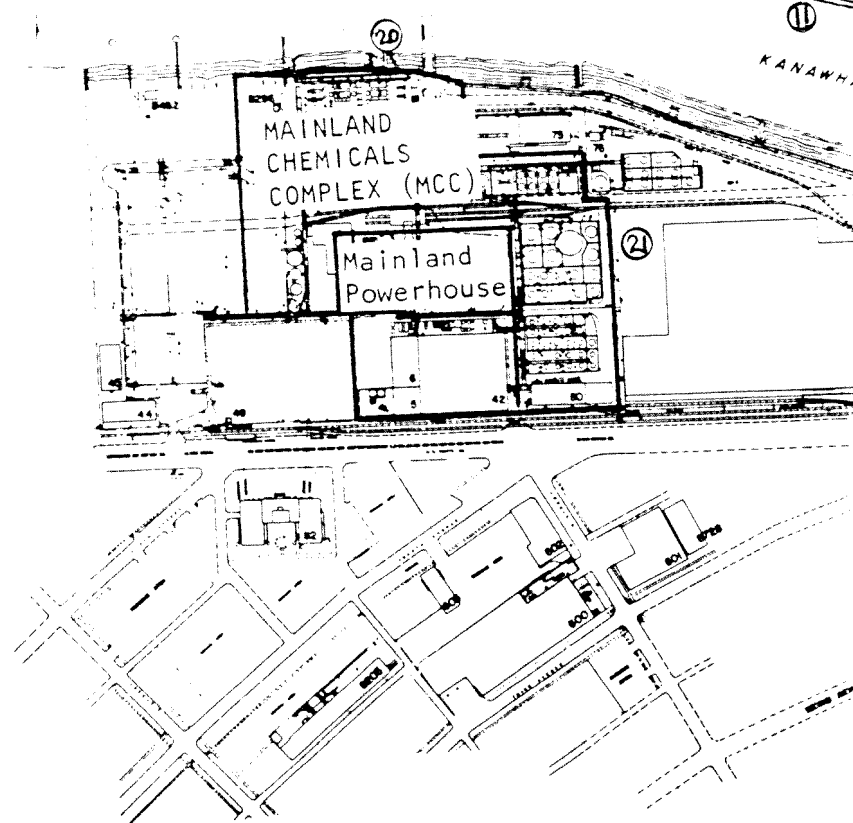
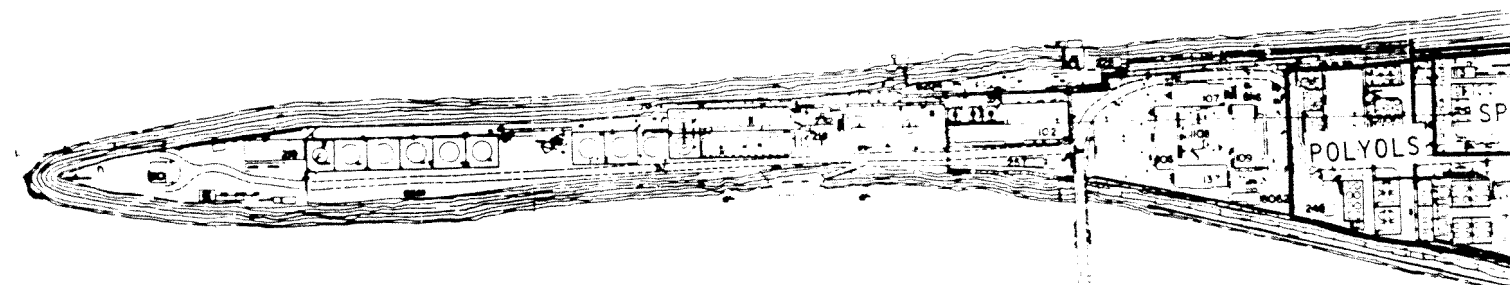


Figure 2. Plot Plan - South Charleston Plant
Union Carbide Corporation





KANAWHA



which is owned by Union Carbide and operated by the Institute facility. Flyash, secondary solids from the SCWWTF and the Institute wastewater treatment facility sludge are lagooned in Holz Pond.

POLYVINYL ACETATE, SOLUTION RESINS AND AYAC RESINS PRODUCTION

Polyvinyl acetate, solution resins and Ayac (chewing gum) resins are produced by a process considered confidential. Process equipment includes a reactor product drying system, packaging system and a solvent recovery system. The solvent recovery system is a common system located in the solvent-vinyl-resins area.

Wastewater from the solvent recovery system is discharged to the process sewer and all non-contact cooling water is discharged to the river through Outfall 023.

Some scrap polyvinyl acetate is disposed, once every six months in the Fillmont Landfill. There are no other solid waste or air emissions sources from this process. Solvent storage tanks are nitrogen blanketed and have conservation vents.

2-ETHYL HEXANOIC ACID PRODUCTION

The product 2-ethyl hexanoic acid is formed by reacting 2-ethyl hexaldehyde with oxygen. The crude acid is refined in vacuum distillation columns. This product is produced on a semi-continuous basis 7 times per year for about 20 days per run. The process schematic is considered confidential.

At the time of the inspection, two vacuum jets were used on the distillation columns and the wastewater from the jets was discharged to the process sewer. Company officials stated that the two jets

would be replaced by surface condensers by June 1978. At that time, the unit would use only non-contact cooling water that would discharge through Outfall 027.

Air emissions are the inerts from the vacuum jets; however these emissions will be eliminated in June.

2-DIMETHYL AMINO ETHANOL AND OTHER ALKANOL AMINES PRODUCTION

The products 2-dimethyl amino ethanol and other alkanol amines are formed by reacting oxygen with dimethylamine in a reactor and refining unit. This process schematic is considered confidential.

Wastewater sources include the reactor wash water and the reactor scrubber water. These wastewaters contain about 90 kg (200 lb)/day of TOC and are discharged to the process sewer. Company officials stated that the reactor water scrubber will be replaced with an acetic acid scrubber by September 1978. The scrubbing media will be burned in the powerhouse, thus eliminating 68 kg (150 lb)/day of TOC from this process. Non-contact cooling water, 28 m³/min (7,500 gpm), is discharged through Outfall 074.

The air emissions from the reactor scrubber, containing amines, are discharged to the atmosphere.

PROPYLENE AND DIPROPYLENE GLYCOL PRODUCTION

Propylene and dipropylene glycol are produced continuously in a reactor and refining system by reacting propylene oxide and water.

Wastewater, 142 m³ (37,600 gal)/day, from the reactor, containing 86 kg (190 lb)/day of TOC is discharged along with vacuum jet water to the process sewer.

The only air emission source is the vacuum jet off-gas. Residue from the refining system is burned at the powerhouse.

ACETIC ANHYDRIDE PRODUCTION

Acetic anhydride and the ketene by-product are produced by thermal cracking acetic acid in the presence of a triethylphosphate catalyst. Equipment includes thermal cracking furnaces and a refining system.

Wastewaters from the cracking furnace clean-out and area cleanup are discharged to the process sewer. Non-contact cooling water is discharged through Outfalls 035 and 036. Residue from the refining unit are burned at the Island powerhouse. There are no air emission or solid waste sources.

DIACETONE ALCOHOL PRODUCTION

Diacetone alcohol is produced by a catalytic reaction of acetone with acetone [Figure 3].

Vacuum jet water and area wash-up are discharged to the process sewer. Non-contact cooling water is discharged through Outfalls 040 and 042.

The only air emissions are from storage and mixing tanks [Figure 3]. These emissions are controlled by conservation vents. Distillation column residue is burned at the Island powerhouse and the metal catalyst recovered and returned to the vendor.

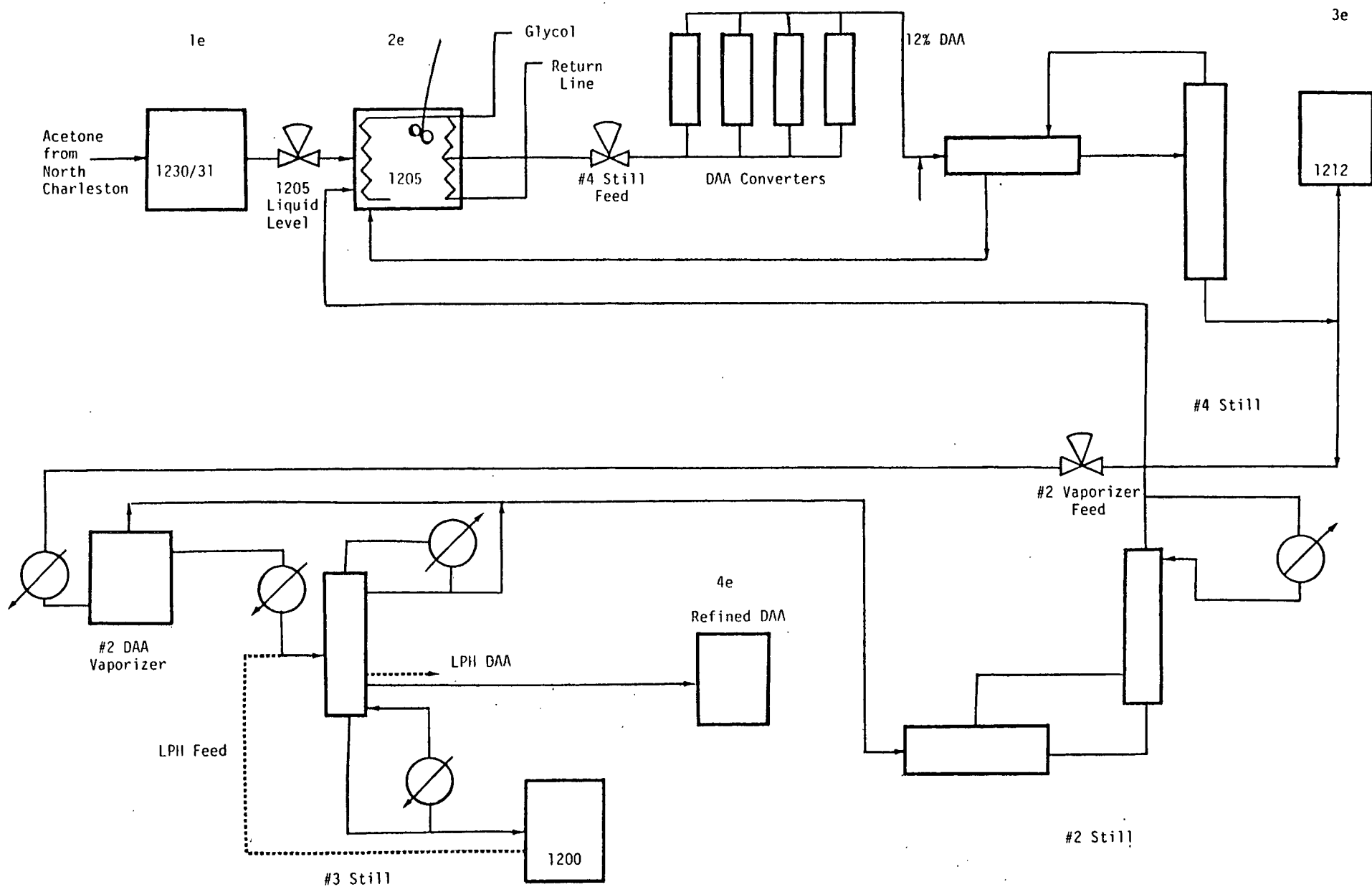


Figure 3. Diacetone Alcohol Schematic

VINYL ACETATE-VINYL CHLORIDE COPOLYMERS AND FINISHED RESINS PRODUCTION

Approximately 10 to 15 different vinyl acetate-vinyl chloride copolymers, terpolymers and finished resins are made in these processes [Figures 4 and 5]. Raw materials for these processes are methanol, methyl acetate, acetone and vinyl chloride.

Wastewaters discharged to the process sewer from the No. 1 and No. 2 solvent vinyl resin units [Figures 4 and 5] include the vinyl chloride vent scrubber media, slurry tank filter wash and still contents during an upset. During a No. 1 unit still upset, vinyl chloride at 400 ppm/lb product could be discharged to the process sewer; however Company officials stated that most of the vinyl chloride would go to the atmosphere. Non-contact cooling water is discharged through Outfalls 023 and 025.

Air emission sources include the vinyl chloride vent scrubber, slurry tanks, centrifuge, resin collector and storage areas, cyclones, stills, storage tanks and baghouses. The air emissions inventory* indicates that as much as 135 kg (300 lb)/hr acetone, 3.6 kg (8 lb)/hr vinyl acetate, 3.6 kg (8 lb)/hr acetaldehyde, 13.5 kg (30 lb)/hr isopropanol 3.7 kg (7 lb)/hr vinyl chloride, 132 kg (290 lb)/hr methyl acetate and 75 kg (165 lb)/hr methanol are emitted during normal operations.

Solid scrap product is hauled to the Fillmont landfill.

NIAX POLYOLS PRODUCTION

The Niox polyols units (2) are used to produce Niox polyols, Niox diols (flexible), Niox triols, blocked and capped polyols, polymer

* Data source is a UCSC-developed air emissions inventory. All air emissions discussion is based on this inventory.

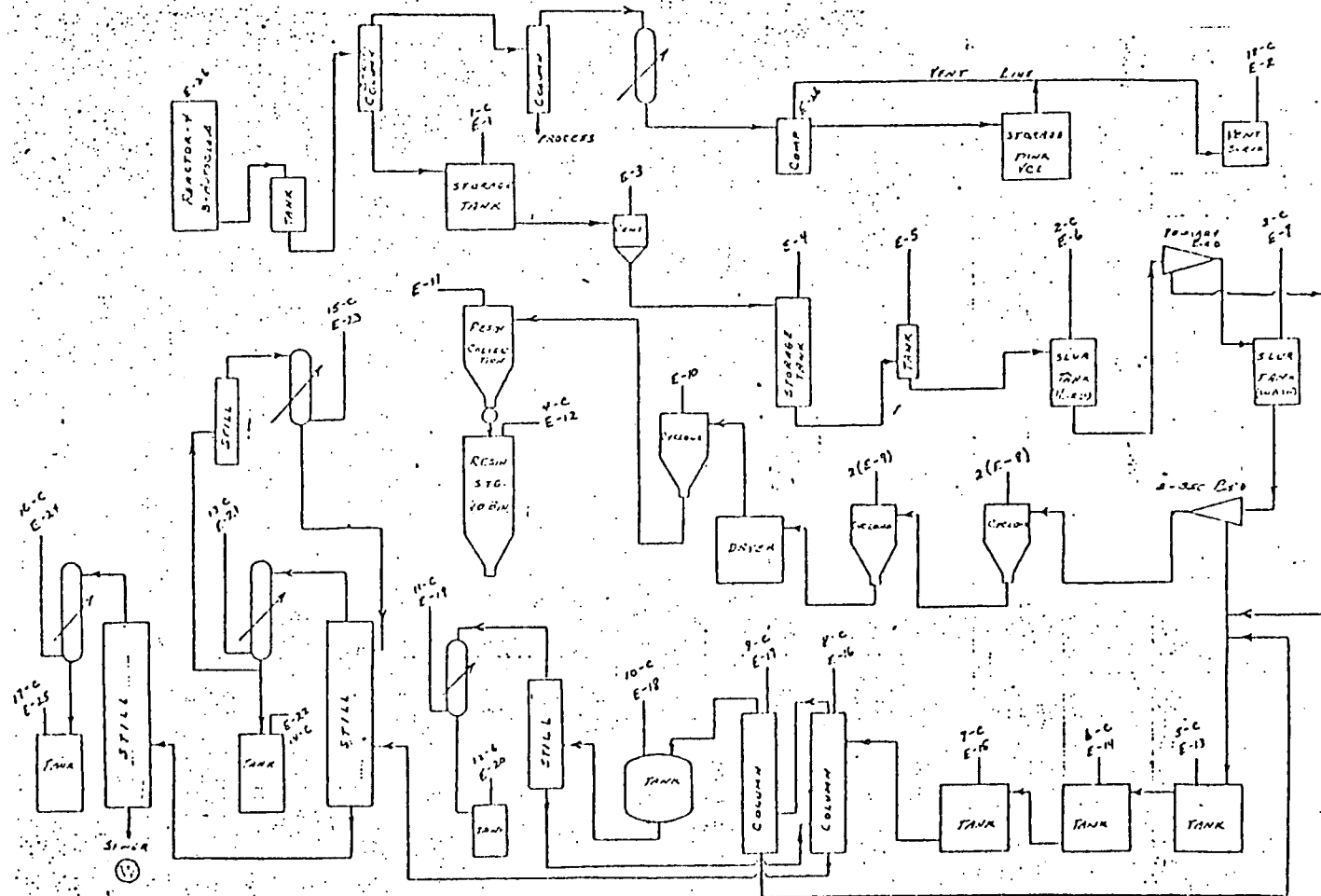


Figure 4. South Charleston No. 1 Solvent Vinyl Resins Schematic

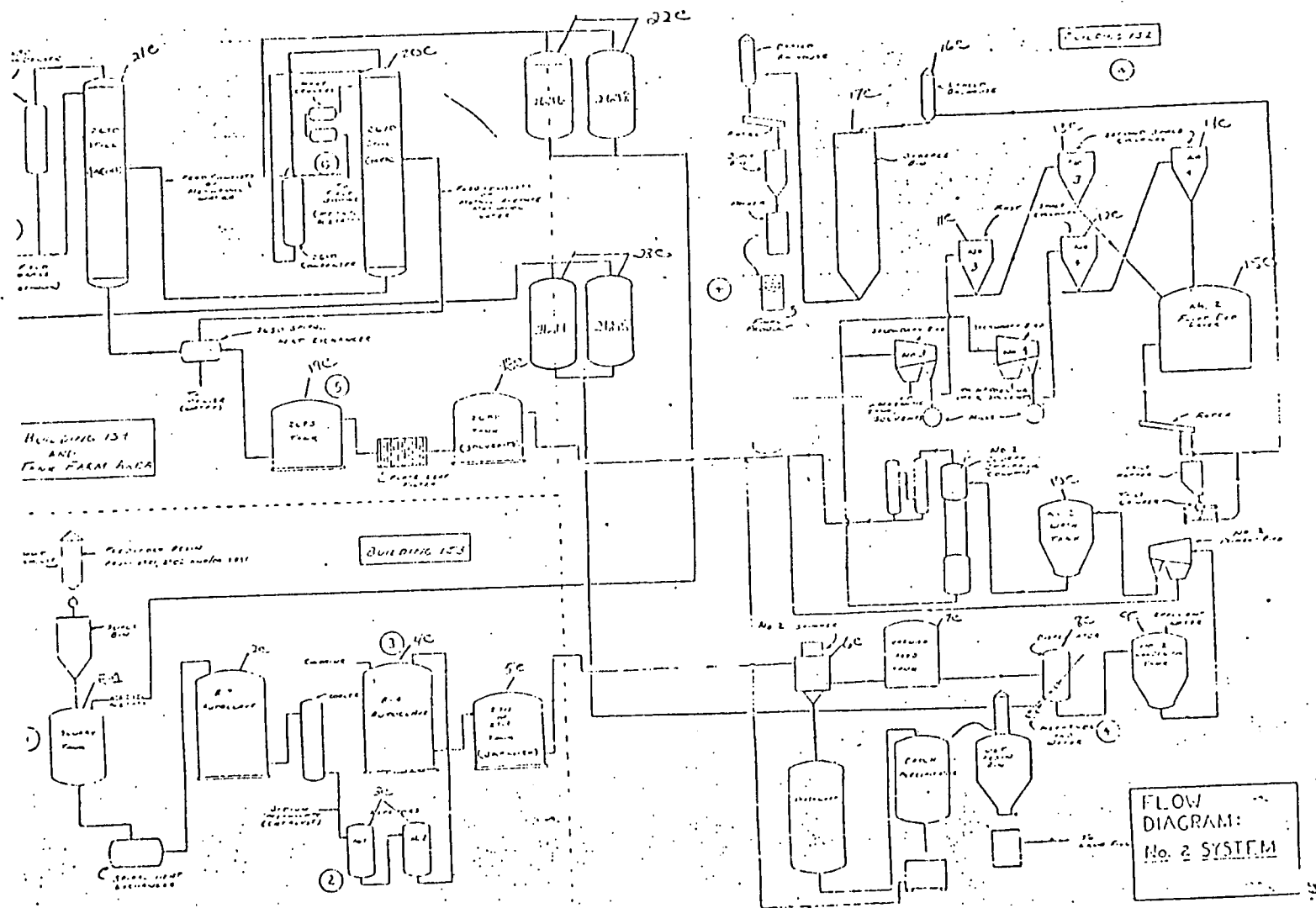


Figure 5. Solvent Vinyl Resins Unit #2 Schematic

polyols and resin blends. Polyols contain styrene and acrylonitrile.

Wastewaters from the vacuum jets and area clean-up are discharged to the process sewer. Non-contact cooling water and tank farm runoff are discharged through Outfalls 014, 015 and 023.

Air emissions from the reactors and the jet system are discharged uncontrolled. All storage tanks emissions are controlled by conservation vents. The air emissions inventory indicates that 90 kg (200 lb)/hr of propylene oxide and 1.4 kg (3 lb)/hr acrylonitrile are emitted from these sources.

Semi-liquid residue waste is put into containers and hauled to the powerhouse for burning.

HEXANE GLYCOLS PRODUCTION

Hexane glycols are produced in batches on an infrequent basis by hydrogenation of diacetone alcohol. Company officials stated that one more batch of hexane glycol would be produced in late 1978. The only wastewater source, the vacuum jet water, is discharged to the process sewer.

All water and residue from the distillation unit is burned in the powerhouse. There are no air emission or solid waste sources in this process.

ORGANIC ACID AND HYDROGENATED CROTON OIL ALCOHOL PRODUCTION

Organic acids (C_4 , C_5 and C_8) are produced by the oxidation of organic aldehydes (C_4 , C_5 and C_8) and purified by vacuum distillation. Company officials stated that organic acid production would

stop by mid 1979. Hydrogenated croton oil alcohol is received from the Institute plant and refined in the vacuum distillation unit.

Wastewaters from the vacuum jets and distillation column and storage tanks clean-up are discharged to the process sewer. Non-contact cooling water is discharged through Outfall 028.

The only air emission sources are the vacuum jet inerts. All distillation residues are burned in the powerhouse.

