

ENVIRONMENTAL PROTECTION AGENCY

OFFICE OF ENFORCEMENT

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Compliance Evaluation
And
Wastewater Characterization
Union Carbide Company
Institute, West Virginia

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

DENVER, COLORADO

AND

REGION III PHILADELPHIA

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Environmental Protection Agency
Office of Enforcement
EPA-330/2-79-014a

COMPLIANCE EVALUATION
AND
WASTEWATER CHARACTERIZATION

UNION CARBIDE COMPANY
INSTITUTE, WEST VIRGINIA

REGION III LIBRARY
ENVIRONMENTAL PROTECTION AGENCY

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March 1979 - Rev. May 1979

National Enforcement Investigations Center - Denver
and
Region III - Philadelphia

II. PROCESS OPERATIONS AND POLLUTION SOURCES

Union Carbide produces about 40 products from approximately 350 raw materials. Twenty-eight of the products or raw materials are on the Toxic Pollutant List [Table 1]. Production rates and process capacities are considered confidential and are not included in this report. Figures 1, 2 and 3 show the general flow of materials at this plant and Figures 4 and 5 show the relationship of process units and wastewater disposal routes.

Kanawha River water, 720,000 m³ (190 mgd), is used for cooling and process water at the plant. All process wastewater, domestic waste, and storm runoff are discharged after treatment in the activated sludge wastewater treatment facility (WWTF) to the Kanawha River through Outfall 001. Non-contact cooling water is discharged untreated through 5 permitted outfalls (002, 003, 004, 005 and 008). The Company has installed specific organics monitors on two major cooling water discharges (Outfalls 003 and 005). The NPDES limitations [Tables 2 and 3] for these discharges were effective July 1, 1977.

OLEFINS PRODUCTION

The olefins unit is currently used only for removing contaminants from methane. The major process equipment is the distillation column. The only wastewater associated with this unit is the non-contact cooling water that is discharged through Outfall 004. All still column residues are burned at the powerhouse. There are no air emission sources from this unit.

Table 1

UNION CARBIDE, INSTITUTE PLANT
MATERIALS ON TOXIC POLLUTANT LIST

Acrolein	Isophorone [#]
Acrylonitrile	Isophorone residue
Antimony Trichloride	Methyl Chloride
Benzene	Naphthalene
Carbon Tetrachloride	Nickel
Carbaryl	Nickel Nitrate
Chlorobenzene	Nickel Sulfate
Chloroform	Phenol
Chlorophenol	Ramey Nickel
Chrome nitrate	Silver
Copper	Silver Oxide
Dichlorobenzene	Toluene
Dichlorophenol	Trichlorobenzene
Dinitrotoluene	Trichlorophenol

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I. INTRODUCTION

Union Carbide Corporation, Chemicals and Plastic Division, operates a petrochemical plant at Institute, West Virginia. On the 314 hectare (775 acre) plant site, Union Carbide Institute (UCI) produces about 40 different chemicals, the largest volume associated with the insecticide SEVIN®.

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 of in the Kanawha Valley with 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 to the Kanawha River, b) evaluate pollution abatement practices, and c) determine if NPDES* permit requirements were being met. NEIC conducted a detailed plant inspection and a subsequent field survey. The inspection results are summarized in this report, and presented in full context in Appendix A.

* 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 objectives of the April 1978 plant inspection were to:

1. inspect process operations
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 9 to 18, 1978 survey were to:

1. measure all wastewater discharge flows.
2. determine if NPDES permit No. WV0000086 effluent limitations were being met.
3. collect wastewater and water intake samples 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 UCI 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 9 to 18, 1978, NEIC personnel conducted a survey at the UCI facility. Cooling water flows were measured using lithium chloride dilution procedures. Twenty-four hour composite samples of the wastewater treatment plant and 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. Air pollution emissions are controlled by scrubbers, flares, baghouses and electrostatic precipitators. Combustible gases are collected and burned.

Solid wastes disposal in the Goff Mountain Chemical Landfill and Holz pond appears to be adequate. According to Union Carbide officials, most hazardous chemical wastes are trucked to the Goff Mountain Chemical Landfill for disposal. Waste containing heavy metals and toluene diisocyanate are contract disposed. Waste activated sludge from the wastewater treatment facility (WWTF), are pumped to Holz pond.

Even though time-weighted composite, instead of flow-weighted composite, samples are collected from the non-contact cooling water and WWTF discharge, sample collection procedures are adequate. Cooling water flows and the WWTF discharge remain fairly constant over any given 24-hour period. Therefore, Company time-weighted samples are equivalent to flow-weighted composites.

Measurement of WWTF effluent flow is inadequate. The Marsh McBirney Model 250 flow meter was not operating. Flow was being calculated based on the amount of water entering the aeration basin less the amount of sludge wasted. The NPDES permit requires that the flow be continuously measured and recorded.

Sample preservation is inadequate. Samples for phenolic analysis are not preserved during collection. TOC, phenol, NH_3 and TKN samples collected during weekends and not analyzed until Monday are only kept cool, not preserved as required by EPA prescribed procedures.

In general, chemical analyses are adequate with most analysis performed according to EPA approved methods. The TDS concentrations were being calculated by subtracting the difference between total and suspended solids. However, this is not an approved method and would result in TDS values greater than actual values. Analyses of standard reference samples provided by NEIC showed TOC, TKN and TDS results were not within acceptance limits.

In general, bioassay procedures are adequate. Discrepancies observed include: a) not starting tests within 8 hours as recommended by Standard Methods, b) using city dechlorinated tap water as 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.

According to DMRs, the Company exceeded NPDES permit limitations during the period October 1977 through March 1978. The 001 discharge exceeded either monthly average or daily maximum BOD, TSS, TOC and chloride limitations 3, 6, 2 and 1 months, respectively. TOC limitations were exceeded on each cooling water outfall. TKN violations were reported from Outfalls 004, 005 and 008, NH_3 from Outfalls 004 and 005 and pH from 002, 004, 005 and 008. Flow values were not reported on the DMRs.

The WWTF effluent contained 13 priority pollutants.* Seven of these, 1,2-dichlorobenzene, 1,4-dichlorobenzene, 2,6-dinitrotoluene, 1,4-diphenylhydrazine, methylene chloride, naphthalene, and bis-(2-ethylhexyl) phthalate, had concentrations greater than 10 $\mu\text{g/l}$.

SURVEY

The Company flow measurements using lithium chloride are not accurate because of discrepancies in the procedures. The lithium chloride injection pump discharge rate changes during the test such that the volume of concentrate added to the wastewater decreases. Company personnel use the average discharge rate to calculate flow which assumes linear variation during the test. In using tracer technique it is necessary that the tracer be introduced at a constant rate otherwise the calculated flow can be in error.

The Company installed flow device (March McBirney Model 250) on the WWTF effluent is not accurate. Instantaneous measurement showed that the meter recording flow was approximately 50% higher than actual.

The WWTF effluent (Outfall 001) was in compliance with permit limitations. Outfall 001 had a daily averaged discharge of 220 kg/day BOD, 480 kg/day TSS, 1,600 kg/day TOC, 270 kg/day TKN, 150 kg/day NH_3 and 23,000 kg/day chloride. These values were only 10, 38, 35, 18, 22 and 22%, respectively, of the NPDES limitations. Fecal coliform bacteria densities ranged from 5 to 33/100 ml (permit limitation of 400/100 ml).

Outfall 001 discharge was determined to be mutagenic by the Ames test. The basic extract displayed a mutagenic activity ratio greater than 2.5 which correlates closely (>90% probability) with inducement of cancer in laboratory animals.

The WWTF effluent was acutely toxic as demonstrated by bioassay tests. These test results show that the 96-hour LC_{50} was 80.5% effluent in Kanawha River dilution water.

Except for Outfall 002, cooling water discharges complied with NPDES limitations. The 002 discharge exceeded TOC, TKN and NH_3 permit limitations. The daily maximum net TOC, TKN and NH_3 concentrations ranged from 12 to 15, 0.2 to 1.6 and 0 to 0.8 mg/l, respectively. The NPDES TOC, TKN and NH_3 limitations were exceeded 2, 1 and 1 day respectively.

A total of 53 organic (38 confirmed) compounds were identified in the UCI discharges. Concentrations of the confirmed compounds ranged from <1 to 140,000 $\mu\text{g/l}$. Seventeen of the 53 compounds are

* For explanation of Priority Pollutant see Section IV.

priority pollutants. Four of the priority pollutants benzene, carbaryl, carbon tetrachloride and chloroform are known carcinogens. Benzene is carcinogenic to humans; the others are carcinogenic to animals.

III. INSPECTION METHODS AND RESULTS

During the April 1978 inspection, NEIC personnel obtained information on process operations and associated pollution sources, pollution abatement practices and self-monitoring techniques [Appendix A]. According to Company personnel, the plant operates continuously with little production variation. Production rates and process capacities are not included in this discussion because of their confidential status.

POLLUTION SOURCES AND DISPOSAL METHODS

Process, boiler feed and cooling waters are obtained from the Kanawha River. All process and domestic wastewater and supernatant from Goff Mountain Chemical Landfill are collected and treated in a conventional activated sludge wastewater treatment facility (WWTF) [Figure 1]. The treatment system includes primary clarification, flow equilization, biological treatment (aeration basins) and final sedimentation. The effluent is discharged into the Kanawha River through Outfall 001. Non-contact cooling water is discharged into the Kanawha River through five outfalls (002, 003, 004, 005 and 008) [Figure 2]. Methylisocyanate unit residues are hydrolyzed and burned in a liquid incinerator.

The Company has an extensive in-plant wastewater monitoring program. Unit process wastewaters are monitored with in-plant samplers and total carbon (TCA) and gas chromatographic (GC) analyzers to identify and locate spills and/or leaks. Each outfall is equipped with a total TCA which is telemetered to the WWTF. When the TCA value exceeds a specific value, grab samples are collected and analyzed on a GC to

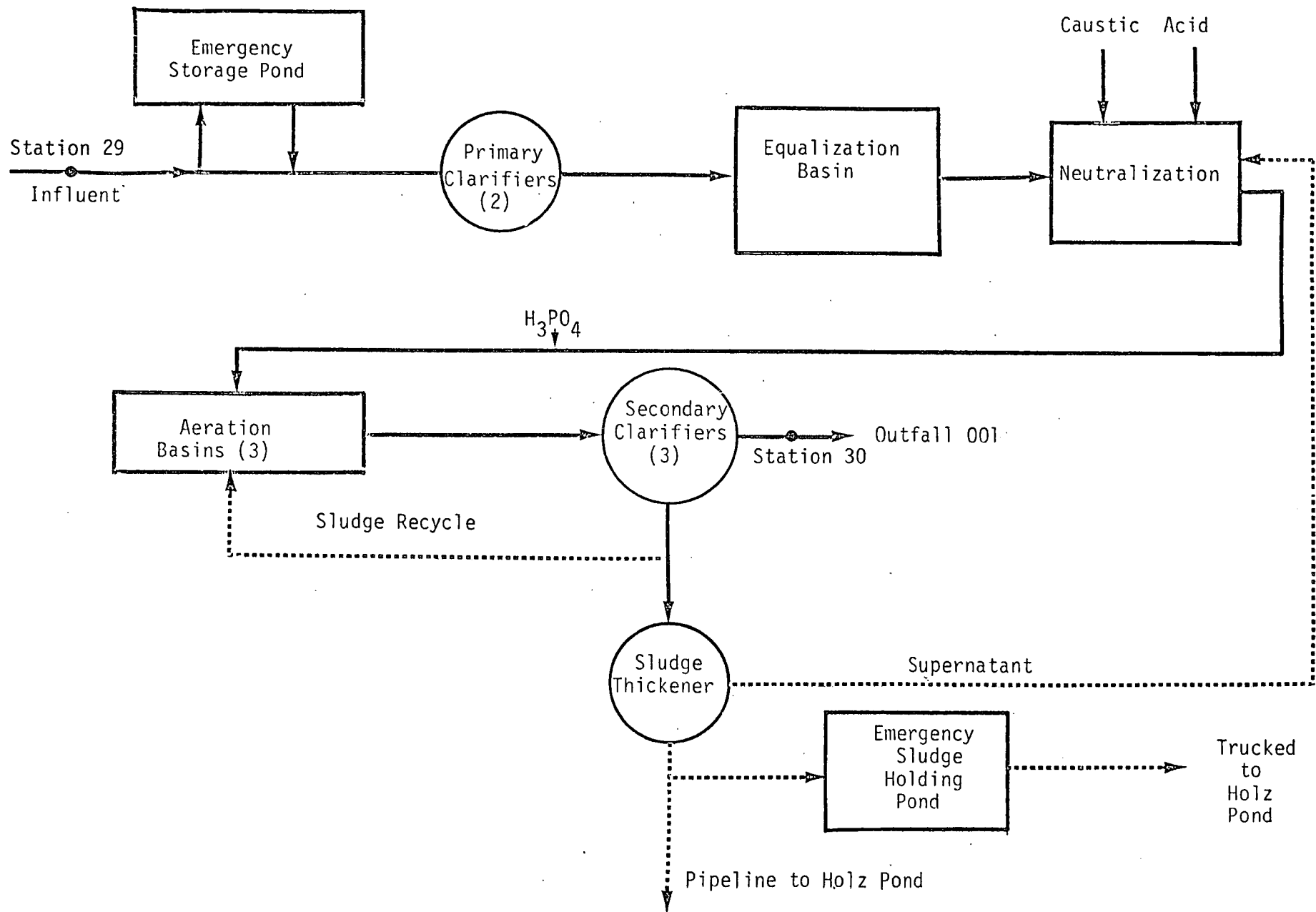


Figure 1. Schematic of Union Carbide Institute Wastewater Treatment Facilities

PLANT COOLING WATER SEWER SYSTEM

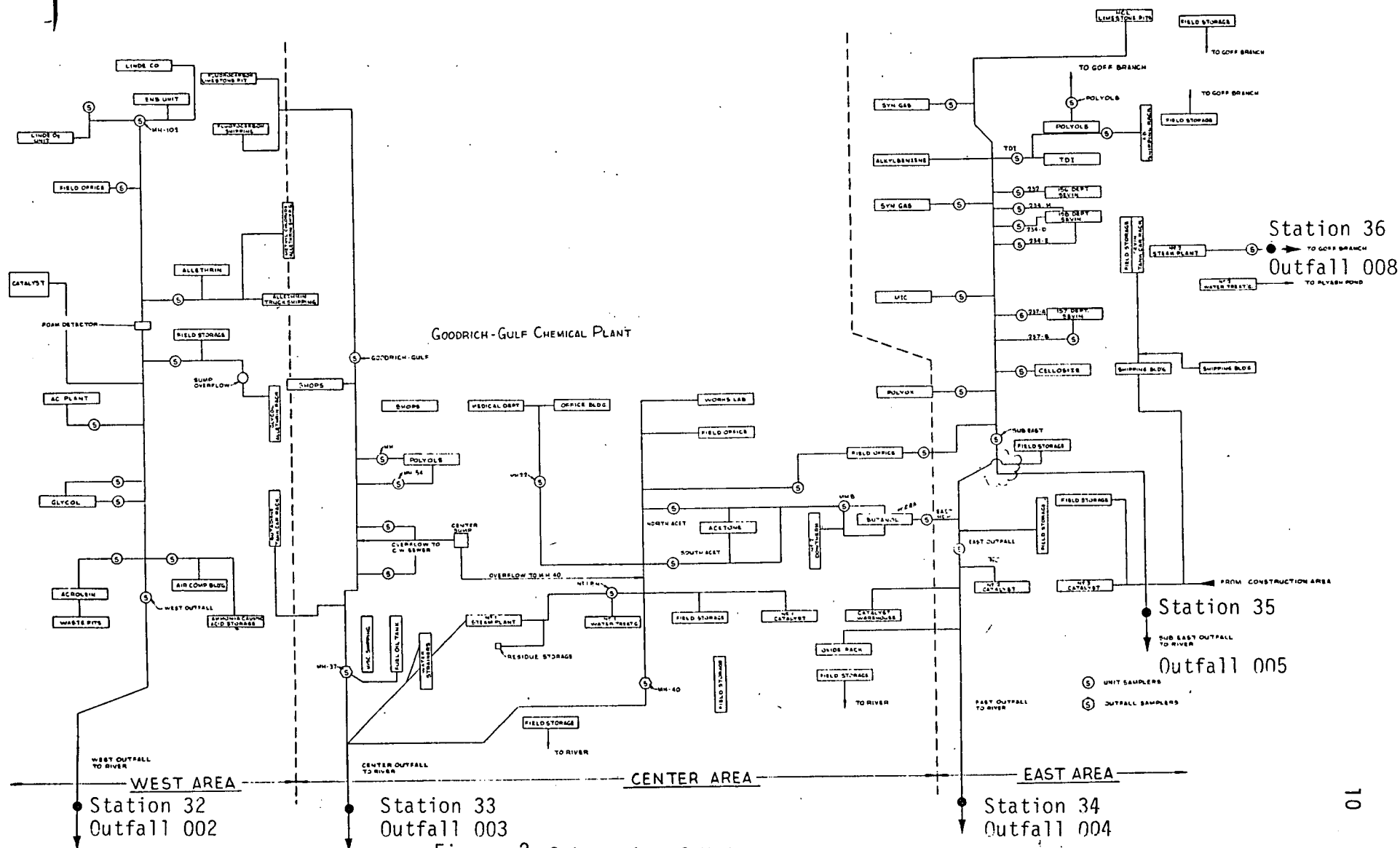


Figure 2. Schematic of Union Carbide Institute Cooling Water Sewer System

identify the pollutant source. Specific organics monitors were installed on cooling water Outfalls 003 and 005 during the inspection.