UCON AND UCAR PRODUCTION

Ucon and Ucar, Tergitol precursor and Ucon HB 50 series polymers are made using ethylene and propylene oxide, butanol, diethylene glycol and isopropanol.

The ion exchange unit beds used in the process are flushed and regenerated once per week with sulfuric acid and sodium hydroxide. This is the only wastewater from the system and it is discharged to the process sewer. Non-contact cooling water is discharged through Outfall 075.

This system operates under pressure; therefore the only possible air emission sources are from pressure relief valves on the equipment.

Ion-exchange bed filter paper is disposed in the Union Carbide Goff Mountain Chemical Landfill. No other solid waste is generated.

BUTRALDOL AND METHYL PENTENAL PRODUCTION

Butraldol and methyl pentenal are produced from butryaldehyde, propionaldehyde, isobutryaldehyde and caustic. Equipment includes an oxidizer and vacuum distillation unit.

Water from the vacuum jets and area cleaning is discharged to the process sewer. Non-contact cooling water is discharged through Outfall 032.

Emissions from the oxidizer vent and the distillation residues are burned at the powerhouse.

VINYL METHYL ETHER PRODUCTION

Vinyl methyl ether is produced [Figure 6] by reacting acetaldehyde and methanol. Vacuum for the vacuum distillation is created by a barometric condenser.

The barometric condenser, other process and clean-up waters are discharged to the process sewer. Non-contact cooling water is discharged through Outfall 039.

All air emission sources are controlled by pressure relief valves. Distillation residue is burned in the powerhouse.

VACUUM AND ATMOSPHERIC ESTER PRODUCTION

Vacuum (high molecular weight) and atmospheric (low molecular weight) esters are produced by esterification and distillation of glycol esters, alcohols and acetic anhydride.

Vacuum jets and esterification reaction waters are discharged to the process sewer. Non-contact cooling water is discharged through Outfall 031.

The only air emission source is the inerts discharged from the vacuum jets. Distillation residues are burned in the Island powerhouse.

PLASTICIZER PRODUCTION

A variety of plasticizers are made from organic acids in a confidential process that includes vacuum distillation. The vacuum jet water, the only process wastewater source, is discharged to the process sewer and the cooling water is discharged through Outfall 023.

Air emissions are the inerts from the vacuum jets. Distillation residues are collected and hauled to the powerhouse for burning.

ESTER DIOL 204 AND DIENE 234 PRODUCTION

Ester Diol 204 and Diene 234 are produced by reacting formaldehyde, isobutyraldehyde and tetrabenzaldehyde followed by vacuum distillation for refining.

The process wastewater from the vacuum jets is discharged to the process sewer and the non-contact cooling water is discharged through Outfall 028.

The vacuum jet inerts are the only emissions from the units. Distillation residues are burned at the Island powerhouse.

DIISOBUTYL CARBINOL AND 2-ETHYL HEXANE DIOL PRODUCTION

Diisobutyl carbinol and 2-ethyl hexane diol are produced by hydrogenation of diisobutyl ketone and butyraldehyde followed by refining in a batch vacuum distillation unit.

The vacuum jet water and process equipment wash waters are discharged to the process sewer. Non-contact cooling water is discharged through Outfall 017. The air emission sources are the vacuum jets and the storage tanks.

The vacuum jets discharge directly to the atmosphere and the storage tank emissions are controlled by nitrogen blanketing. Distillation column bottoms, water and residue, are burned in the powerhouse.

POLYGLYCOL-DIAMINE PRODUCTION

Polyglycols and diamines are produced by reacting various diamines, diethylene glycol, hydrogen and acrylonitrile. The reaction is followed by vacuum distillation and hydrogenation.

Wastewater sources include vacuum jets, equipment clean-up and a caustic scrubber. This wastewater is discharged to the process sewer and cooling water is discharged through Outfall 017.

Air emission sources include the vacuum jets and the amine handling and storage system. The vacuum jet gases are discharged uncontrolled to the atmosphere. The amines which are very volatile, are controlled by two systems emissions. The first system collects and sends the amines to the powerhouse for burning. In the event a malfunction of the first system occurs, the amines are collected, scrubbed with caustic and emitted to the atmosphere. In addition, the distillation bottoms, water and residue, is burned in the powerhouse.

NIAX CATALYST PRODUCTION

The basic catalyst (A-99), an organic catalyst, is manufactured by reacting amino ethyl ethanol amine, Chlorex (dichloroethylether) and trimethylamine. After the reaction, the precursor is dehydrated and refined (batch, vacuum distillation). In addition, Niaux catalysts A-1, A-397, ESN and others are made by blending A-99 with water and other materials.

The wastewater sources include an acetic acid scrubber, vacuum jets, process clean-up and the dehydration unit. These wastewaters are discharged to the process sewer. The dehydration water, about 19 m³ (5,000 gal)/month, contains approximately 1% organics and is discharged intermittently (once per month) to the process sewer. Non-contact cooling water is discharged through Outfalls 023 and 025.

Air emission sources are the vacuum jets, storage tanks and process vents. The vacuum jets discharge to the atmosphere and the vapors from the process and storage tank vents are collected and burned at the powerhouse. In the event that the process and tank vent collection system cannot discharge to the powerhouse, these gases are scrubbed in the acetic acid scrubber and then discharged to the atmosphere. The storage tanks are pressure vessels and emissions are controlled by pressure relief valves.

ETHYL SILICATES PRODUCTION

Ethyl silicates are produced by reacting silicon tetrachloride with ethanol and then filtered to remove solids. Hydrochloric acid (HCl) vapor is released during the reaction and removed in a water scrubber.

Wastewaters from the HCl scrubber and equipment clean-up are discharged to the process sewer. Cooling water is discharged through Outfall 025.

The only air emission source is the HCl scrubber. After scrubbing to remove HCl these gases are discharged to the atmosphere.

Solid wastes from the filter, one dumpster per month, are disposed in the Fillmont Landfill.

GLYCOL ETHERS PRODUCTION

Glycol ethers are produced by reacting alcohol with ethylene or propylene oxide [Figure 7].

Wastewaters from refining neutralization, and distillation are discharged to the process sewer. Non-contact cooling water is discharged through Outfall 074.

Air emission sources are from the refining unit and storage units. The refining unit vents are uncontrolled and the storage tank emissions are controlled by nitrogen blanketing of the tanks. Distillation column residues are burned at the powerhouse.

BUTYL CHLORIDE PRODUCTION

Butyl chloride is manufactured by reacting butanol with hydrogen chloride [Figure 8].

Wastewater sources are from the reactor and the distillation system. The reactor water is collected and burned in the powerhouse. Approximately 1,500 liter (400 gal)/day of water is discharged from the production unit to the process sewer along with the scrubber water. Cooling water is discharged through Outfall 014.

Air emissions from the reactor, still and storage tanks are collected, water scrubbed and are then discharged to the atmosphere.

DRUMMING AND MIXING AREA

A variety of products are handled, mixed, blended, placed in drums, etc. in this area.

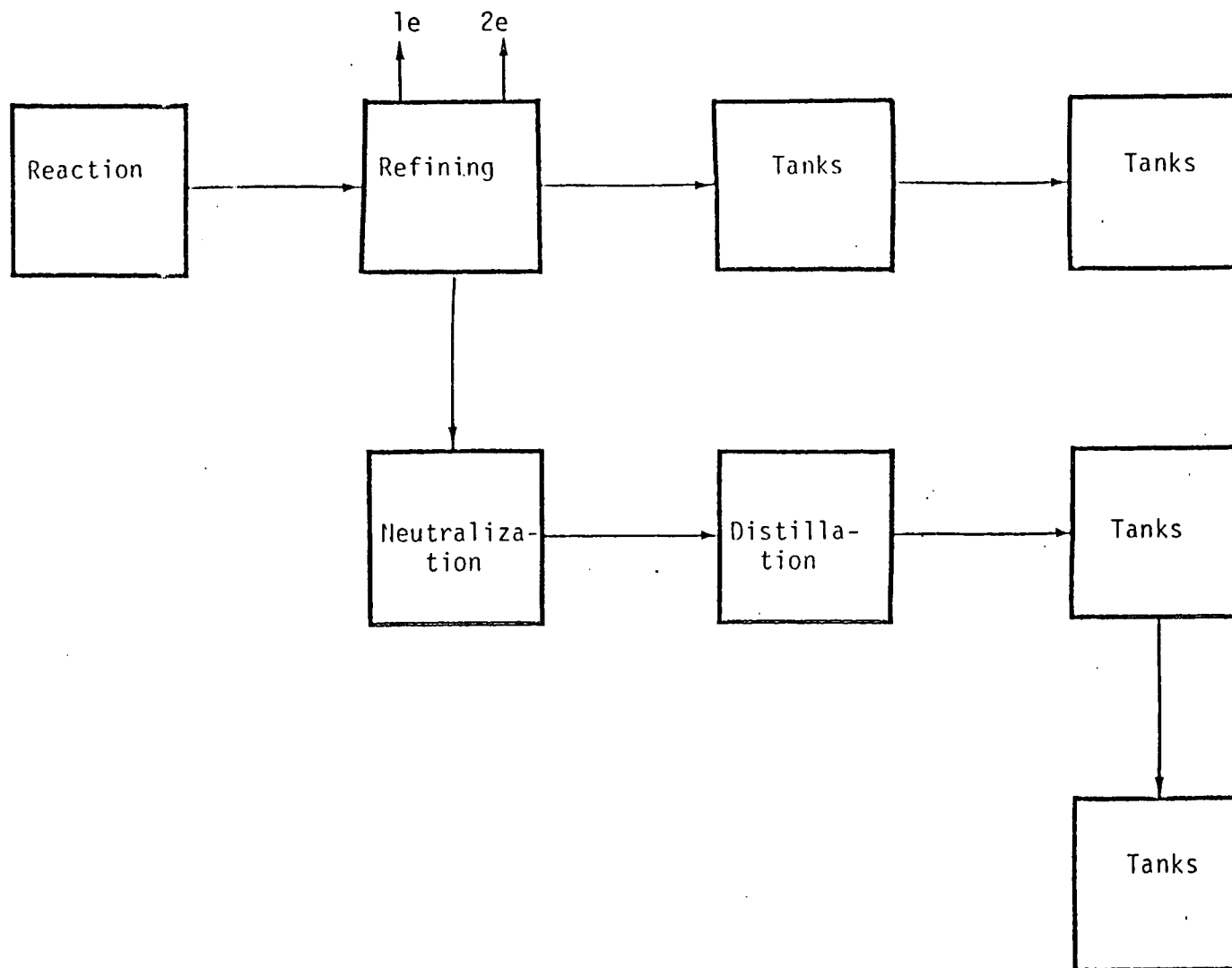


Figure 7. Glycol Ethers Process Schematic

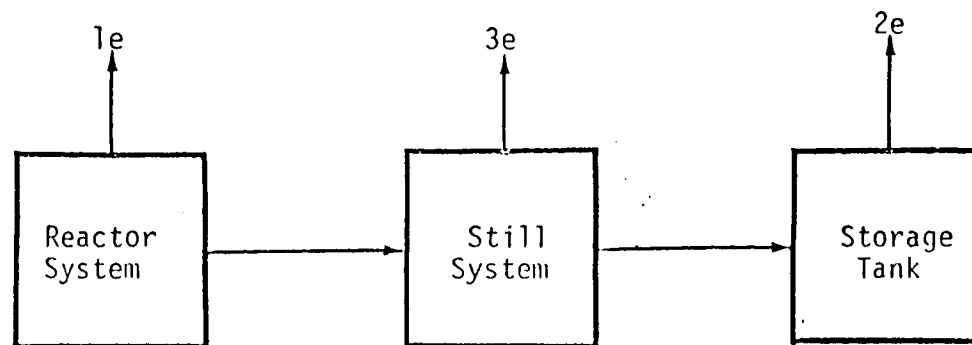


Figure 8. Butyl Chloride System Schematic

Area clean-up (spills, etc.) water is discharged to the process sewer.

The major air emission sources are building ventilation and storage tanks. Emissions due to local ventilation are discharged directly to the atmosphere. The storage tank emissions are controlled by nitrogen blanketing or conservation vents.

ENERGY PRODUCTION

The two powerhouses, one on the mainland and one on the island, burn coal, natural gas, natural gas concentrates and distillation residues. Company officials estimate the residue burned to be 5% of the total heat requirement. The mainland powerhouse consists of two 68,100 kg (150,000 lb) steam/hr boilers that discharge through one stack. The island powerhouse consists of two boilers rated at 68,100 kg (150,000 lb) steam/hr, two at 45,500 kg (100,000 lb) steam/hr and one at 131,600 kg (290,000 lb) steam/hr that discharge through 3 stacks.

Particulate emissions from each boiler are controlled by an electrostatic precipitator (ESP). A Honeywell transmissometer is used to monitor each boiler outlet. There are no monitors on the stacks, but the Company plans to install a Lear Siegler transmissometer on each stack. Visual monitoring via a camera is conducted by the operators. Sulfur dioxide (SO_2) emissions are controlled by burning low sulfur coal. Particulate source tests were conducted at the time of installation; however, none have been conducted since that time; also no SO_2 source tests have been conducted. Visible emissions from these stacks were in compliance with the State regulation (i.e. <10% opacity) at the time of the NEIC inspection.

Wastewater sources include the storm water from the diked area around the powerhouses and water treatment waste. The storm water is

checked for total carbon and if the total carbon is less than a given value, (not specified), the water is discharged to the river. If the total carbon is high, the water is trucked to the South Charleston Sewage Treatment Company for treatment.

Boiler and process waters are coagulated, settled, filtered and zeolite softened. The zeolite is washed with caustic and regenerated with H_2SO_4 . This regeneration waste and the settled solids are discharged to the river through Outfall 009.

Flyash from the boilers is pumped to Holz pond. Bottom ash is collected and used as fill cover at the Fillmont landfill.

III. POLLUTION ABATEMENT AND WASTE DISPOSAL PRACTICES

There is no wastewater treatment at this plant.

The NPDES permit issued to Union Carbide Corporation - South Charleston Plant authorizes the discharge of non-contact cooling water to the Kanawha River through 22 outfalls. All process and domestic wastewaters are collected and flumed to the South Charleston Sewage Treatment Company (SCSTC).

The Company has installed 22 monitoring stations* on their process sewers [Table 2], which monitor flow and total carbon. Selected stations measure pH, temperature, and specific organics. These monitoring stations are used to identify the processing area responsible for spills and/or poor housekeeping practices.

The two largest cooling water discharges (Outfalls 023 and 025) are each equipped with organic spill detectors, calibrated at 50 ppm isopropanol. When the discharge exceeds this value, an alarm is set and the spill detection unit collects a sample. The sample is immediately taken to the Company laboratory and analyzed with a gas chromatograph to determine what compound(s) was (were) discharged.

All outfalls are equipped with Union Carbide designed automatic samplers which collect time composites. The Company stated that the flow through these cooling water outfalls remains constant, therefore the composite samples are flow proportional.

* In addition, the Company also installed a station on the industrial influent to the SCSTC. This station is equipped with a total carbon analyzer, pH and temperature recorders, and a biomonitor.

The Company monitors on a daily basis for 43 specific organic chemicals in the process wastewater and cooling water [Attachment A]. During the inspection, NEIC personnel collected a grab sample from the process waste stream discharged to the SCSTC to screen for toxic pollutants and other organics. The analyses identified a total of 39 organic chemicals in this sample [Attachment B]. Fourteen of these were on the Toxic Pollutant List and ten had concentrations of greater than 10 µg/l. These are: benzene (130 µg/l), chlorobenzene (12 µg/l), 1,2-dichloroethane (48 µg/l), chloroform (22 µg/l), 1,2-dichlorobenzene (19 µg/l), ethylbenzene (470 µg/l), methylene chloride (30 µg/l), isophorone (57 µg/l), 2,4-dinitrophenol (11 µg/l) and toluene (200 µg/l). No nitrosamines were detected.

The air pollution controls at this facility are extensive and include scrubbers, electrostatic precipitators, nitrogen blanketing and conservation vents on tanks, and collection and burning of all burnable wastes. Union Carbide has installed 11 ambient-air monitoring stations in the plant and the surrounding area. The air emissions inventory lists the emissions of hydrocarbons and NO_x for each source in the plant. This inventory indicates that only 9 hydrocarbons are emitted at greater than 9 kg (20 lb)/hr. These are pentane (21 kg/hr), acetone (158 kg/hr), isopropanol (72 kg/hr), methylacetate (132 kg/hr), methanol (80 kg/hr), diethylamine (16 kg/hr) butylchloride (27 kg/hr), propylene oxide (53 kg/hr) and ethanol (32 kg/hr).

Solid wastes are placed in the Fillmont Landfill, Goff Mountain Chemical Landfill, and Holz pond. Non-chemical (lumber, paper, scrap polymer, etc.) solid wastes are disposed in the Fillmont Landfill operated by Union Carbide. The waste is put into the landfill and covered daily with bottom ash from the boilers. Chemical wastes are trucked to the Goff Mountain Chemical Landfill* for disposal. The Goff Mountain landfill is owned by Union Carbide and operated by Institute plant personnel. Both landfills are State approved.

* See Union Carbide Institute report for discussion of this landfill.

Holz Pond, an anerobic lagoon owned and operated by the Company, has a capacity of 760,000 m³ (200x10⁶ gal). The Company plans to expand the pond to provide capacity until the year 2000. Details of construction for the pond were not provided. This pond receives 9,000 kg (20,000 lb)/day of sludge from the South Charleston waste treatment facility, 13,600 kg (30,000 lb)/day of sludge from the Union Carbide Institute wastewater treatment facility, 45,400 kg (100,000 lb)/day of flyash from the South Charleston plant and 11,800 kg (26,000 lb)/day of lime.

The supernatant from Holz Pond is collected and returned to the SCSTC for treatment. At the time of the inspection, there was no evidence of leaking or leaching from Holz pond.

IV. EVALUATION OF SELF-MONITORING PRACTICES

BIOASSAY PROCEDURES

The bioassay evaluation [Attachment C], conducted on April 11, 1978, showed that the Company bioassay facilities are maintained at the Union Carbide Technical Center in South Charleston. The facility is environmentally controlled and properly equipped for bioassay testing. The bioassays and the associated chemical tests are performed according to Standard Methods except as noted below:

1. The effluent sample collected for bioassay testing is a 24-hour equal-volume composite rather than a 24-hour flow-proportional composite as required by the NPDES permit.
2. The bioassay tests do not always commence within eight hours after sample collection as recommended by Standard Methods.
3. Dechlorinated city tap water is used as dilution water rather than Kanawha River water as required by the NPDES permit.
4. The bioassay tests are not done in duplicate as recommended by Standard Methods.
5. All bioassays are aerated throughout the 96-hour test period. Aeration should be discontinued except in cases where BOD or COD are sufficiently high that adequate dissolved oxygen concentrations cannot be maintained.

6. The laboratory depends on controlled ambient air temperature to maintain a constant test temperature. It is advisable but not required, that a constant temperature water bath be used to maintain test temperatures.

ANALYTICAL PROCEDURES

The Company performs all the analyses required by the NPDES permit. The analysis are performed according to EPA-approved methods. Analytical quality control procedures consisting of routine and blind duplicates as well as spikes and reference samples are routinely performed and the test results are well documented.

Company laboratory personnel analyzed standard reference TOC samples provided by NEIC. The results were in close agreement with the true values [Attachment D].

SAMPLING PRACTICES

Some of the automatic sampling units contained algal growth, flaking paint and an accumulation of solids which could contaminate the samples. Officials indicated that the Company does not have a preventative maintenance program to clean and repaint these samplers. Because of the poor maintenance practices the samples collected with these units may not be representative.

The samples are reportedly 24-hour composites, as required by the NPDES permit, with the first aliquot collected at approximately 5:00 a.m. each day. Observations showed that at 1:30 p.m. several of the sample containers were already full and others were more than 2/3 filled. The samples, therefore, are not 24-hour composites as specified in the permit. Furthermore, the samples are not refrigerated

during collection, which is contrary to the requirement of maintaining the sample at 4°C. Adequate container capacity or a reduction in the sample aliquot size plus adequate temperature controls are necessary.

SELF-MONITORING DATA

Discharge Monitoring Reports (DMR's) from October through December 1977, for the cooling water discharges are contained in Tables 3 through 9. The DMR's show, that during this last quarter of 1977, the Company exceeded both daily average and daily maximum TOC limitations on the total discharge from 16 small discharges [Table 3]. In addition, daily maximum TOC violations were reported for Outfalls 025, 032, 035 and 074.

Table 3
 SUMMARY OF DISCHARGE MONITORING REPORTS*
 SOUTH CHARLESTON UNION CARBIDE
 OUTFALLS 009, 014, 015, 016, 017, 024,
 027, 028, 031, 036, 039, 040, 042, 048, 075, 076

Parameter	Permit Limitations		October		November		December	
	Daily Ave.	Daily Max.	Ave.	Max.	Ave.	Max.	Ave.	Max.
Flow - m ³ /day (10 ³) MGD	N/A	N/A	110.9	-	123.0	-	116.2	-
	N/A	N/A	29.3	-	32.5	-	30.7	-
Total Organic Carbon mg/l	4	12	2	8	4	71	5	67
Temperature (°C)	N/A	43.3	-	23.3	-	22.8	-	11.1
pH range	6.0-9.0		7.1-7.3		7.2-7.7		7.4-8.6	

* m³/day and °C are not reported by the plant; values are computed by NEIC.

Table 4
SUMMARY OF DISCHARGE MONITORING REPORTS*
SOUTH CHARLESTON UNION CARBIDE - OUTFALL 23

Parameter	Permit Limitations		October		November		December	
	Daily Ave.	Daily Max.	Ave.	Max.	Ave.	Max.	Ave.	Max.
Flow - m ³ /day (10 ³) MGD	N/A	N/A	67.0	-	74.2	-	70.0	-
	N/A	N/A	17.7	-	19.6	-	18.5	-
Total Organic Carbon mg/l	4	12	1	7	0.6	5	0.3	3
Temperature (°C)	N/A	43.3	-	20	-	21.1	-	11.7
Vinyl Chloride Monomer	N/A	N/A	-	-	0	-	-	-
pH range	6-9		7.2-7.4		7.4-7.6		7.3-8.0	

* m³/day and °C are not reported by the plant; values are computed by NEIC.

Table 5
SUMMARY OF DISCHARGE MONITORING REPORTS*
SOUTH CHARLESTON UNION CARBIDE - OUTFALL 25

Parameter	Permit Limitations		October		November		December	
	Daily Ave.	Daily Max.	Ave.	Max.	Ave.	Max.	Ave.	Max.
Flow - m ³ /day (10 ³) MGD	N/A	N/A	58.7	-	64.7	-	61.3	-
	N/A	N/A	15.5	-	17.1	-	16.2	-
Total Organic Carbon mg/l	4	12	3	12	4	28	5	10
Temperature (°C)	N/A	43.3	-	23.3	-	25.6	-	16.7
Vinyl Chloride Monomer	N/A	N/A	-	-	0	-	0	-
pH range	6.0-9.0		7.2-10.2		7.3-7.9		7.1-7.7	

* m³/day and °C are not reported by the plant; values are computed by NEIC.

Table 6
SUMMARY OF DISCHARGE MONITORING REPORTS*
SOUTH CHARLESTON UNION CARBIDE - OUTFALL 32

Parameter	Permit Limitations		October		November		December	
	Daily Ave.	Daily Max.	Ave.	Max.	Ave.	Max.	Ave.	Max.
Flow - m ³ /day (10 ³) MGD	N/A	N/A	15.1	-	16.7	-	15.9	-
	N/A	N/A	4.0	-	4.4	-	4.2	-
Total Organic Carbon mg/l	4	12	0.7	3	3	15	0.4	3
Temperature (°C)	N/A	43.3	-	24.4	-	27.2	-	22
pH range	6.0-9.0		8.5-9.0		8.1-9.0		7.3-9.5	

* m³/day and °C are not reported by the plant; values are computed by NEIC.

Table 7
SUMMARY OF DISCHARGE MONITORING REPORTS*
SOUTH CHARLESTON UNION CARBIDE - OUTFALL 35

Parameter	Permit Limitations		October		November		December	
	Daily Ave.	Daily Max.	Ave.	Max.	Ave.	Max.	Ave.	Max.
Flow - m ³ /day (10 ³) MGD	N/A	N/A	14.8	-	16.3	-	15.5	-
	N/A	N/A	3.9	-	4.3	-	4.1	-
Total Organic Carbon mg/l	4	12	1	21	2	10	2	39
Temperature (°C)	N/A	43.3	-	22.8	-	22.2	-	9.4
pH range	6.0-9.0		7.4-7.9		6.6-7.6		7.3-8.0	

* m³/day and °C are not reported by the plant; values are computed by NEIC.

Table 8
SUMMARY OF DISCHARGE MONITORING REPORTS*
SOUTH CHARLESTON UNION CARBIDE - OUTFALL 74

Parameter	Permit Limitations		October		November		December	
	Daily Ave.	Daily Max.	Ave.	Max.	Ave.	Max.	Ave.	Max.
Flow - m ³ /day (10 ³) MGD	N/A	N/A	23.5	-	26.1	-	24.6	-
	N/A	N/A	6.2	-	6.9	-	6.5	-
Total Organic Carbon mg/l	4	12	0.6	5	0.8	4	2	13
Temperature (°C)	N/A	43.3	-	25	-	24.4	-	10
pH range	6.0-9.0		7.1-8.8		7.4-8.9		7.5-7.7	

* m³/day and °C are not reported by the plant; values are computed by NEIC.

Table 9
SUMMARY OF DISCHARGE MONITORING REPORTS
SOUTH CHARLESTON UNION CARBIDE - TOTAL ALL OUTFALLS

Parameter	Permit Limitations		October		November		December	
	Daily Ave.	Daily Max.	Ave.	Max.	Ave.	Max.	Ave.	Max.
Flow - m ³ /day (10 ⁴)	N/A	N/A	30	-	32.1	-	30.3	-
MGD	N/A	N/A	76.6	-	84.8	-	80.1	-
Chlorides - mg/l	N/A	N/A	5.5	21.99	2.06	4.0	2.1	4.0
lb/day	12,718	19,427	421	1,684	1,456	2,828	1,411	2,672
kg/day	5,774	8,820	191	765	659	1,284	639	1,213
Phenolics - mg/l	N/A	N/A	0.004	0.008	-	-	-	-
lb/day	N/A	N/A	2.5	5.1	-	-	-	-
kg/day	N/A	N/A	1.1	2.3	-	-	-	-
Dissolved Solids								
mg/l	N/A	N/A	6	23	7.7	13	15	37
lb/day	N/A	N/A	475	1,762	5,479	9,191	9,886	24,716
kg/day	N/A	N/A	215	800	2,481	4,178	4,477	11,221
Kjeldahl Nitrogen								
mg/l	N/A	N/A	0.21	0.60	0.07	0.14	0.09	0.19
lb/day	492	1,014	16	46	49	99	63	127
kg/day	223	461	7.2	21	22	45	29	58
Organic Nitrogen								
mg/l	N/A	N/A	0.09	0.25	0	0	0.01	0.05
lb/day	436	872	7	19	0	0	7	33
kg/day	198	396	3.2	8.6	0	0	3	15

* kg/day and m³/day not reported by plant, values computed by NEIC.

ATTACHMENT A

COMPANY ORGANIC CHEMICAL MONITORING



UNION CARBIDE CORPORATION
CHEMICALS AND PLASTICS

P.O. BOX 2004, SOUTH CHARLESTON, W. VA. 26060

1 CHARLESTON PLANT

August 9, 1977

Dr. T. E. Fielding
Environmental Protection Agency
Region III
West Virginia Section, Enforcement Division
6th and Walnut Streets
Philadelphia, Pennsylvania 19106

Ref.: Permit WV 0000078

Ref.: Letter, J. L. Worstell
to Dr. T. E. Fielding,
June 23, 1977

Dear Dr. Fielding:

A review of the information sent to you on June 23 has revealed an error. The substance ethylene chlorohydrin was identified as being present in the industrial influent to the South Charleston Waste Treatment Works to the extent of 153 ppm (average concentration for the period June 1976 through November 1976, inclusive).

The correct identification is noted on the attached revised table.

Ethylene chlorohydrin is confined to one section of our plant.* The waste water from this area is chromatographed almost on a daily basis, and these analytical results have always shown virtually no ethylene chlorohydrin. This gives us an excellent cross-check.

If you have any question about this, please contact me.

Very truly yours,

J. L. Worstell
Environmental Protection
Coordinator

JLW/pl

Attachment

cc: Mr. J. D. Moore

West Virginia Department of Natural Resources
Division of Water Resources

Chemical*

acetaldehyde	10
acetone	456
acrylonitrile/MVA/DEK/Valeraldehyde	192
butanol	289
butyl Acetate	204
butyl Acrylate	44
butyl Carbitol	115
butyl Carbitol Acetate	42
butyl Cellosolve Acetate/Diethyl Carbitol	67
butyraldol	2
carbitol	6
carbitol Acetate	14
cellosolve Acetate	77
diacetone Alcohol	67
diisobutyl Cellosolve	28
diisobutyl Ketone/2-Ethylhexaldehyde	2
ethanol	400
ethyl Butanol	4
2-Ethyl Hexanol	200
2-Ethyl Hexyl Chloride	2
2-Ethyl Pentaldehyde/Methyl Vinyl Acetate	153
ethylene Glycol	4
1-Heptanol	18
n-Hexanol	75
Hexyl Cellosolve	10
isobutyl Carbitol	4
isobutyl Cellosolve	100
isopropanol	954
mesityl Oxide	67
methanol	365
methoxy Acetone	46
methyl Amyl Alcohol	46
methyl Carbitol	40
methyl Cellosolve Acetate/Propasol P	46
methyl Isobutyl Ketone/N-Propanol/Ethyl Carbitol	97
2-Methyl Pentenal/Ethyl Butyl Ketone	87
pentanedione	137
pinacolone	2
Propasol B/n-Hexanol/Diisobutyl Carbitol	2
Propasol DM	2
Propasol M/n-Butanol	2
Propiophenone	40
propylene Glycol	100
propyl Ethyl Ether	8

*The specific chemical analysis is done by gas chromatography and the above identification is by retention time on the chromatograph.

Mass spectrometry is used for identification of any one constituent is after 100 ppm.



UNION CARBIDE CORPORATION
CHEMICALS AND PLASTICS

P.O. BOX 8004, SOUTH CHARLESTON, WEST VIRGINIA 25311

SOUTH CHARLESTON PLANT

August 9, 1977

Environmental Protection Agency
Region III
Permits Application Section
6th and Walnut Streets
Philadelphia, Pennsylvania 19106

Attn: Dr. T. E. Fielding

Subject: NPDES Permit
WV 0000078

Dear Dr. Fielding:

As required by Part III, No. 3, page 24 of our NPDES Permit, attached is an analytical characterization of the cooling water discharges from the South Charleston Plant.

Very truly yours,

A handwritten signature in dark ink, appearing to read "J. L. Worstell".

J. L. Worstell
Environmental Protection
Coordinator

JLW/pl

Attachment

cc: West Virginia Department of Natural Resources
Division of Water Resources
1201 Greenbrier Street
Charleston, West Virginia 25311
Attn: Mr. R. M. Sovic

Mr. J. D. Moore - UCC

FROM COOLING WATER OUTFALLS

Chemical	Concentration	No. of Times**
	Range (PPM)	Detected
Acetone	0 - 335	23
Butanol/Propasol M	0 - 12	7
Ethyl Acetate	0 - 12	14
Ethyl Carbitol	0 - 95	17
Ethyl Carbitol Acetate	0 - 18	4
Ethyl Cellosolve	0 - 1	2
Ethyl Cellosolve Acetate/Diethyl Carbitol	0 - 2	1
Isobutyl	0 - 7	7
Isobutyl Acetate	0 - 3	1
Cellosolve	0 - 30	6
Acetone Alcohol	0 - 24	1
Ethyl Ethanolamine/Dimethyl Ethanolamine	0 - 5	1
Isobutyl Carbinol	0 - 3	1
Isobutyl Ketone/2-Ethylhexaldehyde	0 - 6	1
Propylene Glycol	0 - 38	3
Ethyl Hexanol	0 - 6	2
Glycol	0 - 3	1
Hexanol	0 - 1	1
Ethyl Cellosolve	0 - 2	1
Isobutyl Carbitol	0 - 6	1
Isopropanol	0 - 110	13
Ethyl Oxide/Methyl Amyl Acetate	0 - 24	11
Ethoxy Acetone	0 - 2	1
Ethyl Carbitol	0 - 10	1
Ethyl Isobutyl Ketone/N-Propanol/Ethylbutyraldehyde	0 - 1	3
Ethyl Vinyl Acetate	0 - 99	1
Isopropanol	0 - 1	1
Isophenone	0 - 3	2
Propylene Glycol	0 - 100	1

The specific chemical analyses are done by gas chromatography and the above identification is by retention times on the chromatograph.

Number of times detected from any one of the cooling water outfalls over a 180 day time period. Potentially, any given compound could be detected over 3,000 times over this time period.

SPECIFIC ORGANIC CHEMICALS TO RIVER
FROM COOLING WATER INTAKES

<u>Chemical*</u>	<u>AVG. PPM*</u>
acetone	0.180
Butanol/Propasol M	0.020
ethyl Acetate	0.020
ethyl Carbitol	0.070
ethyl Carbitol Acetate	0.003
ethyl Cellosolve	0.022
ethyl Cellosolve Acetate/Diethyl Carbitol	0.001
Carbitol	0.007
Carbitol Acetate	0.003
Cellosolve	0.040
Acetone Alcohol	0.020
ethyl Ethanolamine/Dimethyl Ethanolamine	0.009
isobutyl Carbinol	0.003
isobutyl Ketone/2-Ethylhexaldehyde	0.020
propylene Glycol	0.080
ethyl Hexanol	0.007
Glycol	0.001
Hexanol	0.001
ethyl Cellosolve	0.006
isobutyl Carbitol	0.001
isopropanol	0.050
isobutyl Oxide/Methyl Amyl Acetate	0.020
ethoxy Acetone	0.001
ethyl Carbitol	0.020
ethyl Isobutyl Ketone/N-Propanol/Ethylbutyraldehyde	0.003
ethyl Vinyl Acetate	0.020
Propanol	0.001
propionophenone	0.004
propylene Glycol	0.090

The specific chemical analyses are done by gas chromatography and the above identification is by retention times on the chromatograph.

The concentration (in ppm) of any chemical averaged over the total flow of cooling water from the plant.

WASTEWATER MONITORING STATIONS

<u>Station #</u>	<u>Area Monitored</u>	<u>Parameters Monitored</u>
1	UICC	Flow, TC (1,2)
2	UICC	Flow, TC (1,2)
3	Distribution	Flow, TC (2)
4	UICC	Flow, TC (1,2)
5	EPD (Main Flume)	Flow, TC (1,2), pH
6	MICC	Flow, TC (1,2) pH
7	MICC	Flow, TC (1,2)
8	EPD	Flow, TC (2)
9	EPD (Main Flume)	Flow, TC (1,2) pH
10	EPD (Main Flume)	Flow, TC (1,2) pH
11	Polyols	Flow, TC (2)
12	Chemical Mixing	Flow, TC (2)
13	Solvent Vinyl Resins	Flow, TC (2)
14	EPD (Main Flume)	Flow, TC (1,2) Specific Organics Analyzer (SOA), pH, Temperature
15	Distribution	Flow, TC (2)
16	Specialty Chemicals LPH	Flow, TC (2)
17	Specialty Chemicals Modernization	Flow, TC (2)
18	Specialty Chemicals	Flow, TC (H2)
19	Polyols	Flow, TC (1&2)
20	Mainland Chemicals	Flow, TC (1&2) pH
21	EPD (Holz Return)	Flow
22	Tech Center	(3)
23	SCWTW	Flow, TC (1,2), pH, Biomonitor, Temperature

- (1) Continuous Total Carbon Analysis
 (2) Daily Composite Total Carbon Analysis

JLW
 1/78

SPECIFIC ORGANIC										No. of Times Detected		MAX ppm		AVE ppm	
1. Acetaldehyde															
2. Acetone										31		144		41	
3. Acrylonitrile, Methyl Vinyl Acetate, Valeraldehyde "Area"										26		70		17	
4. Ally Isopropylsolv #1										2		23		1	
5. Ally Isopropylsolv #2															
6. Ally Isopropylsolv #3										2		65		3	
7. Butanol "Area"										14		47		8	
8. Butyl Acetate "Area"										22		10		4	
9. Butyl Carbitol										6		45		6	
10. Butyl Carbitol Acetate "Area"										1		63		2	
11. Butyl Cellosolve "Area"										3		31		2	
12. Butyl Cellosolve Acetate										1		67		2	
13. Carbitol Acetate															
14. Cellosolve										9		270		17	
15. Cellosolve Acetate															
16. Chlorex "Area"										7		26		2	
17. Diacetone Alcohol "Area"										5		41		4	
18. Dimethyl Acetate															
19. Diethylene Glycol										3		50		4	
20. Ethanol										23		132		9	
21. Ethyl Butanol "Area"															
22. Ethylhexaldehyde "Area"										4		18		2	
23. Ethylhexanol "Area"										19		250		34	
24. Ethyl Butyl Ketone "Area"										5		18		2	
25. Ethyl Propylacrolein															
26. Glycol Diacetate										4		176		16	
27. Heptanol "Area"										4		41		3	
28. Hexanol "Area"										3		177		7	
29. Hexyl Cellosolve "Area"										14		50		11	
30. Isobutyl Carbitol															
31. Isopropanol										31		971		151	
32. Isopropyl Acetate "Area"															
33. Methanol										31		147		31	
34. Methyl Amyl Acetate															
35. Methyl Cellosolve										5		35		6	
36. Methyl Cellosolve Acetate "Area"										3		16		1	

[illegible]

SPECIFIC ORGANIC										No. of Times Detected		MAX ppm		AVE ppm	
1. Acetaldehyde										31		151		49	
2. Acetone															
3. Acrylonitrile, Methyl Vinyl Acetate, Valeraldehyde "Area"										31		110		25	
4. Ally Isopropylsolv #1										1		36		1	
5. Ally Isopropylsolv #2															
6. Ally Isopropylsolv #3															
7. Butanol "Area"										21		39		15	
8. Butyl Acetate "Area"										25		25		6	
9. Butyl Carbitol										7		50		6	
10. Butyl Carbitol Acetate "Area"										1		24		*	
11. Butyl Cellosolve "Area"										4		20		2	
12. Butyl Cellosolve Acetate															
13. Carbitol Acetate															
14. Cellosolve										17		139		17	
15. Cellosolve Acetate															
16. Chlorex "Area"										17		29		4	
17. Diacetone Alcohol "Area"										16		94		12	
18. Dimethyl Acetate															
19. Dimethylene Glycol										7		140		13	
20. Ethanol										27		38		12	
21. Ethyl Butanol "Area"															
22. Ethylhexaldehyde "Area"										2		21		1	
23. Ethylhexanol "Area"										9		47		6	
24. Ethyl Butyl Ketone "Area"										1		27		1	
25. Ethyl Propylacrolein															
26. Glycol Diacetate															
27. Heptanol "Area"										2		16		1	
28. Hexanol "Area"										3		53		3	
29. Hexyl Cellosolve "Area"										15		30		9	
30. Isobutyl Carbitol															
31. Isopropanol										31		376		100	
32. Isopropyl Acetate "Area"										2		53		2	
33. Methanol										31		166		42	
34. Methyl Amyl Acetate															
35. Methyl Cellosolve										2		27		2	
36. Methyl Cellosolve Acetate "Area"										2		14		4	

SPECIFIC ORGANIC										No. of Times Detected			MAX ppm		AVE ppm	
1. Acetaldehyde																
2. Acetone												30		267		52
3. Acrylonitrile, Methyl Vinyl Acetate, Valeraldehyde "Area"												29		61		29
4. Ally Isopropylsolve #1												2		17		1
5. Ally Isopropylsolve #2																
6. Ally Isopropylsolve #3																
7. Butanol "Area"												12		39		8
8. Butyl Acetate "Area"												25		14		3
9. Butyl Carbitol												1		281		9
10. Butyl Carbitol Acetate "Area"												2		47		3
11. Butyl Cellosolve "Area"												5		359		17
12. Butyl Cellosolve Acetate												1		10		*
13. Carbitol Acetate												1		27		*
14. Cellosolve												9		244		19
15. Cellosolve Acetate																
16. Chlorex "Area"												3		8		*
17. Diacetone Alcohol "Area"												14		197		22
18. Dimethyl Acetate																
19. Diethylene Glycol												10		91		12
20. Ethanol												28		592		38
21. Ethyl Butanol "Area"												1		87		3
22. Ethylhexaldehyde "Area"												9		121		19
23. Ethylhexanol "Area"												12		101		9
24. Ethyl Butyl Ketone "Area"												1		12		*
25. Ethyl Propylacrolein																
26. Glycol Diacetate																
27. Heptanol "Area"												1		15		*
28. Hexanol "Area"												7		64		6
29. Hexyl Cellosolve "Area"												12		103		13
30. Isobutyl Carbitol																
31. Isopropanol												30		561		132
32. Isopropyl Acetate "Area"												12		2454		108
33. Methanol												30		665		48
34. Methyl Amyl Acetate																
35. Methyl Cellosolve												6		264		20
36. Methyl Cellosolve Acetate "Area"												1		101		3

* Less than 1 ppm

SPECIFIC ORGANIC										No. of Times Detected		MAX ppm		AVE ppm	
1. Acetaldehyde															
2. Acetone										31		486		79	
3. Acrylonitrile, Methyl Vinyl Acetate, Valeraldehyde "Area"										31		263		55	
4. Ally Isopropylsolv #1															
5. Ally Isopropylsolv #2										2		4		*	
6. Ally Isopropylsolv #3															
7. Butanol "Area"										24		93		22	
8. Butyl Acetate "Area"										28		12		4	
9. Butyl Carbitol										5		45		3	
10. Butyl Carbitol Acetate "Area"										2		33		1	
11. Butyl Cellosolve "Area"										13		149		14	
12. Butyl Cellosolve Acetate										4		165		6	
13. Carbitol Acetate															
14. Cellosolve										13		51		9	
15. Cellosolve Acetate															
16. Chlorex "Area"										8		150		6	
17. Diacetone Alcohol "Area"										6		18		2	
18. Dimethyl Acetate															
19. Diethylene Glycol															
20. Ethanol										21		195		20	
21. Ethyl Butanol "Area"															
22. Ethylhexaldehyde "Area"										9		187		9	
23. Ethylhexanol "Area"										15		189		20	
24. Ethyl Butyl Ketone "Area"										6		44		3	
25. Ethyl Propylacrolein										5		38		2	
26. Glycol Diacetate										4		30		2	
27. Heptanol "Area"										4		18		1	
28. Hexanol "Area"										6		67		3	
29. Hexyl Cellosolve "Area"										14		134		14	
30. Isobutyl Carbitol										3		30		1	
31. Isopropanol										31		1192		265	
32. Isopropyl Acetate "Area"										2		254		9	
33. Methanol										29		446		62	
34. Methyl Amyl Acetate										4		40		4	
35. Methyl Cellosolve										5		100		2	
36. Methyl Cellosolve Acetate "Area"										9		42		5	

* Less than 1 ppm

SPECIFIC ORGANIC										No. of Times Detected		MAX ppm		AVE ppm	
1. Acetaldehyde										25		385		55	
2. Acetone										30		407		105	
3. Acrylonitrile, Methyl Vinyl Acetate, Valeraldehyde "Area"										1		5		*	
4. Ally Isopropylsolv #1										4		24		2	
5. Ally Isopropylsolv #2															
6. Ally Isopropylsolv #3										30		159		47	
7. Butanol "Area"										22		17		3	
8. Butyl Acetate "Area"										1		20		*	
9. Butyl Carbitol															
10. Butyl Carbitol Acetate "Area"										7		112		7	
11. Butyl Cellosolve "Area"										4		54		4	
12. Butyl Cellosolve Acetate															
13. Carbitol Acetate										14		57		8	
14. Cellosolve															
15. Cellosolve Acetate															
16. Chlorex "Area"										8		30		4	
17. Diacetone Alcohol "Area"															
18. Dimethyl Acetate										1		26		*	
19. Diethylene Glycol										20		229		24	
20. Ethanol															
21. Ethyl Butanol "Area"										14		239		28	
22. Ethylhexaldehyde "Area"										19		83		18	
23. Ethylhexanol "Area"										2		5		*	
24. Ethyl Butyl Ketone "Area"										9		615		32	
25. Ethyl Propylacrolein										5		12		1	
26. Glycol Diacetate										6		21		2	
27. Heptanol "Area"															
28. Hexanol "Area"										18		58		17	
29. Hexyl Cellosolve "Area"										1		57		1	
30. Isobutyl Carbitol										29		561		154	
31. Isopropanol										9		70		11	
32. Isopropyl Acetate "Area"										15		81		18	
33. Methanol															
34. Methyl Amyl Acetate										5		202		15	
35. Methyl Cellosolve										3		16		1	
36. Methyl Cellosolve Acetate "Area"															

[illegible]

SPECIFIC ORGANIC										No. of Times Detected		MAX ppm		AVE ppm	
1. Acetaldehyde										2		110		4	
2. Acetone										31		1836		183	
3. Acrylonitrile, Methyl Vinyl Acetate, Valeraldehyde "Area"										29		620		48	
4. Ally Isopropylsolve #1															
5. Ally Isopropylsolve #2															
6. Ally Isopropylsolve #3															
7. Butanol "Area"										27		129		27	
8. Butyl Acetate "Area"										24		71		9	
9. Butyl Carbitol										5		115		9	
10. Butyl Carbitol Acetate "Area"										1		19		*	
11. Butyl Cellosolve "Area"										5		105		6	
12. Butyl Cellosolve Acetate															
13. Carbitol Acetate										2		51		1	
14. Cellosolve										19		104		23	
15. Cellosolve Acetate															
16. Chlorex "Area"															
17. Diacetone Alcohol "Area"										5		129		7	
18. Dimethyl Acetate										4		47		4	
19. Dimethylene Glycol										3		52		3	
20. Ethanol										22		152		20	
21. Ethyl Butanol "Area"															
22. Ethylhexaldehyde "Area"										5		279		12	
23. Ethylhexanol "Area"										10		154		11	
24. Ethyl Butyl Ketone "Area"										3		18		1	
25. Ethyl Propylacrolein										1		5		*	
26. Glycol Diacetate															
27. Heptanol "Area"										3		114		4	
28. Hexanol "Area"										5		21		2	
29. Hexyl Cellosolve "Area"										24		119		22	
30. Isobutyl Carbitol										2		33		1	
31. Isopropanol										30		224		63	
32. Isopropyl Acetate "Area"										26		213		55	
33. Methanol										28		359		87	
34. Methyl Amyl Acetate										1		7		*	
35. Methyl Cellosolve										1		17		*	
36. Methyl Cellosolve Acetate "Area"										3		44		3	

[illegible]

ATTACHMENT B

ORGANIC CHEMICALS SCREENING ANALYSIS

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER
DENVER, COLORADO 80225

TO : Dr. Wayne Smith
Process Control Branch

DATE: June 2, 1978

FROM : O. J. Logsdon

SUBJECT: Organics Analysis Results: Union Carbide Institute Plant and South Charleston
WWTC Recon Samples

Summary:

Water samples from Union Carbide Institute Plant and South Charleston WWTC were received under chain-of-custody procedures and analyzed for organics characterization and selected priority pollutants. These reconnaissance samples contained numerous chemicals, some of which were priority pollutants.