The air pollution emission from the various processes are controlled by scrubbers, flares, baghouses, and electrostatic precipitators. These control devices appear to adequately control air emissions from this plant. According to information provided by Company personnel, most combustible gases are collected and burned in the flares [Appendix A].

Solid wastes are disposed of in Goff Mountain Chemical Landfill,* Holz Pond* or the Regional Development Authority Landfill. Hazardous chemical wastes and toxic substance from UCI and Union Carbide South Charleston (UCSC) are hauled to the Goff Mountain Landfill. Leachate from the landfill is collected in underdrain piping network and treated in the wastewater treatment facility. Waste-activated sludge is pumped to Holz pond, an anaerobic lagoon. Non-chemical solid wastes (lumber, paper, scrap polymer, etc.) are disposed of in the Regional landfill.

SELF-MONITORING EVALUATION

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

The NPDES permit requires the collection of 24-hour flow-proportional composite samples. Company personnel collect time-weighted composite samples from the non-contact cooling water and WWTF discharges. Company personnel indicated that flows remain fairly constant, therefore, the samples are essentially flow-proportional.

* Goff Mountain landfill is operated by Union Carbide Institute personnel. Holz Pond is operated by Union Carbide, South Charleston personnel.

The flow measurement device on the WWTF effluent (Outfall 001) was not operating during the April inspection [Appendix A]. Flows were being calculated based on an instantaneous measurement of the amount of water entering the aeration basin less the amount of sludge wasted. The NPDES permit requires that the WWTF effluent (Outfall 001) be continuously monitored and recorded. Flow data have not been reported on DMRs.

WWTF effluent samples are collected from a manhole at the point where the clarifier discharges join. Company personnel performed tests subsequent to the inspection to determine if representative samples can be collected at this point. Test data show the samples are representative.

Samples for phenolic analysis are aliquoted from composite samples and not preserved during collection. Samples collected during weekends are not analyzed until Monday. These samples are only kept cool and not preserved as required by EPA prescribed procedures. TOC, phenolic, $\text{NH}_3\text{-N}$, and TKN samples require acid preservation, as well as cooling to assure stability.

Company personnel also analyzed standard reference BOD, TOC, TSS, chlorides, TKN, pH, TDS, total alkalinity, $\text{NH}_3\text{-N}$, sulfate, fluoride and orthophosphorous samples provided by NEIC. All of these analyses, except TDS, were performed according to EPA-approved methods. TDS were being calculated by subtracting the difference between total and suspended solids. Company reference sample results show that TOC, TKN and TDS values were not within acceptance limits.

Bioassay testing is conducted quarterly on all outfalls as required by the NPDES permit. Discrepancies noted in the Company's bioassay procedures were: a) collection of time rather than flow-proportional samples, b) testing not started within 8 hours as recommended by Standard

Methods, c) city dechlorinated tap water was used as dilution water instead of Kanawha River water, d) not running tests in duplicate, and e) aerating samples throughout the 96-hour test.

It is advisable, though not required, that the laboratory use a constant-temperature water bath to maintain test temperatures rather than depend on ambient air temperatures.

Discharge Monitoring Reports [Appendix A, Tables 4 and 5] show that for the period October 1977 through March 1978 the Company was in violation of monthly average or daily maximum permit limitations as follows:

Parameter	No. of Months Limitation Exceeded					
	Outfall					
	001	002	003	004	005	008
BOD	3	N/A ^a	N/A	N/A	N/A	N/A
TSS	6	N/A	N/A	N/A	N/A	N/A
TOC	2	1	4	5	3	2
TKN	0	0	0	1	1	1
NH ₃ -N	0	0	0	1	2	0
Chlorides	1	N/A	N/A	N/A	N/A	N/A
pH	0	2	0	3	5	2

a N/A - not applicable.

A composite sample representing combined discharges of Outfalls 002, 003, 004, 005 and 008 also violated the daily maximum phenol limitation in January 1978.

NEIC SAMPLE ANALYSIS

NEIC personnel collected a grab sample from the UCI WWTF effluent.

This sample was analyzed for priority pollutants* and other organics [Appendix A]. A total of 30 organic chemicals, including 13 priority pollutants, were identified. Of the priority pollutants, 7 had concentrations of 10 µg/l or greater; these were 1,2-dichlorobenzene, 1,4-dichlorobenzene; 2,6-dinitrotoluene; 1,4-diphenylhydrazine; methyl chloride; naphthalene and bis-(2-ethylhexyl) phthalate. In addition, the pesticide Carbaryl was detected.

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

IV. SURVEY METHODS AND RESULTS

During August 1978 NEIC personnel measured wastewater flow, determined compliance with NPDES permit effluent limitations and characterized the wastewater discharged and the water intake. Permit compliance was based on the following effluent limitations, which became effective July 1, 1977.

OUTFALL 001

Paramter	Discharge Limitations	
	kg/day (lbs/day)	
	Daily Average	Daily Maximum
Flow m ³ /day	NA	NA
BOD ₅ May-Oct	2,270 (5,000)	5,000 (11,000)
Nov-Apr	3,860 (8,500)	8,600 (19,000)
TSS	1,270 (2,800)	1,910 (4,200)
TOC May-Oct	4,540 (10,000)	10,000 (22,000)
Nov-Apr	7,720 (17,000)	17,250 (38,000)
TKN	1,500 (3,300)	3,000 (6,600)
NH ₃ -N	680 (1,500)	1,360 (3,000)
Chlorides	104,000 (228,000)	136,000 (300,000)
Fecal Coliforms/100 ml	200	400
Threshold Odor No.	512	1,024
Temperature °C (°F)	NA	43.3 (110)
pH	Range 6-9	

OUTFALLS 002, 003, 004, 005 and 008

Parameter	Discharge Limitations (Net)			
	kg/day (lbs/day)		Other Units	
	Daily Avg.	Daily Max.	Daily Avg.	Daily Max.
TOC	NA	NA	4 mg/l	12 mg/l
TKN	NA	NA	0.5 mg/l	1.5 mg/l
NH ₃ -N	NA	NA	0.2 mg/l	0.6 mg/l
Chlorides	137,000 (302,000	182,000 (400,000)	NA	NA
Phenolics	30 (70)	82 (180)	NA	NA
Threshold Odor No.	NA	NA	128	256
Temperature °C (°F)	NA	NA	NA	43.3 (110)
Sulfides	NA	NA	NA	NA
(O.F. 005 only)				
pH	NA	NA	Range 6-9 ^a	

- a For Outfalls 002, 003, 004 and 005, deviations from the pH range 6-9 which (1) do not exceed 15 minutes for any single instances, (2) do not exceed a total of 30 minutes in any calendar day, (3) do not exceed a total of 10 hours in any calendar month, and (4) are within the pH range of 4.0-10.5 units, are not considered violations of this permit.

The permit requires that toxicity be monitored by quarterly bioassays.

The above limitations, except for temperature, pH, sulfides and fecal coliform organisms are based on 24-hour composite samples. Sulfide, fecal coliform organisms and pH are based on grab samples. Temperature is to be measured in the effluent stream.

FLOW MEASUREMENTS

The NPDES permit requires that cooling water flows be determined from quarterly water meter balances and that the WWTF discharge be continuously measured and recorded. Company personnel determine relative percent of water meter flow being discharged through each cooling water outfall using the lithium chloride dilution technique. Instantaneous cooling water discharge rates are measured quarterly. NEIC personnel

measured the flow through each cooling water outfall August 9, 1978 using the lithium chloride dilution technique [Appendix B]. Results [Table 1] show a total instantaneous cooling water discharge of 974,000 m³/day (257 mgd). Average recorded water intake for this date was reported as 883,000 m³/day (233 mgd), 9% less than the measured value. Company data for these outfalls [Table 1] show a total discharge of 883,000 m³/day (220 mgd), 14% less than measured by NEIC.

Observations of Company procedures showed them to be similar to NEIC procedures. The major difference observed was that, during the test, the volume of lithium chloride discharged from the Company's injection pump did not remain constant. Company personnel average the initial and final lithium chloride injection rates to determine flow. Unless the injection rate varies linearly during the test period, the procedure will result in erroneous flow calculations.

The WWTF discharge (Outfall 001) flow-meter is monitored with a Marsh McBirney Model 250 meter. This flow-meter was checked by NEIC using lithium chloride. Results are tabulated below:

Date (August)	Time (Approx.)	NEIC Lithium Chloride Flow		Company Meter Flow	
		m ³ /day	mgd	m ³ /day	mgd
9	1145	27,600	7.3	22,700	6.0
16	1825	17,800	4.7	30,700	8.1
19	0950	18,900	5.0	29,900	7.9
19	1110	18,900	5.0	29,900	7.9
19	1230	18,900	5.0	31,800	8.4
--	Avg.	20,400	5.4	29,000	7.7

Table 1
COOLING WATER FLOWS^a
UNION CARBIDE INSTITUTE

Outfall No.	NEIC		Company	
	m ³ /day	mgd	m ³ /day	mgd
002	216,000	57	160,410	42.4
003	178,000	47	218,050	57.6
004	22,300	5.9	36,710	9.7
005	553,000	146	411,810	109
008	4,500	1.2	5,680	1.5
Total	974,000	257	833,000	220

a Flows were measured using lithium chloride. NEIC data for 8/9/78 and company data 7/29/78 (Outfalls, 002, 003, 004 and 005) and 8/4/78 (Outfall 008).

During a power outage August 18, the flow-meter was registering approximately 9460 m³/day (2.5 mgd). However no wastewater was being discharged. This discrepancy was reported to Company personnel.

Based on Marsh McBirney chart readings, the flows average 28,000, 29,500 and 29,100 m³/day (7.4, 7.8 and 7.7 mgd), respectively, August 16, 17 and 18, 1978. These values show that, during the survey, the flow remained fairly constant.* The average of the five instantaneous lithium chloride dilution measurement noted above show a flow of 20,400 m³/day (5.4 mgd), compared to an average meter reading of 29,000 m³/day (7.7 mgd).

Company personnel can also measure process wastewater from the in-plant collection sump (orifice plates), influent to WWTF (venturi meter) and the effluent from aeration basins (weirs). After the above discrepancy was reported, the flow values for these measuring monitoring points were compared and none agreed.

As previously noted, Company personnel have only been reporting Outfall 001 analytical data on DMRs and not flow values as required by the permit. Data submitted to the State however, show average monthly flow values for October 1977 through July 1978 ranging from 17,000 to 21,200 m³/day (4.5 to 5.6 mgd). Based on these results, a conservative flow of 19,000 m³/day (5 mgd) was used by NEIC to determine compliance with NDPEs limitations.

Sampling

Sampling was performed by NEIC at Union Carbide Institute August 15 to 18, 1978. Samples were collected from the river intake, cooling water discharges (Outfalls 002, 003, 004, 005 and 008) [Figure 2] and

* According to Company personnel, wastewater from the equalization basin is discharged into the aeration basins at a constant rate.

WWTF influent and effluent (Outfall 001) [Figure 1]. Sample aliquots were manually collected from the cooling water discharges and river intake every two hours and continually composited on a time-weighted basis, except for volatile organics. Volatile organic samples were collected three* times per day. The influent to the WWTF was manually sampled hourly and continually composited on a time-weighted basis. Grab samples were collected three times each day for volatile organic and direct aqueous injection analysis. The WWTF effluent was also manually sampled hourly and continually composited on a flow-weighted basis for all parameters, except for volatile organics, direct aqueous injection and fecal coliform analyses. Three grab samples were collected daily for these parameters. Samples were collected over the period 7 a.m. to 7 a.m. which corresponds to the production day at Union Carbide Institute. The parameters monitored and the sample type of each station are shown in Table 2. Chain-of-custody procedures [Appendix C] were followed for the collection of the samples and for laboratory analyses. All samples were analyzed by procedures discussed in Appendices D, E and F.

Flow-through bioassays were conducted August 15 to 19, 1978 on the WWTF effluent [Appendix G]. The effluent wastewater was continually composited on a equal-volume basis. During collection, the wastewaters for bioassay were not preserved or iced. Dilution water was obtained from the Kanawha River at a point approximately 3 km (2 miles) upstream of the mouth of the Elk River.

* Four volatile organic samples were collected on the first day of the survey from Outfall 005.

Table 2
DESCRIPTION OF MONITORING STATIONS
UNION CARBIDE INSTITUTE

Station ^a	Description	Type of Sample	Parameter ^b
29	Influent to WWTF	24-hour composite	chlorides; TOC; NH ₃ ; TKN; organics
		Grab	volatile organics ^c ; direct aqueous injection ^c
30	Effluent from WWTF (Outfall 001)	24-hour composite	BOD; TSS, chloride; TOC; NH ₃ ; TKN; metals; phenol; organics; mutagens ^d
		Grab	volatile organics; direct aqueous injections ^c ; fecal coliforms ^c
32	Cooling Water (Outfall 002)	24-hour composite	TOC; NH ₃ ; TKN; organics
		Grab	volatile organics
33	Cooling Water (Outfall 003)	24-hour composite	TOC, NH ₃ ; TKN; organics
		Grab	Volatile organics
34	Cooling Water (Outfall 004)	24-hour composite	TOC; NH ₃ ; TKN; organics
		Grab	Volatile organics
35	Cooling Water (Outfall 005)	24-hour composite	TOC; NH ₃ ; TKN; organics
		Grab	Volatile organics
36	Cooling Water (Outfall 006)	24-hour composite	TOC; NH ₃ ; TKN; organics
		Grab	Volatile organics
37	Water Intake	24-hour composite	TOC; NH ₃ ; TKN; organics
		Grab	Volatile organics

a Figures 1 and 2 show station location.

b Temperature and pH were measured periodically at all stations.

c Grab samples collected three time each day for this parameter, except for voaltile organics at Outfall 005 which was collected four times on the first day of sampling.

d Mutagen samples were collected twice during the survey. Only one of the mutagen samples was analyzed.

Plant production* for the week prior to and during the survey are summarized below.

Date(s) (August)	Total Plant Operating Rate (% of Capacity)
6-12	47.49%
15	49.10%
16	50.20%
17	51.76%
Units reported down during this time period were:	
6-12	Methyl Chloride
15	SEVIN
16	Methyl Chloride and MIC

Following is a discussion of sampling results by individual outfalls.

Outfall 001 (Station 30)

Outfall 001, a submerged discharge, contains the effluent from the activated sludge wastewater treatment facility (WWTF). The WWTF treats all process wastewaters and leachate from the Goff Mountain Chemical Landfill. Influent and effluent samples were collected respectively at the splitter box just upstream of the primary clarifiers and from the last manhole on the discharge line.

Results [Table 3] show that the WWTF influent contained an average of 18,000, 700, 48 and 25,000 kg/day respectively, of TOC, TKN, NH_3 , and chloride. The final effluent contained an average of 1,600 kg/day

* Actual production figures are considered confidential by Company personnel. Percentage figures were provided by the Company.

Table 3
SUMMARY OF FIELD MEASUREMENTS AND ANALYTICAL DATA
UNION CARBIDE INSTITUTE

Parameters (August)	16	17	18	Average
Station 29 Influent to WWTF				
<u>Flow^a</u>				
m ³ /day x 10 ³	19.0	19.0	19.0	19.0
mgd	5.0	5.0	5.0	5.0
Temperature °C Range	39-44	38-43	39-46	
pH Range	3.9-12.2	3.5-10.5	1.5-10.8	
<u>TOC</u>				
mg/l	500	740	1,600	950
kg/day	9,500	14,000	30,000	18,000
lb/day	21,000	31,000	67,000	40,000
<u>TKN</u>				
mg/l	16	59	37	37
kg/day	300	1,100	700	700
lb/day	670	2,500	1,500	1,600
<u>NH₃-N</u>				
mg/l	1.0	5.3	1.3	2.5
kg/day	19	100	25	48
lb/day	42	220	54	110
<u>Chloride</u>				
mg/l	850	1,100	2,000	1,300
kg/day	16,000	21,000	38,000	25,000
lb/day	35,000	46,000	83,000	55,000

a Conservative estimate based on effluent flows measured with lithium chloride dilution techniques.

TOC, 270 kg/day TKN, 150 kg/day NH_3 and 23,000 kg/day chloride [Table 4]. These data show that the WWTF removed 91% of the TOC, 61% of the TKN and 8% of the chloride. The amount of NH_3 discharged was more than 3 times that contained in the influent.