Recommendations:

Analysis of the reconnaissance samples showed many organic solvent components, cellosolves, etc. If a full survey is conducted, analysis by direct aqueous injection techniques will be required to acquire accurate quantitative data for these compounds. Only small amounts of phenol and 2,4-dinitrophenol and some low molecular weight acids were detected in the acidic fractions. Therefore, with only a minor compromise in data quality, samples from the survey should be analyzed for neutrals extractables instead of the time consuming acids and base/neutrals procedure for these two locations unless priority pollutants are specifically requested.

Union Carbide Institute Plant

Table I^{*} shows the results of organics characterization analysis of sample number 003-30-A-4-11-78-0900. Seven chemicals were confirmed by comparison of their mass spectra to in-house reference spectra. Ten additional compounds were identified but not confirmed. Priority pollutant analysis detected 12 compounds, 7 of which exceeded 10 ug/l. The data are attached.

This sample was also analyzed for extractable nitrosamines. Attached is Mr. Nottingham's memo describing the analysis. None of the following nitrosamines were detected: dimethyl, diethyl, methyl ethyl, methyl propyl, ethyl propyl, dipropyl, ethyl butyl, propyl butyl, methyl amyl, dibutyl, and diamyl nitrosamines, nitrosopiperidine, nitrosopyrrolidine, and nitrosomorpholine.

The sample was also subjected to analysis by high performance liquid chromatography. The herbicide Carbaryl was detected at 260 ug/l. Mr. Nottingham's discussion of the analysis is attached.

* Table I not included in this report; available upon request from NEIC.

- 2 -

South Charleston WWTIC

Tables II and III show the results of the organics characterization analysis of samples: 003-40-A-4-12-78-1115, 003-41-A-4-12-78-1030, 003-43-A-4-12-78-1100, and 003-45-A-4-12-78-1050. Thirteen chemicals were identified and confirmed by GC/MS analysis. Thirty other chemicals were identified but not confirmed. Numerous other components were not identified; however, many mass spectra had the characteristics of alkyl ethers and alcohols. Available self-monitoring data (SCSTW influent and effluent July - December, 1977) show numerous oxygenated solvents e.g. cellosolves, acetates, and alcohols, a few of which have been identified in these samples.

Priority pollutant analysis was limited to acid and base/neutrals extractables and volatile organics. Numerous compounds were detected and are reported in the attached tables. In addition, sample 003-45-A-4-12-78-1080 was analyzed for nitrosamines. None of the following nitrosamines were detected: dimethyl, diethyl, methyl ethyl, methyl propyl, ethyl propyl, dipropyl, ethyl butyl, propyl butyl, methyl amyl, dibutyl, and diamyl nitrosamines, nitrosopiperidine, nitrosopyrrolidine, and nitrosomorpholine.


O. John Logsdon

Attachments

cc: Hatheway
Young
Masse

Table II
SOUTH CHARLESTON WWT
GRAB SAMPLES COLLECTED 4/12/78. COMPOUNDS CONFIRMED.
CONCENTRATION IN $\mu\text{g/l}$

Name	003-40-A	003-41-A	003-43-A	003-45-A
acrylonitrile	a	55		
benzothiazole			c	
bis-(2-chloroisopropyl) ether			2.7	
n-butanol	a			
2-n-butoxy-ethanol	3,700			
dichloromethane (methylene chloride)		55	c	180
2-ethyl-1-butanol	1,500			
2-ethyl hexanoic acid	9,900			
2-propanone (acetone)	a	a		
trichloromethane (chloroform)			43	
vinyl benzene ^b (styrene)	4,000			
ethyl benzene ^b	470			

a could not quantitate - does not purge quantitatively during volatile organics analysis.

b quantity based on priority pollutant analysis.

c unable to quantitate.

Table III
SOUTH CHARLESTON WWTC
GRAB SAMPLES COLLECTED 4/12/78. COMPOUNDS IDENTIFIED BUT NOT CONFIRMED.

Name	003-40-A	003-41-A	003-43-A	003-45-A
acetyl-(beta)-methylcholine	X			
benzoic acid	X		X	
butanoic acid	X		X	
2-butanone (methyl ethyl ketone)	X			
n-butyl chloride	X	X		
2-butoxyethoxy ethanol isomer	X	X		X
2-butyl-1-octanol	X			
carbon disulfide		X		X
diethylether		X	X	X
diisopropylether		X	X	X
1,1-dimethoxy ethane		X		
N,N-dimethyl butylamine	X			
4-ethyl-2-octene		X		
heptanoic acid	X		X	
isopropanol		X		
2-(hexyloxy)-ethanol	X			
methylcyclopentane isomer	X			
2-methyl-1,3-dioxolane		X		
2-methyl-2-pentanol				
2-methyl propanenitrile	X	X		X
methoxyacetone		X		
1[1-methyl-2-(2-propenyloxy)]ethoxy- 2-propanol	X		X	
octadecanoic acid (stearic acid)			X	
1,1'-oxybis-(2-ethoxy)ethane		X		X
3-pentanone	X			
phenylacetic acid	X			
2-n-propoxyethyl acetate		X		X
1,trans-2,cis-4-trimethyl cyclopentane	X			
2,2,4-trimethyl pentane (isooctane)		X		
trioctyl phosphate		X		X

A-72

CHARLESTON WTC

CHARLESTON WV

FOREI NO

STATION LOCATION: INF TO BASIN

LABORATORY SAMPLE NO: 003-40

DATE & TIME OF SAMPLE COLLECTION: 12APR78 1115 HRS

COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC.
01. ACENAPHTHENE	ND
02. ACROLEIN	NA
03. ACRYLONITRILE	NA
04. BENZENE*	130.
05. BENZIDINE	NA
06. CARBON TETRACHLORIDE (TETRACHLOROMETHANE)*	ND
07. CHLOROBENZENE*	12.
08. 1,2,4-TRICHLOROBENZENE	ND
09. HEXACHLOROBENZENE	ND
10. 1,2-DICHLOROETHANE*	48.
11. 1,1,1-TRICHLOROETHANE*	ND
12. HEXACHLOROETHANE	ND
13. 1,1-DICHLOROETHANE*	ND
14. 1,1,2-TRICHLOROETHANE*	ND
15. 1,1,2,2-TETRACHLOROETHANE*	ND
16. CHLOROETHANE*	NA
17. BIS(CHLOROMETHYL) ETHER*	NA
18. BIS(2-CHLOROETHYL) ETHER	ND
19. 2-CHLOROETHYL VINYL ETHER (MIXED)*	ND
20. 2-CHLORONAPHTHALENE	ND
21. 2,4,6-TRICHLOROPHENOL	ND
22. PARACHLOROMETA CRESOL	ND
23. CHLOROFORM (TRICHLOROMETHANE)*	22.
24. 2-CHLOROPHENOL	ND
25. 1,2-DICHLOROBENZENE	19.
26. 1,3-DICHLOROBENZENE	ND
27. 1,4-DICHLOROBENZENE	ND
28. 3,3'-DICHLOROBENZIDINE	NA
29. 1,1-DICHLOROETHYLENE*	3.1
30. 1,2-TRANS-DICHLOROETHYLENE*	ND

VGA ANALYSES (UNPRESERVED/PRESERVED)

TRACE

1 NOT ANALYZED FOR

2 NOT DETECTED

31 NOT ABLE TO ANALYZE DUE TO INTERFERENCE

CHARLESTON WTC

CHARLESTON WY

0001 NO

ATION LOCATION: INF TO BASIN

LABORATORY SAMPLE NO: 003-40

DATE & TIME OF SAMPLE COLLECTION: 12APR78 1115 HRS

COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC.
1. 2,4-DICHLOROPHENOL	ND
2. 1,2-DICHLOROPROPANE*	ND
3. 1,3-DICHLOROPROPYLENE*	ND
4. 2,4-DIMETHYLPHENOL	ND
5. 2,4-DINITROTOLUENE	ND
6. 2,6-DINITROTOLUENE	ND
7. 1,2-DIPHENYLHYDRAZINE	ND
8. ETHYLBENZENE*	470.
9. FLUORANTHENE	ND
10. 4-CHLOROPHENYL PHENYL ETHER	NA
11. 4-BROMOPHENYL PHENYL ETHER	ND
12. BIS(2-CHLOROISOPROPYL) ETHER	ND
13. BIS(2-CHLOROETHOXY) METHANE	ND
14. METHYLENE CHLORIDE (DICHLOROMETHANE)*	30.
15. METHYL CHLORIDE (CHLOROMETHANE)*	NA
16. METHYL BROMIDE (BROMOMETHANE)*	NA
17. BROMOFORM (TRIBROMOMETHANE)*	ND
18. DICHLOROBROMOMETHANE*	ND
19. TRICHLOROFLUOROMETHANE*	ND
20. DICHLORODIFLUOROMETHANE	NA
21. CHLORODIBROMOMETHANE*	ND
22. HEXACHLOROBUTADIENE	ND
23. HEXACHLOROCYCLOPENTADIENE	ND
24. ISOPHORONE	57.
25. NAPHTHALENE	ND
26. NITROBENZENE	ND
27. 2-NITROPHENOL	ND
28. 4-NITROPHENOL	ND
29. 2,4-DINITROPHENOL	11.
30. 4,6-DINITRO-O-CRESOL	ND

VCA ANALYSES (UNPRESERVED/PRESERVED)

TRACE

NOT ANALYZED FOR

NOT DETECTED

NOT ABLE TO ANALYZE DUE TO INTERFERENCE

A-74

CHARLESTON WWTG

CHARLESTON WV

TORREY RD

LOCATION: INF TO BASIN

LABORATORY SAMPLE NO: 003-40

DATE & TIME OF SAMPLE COLLECTION: 12APR78 1115 HRS

COMPOSITE TIME: 00 HR

COMPOUND NAME

UG/L
CONC.

51. N-NITROSODIMETHYLAMINE	ND
52. N-NITROSODIPHENYLAMINE	ND
53. N-NITROSODI-N-PROPYLAMINE	ND
54. PENTACHLOROPHENOL	ND
55. PHENOL (4AAP)	NA
56. BIS(2-ETHYLHEXYL) PHTHALATE	3.4
57. BUTYL BENZYL PHTHALATE	ND
58. DI-N-BUTYL PHTHALATE	1.1
59. DI-N-OCTYL PHTHALATE	ND
60. DIETHYL PHTHALATE	ND
61. DIMETHYL PHTHALATE	ND
62. BENZO(A)ANTHRACENE (1,2 BENZANTHRACENE)	ND
63. BENZO(A)PYRENE (3,4-BENZOPYRENE)	ND
64. 3,4-BENZOFLUORANTHENE	ND
65. BENZO(K)FLUORANTHENE (11,12-BENZOFLUORANTHENE)	ND
66. CHRYSENE	ND
67. ACENAPHTHYLENE	ND
68. ANTHRACENE	ND
69. BENZO(G,H,I)PERYLENE (1,12-BENZOPERYLENE)	NA
70. FLUORENE	ND
71. PHENANTHRENE	ND
72. DIBENZO (A,H)ANTHRACENE	NA
73. INDENO (1,2,3-CD)PYRENE	NA
74. PYRENE	ND
75. TETRACHLOROETHYLENE*	3.4
76. TOLUENE*	200.
77. TRICHLOROETHYLENE*	ND
78. VINYL CHLORIDE (CHLOROETHYLENE)*	NA
79. ALDRIN	NA
80. DIELDRIN	NA

VOC ANALYSES (UNPRESERVED/PRESERVED)

TRACE

1. NOT ANALYZED FOR

2. NOT DETECTED

3. NOT ABLE TO ANALYZE DUE TO INTERFERENCE

CHARLESTON WWTG

CHARLESTON WV

SITE NO

LOCATION: INF TO BASIN

LABORATORY SAMPLE NO: 003-40

TIME OF SAMPLE COLLECTION: 12AFR78 1115 HRS

COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC
1. CHLORANE (TECH, MIXTURE & METABOLITES)	NA
2. 4,4'-DDT	NA
3. 4,4'-DDE	NA
4. 4,4'-DDD (P,P')-TDE	NA
5. A-ENDOSULFAN-ALPHA	NA
6. B-ENDOSULFAN-BETA	NA
7. ENDOSULFAN SULFATE	NA
8. ENDRIN	NA
9. ENDRIN ALDEHYDE	NA
10. HEPTACHLOR	NA
11. HEPTACHLOR EPOXIDE	NA
12. A-BHC-ALPHA	NA
13. B-BHC-BETA	NA
14. G-BHC-(LINDANE)-GAMMA	NA
15. G-BHC-DELTA	NA
16. PCB-1242 (AROCOR 1242)	NA
17. PCB-1254 (AROCOR 1254)	NA
18. PCB-1221 (AROCOR 1221)	NA
19. PCB-1232 (AROCOR 1232)	NA
20. PCB-1248 (AROCOR 1248)	NA
21. PCB-1260 (AROCOR 1260)	NA
22. PCB-1016 (AROCOR 1016)	NA
23. TOXAPHENE	NA
24. ANTIMONY (TOTAL)	NA
25. ARSENIC (TOTAL)	NA
26. ASBESTOS (FIBROUS)	NA
27. BERYLLIUM (TOTAL)	NA
28. CADMIUM (TOTAL)	NA
29. CHROMIUM (TOTAL)	NA
30. COPPER (TOTAL)	NA

VOA ANALYSES (UNPRESERVED/PRESERVED)

TRACE

NOT ANALYZED FOR

NOT DETECTED

NOT ABLE TO ANALYZE DUE TO INTERFERENCE

A-76

CHARLESTON WWTIC

CHARLESTON WV

LORET NO

ATION LOCATION: INF TO BASIN

LABORATORY SAMPLE NO: 003-40

TE & TIME OF SAMPLE COLLECTION: 12APR78 1115 HRS

COMPOSITE TIME: 00 HR

COMPOUND NAME

UG/L
CONC.

21. CYANIDE (TOTAL)
22. LEAD (TOTAL)
23. MERCURY (TOTAL)
24. NICKLE (TOTAL)
25. SELENIUM (TOTAL)
26. SILVER (TOTAL)
27. THALLIUM (TOTAL)
28. ZINC (TOTAL)
29. 2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN (TCDD)
30. PHENOL (BY GC/FID OR GC/MS)

NA
NA
NA
NA
NA
NA
NA
NA
NA
ND

VSA ANALYSES (UNPRESERVED/PRESERVED)

TRACE

A NOT ANALYZED FOR

D NOT DETECTED

AI NOT ABLE TO ANALYZE DUE TO INTERFERENCE

ATTACHMENT C

BIOASSAY PROCEDURES EVALUATION

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER
DENVER, COLORADO 80225

TO : Wayne Smith

DATE: May 16, 1978

FROM : Bruce Binkley

SUBJECT: Laboratory Evaluation Inspection of Industries in the Kanawha Valley,
Charleston, West Virginia

On April 11, 1978, NEIC conducted a laboratory evaluation inspection of the Union Carbide Corp. plant at Charleston, West Virginia. The purpose of this inspection was to determine whether laboratory facilities and test procedures were adequate to satisfy the self-monitoring bioassay requirements of NPDES Permit Number WVC000078.

The bioassay facilities are maintained at the Union Carbide Corporations South Charleston Technical Center. In general, this laboratory is adequately equipped and staffed to perform static bioassay tests. The testing area is environmentally controlled for temperature and photoperiod; however, it appeared to be somewhat limited in adequate working space. Bioassay, physical and chemical tests are performed according to recognized standard methods. Procedural inconsistencies and recommendations for improvement of this testing facility are as follows:

1) Expansion of existing floor space could be utilized to provide more efficient working areas.

2) Effluent samples for bioassay consist of 24-hour equal volume composites. This is inconsistent with the permit limitation which specifies a 24-hour flow proportioned composite for bioassay testing. The current sampling method should be modified to reflect NPDES Permit specifications.

3) It was reported that bioassay tests do not always commence within eight hours of the sample collection. These tests must be initiated within eight hours of the completion of composite sampling.

-2-

4) Dechlorinated city tap water is used for holding test organisms and as the dilution water for bioassay testing. This water supply is acceptable for long-term holding of test fish; however, dilution water for bioassay testing should consist of Kanawha River water. Test fish should be acclimated to Kanawha River water at least four days prior to bioassay testing.

5) Bioassay tests are not done in duplicate. It is recommended that all bioassay tests be done in duplicate.

6) Physical and chemical parameters (dissolved oxygen concentration, pH, and temperature) are monitored daily. Because ammonia buildup can be a problem in static bioassay testing, measurements for total ammonia-N should be included. Measurements for total ammonia-N should be made at the high and low test concentrations at the beginning and end of the test period. Calculations for un-ionized ammonia concentrations should then be made.

7) This laboratory currently conducts bioassays on a 12-hour light photoperiod. This photoperiod should be increased to a 16-hour light and 8-hour dark interval.

8) All bioassays are aerated throughout the 96-hour test period. Aeration should be discontinued except in cases where B.O.D. and/or C.O.D. are sufficiently high that adequate dissolved oxygen concentrations cannot be maintained. Any use of aerated test water must be documented on the bioassay bench sheets.

9) Washing procedures for bioassay test chambers should include a solvent rinse. Acetone is an acceptable solvent for this purpose.

10) This laboratory depends on controlled ambient air temperature to maintain a constant test temperature in bioassay test solutions. It is advisable that test chambers be placed in a constant temperature water bath for more precise temperature control.

cc: J. Hatheway
R. Harp
Biology Branch Files

BIOASSAY LABORATORY EVALUATION

Laboratory or Industry Huon Carbide Corp.
Location Tech. Center Charleston, West Virginia
Date 11 April 1978
Investigator Bruce A. Binkley
Company Representative Jack Dawson (Asst. to Gene Waddy)
Test Method 9th Edition - S.M. and Standard
Methods 11th Edition - Just received EPA 600/4-78-012
January 1978

Dilution Water

Source D-Charleston City tap water (Charcoal filtered)
Should use Kennebec River water
Chemical Analyses Performed None

Pretreatment Carbon filtration

Effluent Water

Source Union Carbide Dischargers C23, C25, C32, C35, C37, C34
(C19, C14, C15, C16, C17, C24, C27, C28, C31, C36, C39) Aggregate
 Retention time NONE

Sampling technique 24 hour equal volume Composite - Sampled
every two hours

Holding time and conditions Never exceeds 24-hours. Occasionally
exceeds 6-hours.

Pretreatment NONE

Chemical Analyses Performed pH, Dissolved Oxygen Concentration,
temperature

Test Organisms

Species Fathead Minnow

Life stage Viable Adult

Source Kurtz's Fish Hatchery

Holding facilities Stainless Steel - flow through system utilizing
dechlorinated tap water

Acclimation Procedure 7-days minimum

Treatment tetracycline and Methylene Green - formaldehyde
Routine for all newly Arrived fish

Experimental DesignTest Chambers

Construction Material Glass

Dimensions Cylinder (Butterfly jar type)

Volume 15 liter

Volumetric exchange rate N/A

Test concentrations 5 test Concentrations of effluent plus
a Control (100, 50, 25%)

Number of organisms per concentration 10 fish per Concentration
Not done in duplicate.