Daily maximum discharges of BOD, TSS, TOC, TKN, NH_3 and chloride were only 5, 31, 18, 10, 14 and 17% respectively, of NPDES limitations. As previously noted, compliance was based on a conservative flow of 19,000 m^3/day . However, if the WWTF had been operated at a design flow* of 24,600 m^3/day (6.5 mgd), the discharge would still have met permit limitations.

Composite samples were also analyzed for metals (Zn, Al, Ni, Sn, As, Cd, Cr and Cu) [Appendix D]. With the exception of zinc and aluminum, the concentrations were below detectable limits. Zinc concentrations ranged from 0.03 to 0.06 mg/l and aluminum from 0.4 to 1.0 mg/l.

DMR data for August to October 1978 show that the TSS maximum limitation was violated on two different days [Appendix A]. The reported DMR data are generally higher than those obtained during the survey.

Bacteriological analyses [Appendix E] show that, during the survey, the WWTF effluent contained fecal coliform bacteria densities ranging from 5 to 33/100 ml (geometric mean of 19/100 ml) which is well within NPDES permit limitations (daily average 200/100 ml; daily maximum 400/100 ml).

Analyses for mutagenic activity [Appendix F] were performed on one composite sample from Outfall 001. The Ames Bacterial Assay for Mutagenicity¹ was performed on sample concentrates using the agar

* Observations and instantaneous lithium chloride flow checks show that the plant was operating at less than design flow.

Table 4
SUMMARY OF FIELD MEASUREMENTS AND ANALYTICAL DATA
UNION CARBIDE INSTITUTE

Parameters (August)	16	17	18	Average
Station 30 WTF Discharge (Outfall 001)				
<u>Flow^a</u>				
m ³ /day x 10 ³	19.0	19.0	19.0	19.0
mgd	5.0	5.0	5.0	5.0
Temperature °C. Range	29-32	27-31	28-31	
pH Range	6.3-8.5	5.6-7.8	6.0-7.4	
<u>BOD</u>				
mg/l	9	13	13	12
kg/day	170	250	250	220
lb/day	380	540	540	490
<u>TSS</u>				
mg/l	20	24	32	25
kg/day	380	450	610	480
lb/day	830	1,000	1,300	1,000
<u>TOC</u>				
mg/l	80	80	95	85
kg/day	1,500	1,500	1,800	1,600
lb/day	3,300	3,300	4,000	3,500
<u>TKN</u>				
mg/l	14	15	14	14
kg/day	260	280	260	270
lb/day	580	630	580	600
<u>NH₃-N</u>				
mg/l	5.5	9.8	8.4	7.9
kg/day	100	190	160	150
lb/day	230	410	350	330
<u>Chloride</u>				
mg/l	1,200	1,200	1,200	1,200
kg/day	23,000	23,000	23,000	23,000
lb/day	50,000	50,000	50,000	50,000
<u>Phenolic Compounds</u>				
µg/l	29	26	57	37
kg/day	550	490	1,100	710
lb/day	1,200	1,100	2,400	1,600

a Conservative estimate based on results obtained by the lithium chloride dilution technique.

plate incorporation method [Appendix F]. The standard Ames Test determines mutagenic activity through use of bacteria as indicator organisms; this information correlates closely ($>90\%$ probability) with inducement of cancer in laboratory animals by organic compounds.^{2,3,4} Extrapolation of this information to higher organisms (such as humans) is warranted because mutagens may alter genetic material (deoxyribonucleic acid) in a similar manner in other life forms. If a compound is mutagenic in any organism, it should not be exposed to the human population. Only one molecule of a mutagen is potentially sufficient to cause a mutation that is also likely to be carcinogenic. Because genetic repair systems are not completely effective, safe doses of mutagens and carcinogens cannot be projected.^{5,6}

Acidic and basic sample extracts were prescreened for mutagenic activity using four standard Salmonella test strains, TA 98, TA 100, TA 1535 and TA 1537. Samples were first tested individually and then subjected to metabolic activation by addition of rat liver homogenate.

The sample failed to show evidence of mutagenic activity when screened individually with the four Salmonella tester strains. When the Union Carbide Institute WWTF discharge sample extract was treated with the rat liver preparation [Appendix F], Salmonella tester strain TA 98 showed obvious mutagenic activity. The basic extract from this sample displayed a mutagenic activity ratio* higher than 2.5. Table 5

* The mutagenic activity ratio is a measure of the tester strain mutation rate compared to control rates. A mutagenic activity ratio of 2.5 or greater correlates closely ($>90\%$ probability) with inducement of cancer in laboratory animals.^{2,3,4} If the activity ratio is 2.5 or greater and a typical dose response relationship can be demonstrated between the tester strain and increasing concentrations of sample, the results are considered positive (i.e., the substance is a mutagen). The mutagenic activity ratio is defined as $(E-C)/c$ where E is the average number of mutant colonies per test with the sample added; C is the corresponding value for the control, and c is the historical control value of 40 averaged over 100 or more tests.

Table 5
MUTAGENIC ACTIVITY OF UNION CARBIDE INSTITUTE DISCHARGE
ON SALMONELLA TESTER STRAIN TA 98
INSTITUTE, WEST VIRGINIA
August 15-21, 1978

Station Number Description	Sample ^a Type	Date-Time Collected	Extract pH	Volume of Sample ^b Concentrate Tested (μ l)	Equivalent Volume of Sample (ml)	No. of Revertant Colonies Per Plate		Mutagenic Activity Ratio ^e
						Control ^c	Experimental ^d	
30 Union Carbide Institute WWTF Discharge	Composite	8/18/78 0608	Base	500	63.3	40	346	7.7
				400	50.7		321	7.0
				300	38.0		339	7.5
				200	25.3		268	5.7
				100	12.7		218	4.5
				50	6.3		103	1.6
				25	3.2		56	<1.0
				10	1.3		39	<1.0

a Composite Samples - Compositing was hourly for each 24-hour period; date and time listed is date and time that period ended.

b Rat-liver homogenate (S-9 mix) added.

c Value based on average of 30 control values.

d Average of 2 plates

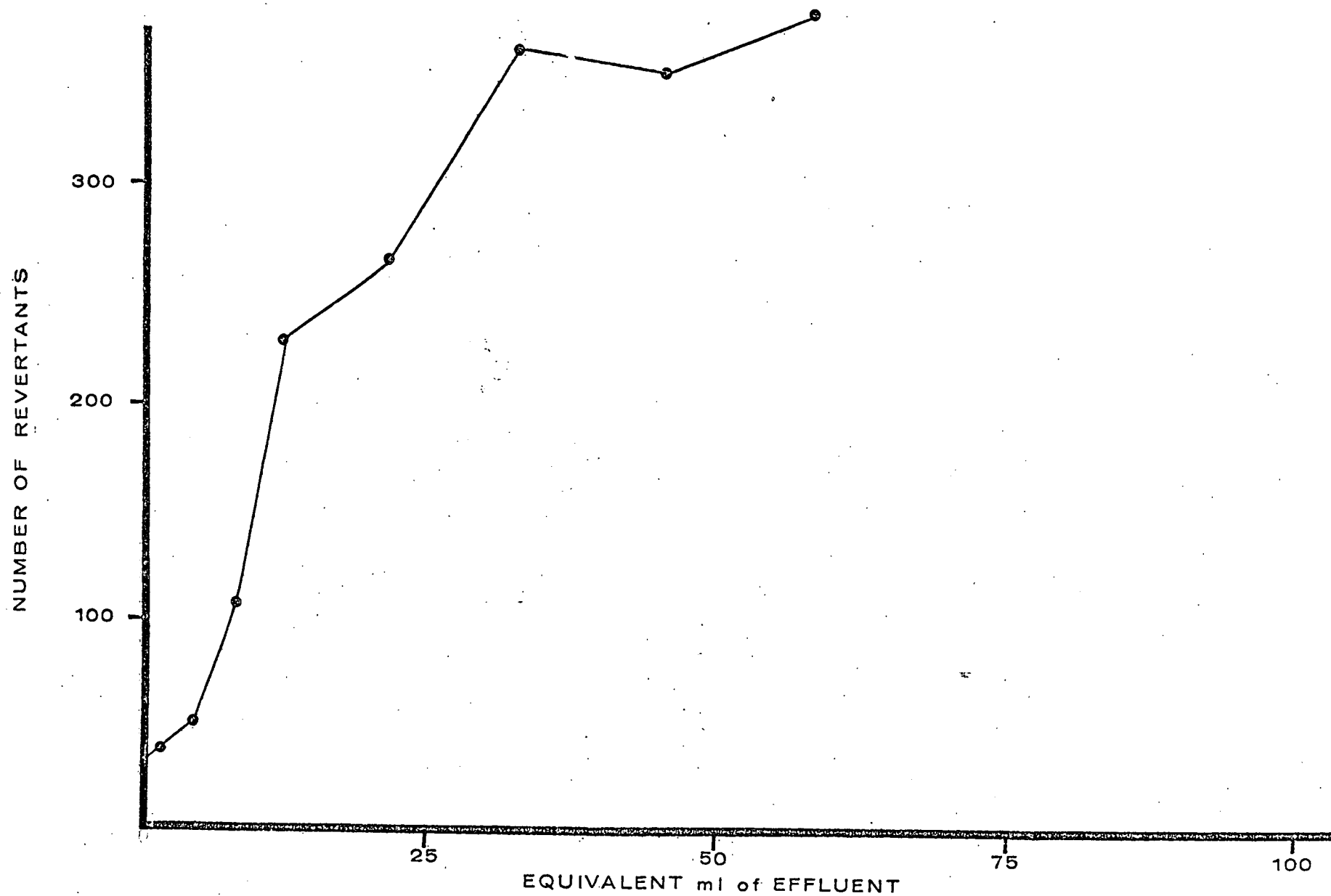
e Mutagenic Activity Ratio = $(E-C)/\bar{c}$, where E is the no. of colonies/experimental plate, C is the no. of colonies/control plate and \bar{c} is the historical control value of 40 averaged over 100 tests.

shows the volume of extract tested, the equivalent sample volume and the resultant mutagenic effect on tester strain TA 98 for the sample collected on August 18, 1978. Further testing showed a typical dose-response relationship [Figure 3] between tester strain TA 98 and the concentrated extract, illustrating an increasing number of revertant colonies with increasing concentrations of sample. The optimum concentration causing highest reversion rates was 500 μ l (63.3 ml equivalent volume of sample) causing a mutagenic activity ratio of 7.7. Volumes larger than 500 μ l could not be tested because they exceed the limits of the assay procedures. Acidic sample extracts from the UCI WWTF discharge did not satisfy the Ames Test requirements for mutagenicity. Data for test results that did not exhibit elevated reversion rates (negative mutagenic activity) are not presented in this report.

A flow-through bioassay was conducted on Outfall 001 to determine whether the wastewater was acutely toxic to fish. Juvenile fathead minnows (Pimephales promelas Raginesque) averaging 4 cm in length were used as test organisms [Appendix G]. Results show that the effluent is acutely toxic. The 96-hour LC_{50} * for this discharge was calculated to be a mixture of 80.5% effluent in Kanawha River dilution water. Because of the limited mortality of test fish [Table 6], the confidence limits for the LC_{50} were not calculated.

Chemical analysis indicate that this effluent contains a complex mixture of organic [Tables 7, 8, 9] and inorganic compounds [Table 4], as well as aluminum and zinc. Toxicity to fish could have been due to the presence of a single chemical component in lethal concentration or resulted from additive or synergistic effects of two or more chemical compounds individually present at sub-lethal concentrations. From the scope of the analysis, the exact cause could not be determined.

* LC_{50} indicates the concentration (actual or interpreted) at which 50% of the test organisms died or would be expected to die.



*Figure 3. Union Carbide Institute Discharge
Mutagen Testing Dose Response Curve (Basic Extract)*

Table 6
96-HOUR FLOW-THROUGH SURVIVAL DATA
UNION CARBIDE INSTITUTE
August 1978

Time Period	% Survival Effluent Concentration (%)						
	Control (Kanawha River Water)	10	18	32	56	75	100
24-hour	100	100	100	100	100	95	95
48-hour	100	100	100	100	100	95	70
72-hour	100	100	100	100	100	95	30
96-hour	100	100	100	100	100	75	0

Organic data [Tables 7, 8, 9] show that the influent to the WWTF contained 27 organic compounds with concentrations ranging from 2 to 140,000 µg/l. Of these, 13 are priority pollutants. Ten additional compounds were identified; 9 of which could not be confirmed.

The majority of these compounds were effectively removed in the WWTF. The effluent (Outfall 001), however did contain o- and p-dichlorobenzene (5 to 19 µg/l), N,N-dimethylaniline (3,500 µg/l), isophorone (55 µg/l) and chloroform (10 to 100 µg/l). Several other compounds - m-dichlorobenzene, 4-ethylacetophenone, butyloctenal, ethylmethylpyridine, trimethylisocyanurate, (1-butyloctyl)-benzene and (1-ethyldecyl)-benzene were identified but either could not be quantified because of interfering compounds or confirmed because pure standards were unavailable. Chloroform, o- and p- dichlorobenzene, isophorone and m-dichlorobenzene are priority pollutants.

Two samples from Outfall 001 were also analyzed for carbaryl. This compound was detected in both samples at a maximum concentration of 26 µg/l.

Outfall 002 (Station 32)

The discharge from Outfall 002 contains non-contact cooling water originating from ethylideneornbornene and vinylornbornene production, ethylene glycol recovery, the allethrin unit, acrolein derivatives production, anhydrous isopropanol recovery, and the LINDE[®] System.

Sampling results [Table 10] show that this discharge contained net concentrations of 12 to 15 mg/l TOC, 0.2 to 1.6 mg/l TKN, and 0 to 0.8 mg/l NH₃. These data show that TOC, TKN and NH₃ NPDES daily maximum limitations (12, 1.5 and 0.6 mg/l), respectively, were exceeded. The average net TOC, TKN and NH₃ concentrations for the three-day survey were respectively 3.5, 1.8 and 2 times the allowable daily average permit limitations.

Table 7
NEUTRAL EXTRACTABLE ORGANICS SAMPLING DATA
Concentration in µg/l (Gross)
UNION CARBIDE INSTITUTE

Chemical Name	Date (August)	Station 29 - Influent to WWTF			Station 30 - Outfall 001			Station 32 Outfall 002	
		→ 16	17	18	16	17	18	16	18
Aniline		600	ND ²	ND	ND	ND	ND	ND	ND
Biphenyl		NA ³	NA	NA	NA	NA	NA	<1 ⁵	3
m-Dichlorobenzene ¹		ND	190	2,600	MS	MS	ND	MS	MS
o- and p-Dichlorobenzenes ¹		3,600	MS	39,000	19	5	MS	22	15
3,4-Dihydro-1(2H)-Naphthalenone		MS	5,800	28,000	ND	ND	ND	ND	MS
N,N-Dimethylaniline		1,100	530	140,000	ND	ND	3,500	ND	15
2,6-Dinitrotoluene ¹		NA	NA	NA	NA	NA	NA	710	700
4-Ethylacetophenone		140	MS	ND	MS	MS	ND	ND	ND
2-Ethyl-1-Hexanol		ND	MS	MS	ND	ND	ND	84	ND
Isophorone ¹		12,000	3,700	MS	ND	55	ND	3	8
N-Methylaniline		200	MS	3,600	ND	ND	ND	ND	ND
2-Methyl-4-Octanone		2,600	ND	ND	ND	ND	ND	ND	ND
2-Methylnaphthalene		ND	ND	ND	ND	ND	ND	ND	ND
Naphthalene ¹		1,900	4,400	39,000	ND	ND	ND	1	13
2-Naphthalenol		11,000	14,000	19,000	ND	ND	ND	ND	ND
p-Nitrotoluene		NA	NA	NA	NA	NA	NA	ND	ND
Phenyl Ether		73	ND	5,900	ND	ND	ND	2	5
alpha-Terpineol		NA	NA	NA	NA	NA	NA	ND	ND
1,2,3,4-Tetrahydronaphthalene		2,200	4,500	120,000	ND	ND	ND	ND	ND
1,2,3,4-Tetrahydronaphthalen-1-ol		MS	100,000	ND	ND	ND	ND	ND	ND
5,6,7,8-Tetrahydronaphthalen-1-ol		930	MS	4,200	ND	ND	ND	ND	ND
Butyloctenal		NC ⁶	NC	NC	ND	NC	ND	ND	NC
Chloronaphthalene		ND	ND	NC	ND	ND	ND	ND	ND
Dinitrotoluene (other than 2,6)		NA	NA	NA	NA	NA	NA	NC	NC
Ethylmethylpyridine		NC	NC	NC	ND	NC	NC	NC	NC
1-Methylnaphthalene		ND	ND	ND	ND	ND	ND	ND	ND
Methylnitroaniline		NA	NA	NA	NA	NA	NA	NC	NC
Tetrahydromethylnaphthalene		ND	ND	NC	ND	ND	ND	ND	ND
Dihydrotetramethylnaphthalenone		NA	NA	NA	NA	NA	NA	ND	ND
Trimethylisocyanurate		ND	ND	ND	NC	NC	NC	ND	ND
(1-Butylhexyl)-Benzene		ND	NC	NC	ND	ND	ND	ND	ND
(1-Methylnonyl)-Benzene		ND	ND	NC	ND	ND	ND	ND	NC
(1-Butyloctyl)-Benzene		ND	NC	NC	NC	NC	NC	ND	ND
(1-Propylnonyl)-Benzene		ND	NC	NC	ND	ND	ND	ND	ND
(1-Ethyldecyl)-Benzene		ND	NC	NC	ND	ND	NC	ND	ND
(1-Methylundecyl)-Benzene		ND	NC	NC	ND	ND	ND	ND	NC