Loading rate Aug at free fish conc. 1.2g \rightarrow 12 grams / 15 liter

Test temperature-average and range 18-22 $^{\circ}$ \pm 2 $^{\circ}$ C Adverse use of Constant temperature water bath.

Chemical parameters monitored and frequency Dissolved Oxygen Concentration pH temperature Monitored daily - Expected to include total NH₃-N at high & low test concentrations at beginning and end of test.

Duration and frequency of test Directly 96-hour static

Definition of adverse effect Death

Frequency of observations 24-hour intervals

Method of calculating EC₅₀ Straight line Graphical Interpolation - Process of going to Computerized Program.

Special conditions photoperiod 12 light 12 dark - Modified to 16 light and 8 dark

Methods used for all chemical analyses EPA approved Standard Methods

Other relevant information All test chambers are painted by gross bubblers - Percentage iterations retest BID and on test days excessively high Concentrations and Volatiles. Dilutions performed three fish randomly added. Washing test chambers - Hot water detergent, Water Rinse - Will prefer to Hard Rinse - Should include at Silent Rinse (Acetone acceptable)

ATTACHMENT D

CHEMICAL LABORATORY PROCEDURES EVALUATION

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER
DENVER, COLORADO 80225

TO : Dr. Wayne C. Smith
Process Control Branch

DATE: June 1, 1978

FROM : Technical Coordinator for
Inorganics and Air Analysis

SUBJECT: Compliance Monitoring Inspections for Union Carbide - Institute, West Virginia; South Charleston WWTP - South Charleston, West Virginia; and Union Carbide - South Charleston, West Virginia

Attached are my evaluations of the two above-mentioned Union Carbide facilities as well as the South Charleston Wastewater Treatment Facility along with completed "Self-Monitoring Program" sheets.

If there are any questions concerning the inspections, please contact me.



D. David Vietti

Attachments

cc: Meiggs
Carter
Slovinski
Masse

UNION CARBIDE
South Charleston, West Virginia

April 12, 1978

<u>Inspection Attendees</u>	<u>Affiliation</u>
Jack Worstell	Department Head, Environmental Protection Department, Union Carbide
M. E. Griffith	Department Head, Analytical Section, Union Carbide
Connie McCorkle	Chemist, Union Carbide
Gilbert Melln	Chemist, Union Carbide
David Vietti	Chemist, EPA-NEIC

Introduction

The laboratory was inspected for its skill to produce and report reliable NPDES self-monitoring data. The evaluation consisted of a review of the testing methods in use, laboratory techniques, instrument maintenance and calibration, sample preservation and holding times, data handling and reduction, as well as bookkeeping and quality control practices and documentation being followed.

The department heads and analysts all had a number of years of experience with Union Carbide in many different areas of analytical and instrumental chemistry. They were interviewed concerning chemical procedures.

A standard reference sample for TOC was left with the company and they were asked to return the results by the end of the month.

Based on the observations and findings during the evaluation, the data and information obtained, the inspection form presented on the following pages was completed and conclusions, summary, and recommendations were prepared.

Conclusions and Summary

Proper preservation procedures for composite samples were not being adhered to. Composite samples were not iced during the compositing period.

Excellent method development and implementation has been established for the analysis of the vinyl chloride monomer.

The method that is referenced is "Organics by Purge and Trap-Gas Chromatography" contained in the protocol for Priority Pollutant screening distributed by EPA-EMSL in Cincinnati.

An excellent analytical quality control program consisting of routine and blind duplicates as well as blind and routine spikes and reference samples

is being utilized for all permit parameters. The technical staff has been and is working jointly with the Union Carbide staff on methods development and implementation in regard to the parameters in their NPDES permit.

Results of the reference sample was excellent.

Recommendations

1. The samples should be iced while compositing.
2. Their excellent quality assurance program, quarterly round robins, etc. should continue.
3. Their methods developments for NPDES monitoring in conjunction with the Union Carbide Technical Center with regard to GC/MS work on toxic organics should be augmented.

SELF-MONITORING PROGRAM

On the following items, code 1 = yes, 2 = no, 3 = undetermined,
4 = not applicable.

RECORDS AND REPORTS

- ☒ 1. Properly maintained records of date, exact place and time of sampling.
- ☒ 2. Properly maintained records of the dates samples were analyzed.
- ☒ 3. Properly maintained records of who performed the analyses.
- ☒ 4. Properly maintained records of the analytical techniques and methods used.
- ☒ 5. Properly maintained records of the results of analyses.
- ☒ 6. Records maintained for a minimum of ^{one}~~three~~ years including all original strip chart recordings (continuous monitoring instrumentation calibration, maintenance records).
- ☒ 7. Plant operating records kept including operating logs of each treatment unit.
- ☒ 8. Results of sample analyses correctly calculated and recorded.
- ☒ 9. Self-monitoring frequency and parameters conform to permit requirements.
- ☒ 10. Laboratory records consistent with DMR data.
- ☒ 11. Records maintained of major contributing industries using publicly owned treatment works.
- ☒ 12. Records maintained of major contributing industries' compliance/non-compliance status.
- ☒ 13. Quality assurance records kept including spiked samples, laboratory equipment calibration, etc.

Other Comments on Records and Reports:

2-6. An excellent data and recordkeeping system in use. All the original documents and benchsheets are kept and stored in a central file for one year and then sent to the company's headquarters central file for storage. Each analyst has their own record notebook and they are reviewed periodically by the supervisory chemists.

13. A first class company internal auditing system in existence and in use.

- ☒ 7. When answer to No. 6 is yes, results are being reported in permittee's Discharge Monitoring Form (EPA No. 3320-1).
- ☒ 8. When necessary during compositing, samples are properly iced.
- ☒ 9. Proper preservation techniques used.
- ☒ 10. Flow proportioned samples obtained where required by permit.
- ☒ 11. Sample holding times prior to analyses in conformance with 40 CFR 136.3 regulations.

Other Comments on Sampling Techniques:

8-9. During compositing, the samples are not being iced.

LABORATORY PROCEDURES

- ☒ 1. EPA approved analytical testing procedures used (40 CFR 136.3).
- ☒ 2. If alternate analytical procedures are used, proper approval has been obtained.
- ☒ 3. Parameters other than those required by the permit are analyzed.
- ☒ 4. Commercial laboratory utilized.
Name _____
Address _____
- ☒ 5. Commercial laboratory State certified.
- ☒ 6. Satisfactory calibration and maintenance of instruments and equipment.
- ☒ 7. Quality control procedures used.
- ☒ 8. Duplicate samples are analyzed. 50 % of time.

- and reference
- ☒ 9. Spiked samples are used. 50 % of time.
- ☒ 10. Laboratory records properly maintained.
- ☒ 11. Laboratory employees qualified.

General Comments on Laboratory Procedures:

5. The state of West Virginia does not have a state certification program.

7-9. The laboratory quality assurance program consists of quality control analysis of routine and blind duplicate samples as well as known and unknown spiked and reference samples. Also, round robin samples supplied by the Union Carbide Technical Center coordinator are analyzed quarterly. The program is administered internally within the Union Carbide South Charleston Plant and the results look excellent.

Results of NEIC Quality Control Check Samples Analyzed by Laboratory

<u>Parameter</u>	<u>Union Carbide, South Charleston mg/l</u>	<u>Time mg/l</u>
TOC	1) 39.5 2) 160.0	1) 44.8 2) 165.0

APPENDIX B

LITHIUM FLOW VERIFICATION PROCEDURES AND SAMPLING TECHNIQUES

Lithium Flow Verification Procedures

Flow verification was accomplished with the tracer dilution technique, using lithium as the tracer. The concept employed is that mass is conserved (i.e., mass of tracer in equals mass of tracer out). Fundamental to the use of this technique are the following conditions:

1. A conservative tracer.
2. A constant tracer injection rate and an accurate measurement of the rate.
3. An accurate measurement of the tracer concentrate, background tracer levels, and diluted tracer in the flow stream to be measured.
4. Complete mixing in the flow stream to be measured.

It was determined that all these respective criteria could be met by:

1. Using lithium (Li) in the form of lithium chloride as a tracer. Previous studies have shown that spiking various types of wastewater with known amounts of lithium results in an overall average recovery of 100%.
2. Metering the injected tracer solution with low flow rate, high precision pumps. During verification, injection rate was checked at least twice with a graduated cylinder and stop watch.

3. Measuring Li concentration with a Perkin-Elmer Model 403 Atomic Absorption Spectrophotometer. This instrument was calibrated before each use with lithium standards of known concentration. Concentrate samples were analyzed each time a batch was mixed. Background samples were collected and analyzed each time a flow measurement was performed.
4. Injecting the lithium chloride concentrate solution into the suction side of the effluent pump and monitoring the diluted Li tracer on the discharge side.

Flow was calculated with the following equation:

$$Q = \frac{q C_q F}{C - C_b}$$

where Q is unknown flow (mgd)

q is injection rate (l/min)

C_q is lithium concentration of injection solution (mg/l)

C is lithium concentration downstream of injection (mg/l)

C_b is background concentration of lithium (mg/l)

F is factor to convert l/min to mgd

$$(380.45 \times 10^{-6} \frac{\text{min} - \text{gal}}{\text{day} - \text{liter}})$$

Sampling Techniques

Composite samples were collected by hand at regular intervals throughout a 24-hour period and aliquoted proportional to the volume of the discharge into iced sample containers. For those samples whose nature could change during the collection period chemical preservatives were added to the sample container prior to the start of the collection period. Each of the ^{se} sample aliquots were chemically preserved upon collection. At the end of the sampling period, the chemically unpreserved portion of the sample was transferred into appropriately preserved containers, identified and transported to either NEIC mobile laboratories located at the South Charleston Sewage Treatment Company plant or the NEIC Laboratory Denver, Colorado.

Grab samples were handled as discussed above with the exception that the sample consisted of a single aliquot rather than multiple samplings.

APPENDIX C

CHAIN-OF-CUSTODY-PROCEDURES

CHAIN-OF-CUSTODY PROCEDURES (March 29, 1978)

Due to the evidentiary nature of samples collected during enforcement investigations, the possession of samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. To maintain and document sample possession, Chain-of-Custody procedures are followed.

SAMPLE CUSTODY

A sample is under custody if:

1. It is in your actual possession, or
2. It is in your view, after being in your physical possession, or
3. It was in your physical possession and then you locked it up to prevent tampering, or
4. It is in a designated secure area.

FIELD CUSTODY PROCEDURES

1. In collecting samples for evidence, collect only that number which provides a fair representation of the media being sampled. To the extent possible, the quantity and types of samples and sample locations are determined prior to the actual field work. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are transferred or properly dispatched.
3. Sample tags (see attached) shall be completed for each sample, using waterproof ink unless prohibited by weather conditions.
4. During the course and at the end of the field work, the Project Coordinator determines whether these procedures have been followed, and if additional samples are required.

TRANSFER OF CUSTODY AND SHIPMENT

1. Samples are accompanied by a Chain-of-Custody Record (see attached). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, or to the NEIC laboratory in Denver.
2. Samples will be properly packaged for shipment and dispatched to the appropriate NEIC laboratory* for analysis, with a separate Record prepared for each laboratory (e.g., Mobile Chemistry Lab, Mobile Biology Lab(s), Denver Chemistry Lab, Denver, Biology Lab). Shipping containers will be padlocked for shipment to the Denver laboratory. The "Courier to Airport" space on the Chain-of-Custody Record shall be dated and signed.

* See Appendix B of NEIC Policies and Procedures Manual for Safety Precautions When Accepting Samples From Outside Sources.

3. Whenever samples are split with a facility or government agency, a separate Chain-of-Custody Record is prepared for those samples and marked to indicate with whom the samples are being split.
4. All packages will be accompanied by the Chain-of-Custody Record showing identification of the contents. The original Record will accompany the shipment, and a copy will be retained by the Project Coordinator.
5. If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, a Government Bill of Lading should be used. Receipts from post offices and bills of lading will be retained as part of the permanent documentation.


LABORATORY CUSTODY PROCEDURES

1. A sample custodian or a designated alternate will receive samples for the laboratory and verify that the information on the sample tags matches that on the Chain-of-Custody Record included with the shipment. The custodian signs the custody record in the appropriate space; a laboratory staff member performs this function in the field. Couriers picking up samples at the airport, post office, etc., shall sign in the appropriate space.
2. The custodian distributes samples to the appropriate analysts. The names of individuals who receive samples are recorded in internal Branch records. Laboratory personnel are responsible for the care and custody of samples from the time they receive them until they return them to the custodian. Samples received after normal working hours may be analyzed immediately or stored as appropriate.


3. Once field-sample testing and necessary quality assurance checks have been completed, the unused portion of the sample may be disposed of. All identifying tags, data sheets and laboratory records shall be retained as part of the permanent documentation. Samples forwarded to the Denver laboratory for analysis will be retained after analyses are completed. These samples may be disposed of only upon the orders of the Chief, Enforcement Specialist Office and Assistant Director for Technical Programs, and only after all tags have been removed for the permanent file.

...

SAMPLE TAG

	Proj. Code	Station No.	Sequence No.	Mo./Day/Yr.	Time
	Station Location			Comp.	Grab
	ENVIRONMENTAL PROTECTION AGENCY OFFICE OF ENFORCEMENT NATIONAL ENFORCEMENT INVESTIGATIONS CENTER BUILDING 53, BOX 25227, DENVER FEDERAL CENTER DENVER, COLORADO 80225				1501
	Samplers: (Signature)				

obverse


<u>Sample Type/Preservative(s)</u> 1. General Inorganics/Ice 2. Metals/ HNO_3 3. Nutrients/ H_2SO_4 & Ice 4. Oil & Grease/ H_2SO_4 & Ice 5. Phenolics/ H_3PO_4 & CuSO_4 & Ice 6. Cyanide/ NaOH & Ice 7. Organic Characterization/Ice 8. Volatile Organics/Ice 9. General Organics/Ice 10. Tracer/None 11. Solids - Inorganics/Ice or Freeze 12. Solids - Organics/Ice or Freeze 13. Biol. - Inorganics/Ice or Freeze 14. Biol. - Organics/Ice or Freeze 15. Source Filter/None 16. Probe Wash/None 17. Impinger Catch/None 18. Ambient Filter/None 19. Solid Adsorbant/Ice or Freeze 20. Ambient Impinger/Amb. or Ice 21. Benthos/Ethanol or Formal 22. Bacteriology/Ice 23. Plankton/Formal; HgCl_2 ; Lugol's 24. Chlorophyll/Ice or Freeze 25. Pathogenic Bacteria/Ice 26.
Remarks: <div style="text-align: center;">..</div>
☆GPO 777-941

reverse

0000

APPENDIX D

ANALYTICAL METHODS AND QUALITY CONTROL

CHEMISTRY ANALYTICAL METHODOLOGY AND QUALITY CONTROL

The analytical procedures used by the Chemistry Branch are described in the following sections which are organized by working groups, Inorganics and Organics. The quality control procedures and data used to verify the quality of the analytical data are also discussed.

INORGANICS

The samples from this study were analyzed for the following inorganic parameters: TOC, NH_3 , total Kjeldahl nitrogen, chloride and phenolics. Methods approved by the EPA for the NPDES program (40 CFR 136, Federal Register, December 1, 1976) were used to analyze all samples. The references to the methods for each parameter are listed in Table 1 below.

Table 1

Parameter	Technique	Detection Limit, mg/l	Reference
TOC	Combustion-Infrared	1	Std. Methods ^a pg 532
NH_3	Automated phenolate	0.05	Std. Methods pg 616
Phenolics	4-AAP colorimetric	0.001	Std. Methods pg 574
TKN	Kjeldahl digestion, Automated phenolate	0.2	EPA Manual ^b pg 175 Std. Methods pg 616
Chloride	Mercuric nitrate	1	Std. Methods pg 304

a Std. Methods = "Standard Methods for the Examination of Water and Wastewater", 14th edition (1975).

b EPA Manual = "Methods for Chemical Analysis of Water and Wastes", 1974.

Additional precautions taken during the analysis of the samples are discussed below by parameter.

TOC

Samples were acidified and homogenized before analysis to drive off the inorganic carbon and reduce the particle size. Samples were alternately homogenized and analyzed until two successive results agreed within 2 mg/l. One reference sample was analyzed with an accuracy of 101%. Three samples were spiked with a mean recovery of 107%.

Chloride

Low and high level mercuric nitrate reagents were used for samples below and above 25 mg/l. Eight samples were spiked with a mean recovery of 100%. A reference sample was analyzed on five days with an accuracy of 100%. Fifteen samples were analyzed in duplicate with a mean FSD of 1%.

Ammonia

The auto-analyzer method was adapted to 0-30 mg/l full scale by adding a dilution loop onto the front end of the manifold. Two reference samples were analyzed six times each with accuracies of 98 and 104%. Seven samples were analyzed in duplicate with five samples below the detection limit. The RSD of the two pairs of data is 1.6%.

Phenolics

All absorbance were measured against a chloroform blank. Three samples were spiked with a mean recovery of 98%. One reference sample was analyzed with 92% recovery.

TKN

The method was set up for 20 mg/l TKN-N full scale. Samples over 20 mg/l were diluted and re-digested before analysis. A reference sample was analyzed five times with 92% accuracy.

ORGANICS

Several techniques for the analysis of organic compounds were utilized for the waste source evaluation. Identification of individual organic compounds was made by combined gas chromatography/mass spectrometry (GC/MS) while capillary column gas chromatography (CPGC) was used for quantitation and confirmation of identity. The samples were analyzed for neutral extractables and volatiles. A grab sample collected in April was analyzed for priority pollutants.

NEUTRAL EXTRACTABLE ANALYSIS

GC/MS Identification: Methylene chloride extracts of water, and acetone extracts of the sediment samples were concentrated to small volumes and exchanged with isooctane and analyzed by CG/MS. The initial identification was made using a manual search utilizing reference spectra analyzed under the same instrumental conditions used for the samples.

A library of standard spectra of the commonly occurring compounds was made using a computer assisted evaluation program (1). In those instances where other than the commonly occurring compounds appeared, a more complete search was made utilizing the complete computer library and a followup manual search (2) (3) (4) (5).

Capillary Column Gas Chromatography: All the sample extracts were analyzed by capillary column gas chromatography. Initial screening and quantitation were carried out on this gas chromatograph. Compounds were identified by coincidence of retention times with standards and quantitation was made using peak height measurement.

Packed Column Gas Chromatography: All the extracts were analyzed by packed column gas chromatography using a computer-controlled automatic injector. Initial screening was carried out on this gas chromatograph.

References

1. "INCOS Data System - MSDS Operator's Manual, Revision 3". Finnigan Instruments, March 1978.
2. "Eight Peak Index of Mass Spectra", Mass Spectrometry Data Centre, Aldermaston, Reading, UK. Second Edition 1974.
3. "Registry of Mass Spectral Data", Stenhagen, Abrahamson and McLafferty, John Wiley & Sons, New York 1974.
4. "Atlas of Mass Spectra Data" edited by: Stenhagen, Abrahamson and McLafferty, John H. Wiley & Sons, New York 1969.
5. Computer Assisted Evaluation of Organic Priority Pollutant GC/MS Data - NEIC, September 1978.

Quality Control: Quality control procedures consisted of analysis of selected duplicate samples, analysis of solvent and procedure blanks to identify interferences, and gas chromatographic analysis of standards on a daily basis to confirm the integrity of the GC system. For mass spectrometry, a daily calibration was used to tune the mass spectrometer, and assure the integrity of the complete system. The quality control procedures are documented in the attached methodologies. Attachments 5, 6, 7, 8, 9, 10.

VOLATILE ANALYSIS

GC/MS Identification: An aliquot (5 ml) of a water sample was purged with inert gas. The lower molecular weight purgable organic compounds were stripped from the sample and trapped on a porous polymer. These compounds were then desorbed from the column by reversing the gas flow and rapidly heating the trap. The volatile organics released were collected on an analytical GC column at room temperature. After collection, the GC column oven was heated at a uniform rate and the eluted compounds analyzed by the mass spectrometer. The common volatile organic solvents are all identified using this technique and it also includes the identification of the volatile priority pollutants. This procedure is the method recommended for the priority pollutants (1). The identification again was made using a computer-assisted evaluation program as for the neutral extractables (2). A library of standard spectra was created by analyzing all the commonly occurring organics in the UCSC samples, and adding these to the library. The samples were routinely searched for these compounds for each sample analyzed by GC/MS.

Quantitative results were obtained using an internal standard computer technique (2) (3).

REFERENCES

1. "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.
2. "INCOS Data System - MSDS Operator's Manual - Revision 3", Finnigan Instruments, March 1978.
3. Computer Assisted Evaluation of Organic Priority Pollutant GC/MS Data - NEIC, September 1978.

Quality Control: Quality control procedures consisted of daily routine calibration of the GC/MS, analysis of an organics free-water blank, and a standard mix at a concentration near midpoint of the standard calibration curve. The calibration curve was previously established by analyzing each standard over a typical working range of 20 to 200 ppb concentration, with response factors calculated relative to an internal standard. Field blanks were analyzed with each set of samples. Replicate analyses were run on at least two samples for every set of twenty samples or less.

QUALITY CONTROL

Blanks

One contaminant, methylene chloride, appeared consistently in the blank results. Blanks for the fifteen days of analysis gave a methylene chloride value of 3 ± 2 $\mu\text{g/l}$.

Summary of blank results ($\mu\text{g/l}$)

Compound	Times Detn 15 samples	Range of Values	Average
Methylene chloride	12	2-13	3 ± 2
Toluene	2	2-5	nil
1,1,1-Trichloroethane	1	3	nil

Duplicates

Nine samples, six of them composites, were analyzed in duplicate. Ten compounds of interest were determined in these analyses. The results are summarized as follows:

Compound	Times Detn (9 samples)	Deviation
Benzene	2	+ 8%
Bromodichloromethane	1	+ 100%
Carbon tetrachloride	1	+ 50%
Chloroform	6	+ 27%
1,2-Dichloroethane	1	+ 20%
Ethylbenzene	1	+ 80%
Methylene chloride	6	+ 45%
Tetrachloroethene	1	+ 25%
Toluene	2	+ 48%
1,1,1-Trichloroethane	1	+ 17%

Recoveries

Four samples were spiked with standard mix to give each component at a concentration of 200 $\mu\text{g/l}$. Recoveries are listed below.