Chemical Name	Date (August)	Station 33 - Outfall 003			Station 34 - Outfall 004			Station 35 Outfall 005		
		→ 16	17	18	16	17	18	16	17	18
Aniline		ND	ND	ND	ND	ND	ND	ND	ND	ND
Biphenyl		ND	MS	6	<1	1	2	2	9	8
m-Dichlorobenzene ¹		MS	ND	MS	MS	1	<1	ND	ND	9
o- and p-Dichlorobenzenes ¹		1	MS	3	1	2	130	47	ND	120
3,4-Dihydro-1(2H)-Naphthalenone		<1	MS	95	ND	5	29	26	3	25
N,N-Dimethylaniline		ND	ND	<1	ND	ND	MS	ND	ND	10
2,6-Dinitrotoluene ¹		ND	ND	ND	ND	ND	ND	ND	950	ND
4-Ethylacetophenone		ND	ND	<1	ND	ND	ND	ND	ND	ND
2-Ethyl-1-Hexanol		ND	MS	ND	ND	ND	12	ND	ND	ND
Isophorone ¹		38	11	19	ND	ND	300	ND	ND	ND
N-Methylaniline		ND	ND	ND	ND	ND	ND	ND	ND	2
2-Methyl-4-Octanone		<1	ND	ND	ND	ND	ND	ND	ND	ND
2-Methylnaphthalene		ND	ND	ND	ND	<1	ND	ND	ND	MS
Naphthalene ¹		ND	2	ND	ND	1	7	35	6	71
2-Naphthalenol		ND	ND	ND	ND	ND	39	ND	ND	ND
p-Nitrotoluene		ND	ND	ND	ND	ND	ND	ND	62	ND
Phenyl Ether		<1	6	14	<1	1	5	4	20	14
alpha-Terpineol		ND	ND	ND	37	ND	31	ND	ND	ND
1,2,3,4-Tetrahydronaphthalene		ND	26	3	ND	49	8	28	14	130
1,2,3,4-Tetrahydronaphthalen-1-ol		ND	ND	ND	ND	ND	ND	ND	ND	ND
5,6,7,8-Tetrahydronaphthalen-1-ol		ND	ND	ND	ND	ND	MS	ND	ND	ND
Butyloctenal		ND	NC	NC	ND	ND	ND	ND	NC	ND
Chloronaphthalene		ND	ND	ND	ND	ND	NC	ND	ND	ND
Dinitrotoluene (other than 2,6)		ND	ND	ND	NC	NC	NC	ND	NC	ND

Table 7 (Cont'd.)
 NEUTRAL EXTRACTABLE ORGANICS SAMPLING DATA
 Concentration in µg/l (Gross)
 UNION CARBIDE INSTITUTE

Chemical Name	Date (August)	Station 33 - Outfall 003			Station 34 - Outfall 004			Station 35 Outfall 005		
		16	17	18	16	17	18	16	17	18
Ethylmethylpyridine		ND	ND	NC	NC	NC	NC	NC	NC	ND
1-Methylnaphthalene		ND	ND	ND	ND	NC	NC	ND	ND	NC
Methylnitroaniline		ND	ND	NC	ND	ND	ND	ND	NC	ND
Tetrahydromethylnaphthalene		ND	ND	ND	ND	NC	ND	NC	ND	NC
Dihydrotetramethylnaphthalenone		NC	NC	NC	NC	NC	NC	ND	ND	ND
Trimethylisocyanurate		ND	ND	ND	ND	ND	NC	NC	ND	NC
(1-Butylhexyl)-Benzene		ND	ND	ND	ND	NC	ND	ND	ND	ND
(1-Methylnonyl)-Benzene		ND	ND	ND	NC	NC	NC	ND	NC	ND
(1-Butyloctyl)-Benzene		ND	ND	ND	NC	NC	NC	ND	ND	ND
(1-Propylnonyl)-Benzene		ND	ND	ND	NC	NC	NC	ND	ND	ND
(1-Ethyldecyl)-Benzene		ND	ND	ND	NC	NC	NC	ND	ND	ND
(1-Methylundecyl)-Benzene		ND	ND	ND	NC	NC	NC	ND	ND	NC

Chemical Name	Date (August)	Station 36 - Outfall 008			Station 37 Water Intake	
		16	17	18	17	18
Aniline		ND	ND	ND	ND	ND
Biphenyl		ND	1	<1	<1	ND
m-Dichlorobenzene ¹		MS	MS	MS	<1	<1
o- and p-Dichlorobenzenes ¹		2	2	3	1	<1
3,4-Dihydro-1(2H)-Naphthalenone		6	11	4	<1	ND
N,N-Dimethylaniline		ND	ND	ND	ND	ND
2,6-Dinitrotoluene ¹		ND	ND	ND	ND	ND
4-Ethylacetophenone		ND	ND	ND	ND	ND
2-Ethyl-1-Hexanol		ND	ND	ND	ND	ND
Isophorone ¹		MS	9	3	<1	ND
N-Methylaniline		ND	ND	ND	ND	ND
2-Methyl-4-Octanone		ND	ND	ND	ND	ND
2-Methylnaphthalene		ND	<1	ND	ND	ND
Naphthalene ¹		4	30	3	2	ND
2-Naphthalenol		ND	87	ND	ND	ND
p-Nitrotoluene		ND	ND	ND	ND	ND
Phenyl Ether		ND	1	<1	<1	ND
alpha-Terpineol		ND	ND	ND	ND	ND
1,2,3,4-Tetrahydronaphthalene		6	95	4	25	<1
1,2,3,4-Tetrahydronaphthalen-1-ol		ND	ND	ND	ND	ND
5,6,7,8-Tetrahydronaphthalen-1-ol		ND	ND	ND	ND	ND
Butyloctenal		ND	ND	ND	ND	ND
Chloronaphthalene		ND	ND	ND	ND	ND
Dinitrotoluene (other than 2,6)		ND	ND	ND	ND	ND
Ethylmethylpyridine		ND	NC	NC	ND	ND
1-Methylnaphthalene		ND	ND	ND	ND	ND
Methylnitroaniline		ND	ND	ND	ND	ND
Tetrahydromethylnaphthalene		ND	NC	ND	NC	ND
Dihydrotetramethylnaphthalenone		ND	ND	NC	ND	ND
Trimethylisocyanurate		ND	ND	ND	ND	ND
(1-Butylhexyl)-Benzene		ND	ND	ND	ND	ND
(1-Methylnonyl)-Benzene		ND	ND	ND	ND	ND
(1-Butyloctyl)-Benzene		ND	ND	ND	ND	ND
(1-Propylnonyl)-Benzene		ND	ND	ND	ND	ND
(1-Ethyldecyl)-Benzene		ND	ND	ND	ND	ND
(1-Methylundecyl)-Benzene		ND	ND	ND	ND	ND

Chemical is a priority pollutant (NRCDVS Train Consent decree June 1976).

ND means not detected by computerized mass spectrometric data analysis.

3. NA means not specifically searched for during computerized mass spectrometric data analysis.

4. MS means the chemical was identified from its mass spectrum but interfering compounds or difficulties in correlation to the flame ionization chromatogram prevented quantitation.

"<" -less than- means the chemical was identified and quantitated but the result was below an estimated detection limit of 1 µg/l.