Compound	Percent Recovery
Benzene	60
Bromodichloromethane	108
Bromoform	127
Carbon tetrachloride	80
Chlorobenzene	86
2-Chloroethylvinyl ether	125
Chloroform	88
Chlorodibromomethane	113
1,2-Dichloroethane	114
1,1-Dichloroethene	81
trans-1,2-Dichloroethene	77
1,2-Dichloropropane	84
Ethylbenzene	72
Methylene chloride	93
1,1,2,2-Tetrachloroethane	140
Tetrachloroethene	83
Toluene	87
1,1,1-Trichloroethane	78
1,1,2-Trichloroethane	121
Trichloroethene	85
Vinyl chloride	97
Average	95

EPA Quality Control Sample

An internal quality control sample, prepared by the EPA Environmental Monitoring and Support Laboratory Quality Assurance Branch, Cincinnati, was analyzed in triplicate. This QC sample, containing volatile organics, was No. 1276 WS.

Compound	Analytical Results	"True" Values	Error
Bromochloromethane (IS)	180 \pm 20 μ g/l	200	10%
Bromodichloromethane	13 \pm 2	12	8
Bromoform	13 \pm 1	14	8
Carbon tetrachloride	9 \pm 1	13	31
Chloroform	60 \pm 7	68	12
Chlorodibromomethane	12 \pm 1	17	29
1,2-Dichloroethane	23 \pm 2	27	15
Tetrachloroethene	8 \pm 1	9	11
1,1,1-Trichloroethane	9 \pm 1	11	18
Trichloroethene	17 \pm 2	19	11

ATTACHMENT I

Neutral Extraction Technique for Organics Analysis
September 1978

1.0 Scope and Application

- 1.1 This procedure is applicable for analysis of water and wastewater samples for a broad spectrum of organic pollutants.

2.0 Summary of Method

- 2.1 Water and wastewater samples are extracted with CH_2Cl_2 (dichloromethane) at a neutral pH. The extract is dried and concentrated with the addition of acetone and iso-octane to exchange solvents. The resultant extract concentrate is subjected to GC and GC/MS analysis to identify and quantitate the organic pollutants present.

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 Definitions and Comments

5.0 Interferences

- 5.1 Solvents, glassware and reagents could be sources of contamination. Therefore, at least one "Reagent Blank" must be prepared contacting the solvent with all potential sources of contamination. This blank should then be processed through the same analytical scheme as the associated samples.
- 5.2 Typical interferences from reagents are:
4-methyl-4-hydroxy-2-pentanone (diacetone alcohol)
from acetone, phthalate esters from Na_2SO_4 ,
cyclohexene from dichloromethane.

6.0 Apparatus

- 6.1 Separatory funnels: 2l and 4l glass with glass or teflon stoppers and stopcocks. No stopcock grease used.
- 6.2 Drying column: All glass 3 cm x 50 cm with attached 250 ml reservoir.
-

- 6.3 Concentrator: 250 or 500 ml Kuderna-Danish evaporative concentrator equipped with a 5 or 10 ml receiver ampule and a 3 ball Snyder column.

7.0 Reagents

- 7.1 Extraction solvent: Pesticide analysis grade CH_2Cl_2 (dichloromethane) (Burdick and Jackson or equivalent)

7.2 Exchange solvents

- 7.2.1 Exchange solvent: Pesticide analysis grade acetone (Burdick and Jackson or equivalent)

- 7.2.2 Exchange solvent: Iso-octane suitable for pesticide analysis (Burdick and Jackson or equivalent)

- 7.3 Drying agent: Analytical reagent grade granular anhydrous Na_2SO_4 (sodium sulfate). Washed with CH_2Cl_2 prior to use.

- 7.4 Glass wool that has been extracted with CH_2Cl_2 prior to use.

- 7.5 6N NaOH for pH adjustment.

- 7.6 6N HCl for pH adjustment.

- 7.7 pH paper for pH measurement.

8.0 Procedure

- 8.1 If low concentrations of pollutants are expected, measure 3 l of sample for extraction. Otherwise, one l is sufficient.
- 8.2 Measure and record the initial pH. Adjust the pH to 6-8 if necessary, and record the adjusted pH.
- 8.3 Extract the sample with 3 successive extractions of 100, 50 and 50 ml of CH_2Cl_2 for 1 liter samples and 200, 100, 100 ml of CH_2Cl_2 for 3 liter samples.

If emulsions form, use a wire or stirring rod to break it, pass the emulsion through glass wool or centrifuge if necessary. Combine the extracts and measure the volume recovered. 85 percent constitutes an acceptable recovery.

- 8.4 Place a glass wool plug in a drying column and add ca 10 cm of Na_2SO_4 . Wash the 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_2 and acetone and transfer to a KD assembly. Add 5 ml of iso-octane for 1 liter extracts and 5 ml iso-octane for 3 liter extracts.
- 8.5 Concentrate on a hot water bath at 80-90°C until the extract stops boiling. Quantitatively transfer the receiving tube contents to a graduated centrifuge tube. Adjust the volume to 2 or 5 ml by either adding more iso-octane or evaporating the excess iso-octane under a gentle stream of carbon filtered air. Transfer to a 12 ml vial and cap with a teflon lined cap. (Note: The final extract volume should depend on the sample. Extracts containing high concentrations of pollutants may not require concentrations to 5 ml while cleaner samples may require a final volume of 2 ml).

9.0 Quality Control

- 9.1 A representative group of the organic pollutants of interest should be spiked into water and carried through the extraction procedure, recoveries calculated and compared to literature values (if available).

10.0 Calculations

- 10.1 Solvent Recovery:
 $\% \text{ recovery} = \text{Volume recovered (ml)} * 100 / \text{volume added (ml)}$
- 10.2 Pollutant Recovery:
 $\% \text{ recovery} = \frac{(\text{Concentration measured} - \text{initial concentration}) * 100}{\text{Concentration added}}$

11.0 Precision and Accuracy

- 11.1 Precision and accuracy vary with the pollutants being measured. Recoveries range from 48 - 119 percent and precision values range from 1 to 9 percent relative standard deviation (% RSD). Typical values are ± 5 % RSD.

12.0 References

- (1) "An EPA GC/MS Procedural Manual-Review Copy", Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

ATTACHMENT II

Summary of Recovery Data
for Neutrals Extractable Organics
in Kanawha River Project

Background

A number of organic compounds were identified in the Kanawha River Project reconnaissance samples. Some of these compounds were available and synthetic sample recoveries were measured to help validate the extraction methods used. Even though few of the compounds used in this evaluation were found in subsequent survey samples, the diversity of the compounds used illustrate the method's capability to recover a broad spectrum of pollutants.

Experimental

A standard mix was prepared containing 50 ng/ul of each compound in acetone. One and 3 l tap water samples were spiked with the standard mix resulting in concentrations of 2500 and 10 ug/l respectively. The samples were then extracted with CH_2Cl_2 and concentrated with the addition of iso-octane as an exchange solvent in Kuderna-Danish evaporative concentrators. The final volumes were 5 and 1 ml for the 1 and 3 l samples respectively. The extracts were then analyzed by gas chromatography with a flame ionization detector using a 6 ft x 2 mm glass column packed with 60/80 mesh GC-Q coated with 6% OV101. The response of each component was measured by area integration using a computerized data reduction system.

Results & Discussion

The nine compounds and their recoveries are listed in Table 1. The 1 l samples at high concentrations show good recoveries. The large variation of butyl carbitol acetate may be attributable to a data system error. Results for 3 l samples at 10 ug/l show large variations and a

Table I. Recoveries for selected organics from tap water for neutral pH extractions.

Name	1 l extraction - 2500 ug/l % Recovery ^a	3 l extraction 10 ug/l % Recovery ^b
methyl cellosolve acetate	79 ± 9	16 ± 0.3
styrene	99 ± 1	167 ± 25
anisole	119 ± 4	328 ± 20
phenol	48 ± 3	0
o-cresol	98 ± 4	105 ± 0.1
N,N-dimethyl aniline	108 ± 5	88 ^c
benzothiazole	103 ± 4	27 ^c
butyl carbitol acetate	86 ± 69	83 ^c
2,6-dinitrotoluene	119 ± 53	217 ± 2

a = Values represent results of 3 replicate sample analyses

b = Values represent results of 2 replicate sample analyses.

c = No recovery in one sample, value is result where recovery was observed.

number of cases of no recoveries. The limiting factor for detection is most likely the use of packed column gas chromatography and could account for a large part of the variation. Recoveries at low levels, however, can be expected to be more variable due to the larger samples and extreme concentration factors required.

Conclusion

Extraction recoveries can be expected to be quite good at high component concentrations. At low levels, 10 ug/l, the variation will be larger and with packed column gas chromatography, may be unacceptable.*

*Note: GlassCapillary column gas chromatography (GC) was used for quantitation of survey samples lowering the effective GC detection limit by a factor of ca 10.

and acrylonitrile were prepared in a separate standard mix. D-15

For gaseous standards - only vinyl chloride in this procedure - a primary standard solution was prepared by bubbling the gas into a tared volumetric flask of suitable solvent (methanol in this instance). The mass increment was measured and the concentration calculated. As with the liquid standards, a calculated volume was then diluted for the standard mix.

For internal standards, 100 mg each of bromochloromethane and 1,4-dichlorobutane were made up to 20 ml in methanol. For each day of analysis, 20 μ l of this solution was diluted to 1.0 ml in water, and 10 μ l of this preparation was added to each 5 ml sample aliquot, to give 200 μ g/l of each component.

Analysis Procedure

The helium purge gas flow on a liquid sample concentrator (LSC) was adjusted to 40 ml/min. and the LSC valve set to the purge position. The VOA sample was removed from cold storage and brought up to ambient temperature. The bottle was carefully opened and the sample water poured into a 5-ml syringe to overflowing. The syringe plunger was replaced and the sample volume adjusted to 5.0 ml, and the syringe valve was closed. A 10 μ l aliquot of the internal standard (IS) mixture was introduced into the sample by opening the valve and injecting the IS into the syringe. An 8-inch needle was attached to the syringe valve, and the sample was injected into the purging chamber of the LSC. The timer of the LSC was set to purge the sample for 12 minutes, with the silica gel-Tenax trap at ambient temperature (20-25°C).

At this time, the oven of the gas chromatograph was brought to near ambient temperature by opening the oven door with the heater off.

After the 12-minute purge time the sample from the trap was injected into the GC by turning the valve to the desorb position and starting a timer for the analysis cycle (time zero). The GC-MS data collection was started at one minute; at four minutes the desorb was ended by turning the valve back to the purge position, and simultaneously the GC oven was closed and the oven temperature was set at 60°C. The temperature program conditions: isothermal at 60°C until 8 minutes; program at 8°C/mm to 170°C; hold at 170°C to the end of the program at 29 minutes.

After the sample purge, and while data was being collected, the trap was baked out at 210°C for ten minutes, then allowed to cool to ambient temperature. Also, the sample tube was removed from the assembly, washed in methanol and baked out, and replaced on the LSC by a clean tube.

ATTACHMENT III

METHODS: VOLATILE ORGANICS ANALYSESPurge and Trap - Gas Chromatography-Mass Spectrometry

This method is basically drawn from "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S.E.P.A. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268, March, 1977, revised April, 1977, and "Volatile Organic Compounds by GC/MS", U.S.E.P.A., NEIC, Denver, Colorado, 80225, July, 1978.

Scope

The Volatile Organics Analyses (VOA) method is designed to determine "priority pollutants" associated with the Consent Decree that are amenable to the purge and trap method. It is a gas chromatographic-mass spectrometric (GC-MS) method intended for the qualitative and quantitative determinations of these compounds.

The purge and trap method is complementary to the liquid-liquid extraction method. There is an area of overlap between the two methods, and some compounds may be analyzed by either method. The efficiency of recovery depends on the vapor pressure and water solubility of each compound. The overlap region in general consists of compounds which boil between 130° and 150°C (1 atmosphere pressure), with a water solubility of approximately two percent. The method of choice for these overlap region compounds is selected according to overall method efficiency and dependability.

Special Apparatus

Tekmar Liquid Sample Concentrator, Model LSC-1; Tekmar Company, P.O. Box 37202, Cincinnati, Ohio, 45222.

Special sorbent trap for LSC: stainless steel tube 1/8-inch O.D. by 17-cm.; packing from inlet, 1 cm glass wool, 5 cm. type 15 silica gel, 8 cm Tenax, 60/80 mesh; 3 cm. glass wool.

GC Column: a 6-ft. by 1/8-inch OD column packed with 0.2% Carbowax 1500 on 60/80 mesh Carbopack C; manufactured by Supelco, Supelco Park, Bellefonte, Pennsylvania, 16823.

Standards

For liquid standards, a primary standard solution for each compound was prepared from 10 ul of the compound in 10 ml of methanol. Concentrations were calculated from the density of each compound, and a standard mix was prepared by diluting a calculated volume of each solution (ca 150 ul) together to a total volume of 10 ml in methanol. Due to instability, acrolein

steps are taken by the operator to stabilize the operation.

To determine the precision of the method, replicate aliquots of environmental samples are analyzed, with at least one set of replicate analyses made for each group of 20 samples or less analyzed. Over the course of a survey, replicate analyses are made on samples which represent the entire range of concentrations and interferences found in that survey.

To determine the recovery of the method, at least one environmental sample for each group of 20 samples or less is re-analyzed after the addition of a spike mixture. The spike concentration should approximately double the background concentration. If the background is negligible, the spike concentration should be five to fifteen times the lower detection limit.

The qualitative and quantitative determinations of the volatile priority pollutants are based upon the characteristic masses and their relative and absolute intensities, from which an extracted ion current profile is obtained for each compound. Details of these determinations are presented in "Computer-Assisted Evaluation of Volatile Organics GC/MS Data", NEIC, July, 1978.

Mass Spectrometer Parameters

The mass spectrometer used was a Finnigan 1015 S/L interfaced to a Systems Industries System 150 data system. The operational parameters include: electron energy, 70 ev; mass range, 20-27 and 33-260 amu; integration time/amu, 17 milliseconds; samples/amu, 1.

GC Column Preparation

The column was connected at the inlet, the helium flow was adjusted, and the column was baked out overnight. This column must be handled with care, due to the fragile character of the Carbopack.

MS Calibration

The mass spectrometer was calibrated daily with perfluorotributylamine (FC 43), according to the Finnigan instrument manual. A further calibration check was made with the first run each day of analysis of a blank with internal standards added. The mass spectrum of bromochloromethane must meet these specifications:

<u>m/e</u>	<u>Relative Intensity</u>
49	100
130	65-98
128	50-75
51	25-35

Quality Assurance

The analysis of blanks is most important in the purge and trap technique, since the purging device and the trap can be contaminated by residues from very concentrated samples and by vapors in the laboratory. Blanks are of low-organic water, prepared by passing distilled water through an activated carbon column. If positive interferences are observed, the blank is repeated; if interferences persist, appropriate measures are taken to eliminate them before analyses are made.

The precision of the method is determined by running blanks dosed with the internal standards, bromochloromethane and 1,4-dichlorobutane. These compounds represent early and late eluters over the range of the Consent Decree compounds and are not on the list.

Each sample is dosed with the internal standards and analyzed by the set procedure. The operator monitors the sensitivity of the system to the internal standards as compared with blank runs; if the deviation is too great, a sample run is repeated. If excess deviation of sensitivity persists, appropriate

ATTACHMENT IV

Computer Assisted Evaluation of
Organic Priority Pollutant GC/MS Data

NEIC - September 1978

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)

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 unambiguous and marginal data may be eliminated.

4.0 Interferences

- 4.1 In some cases, a spectrum may match the library reference sufficiently 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.
-

5.0 Apparatus

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

6.0 Procedure

6.1 Procedure Setup

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

6.2 Library Setup

- 6.2.1 Build user libraries for each analytical class of priority pollutants (VOAs, base-neutrals and phenols). Appendices III, IV and V are library lists of example libraries. The first entry must always be the internal standard and each entry must include the quantitation parameters and relative retention times.
- 6.2.2 Execute PPEVAL, edit the quantitation list for accuracy and update the library parameters using commands in "QUAN".
- 6.2.3 Using the "LIBR" program, generate hard copies of library spectra for reference. Using the library list editor, "EDLL", generate summaries of the entries and quantitation parameters as in Appendices III, IV and V.

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 namelist containing the names of the data files to be processed.
- 6.3.3 Execute the procedure as follows:

PPEVAL library, namelist, yes (no)

Where: library is the appropriate user library name.

 namelist is the list containing the files to be processed.

 yes (no) selects print out of the spectra at a peak that was identified by the procedure.
- 6.3.4 Appendix VI is an example of PPEVAL output for a sample 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 (library list editor) 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", US 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", US EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1978.

Appendices

- I. List of procedures, file names, and functions for PPEVAL
 - II. Trace of PPEVAL
 - III. VOAs library list
 - IV. Base neutrals library list
 - V. Phenols library list
 - VI. Example PPEVAL output
-

PROCEDURE OR METHOD *****	FUNCTION *****
PPEVAL	INITIALIZATION
PPEVA	DATA FILE PROCESSING LOOP
PPEVB	DATA FILE PROCESSING
PPEVC	LOCATING THE INTERNAL STANDARD
PPEVD	INTERNAL STANDARD ERROR HANDLER
PPEVE	COMPOUND LOCATER
PPEVF	NOT DETECTED ERROR HANDLER
PPEVG	IDENTIFICATION CHECK
PPEVH	SPECTRA PRINTING
PRINP1	IDENTIFICATION REPORT HEADER
PRINP2	INTERNAL STANDARD ERROR MESSAGE

APPENDIX IIA.

TRACE OF PROCEDURE PPEVAL

```

* ERASE
* ;[***** PRIORITY POLLUTANT EVALUATION PROCEDURE *****]
* ;[THIS PROCEDURE MAY BE USED TO EVALUATE GC/MS DATA ]
* ;[FOR PRIORITY POLLUTANT (EPA SECTION 307(A)) COMPOUNDS ]
* ;[THE PROCEDURE UTILIZES INTERNAL STANDARDS AND RELATIVE ]
* ;[RESPONSE FACTORS FOR QUANTITATION. THE MSDS OPTION ]
* ;[SEARCH IS USED TO LOCATE AND IDENTIFY PEAKS. THE EPA ]
* ;[IDENTIFICATION CRITERIA, E.G., THREE IONS PER COMPOUND ]
* ;[IS USED TO LOCATE THE COMPOUND OF INTEREST. MORE IONS ]
* ;[HOWEVER MAY BE USED AS THE FIT OF THE SEARCH ROUTINE WILL ]
* ;[YIELD MORE SPECIFICITY FOR THE COMPOUND. THE FULL ]
* ;[SPECTRUM IS OUTPUT IN ORDER TO PROVIDE CONFIRMATION OF ]
* ;[THE PRESENCE OF THE COMPOUNDS. ]
* ;[*****]
* ;[TO USE PPEVAL, BUILD A LIBRARY CONTAINING THE SPECTRA OF ]
* ;[THE COMPOUNDS OF INTEREST. INCLUDE THE QUANTITATIVE DATA]
* ;[THAT IS NECESSARY AS DESCRIBED IN THE MSDS MANUALS. ]
* ;[CREATE A NAMED LIST WITH THE NAMES OF THE FILES TO BE ]
* ;[PROCESSED. EXECUTE THE PROCEDURE AS FOLLOWS: ]
* ;[ PPEVAL LIBRARYNAME, NAMEDLIST, YES(NO) ]
* ;[WHERE YES(NO) SELECTS PRINTED SPECTRA OF ACCEPTABLE ]
* ;[MATCHES. E.G. PPEVAL VO.SAMPLE ]
* ;[ WRITTEN 10AUG78 D.J.LOGSDON II EPA-NEIC 303-234-4661 ]
* ;[ REVISED 05SEP78 D.J.LOGSDON II EPA-NEIC 303-234-4661 ]
* ;SETS PPSCAN;EDLL YES(-;S;W;E);EDLL NO(-;W;E)
* ;SETN $2;SET4 $1;PPEVA;FEED;BEEP;BEEP;BEEP
*
ERASE
SETS PPSCAN
EDLL YES (-;S;W;E)
EDLL NO (-;W;E)
SETN $2
SET4 $1
PPEVA
* ERASE
* ;[PART OF PROCEDURE PPEVAL ]
* ;[GET THE NEXT NAMED LIST ENTRY AND CONTINUE PROCESSING ]
* ;[CAT PPEVB ]
* ;GETN;PPEVB;LOOP
*
ERASE
GETN
PPEVB
* ERASE
* ;[PART OF PPEVAL. THIS PROCEDURE SETS THE LIBRARY ENTRY ]
* ;[POINTER TO THE FIRST ENTRY, WHICH MUST ALWAYS BE THE INTERNAL ]
* ;[STANDARD. PPEVC IS THEN CALLED AND THE INTERNAL FOUND ]
* ;[THE SPECTRUM NUMBER OF THE INTERNAL STANDARD IS ]
* ;[STORED IN I10 FOR FUTURE REFERENCE. THE LIBRARY POINTER ]
* ;[IS THEN RESET TO THE BEGINNING, THE QUANTITATION LIST SET TO ]
* ;[THE FILE NAME AND EMPTIED OUT. PPEVE IS CALLED TO LOCATE EACH ]
* ;[COMPOUND (IF PRESENT). QUAN IS THEN CALLED TO CALCULATE ]
* ;[THE RESULTS AND THE PROCEDURE RETURNS TO PPEVA TO GET THE ]
* ;[NEXT FILE TO PROCESS. ]
* ;FILE(K PRIN.99/N;E)
* ;EDLL PPLIST(-;W;E)
* ;SETI *1;PARA(I;H;E);CHRO(I;H1,1050,350;E);SET4 *1;PPEVC;SETI0 I14;SET4 *0
* ;SETQ $1;EDOL(-;W;E);EDSL(-;W;E);SETL $3;PPEVE;QUAN(I;H;E)
* ;EDLL PPLIST(B!1;E)
* ;PRIN(QP1)
* ;FILE(C PRIN.99/N,M;E)
* ;FEED
* ;BEEP
*
ERASE
FILE (K PRIN.99/N;E)
EDLL PPLIST (-;W;E)
SETI *1
PARA (I;H;E)

```

APPENDIX IIB.

```

CHRO (I:H1.1050.350:E)
SET4 *1
PPEVC
  * ERASE
  * ;[PART OF PPEVAL ]
  * ;[ROUTINE TO FIND AN INTERNAL STANDARD IN A SAMPLE ]
  * ;[USE A REVERSE SEARCH TO LOCATE THE INTERNAL STANDARD]
  * ;SET14 *0
  * ;SEAR/V(I;S;V2500000;N2.10.600;E;D-60.60;E)
  * ;PPEVD
  *
  ERASE
  SET14
  SEAR (I;S;V2500000;N2.10.600;E;D-60.60;E)/V
  PPEVD
    * IF PPEVD ,114
    * ;[PART OF PPEVAL ]
    * ;[NO INTERNAL STANDARD FOUND]
    * ;PRIN(0P2)
    * ;RETU PPEVB
    *
    IF PPEVD,114
    PRIN (0P2)
    RETU PPEVB
SET10 114
SET4
SET0 S1
EDOL (-;W;E)
EDSL (-;W;E)
SETL S3
PPEVE
  * ;[PART OF PPEVAL ]
  * ;[THIS ROUTINE LOCATES COMPOUNDS IN THE ]
  * ;[SAMPLE FILE BY COMPARING THE SPECTRA IN THE LIBRARY ]
  * ;[WITH THE SAMPLE. RELATIVE RETENTION TIMES ARE USED ]
  * ;[AND REFERENCED TO THE INTERNAL STANDARD FOUND EARLIER.]
  * ;[THE LIBRARY POINTER IS BUMPED AND TESTED TO ]
  * ;[SEE IF THE LAST LIBRARY ENTRY HAS BEEN PROCESSED. ]
  * ;[THEN THE CURRENT SCAN NUMBER IS SET TO THE INTERNAL ]
  * ;[STANDARD LOCATION BY RECALLING THE CONTENTS OF 110. ]
  * ;[STORE THE SCAN NUMBER OF ]
  * ;[THE BEST MATCH IN VARIABLE 14 AND ALLOW INTEGRATION ]
  * ;[AT THAT SPECTRUM NUMBER ONLY ]
  * ;[IF THE COMPOUND IS NOT FOUND, PLACE A NOT FOUND ]
  * ;[ENTRY INTO THE QUANTITATION LIST FOR LATER REFERENCE ]
  * ;SET4 14,,*1
  * ;IF 124*1,14
  * ;SET14 *0
  * ;SET1 110
  * ;EDLL PPLIST(S;W;E)
  * ;SEAR/V(I;S;V2500000;N1.10.10;D-20.20;E)
  * ;PRIN/KX(14,2;114,6;115,6;116,7;C;E)
  * ;PPEVF
  * ;LOOP
  *
  SET4 14,,*1
  IF *1124,14
  SET14
  SET1 110
  EDLL PPLIST (S;W;E)
  SEAR (I;S;V2500000;N1.10.10;D-20.20;E)/V
  PRIN (14,2;114,6;115,6;116,7;C;E)/KX
  PPEVF
    * ;[PART OF PPEVAL]
    * ;[IF THE FIT IS LESS THAN OR EQUAL TO 750 ]
    * ;[WRITE A NOT DETECTED, NAMED ENTRY INTO THE]
    * ;[QUANTITATION LIST FOR FUTURE REFERENCE ]
    * ;PPEVG
    * ;EDOL(-;N;*;A;E)
    *

```


APPENDIX IIC.

```

PPEVG
* CPART OF PPEVAL ]
* :CACCESS ANY SCANS IDENTIFIED IN DETECT ]
* :CAND INTEGRATE THEIR AREAS. RECORD THE ]
* :CDATA IN THE QUANLIST ASSIGNED EARLIER. ]
* :CALSO CHECK AND PASS ONLY PEAKS WITH ]
* :CA FIT OF 750 OR GREATER ]
* :IF PPEVG !16,PPEVG *700
* :SETI !14
* :CHRO(I;R;S;*:N1,3;A>5,3;G-4,4;D-5,5;E)
* :PPEVH
* :RETU PPEVF
*
IF PPEVG!16,PPEVG*700
SETI !14
CHRO (I;R;S;*:N1,3;A>5,3;G-4,4;D-5,5;E)
PPEVH
* IF !26 PPEVH,PPEVH
* :SPEC(*;N;H;E)
*
IF PPEVH!26,PPEVH
SPEC (*;N;H;E)
RETU PPEVF
EDOL (-;N;*:A;E)
LOOP
QUAN (I;H;E)
EDLL PPLIST (B!1;E)
PRIN (@P1)
FILE (C PRIN.99/N,M;:E)
FEED
BEEP
LOOP
FEED
BEEP
BEEP
BEEP

```

APPENDIX IID.

PRINP2.ME = C20;T;

C;T;

T;

PRIORITY POLLUTANT EVALUATION;

NO INTERNAL STANDARD WAS FOUND IN SAMPLE ;S1;

;D;F

PRINP1.ME = C2;T; IDENTIFICATION REPORT

S1;C2;T;NO SCAN PURITY FIT

C;E

FILE:

NUM	NAME	RET TIME	BASE	AREA	U.P.*1	U.P.*2
1	FORMULA	MASS	AMT.	REF. PEAK	RESP. FILE	RESP. FACTOR
1	RET. TIME/CAS#					
1:	1,4-DICHLOROBUTANE (INTERNAL STANDARD)					
5	C4.H8.CL2	3:38	55	0.	0.000	0.000
	1.000	55.000200.00	VI	1	C	1.000
2:	BROMOCHLOROMETHANE (INTERNAL STANDARD)					
3	C.H2.CL.BR	0:44	130	0.	0.000	0.000
	0.000	49.000200.00	VO	1	VS	1.000
3:	02 ACROLIEN					
5	C3.H4.O	0:00	56	0.	0.000	0.000
	0.000	56.000200.00	VO	1	VS	1.000
4:	03 ACRYLONITRILE					
3	C3.H3.N	0:00	53	0.	0.000	0.000
	0.000	53.000200.00	VO	1	VS	1.000
5:	04 BENZENE					
3	C6.H6	2:19	70	0.	0.000	0.000
	0.000	70.000200.00	VO	1	VS	1.000
6:	06 CARBONTETRACHLORIDE					
2	C.CL4	1:45	117	0.	0.000	0.000
	0.000	117.000200.00	VO	1	VS	1.000
7:	05 CHLOROBENZENE					
2	C6.H5.CL	3:50	112	0.	0.000	0.000
	0.000	112.000200.00	VO	1	VS	1.000
8:	10 1,2-DICHLOROETHANE					
3	C2.H4.CL2	1:26	62	0.	0.000	0.000
	0.000	62.000200.00	VO	1	VS	1.000
9:	11 1,1,1-TRICHLOROETHANE					
2	C2.H3.CL3	1:41	97	0.	0.000	0.000
	0.000	97.000200.00	VO	1	VS	1.000
10:	13 1,1-DICHLOROETHANE					
3	C2.H4.CL2	0:52	63	0.	0.000	0.000
	0.000	96.000200.00	VO	1	VS	1.000
11:	14 1,1,2-TRICHLOROETHANE					
2	C2.H3.CL3	2:32	97	0.	0.000	0.000
	0.000	03.000200.00	VO	1	VS	1.000
12:	15 1,1,2,2-TETRACHLOROETHANE					
6	C2.H2.CL4	3:27	83	0.	0.000	0.000
	0.000	83.000200.00	VO	1	VS	1.000
13:	23 CHLOROFORM					
0	C.H.CL3	1:20	83	0.	0.000	0.000
	0.000	83.000200.00	VO	1	VS	1.000
14:	29 1,1-DICHLOROETHENE					
6	C2.H2.CL2	0:20	61	0.	0.000	0.000
	0.000	96.000200.00	VO	1	VS	1.000
15:	30 1,2-TRANS-DICHLOROETHENE					
3	C2.H2.CL2	1:01	96	0.	0.000	0.000
	0.000	96.000200.00	VO	1	VS	1.000
16:	32 1,2-DICHLOROPROPANE					
2	C3.H6.CL2	2:11	63	0.	0.000	0.000
	0.000	63.000200.00	VO	1	VS	1.000
17:	33A 1,3-CIS-DICHLORO-1-PROPENE					

APPENDIX IIIA.

110	C3.H4.CL2	0.000	75.000200.00	VO	1	VS	1.000	0.000
VO	18: 330 1,2-TRANS-DICHLORO-1-PROPENE				75	0.	0.000	0.000
110	C3.H4.CL2	2:34			1	VS	1.000	0.000
	0.000	75.000200.00	VO					
VO	19: 30 ETHYLBENZENE				91	0.	0.000	0.000
106	C8.H10	0:00	91.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	20: 44 METHYLENECHLORIDE				84	0.	0.000	0.000
84	C.H2.CL2	0:04	84.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	21: 47 BROMOFORM				173	0.	0.000	0.000
250	C.H.BR3	3:02	173.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	22: 40 BROMODICHLOROMETHANE				93	0.	0.000	0.000
162	C.H.CL2.BR	1:59	83.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	23: 49 TRICHLOROFLUOROMETHANE				101	0.	0.000	0.000
136	C.CL3.F	0:19	101.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	24: 51 DIBROMOCHLOROMETHANE				129	0.	0.000	0.000
206	C.H.CL.BR2	2:32	129.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	25: 65 TETRACHLOROETHENE				166	0.	0.000	0.000
164	C2.CL4	3:22	129.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	26: 86 TOLUENE				91	0.	0.000	0.000
92	C7.H8	3:29	91.000200.00	VO	1	VS	1.000	0.000
	0.000							
VO	27: 87 TRICHLOROETHENE				130	0.	0.000	0.000
130	C2.H.CL3	2:20	95.000200.00	VO	1	VS	1.000	0.000
	0.000							

NAME	RET TIME	BASE	AREA	U.P.*1	U.P.*2
FORMULA	AMT.	PEAK	P.F.I.L.	FACT	
1: D10-ANTHRACENE (INTERNAL STANDARD)	3:44	188	44864.	0.000	0.000
1.000	188.000	20.00	BN	1	1.000
2: 01 ACENAPHTHENE	2:39	154	0.	0.000	0.000
C12.H10	BN	1	1.5	0.586	
0.710	154.000	20.00			
3: 05 BENZIDINE	5:00	184	0.	0.000	0.000
C12.H12.N2	BN	1	1.5	0.047	
1.345	184.000	50.00			
4: 08 1,2,4-TRICHLOROBENZENE	1:10	188	0.	0.000	0.000
C6.H3.CL3	BN	1	1.5	0.102	
0.349	74.000	20.00			
5: 09 HEXACHLOROBENZENE	3:20	284	0.	0.000	0.000
C6.CL6	BN	1	1.5	0.264	
0.893	284.000	20.00			
6: 12 HEXACHLOROETHANE	0:43	201	0.	0.000	0.000
C2.CL6	BN	1	1.5	0.398	
0.192	117.000	20.00			
7: 18 BIS(2-CHLOROETHYL) ETHER	0:37	93	0.	0.000	0.000
C4.H8.O.CL2	BN	1	1.5	0.205	
0.165	93.000	50.00			
8: 20 2-CHLORONAPHTHALENE	2:12	162	0.	0.000	0.000
C10.H7.CL	BN	1	1.5	0.612	
0.589	162.000	20.00			
9: 25 1,2-DICHLOROBENZENE	0:41	146	0.	0.000	0.000
C6.H4.CL2	BN	1	1.5	0.706	
0.183	146.000	20.00			
10: 26 1,3-DICHLOROBENZENE	0:31	146	0.	0.000	0.000
C6.H4.CL2	BN	1	1.5	0.519	
0.138	146.000	20.00			
11: 27 1,4-DICHLOROBENZENE	0:34	146	0.	0.000	0.000
C6.H4.CL2	BN	1	1.5	0.895	
0.152	146.000	20.00			
12: 35 2,4-DINITROTOLUENE	2:59	165	0.	0.000	0.000
C7.H6.O4.N2	BN	1	1.5	0.191	
0.803	165.000	50.00			
13: 36 2,6-DINITROTOLUENE	2:46	165	0.	0.000	0.000
C7.H6.O4.N2	BN	1	1.5	0.184	
0.744	165.000	50.00			
14: 37 1,2-DIPHENYLHYDRAZINE (MEAS. AS AZOBENZENE)	3:06	77	0.	0.000	0.000
C12.H10.N2	BN	1	1.5	1.066	
0.834	77.000	50.00			
15: 39 FLUORANTHENE	4:34	202	0.	0.000	0.000
C16.H10	BN	1	1.5	0.714	
1.223	202.000	20.00			
16: 40 4-CHLOROPHENYL PHENYL ETHER	2:53	204	0.	0.000	0.000
C12.H9.O.CL	BN	1	1.5	0.280	
0.799	204.000	20.00			
17: 41 4-BROMOPHENYL PHENYL ETHER					

APPENDIX IVA.

240	C12.H9.O.NR	0.893	240.000	20.00	BN	1	1.5	0.153	
BN	18: 42 BIS(2-CHLOROISOPROPYL) ETHER	0:43			BN	45	0.	0.000	0.000
170	C6.H12.O.CL2	0.193	45.000	50.00	BN	1	1.5	0.664	
BN	19: 43 BIS(2-CHLOROETHOXY) METHANE	1:20			BN	93	0.	0.000	0.000
172	C5.H10.O2.CL2	0.359	93.000	50.00	BN	1	1.5	0.586	
BN	20: 53 HEXACHLOROCYCLOPENTADIENE	1:20			BN	237	0.	0.000	0.000
270	C5.CL6	0.000	237.000	20.00	BN	1	1.5	1.000	
BN	21: 54 ISOPHORONE	1:09			BN	82	0.	0.000	0.000
130	C9.H14.O	0.309	82.000	50.00	BN	1	1.5	0.984	
BN	22: 55 NAPHTHALENE	1:25			BN	120	0.	0.000	0.000
120	C10.H8	0.379	120.000	20.00	BN	1	1.5	1.287	
BN	23: 56 NITROBENZENE	1:07			BN	77	0.	0.000	0.000
123	C6.H5.O2.N	0.300	77.000	50.00	BN	1	1.5	0.457	
BN	24: 62 N-NITROSODIPHENYLAMINE (MEAS. AS DIPHENYLAMINE)	3:12			BN	169	0.	0.000	0.000
169	C12.H11.N	0.857	169.000	20.00	BN	1	1.5	0.145	
BN	25: 63 N-NITROSODIPROPYLAMINE	0:55			BN	70	0.	0.000	0.000
130	C6.H14.O.N2	0.247	130.000	50.00	BN	1	1.5	0.058	
BN	26: 66 DI-(2-ETHYLHEXYL) PHTHALATE	5:44			BN	149	0.	0.000	0.000
390	C24.H38.O4	1.536	149.000	20.00	BN	1	1.5	0.841	
BN	27: 67 BUTYLBENZYL PHTHALATE	5:27			BN	149	0.	0.000	0.000
312	C19.H20.O4	1.460	149.000	20.00	BN	1	1.5	0.591	
BN	28: 68 DI-N-BUTYL PHTHALATE	4:06			BN	149	0.	0.000	0.000
270	C16.H22.O4	1.098	149.000	20.00	BN	1	1.5	1.732	
BN	29: 69 DI-OCTYL PHTHALATE	6:58			BN	149	0.	0.000	0.000
390	C24.H38.O4	1.866	149.000	20.00	BN	1	1.5	0.580	
BN	30: 70 DIETHYL PHTHALATE	3:04			BN	149	0.	0.000	0.000
222	C12.H14.O4	0.021	149.000	20.00	BN	1	1.5	0.953	
BN	31: 71 DIMETHYL PHTHALATE	2:48			BN	163	0.	0.000	0.000
194	C10.H10.O4	0.714	163.000	20.00	BN	1	1.5	0.817	
BN	32: 72 BENZO(A) ANTHRACENE	6:14			BN	220	0.	0.000	0.000
220	C18.H12	1.670	220.000	20.00	BN	1	1.5	0.120	
BN	33: 76 CHRYSENE	6:14			BN	220	0.	0.000	0.000
220	C18.H12	1.670	220.000	20.00	BN	1	1.5	0.120	
BN	34: 77 ACENAPHTHYLENE	2:33			BN	152	0.	0.000	0.000
152	C12.H8	0.603	154.000	20.00	BN	1	1.5	0.803	

APPENDIX IVB.

35: 78 ANTHRACENE	3:44	178	0.	0.000	0.000
C14.H10	BN	1	:S	1.433	
1.000 178.000 20.00					
36: 80 FLUORENE	3:00	166	0.	0.000	0.000
C13.H10	BN	1	:S	0.573	
0.004 166.000 20.00					
37: 81 PHENANTHRENE	3:44	178	0.	0.000	0.000
C14.H10	BN	1	:S	1.433	
1.000 178.000 20.00					
38: 84 PYRENE	4:34	202	0.	0.000	0.000
C16.H10	BN	1	:S	0.714	
1.223 202.000 20.00					

APPENDIX IVC.

WT	FORMULA	RET TIME	BASE	AREA	U.P.#1	U.P.#2
REL.RET.TIME/CAS#	MASS	ANT.	REF. PEAK	RESP. FILE	RESP. FACTOR	
PH 1: D10-ANTHRACENE (INTERNAL STANDARD)						
188 1.000 188.000 50.00	PH	1	2:43 188	44864.	0.000	0.000
				:S	1.000	
PH 2: 21 2,4,6-TRICHLOROPHENOL						
196 C6.H3.O.CL3	PH	1	1:46 196	0.	0.000	0.000
0.000 196.000 100.00				:S	0.461	
PH 3: 22 4-CHLORO-3-METHYLPHENOL						
142 C7.H7.O.CL	PH	1	2:06 142	0.	0.000	0.000
0.000 142.000 100.00				:S	0.524	
PH 4: 24 2-CHLOROPHENOL						
128 C6.H5.O.CL	PH	1	0:27 128	0.	0.000	0.000
0.000 120.000 100.00				:S	1.014	
PH 5: 31 2,4-DICHLOROPHENOL						
162 C6.H4.O.CL2	PH	1	1:13 162	0.	0.000	0.000
0.000 162.000 100.00				:S	0.714	
PH 6: 34 2,4-DIMETHYLPHENOL						
122 C8.H10.O	PH	1	1:11 122	0.	0.000	0.000
0.000 122.000 100.00				:S	0.617	
PH 7: 57 2-NITROPHENOL						
139 C6.H5.O3.N	PH	1	0:37 139	0.	0.000	0.000
0.000 139.000 100.00				:S	0.534	
PH 8: 58 4-NITROPHENOL						
139 C6.H5.O3.N	PH	1	5:01 139	0.	0.000	0.000
0.000 65.000 100.00				:S	0.000	
PH 9: 59 2,4-DINITROPHENOL						
184 C6.H4.O5.N2	PH	1	2:53 184	543744.	0.000	0.000
1.000 184.000*1000.				:S	0.219	
PH 10: 60 4,6-DINITRO-O-CRESOL						
198 C7.H6.O5.N2	PH	1	2:57 198	781312.	0.000	0.000
1.000 198.000*1000.				:S	0.319	
PH 11: 64 PENTACHLOROPHENOL						
264 C6.H.O.CL5	PH	1	3:12 266	0.	0.000	0.000
0.000 266.000 100.00				:S	0.242	
PH 12: 65A PHENOL						
94 C6.H6.O	PH	1	0:52 94	0.	0.000	0.000
0.000 94.000 100.00				:S	1.025	

APPENDIX VIA.

QUANTITATION REPORT

FILE: SMASA

DATA: SMASA.MI

0:00:00

SAMPLE: VOA STD MIX A W/I.S. SEPT 3, 1978

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	02 ACROLIEN
4	03 ACRYLONITRILE
5	04 BENZENE
6	06 CARBONTETRACHLORIDE
7	07 CHLOROBENZENE
8	10 1,2-DICHLOROETHANE
9	11 1,1,1-TRICHLOROETHANE
10	14 1,1,2-TRICHLOROETHANE
11	15 1,1,2,2-TETRACHLOROETHANE
12	19 2-CHLOROETHYL VINYLETHER
13	23 CHLOROFORM
14	30 1,2-TRANS-DICHLOROETHENE
15	32 1,2-DICHLOROPROPANE
16	38 ETHYLBENZENE
17	44 METHYLENE CHLORIDE
18	47 BROMOFORM
19	48 BROMODICHLOROMETHANE
20	51 DIBROMOCHLOROMETHANE
21	85 TETRACHLOROETHENE
22	86 TOLUENE
23	87 TRICHLOROETHENE
24	88 VINYL CHLORIDE
25	29 1,1-DICHLOROETHENE

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	%TOT
1	55	251	4:11	1	1.000	A 88	1191060.	200.000 PPB	4.55
2	49	75	1:15	1	0.299	A 88	1120800.	200.000 UG/L	4.55
3	NOT FOUND								
4	NOT FOUND								
5	78	175	2:55	1	0.697	A 88	1734110.	200.000 UG/L	4.55
6	117	139	2:19	1	0.554	A 88	1242110.	200.000 UG/L	4.55
7	112	272	4:32	1	1.084	A 88	1944750.	200.000 UG/L	4.55
8	62	117	1:57	1	0.466	A 88	1115510.	200.000 UG/L	4.55
9	97	134	2:14	1	0.534	A 88	1254820.	200.000 UG/L	4.55
10	83	189	3:09	1	0.753	A 88	805200.	200.000 UG/L	4.55
11	83	247	4:07	1	0.934	A 88	1293270.	200.000 UG/L	4.55
12	106	200	3:20	1	0.797	A 88	119382.	200.000 UG/L	4.55
13	83	100	1:48	1	0.430	A 88	1612750.	200.000 UG/L	4.55
14	96	88	1:20	1	0.351	A 88	774512.	200.000 UG/L	4.55
15	63	167	2:47	1	0.665	A 88	1009560.	200.000 UG/L	4.55
16	91	306	5:06	1	1.219	A 88	2419710.	200.000 UG/L	4.55
17	84	45	0:45	1	0.179	A 88	560555.	200.000 UG/L	4.55
18	173	221	3:41	1	0.880	A 88	1034980.	200.000 UG/L	4.55
19	83	153	2:33	1	0.610	A 88	1613140.	200.000 UG/L	4.55
20	129	189	3:09	1	0.753	A 88	1452530.	200.000 UG/L	4.55

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	%TOT
21	129	243	4:03	1	0.968	A 88	1099530.	200.000 UG/L	4.55
22	91	251	4:11	1	1.000	A 88	1079520.	200.000 UG/L	4.55
23	95	178	2:58	1	0.709	A 88	999815.	200.000 UG/L	4.55
24	NOT FOUND								
25	96	63	1:03	1	0.251	M XX	55894.	200.000 UG/L	4.55

QUANTITATION FOR THIS
COMPOUND MANUALLY ADDED
OR

APPENDIX VIB.

NAM	NUM:	WT	FORMULA	NAME
VI	1:	126	C4.H8.CL2	1,4-DICHLOROETHANE (INTERNAL STANDAR
VI	2:	128	C.H2.CL.BR	BROMOCHLOROMETHANE (INTERNAL STANDAR
VI	3:	56	C3.H4.O	02 ACROLIEN
VI	4:	53	C3.H3.N	03 ACRYLONITRILE
VI	5:	78	C6.H6	04 BENZENE
VI	6:	152	C.CL4	06 CARBONTETRACHLORIDE
VI	7:	112	C6.H5.CL	06 CHLOROBENZENE
VI	8:	98	C2.H4.CL2	10 1,2-DICHLOROETHANE
VI	9:	132	C2.H3.CL3	11 1,1,1-TRICHLOROETHANE
VI	10:	132	C2.H3.CL3	14 1,1,2-TRICHLOROETHANE
VI	11:	166	C2.H2.CL4	15 1,1,2,2-TETRACHLOROETHANE
VI	12:	186	C4.H7.O.CL	19 2-CHLOROETHYL VINYLETHER
VI	13:	118	C.H.CL3	23 CHLOROFORM
VI	14:	96	C2.H2.CL2	29 1,1-DICHLOROETHENE
VI	15:	96	C2.H2.CL2	30 1,2-TRANS-DICHLOROETHENE
VI	16:	112	C3.H6.CL2	32 1,2-DICHLOROPROPANE
VI	17:	106	C8.H18	38 ETHYLBENZENE
VI	18:	84	C.H2.CL2	44 METHYLENECHLORIDE
VI	19:	250	C.H.BR3	47 BROMOFORM
VI	20:	162	C.H.CL2.BR	48 BROMODICHLOROMETHANE
VI	21:	206	C.H.CL.BR2	51 DIBROMOCHLOROMETHANE
VI	22:	164	C2.CL4	65 TETRACHLOROETHENE
VI	23:	92	C7.H8	86 TOLUENE
VI	24:	138	C2.H.CL3	87 TRICHLOROETHENE
VI	25:	62	C2.H3.CL	88 VINYL CHLORIDE

IDENTIFICATION REPORT

FILE: D:SMASA.MI

NO	SCAN	PURITY	FIT
1	251	400	864
2	75	819	978
3	53	41	43
4	45	43	204
5	176	615	940
6	139	841	977
7	272	770	960
8	117	673	994
9	134	765	981
10	189	406	979
11	247	606	964
12	200	643	959
13	108	825	984
14	63 44	789	988
15	89	786	977
16	167	726	977
17	307	758	995
18	45	781	976
19	221	798	940
20	153	837	995
21	183	417	945
22	243	835	961
23	251	565	955
24	177	525	981
25	0	0	0

Spectra printouts deleted to conserve paper.

Organic Compound Identification by Glass
Capillary Gas Chromatography/Mass Spectrometry

1. Scope and Application

- 1.1 This method is applicable to surface waters and industrial effluents.
- 1.2 The limit of detection for this method varies from 1 to 10 ug/l (ppb) depending on the type of compound.
- 1.3 The concentration range is from 1 to 100 ug/l (ppb).

2. Summary of Method

- 2.1 Concentrated extracts of 1 to 3 liter water samples are injected into a glass capillary column gas chromatograph directly coupled to a quadrupole mass spectrometer thru a small diameter heated stainless steel glass lined tubing. A splitless injection technique is used. Initial identification is established using a routine computer search of a library of standard reference spectra. The identification is confirmed by comparing the mass spectra of reference standards, analyzed using the same instrumental conditions. The coincidence of the gas chromatography retention times of standards and sample components provides additional confirmation of identity.

3. Interferences

- 3.1 Concentrated solvent extracts often contribute interferences and a method blank is always run to differentiate reagent contamination from sample components.
- 3.2 Common solvent interferences are: diacetone alcohol (4-methyl-4-hydroxy-2-pentanone) from acetone, phthalates from sodium sulfate, and cyclohexene from dichloromethane.

4. Apparatus

- 4.1 Finnigan Model 9500 gas chromatograph equipped with a glass capillary column.
 - 4.1.1 Grob type injector for splitless injection.
 - 4.1.2 Capillary glass column, 25 meters x 0.25 mm ID, OV-101.

4.2 Finnigan Model 3200 electron impact mass spectrometer.

4.2.1 Glass lined stainless steel tubing direct coupling to gas chromatograph.

4.3 Finnigan INCOS data system (1).

5. Procedure

5.1 Gas Chromatography

5.1.1 Inject 1 μ l of sample into the gas chromatograph with the splitter turned off for 1 minute after injection then turn on. (Splitter flow 100 ml/min).

5.1.2 The initial column temperature is equilibrated at 60°C and held for 1 minute after injection, then a temperature program is initiated at 4°C/min. to a final temperature of 220°C and held from 10 to 15 minutes. Column flow is adjusted to give a nominal flow of 1.5 ml/min. at 100°C.

5.2 Mass Spectrometry

5.2.1 The following MS instrumental parameters are used:

Electron multiplier voltage	- 1600 volts
Lens voltage	- 100 volts
Collector voltage	- 35 volts
Extractor voltage	- 6 volts
Ion Energy voltage	- 10 volts
Electron Energy voltage	- 70 volts
Emission Current	- 0.5 ma

5.2.2 The following data acquisition parameters are used:

Scan time	- 2 sec.
Mass Range	- 33-300
Sensitivity	- 10 ⁻⁷ amp.

5.2.3 The data acquisition is initiated immediately upon injection of a sample into the gas chromatograph in a suspended mode with the ionizer turned off. At 4 minutes the ionizer is turned on and at 5 min. the data acquisition is changed from the suspended mode to the centroid mode and actual data collection begun. A normal analysis using the 25 meter capillary OV-101 column will require data collection for 35 to 40 minutes.

5.2.4 A reconstructed ion chromatogram is generated using the MSDS program system and specific spectra are then plotted. A manual computer search of the reference library gives an identification. The initial identification is then confirmed by comparison of sample spectra and reference spectra obtained by analyzing standards under the same instrumental conditions.

6. Quality Control

- 6.1 Daily calibration of the GC/MS is performed before any sample analysis using a standard reference compound. (Pufluorotri-butylamine-FC-43).
- 6.2 The reference compound is metered into the mass spectrometer via a variable leak valve at a constant rate. Several scans are recorded at a scan rate of 3 seconds and a sensitivity of 10^{-6} amps. The calibration is then made utilizing the MSDS system calibration routine.
- 6.3 An ion intensity ratio of 2 to 1 for mass 69 to mass 219 is desirable for good spectra using the capillary system. The ion intensity ratio can vary from 3 to 1 to almost 1 to 1 and still provide legitimate spectra.

7. References

- (1) "INCOS Data System - MSDS Operators' Manual - Revision 3", Finnigan Instruments, March 1978.
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ATTACHMENT VI
COMPUTER ASSISTED EVALUATION OF
ORGANICS CHARACTERIZATION GC/MS DATA

August 1978

1.0 This procedure is applicable to GC/MS data collected under constant analytical conditions for qualitative data analysis.

2.0 Summary of Method

2.1 GC/MS data files are processed by comparing spectra from the sample against spectra of known or suspected pollutants contained in a project related library. If a spectrum matches the project library spectrum sufficiently, an entry is made in a table showing at what spectrum number the match occurred and how good the match was. After completion of the search for each spectrum in the project library, a list of the compounds searched for and the matching results is printed as well as each spectrum that was identified as a probable pollutant. If selected by the user, the procedure will then search the current version of the NB (EPA/NIH/MSDC) library attempting to identify unknown spectra from peaks selected by the Biemann-Billler algorithm in MAP.

3.0 Definitions and Comments

3.1 In some cases, compounds may be identified by comparison to external reference spectra only (1,2,3). These "unconfirmed" compound data may however be useful since the computer matching still traces the presence of selected compounds through each sample analyzed. Therefore, even these "unconfirmed" pollutants can serve to trace a waste stream.

- 3.2 Quantitation of pollutants identified is effected by locating the corresponding GC peaks on GC/FID (flame ionization detector) chromatograms. The areas or peak heights are measured and compared to the response of known amounts of pure standard compounds. The concentrations are then calculated. Since this scheme utilizes two chromatographic systems (GC/MS and GC/FID), in some cases, differences in these systems will allow identification by GC/MS but not allow quantitation. In such cases, "MS" is reported to signify a mass spectrometer identification.
- 3.3 The identities of some components are confirmed by the matching of their mass spectra and GC retention times to the data obtained from the analysis of a pure standard compounds. Such identities are indicated by "CF."
- 3.4 Components not identified by mass spectrometry are reported as "ND" to denote not detected.
- 3.5 Analytical schemes may not allow measurement of some suspected pollutants in all samples and the result is reported as "NA" or not analyzed.

4.0 Interferences

- 4.1 Since absolute GC retention times are used for setting the search windows, the windows must be wide enough to account for slight variations in instrument conditions. This could cause identification errors if compounds with similar spectra (isomers) are in the window. Manually checking each spectrum produced essentially eliminates any error.

5.0 Apparatus

5.1 Finnigan INCOS data system software running revision 3.1 or later version. To initially set up this procedure, the user must understand and be proficient in the use of MSDS (4).

5.2 INCOS "NB" mass spectra library (5).

6.0 Procedure

6.1 Procedure Setup

6.1.1 Load the procedures listed in appendix 1 onto the system disc or create the procedures from the trace of OCEVAL in Appendix 2.

6.2 Library Setup

6.2.1 Obtain spectra of the compounds of interest by running standards under the same analytical conditions to be used for sample analysis.

6.2.2 Using the library editor, create a library containing the standard spectra with chemical names and retention times. Obtain a reference spectrum of each library entry for a permanent record and reference via the library program:

G1; HS; G2; HS;... etc.

6.3 Routine use

6.3.1 Collect mass spectra of samples to be processed under the same conditions as the standards were analyzed.

6.3.2 Using the namelist editor, create a namelist containing the names of the files to be processed.

6.3.3 Execute the procedure:

OCEVAL library, namelist, no (yes)

Where: library is the user library name, namelist is the file containing the names of the data files to be processed and no or yes select a continued search through the NB library.

If the user wants only to perform an NB search, the procedure is initiated as follows:

OCEVAL NB, namelist

6.3.4 Appendix 3 is an example of OCEVAL output consisting of the following:

- (1) The acquisition parameter listing
- (2) A chromatogram with peaks labeled by MAP
- (3) A list of the compounds being searched for and a summary of the search results.
- (4) A collection of the spectra of peaks identified by the procedure
- (5) Library matching results for peaks found by MAP but not identified in the user library.

7.0 Quality Control

7.1 Each identification is manually verified by comparing the sample spectrum to the reference spectrum in the user library. Inaccurate computer results are re-evaluated and the correct data reported.

8.0 Precision and Accuracy

8.1 The auto processing routine's accuracy for correctly identifying compounds is limited by the quality of the original GC/MS data.

9.0 References

- (1) "Eight Peak Index of Mass Spectra," Mass Spectrometry Data Center, Aldermaston, Reading, UK. Second Edition 1974.
- (2) "Registry of Mass Spectral Data," Stenhagen, Abramsson and McLafferty, Wiley & Sons, New York, 1974.
- (3) "Atlas of Mass Spectra Data," edited by: Stenhagen, Abrahams-son and McLafferty, Wiley & sons, New York, 1969.
- (4) "INCOS Data System - MSDS Operators Manual - Revision 3," Finnigan Instruments, March 1978
- (5) "NBS - NIH/EPA/MSDC Library - Revision 3," Finnigan Instruments, March 31, 1978

APPENDIX I.

PROCEDURES AND METHODS REQUIRED FOR OCEVAL

1. OCEVAL
2. OCEV0
3. OCEV1
4. OCEV2
5. OCEV2A
6. OCEV2B
7. OCEV3
8. OCEV5
9. OCEV6
10. OCEV7
11. PRINO1.ME
12. PRINO2.ME


```

TRACE OF PROCEDURE OCEVAL
* [***** OCEVAL ***** JULY 29, 1978 *****]
* ;OCEVAL PROVIDES THE OPERATOR WITH A MEANS OF ]
* ;LOCATING COMPOUNDS THAT ARE SUSPECT BASED ON ]
* ;THEIR RETENTION TIMES AND SPECTRA. THESE]
* ;COMPOUNDS ARE SAVED IN A USER LIBRARY FOR]
* ;ACCESS BY OCEVAL. IF DESIRED, THE USER MAY]
* ;ALSO SELECT THAT ALL OTHER PEAKS LOCATED BY MEANS]
* ;OF BILLER-BIEHMANN IN NMR BE SEARCHED AGAINST THE]
* ;NMR LIBRARY. THE USER LIBRARY MUST CONTAIN]
* ;SPECTRA AND RETENTION TIMES. ALSO, ALL DATA FILES]
* ;[PROCESSED MUST HAVE SCANS AVAILABLE FROM 25]
* ;[BELOW THE EARLIEST ELUTING COMPONENT (OR START AT 0)]
* ;[TO 25 ABOVE THE LATEST ELUTING COMPONENT.]
* ;[TO USER THE PROCEDURE, CREATE A LIBRARY]
* ;[WITH THE SPECTRA AND RETENTION TIMES. CREATE A]
* ;[NAMELIST CONTAINING THE FILE TO BE PROCESSED.]
* ;[
* ;[THEN: >OCEVAL XY,NAMELIST,NO(YES) ]
* ;[
* ;[WHERE: XY IS THE USER LIBRARY NAME OR NB ]
* ;[ NAMELIST IS THE NAMELIST CONTAINING THE FILES]
* ;[ TO BE PROCESSED.
* ;[ NO SELECTS NO NB LIBRARY SEARCH OR YES SELECTS]
* ;[ AN NB SEARCH]
* ;[ IF THE USER SELECTED THE NB LIBRARY]
* ;[ INITIALLY NO ENTRY IS REQUIRED]
* ;[LAST REVISED 9/27/78 OJLOGSDONII ]
* ;SET4 !!
* ;EDLL YES(-;S;U;E);EDLL NO(-;U;E)
* ;SETH OCTEMP;EDNL(-;S1;S2;U;E)
* ;SET11 #0
* ;OCEV0
* ;BEEP;BEEP;BEEP
* ;ERASE
* ;[PROCEDURE OCEVAL IS COMPLETE]
*
SET4 !!
EDLL YES (-;S;U;E)
EDLL NO (-;U;E)
SETH OCTEMP
EDNL (-;S1;S2;U;E)
SET11
OCEV0
* SETH OCTEMP;SETH #0;SETH;SET4 S1
* ;SETH;SETH S1;SETH !!1;SET11 !!1>1;GETN
* ;OCEV1
* ;SETH OCTEMP
* ;EDLL(-;U;E)
* ;FILE(K PR111.99/11;E)
* ;OCEV2
* ;SET12 #0
* ;SETS OCEV2;SETS #0
* ;EDSL(-!12;U;E)
* ;OCEV3
* ;OCEV5
* ;BEEP
* ;LOOP
*
SETH OCTEMP
SETH
GETH
SET4 S1
GETH
SETH S1
SETH !!1
SET11 #1!!1
GETH
OCEV1
* PARA(1;H;E)

```

APPENDIX II. b.

```

* ;SETS OCEV2:EDSL(-;U:E)
* ;SETS OCEV1:EDSL(-;U:E)
* ;MAP(I;F1:U100;V250000:33,300;N>2.5,7;H1,2000,500;E)
*
PARA (I;H;E)
SETS OCEV2
EDSL (-;U:E)
SETS OCEV1
EDSL (-;U:E)
MAP (I;F1:U100;V250000:33,300;N>2.5,7;H1,2000,500;E)
SETL OCTEMP
EDLL (-;U:E)
FILE (K PRIN.99;N;E)
OCEV2
* IF OCEV2 #25000,OCEV2 !24
* ;OCEV2A
* ;PRIN (001)
* ;EDLL (0!1;E)
* ;PRIN (002)
* ;FILE (C PRIN.99,M;N;E)
* ;FEED
*
IF OCEV2=25000,OCEV2!24
OCEV2A
* SET4 !4,,*1;SET!4 *0
* ;IF #!124 OCEV2A,!4 OCEV2A
* ;OCEV2B
* ;LOOP
*
SET4 !4,,*1
SET!4
IF OCEV2A#!124,OCEV2A!4
OCEV2B
* EDLL(S;U:E)
* ;SEAR/V(I;S;0;V250000;N1,200,750;D-25,25;E)
* ;PRIN/KX(!4,6;!14,5;!15,9;!16,6;C;E)
* ;SETS OCEV2:EDSL(!14;U:E)
* ;SETS OCEV1:EDSL(-!14;U:E)
*
EDLL (S;U:E)
SEAR (I;S;0;V250000;N1,200,750;D-25,25;E)/V
PRIN (!4,6;!14,5;!15,9;!16,6;C;E)/KX
SETS OCEV2
EDSL (!14;U:E)
SETS OCEV1
EDSL (-!14;U:E)
LOOP
PRIN (001)
EDLL (0!1;E)
PRIN (002)
FILE (C PRIN.99,M;N;E)
FEED
SET!2
SETS OCEV2
SETS
EDSL (-!12;U:E)
OCEV3
* GETS
* ;SPEC(I;' ;T;H30,350;E)
* ;LOOP
*
GETS
SPEC (I;' ;T;H30,350;E)
LOOP
OCEV3
* SETL 03
* ;OCEV6
* ;SET4 NB
* ;SETS OCEV1;SETS *0
* ;OCEV7

```

APPENDIX II. c.

```

* ;FEED
*
SETL S3
OCEV6
  * IF OCEV6 #25000,OCEV6 !24
  * ;IF OCEV5 !26,OCEV5
  * ;RETU OCEV6
  *
  IF OCEV6#25000,OCEV6!24
  IF OCEV5!26,OCEV5
  RETU OCEV6
SET4 N8
SETS OCEV1
SETS
OCEV7
  * GETS
  * ;LIBR(I;';F;X1.3;HS;E)
  * LOOP
  *
  GETS
  LIBR (I;';F;X1.3;HS;E)
  LOOP
FEED
BEEP
LOOP
BEEP
BEEP
BEEP
ERASE

```

APPENDIX II. d.

PRIND1.ME = C:D:T; ORGANICS CHARACTERIZATION REPORT FILE:
;S1:C2:T;
;D:C2:E

PRIND2.ME = C2:T; NUM SPECs PURITY FIT
;C:E

APPENDIX III.

0/00/00 0:00:00 ORGANICS CHARACTERIZATION REPORT FILE: 0:01:4503N.TI

0/00/00 0:00:00

NAM	NUM:	WT	FORMULA	NAME
39	1:	126	C8.H14.0	2-ETHYL-2-HEXENAL (NC)
39	2:	144	C9.H20.0	2,5-DIMETHYL-4-HEPTANOL OR 5 NONANOL (
39	3:	146	C6.H4.CL2	DICHLOROBENZENE ISOMER (NC)
39	4:	130	C9.H18.0	2-ETHYL-1-HEXANOL (NC)
39	5:	133	C9.H14.0	ISOPHORONE (NC)
39	6:	162	C8.H19.03	BUTYL CARBITOL (NC)
39	7:	0		POLY GLYCOL ETHER (NC UNKNOWN)
39	8:	134	C9.H10.0	1-PHENYL-1-PROPANONE (NC)
39	9:	154	C12.H10	BIPHENYL (NC)
39	10:	170	C12.H10.0	PHENYL ETHER OR HYDROXY BIPHENYL (NC)
39	11:	222	C12.H14.04	DIETHYL PHTHALATE (NC)
39	12:	0		POLY GLYCOL ETHER (NC UNKNOWN)
39	13:	220	C15.H24.0	2,5-DI-TEBT-BUTYL-P-CRESOL (NC)
39	14:	96	C4.H4.0.N2	4(1H)-PYRIMIDINONE (NC)
39	15:	0		UNKNOWN PEAK A
39	16:	0		UNKNOWN PEAK B
39	17:	0		UNKNOWN PEAK C
39	18:	0		UNKNOWN PEAK D
39	19:	0		UNKNOWN PEAK E (A NITRILE?)
39	20:	154	C9.H10.03	1,2-BENZENEDIOL, 4-(2-HYDROXYETHYL)-
39	21:	154	C10.H18.0	2-METHYL-1-NONEN-3-ONE (NC)
39	22:	266	C12.H27.04.P	TRIBUTYLPHOSPHATE (NC)
39	23:	268	C19.H40	PRISTANE (NC)
39	24:	140	C9.H16.0	3,3,5-TRIMETHYL-CYCLOHEXANONE (NC)
39	25:	154	C9.H14.02	2,2,6-TRIMETHYL-1,4-CYCLOHEXANEDIONE (NC)
39	26:	162	C9.H10.03	BIS-(2-ETHOXYETHYL)ETHER (NC)
39	27:	222	C10.H22.05	2,3,8,11,14-PENTAODECANE (NC)

NUM	SPEC	PURITY	FIT
1	0	0	0
2	0	0	0
3	0	0	0
4	221	446	855
5	0	0	0
6	0	0	0
7	0	0	0
8	0	0	0
9	0	0	0
10	0	0	0
11	0	0	0
12	0	0	0
13	0	0	0
14	0	0	0
15	696	411	834
16	696	395	856
17	920	652	974
18	0	0	0
19	0	0	0
20	0	0	0
21	480	361	824
22	602	572	943
23	0	0	0
24	0	0	0
25	0	0	0
26	267	201	630
27	0	0	0

APPENDIX E

TECHNICAL INFORMATION

DATA BASE DESCRIPTION

TECHNICAL INFORMATION
DATA BASE DESCRIPTION

RTECS contains toxicity data for approximately 21,000 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 effects produced by single doses, some lethal and some non-lethal. Substances whose principal toxic effect is from exposure over a long period of time are not presently included. Toxic information on each chemical substance is determined by examining and evaluating the published medical, biological, engineering, chemical and trade information and data for each substance selected.

The Toxline data base contains over 650,000 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 the American Society of Hospital Pharmacists)
- (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 compiled 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)