6. NC means the identification was not confirmed by analysis of pure standards on the same instrument.

Table 8
VOLATILE ORGANICS DATA
UNION CARBIDE INSTITUTE

(August 1978)	Influent to WWTF			WWTF Discharge Outfall 001			Cooling Water Outfall 002	Cooling Water Outfall 003	Cooling Water Outfall 004	Cooling Water Outfall 005				Cooling Water Outfall 008	Water Intake
	15 ^a	16 ^a	17 ^a	15 ^a	16 ^a	17 ^a	16 ^b	16 ^b	15-17 ^c	15 ^d	15 ^d	16 ^d	17 ^d	16 ^b	16 ^b
Compound ^f	concentration in µg/l (Gross)														
Acrolein	ND ^e	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzene	24	270	ND	ND	ND	ND	11	8	ND	ND	260	190	43	22	17
Bromodichloromethane	ND	ND	ND	ND	ND	ND	1	ND	ND	ND	ND	8	ND	11	ND
Bromoform	ND	ND	ND	ND	ND	ND	4	ND	ND	ND	ND	ND	ND	ND	ND
Carbon tetrachloride	24	72	ND	ND	ND	ND	2	2	ND	ND	5	42	2	2	1
Chlorobenzene	2	3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2-Chloroethylvinyl ether	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	66	350	ND	100	71	10	40	31	5	51	91	550	31	110	45
Chlorodibromomethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	2	ND	2	ND
1,2-Dichloroethane	1	2	ND	ND	ND	ND	5	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
trans-1,2-Dichloro- ethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloropropane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	130	510	ND	ND	ND	ND	ND	ND	ND	ND	8	30	3	ND	ND
Methylene chloride	ND	12	5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2,2-Tetrachloro- ethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	ND	250	170	ND	ND	ND	23	27	100	ND	910	350	230	80	49
1,1,1-Trichloroethane	ND	9	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl chloride	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

a Equal volume composite of three grab samples.

b Single grab, grab samples collected on August 15 and 17 not analyzed.

c Equal volume composite of 2 grab samples, one collected on Aug 15 and the other August 17.

d Single grab sample.

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

f All chemicals listed are priority pollutants.

Table 9
DIRECT AQUEOUS INJECTION ORGANIC DATA
UNION CARBIDE INSTITUTE

Station Description	Date ^a (August)	acetone	methylethyl- ketone	acryloni- trile	styrene	isopro- panol	diethyl ketone	isobutro- nitrile	n-butanol	1-chloro- butane	ethanol	4-methyl-2- pentene-2-one	cellosolve acetate
Influent to WWTF	16	ND ^d	ND	ND	ND	210 ^b	ND	ND	ND	ND	ND	100	ND
	17	15	ND	ND	ND	330	ND	ND	ND	ND	ND	36	ND
	18	17 ^c	ND	ND	ND	180 ^{b,c}	ND	ND	25 ^c	ND	ND	ND	ND
WWTF Discharge (Outfall 001)	16	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	17	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	18	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

a Equal volume composite of three grab samples.

b Value exceeded the known linear range for this parameter. A diluted aliquot of the sample was not analyzed.

c Value represents an average of two replicates.

d Not detected.

Table 10
SUMMARY OF FIELD MEASUREMENTS AND ANALYTICAL DATA
UNION CARBIDE INSTITUTE COOLING WATER OUTFALLS

Station No.	Station Description	Date (August)	Flow		Temperature Range °C	Range pH	TOC (mg/l)		TKN (mg/l)		NH ³ -N (mg/l)	
			m ³ /dayx10 ³	mgd			Gross	Net	Gross	Net	Gross	Net
32	Outfall 002	16	196	52	28-29	6.1-7.4	19	12	1.6	1.6	0.5	0.5
		17	171	45	28-29	5.9-7.3	20	15	1.4	1.0	0.8	0.8
		18	183	48	29-31	5.5-9.2	19	15	0.6	0.2	<0.2	0
		Avg.	183	48			19	14	1.2	0.9	0.4	0.4
33	Outfall 003	16	162	43	30-31	6.7-8.4	8	1	0.7	0.7	<0.2	0
		17	141	37	30-31	6.3-8.2	6	1	0.6	0.2	<0.2	0
		18	151	40	31-33	6.6-8.1	6	2	0.6	0.2	<0.2	0
		Avg.	151	40			7	1	0.6	0.4	<0.2	0
34	Outfall 004	16	20	5.4	29-32	7.2-7.8	7	0	1.1	1.1	<0.2	0
		17	18	4.7	28-31	6.6-7.5	12	7	0.9	0.5	<0.2	0
		18	19	5.0	30-36	7.0-7.8	4	0	0.4	0	<0.2	0
		Avg.	19	5.0			8	2	0.8	0.5	<0.2	0
35	Outfall 005	16	502	132	29-31	6.6-8.8	8	1	0.6	0.6	<0.2	0
		17	437	115	30-31	6.6-8.3	8	3	0.4	0	<0.2	0
		18	467	123	31-31	6.6-10.0	6	2	0.5	0.1	<0.2	0
		Avg.	468	123			7	2	0.5	0.2	<0.2	0
36	Outfall 008	16	4.4	1.2	35-49	4.4-7.8	6	0	0.4	0.4	<0.2	0
		17	3.8	1.0	38-41	6.2-7.8	6	1	0.3	0	<0.2	0
		18	4.1	1.1	32-47	6.6-7.8	8	4	0.4	0	<0.2	0
		Avg.	4.1	1.1			7	2	0.4	0.1	<0.2	0
37	Water Intake	16	883	233	25-30	6.5-7.7	7		<0.2		<0.2	
		17	769	203	25-27	6.7-7.5	5		0.4		<0.2	
		18	823	217	25-27	6.8-7.5	4		0.4		<0.2	
		Avg.	825	218			5		0.3		<0.2	

DMR data for October 1977 through March 1978 [Appendix A] show that this outfall exceeded NPDES permit maximum TOC limitations by 25% in December. In addition, UCI reported two pH violations.

Numerous organic compounds were observed in Outfall 002 neutral extractable analyses. Seven compounds [Table 7] were identified, confirmed and quantified with concentrations ranging from <1 to 710 µg/l. Two additional organic chemical (m-dichlorobenzene and 3,4-dihydro-1(2H)-naphthalenone) were identified and confirmed but interfering compounds or difficulty in chromatographic correlation prevented quantification. Five other chemical compounds were identified but could not be confirmed. Of the 9 organic chemicals confirmed 5 (m-dichlorobenzene, o- and p-dichlorobenzene, 2,6-dinitrotoluene, isophorone and naphthalene) are priority pollutants.

Volatile organic analyses [Table 8] showed that this discharge contained benzene (11 µg/l), bromodichloromethane (1 µg/l), bromoform (4 µg/l), carbon tetrachloride (2 µg/l), chloroform (40 µg/l), 1,2-dichloroethane (5 µg/l) and toluene (23 µg/l). Benzene and chloroform concentrations (17 and 45 µg/l), respectively, were higher in the intake water than in the discharge. This is probably due to recycling Outfall 005 wastewater back into the water intake (see discussion of Outfall 005). These volatile compounds are priority pollutants.

Outfall 003 (Station 33)

This outfall contains cooling water from the No. 1 Boilerhouse and from polyol, butanol, ethylbutyraldehyde, acetone, mixed ketones, crude naphthol, isophorone, methyl chloride and catalyst production.

The maximum net TOC and TKN concentrations in the cooling water discharged through Outfall 003 were 2 and 0.7 mg/l respectively, 17 and 47% of the NPDES permit limitations. Net NH₃ concentrations for

the three-day survey were zero. These data are lower than those reported in UCI self-monitoring data with monthly maximum values ranging from 3 to 21 mg/l TOC, 0.09 to 1.46 mg/l TKN and 0 to 0.2 mg/l NH_3 [Appendix A, Table 5].

Outfall 003 neutral extractable organics data [Table 7] show that this discharge contained biphenyl, o- and p-dichlorobenzene, 3,4-dihydro-1(2H)-naphthalenone, N,N-dimethylaniline, 4-ethylacetophenone, isophorone, 2-methyl-4-octanone, naphthalene, phenyl ether and 1,2,3,4-tetrahydronaphthalene at concentrations ranging from <1 to 95 $\mu\text{g/l}$. In addition, m-dichlorobenzene and 2-ethyl-1-hexanol were identified and confirmed but could not be quantified. Butyloc-tenal, methylethylpyridine, methylnitroaniline, and dihydrotetramethyl-naphthalenone were identified but could not be confirmed. Of these 16 organic compounds, 4 (m-dichlorobenzene, o- and p-dichlorobenzene, isophorone and naphthalene) are priority pollutants.

VOA data [Table 8] show that this outfall also contained benzene (8 $\mu\text{g/l}$), carbon tetrachloride (2 $\mu\text{g/l}$), chloroform (31 $\mu\text{g/l}$) and toluene (27 $\mu\text{g/l}$). All of these compounds are priority pollutants. Except for carbon tetrachloride, the concentrations of these compounds were higher in the intake water than in the discharge. The intake, however, is located downstream of Outfall 005. Outfall 005 concentrations were much higher than the intake indicating recycling of wastewater. (See discussion for Outfall 005.)

Outfall 004 (Station 34)

This outfall reportedly contains cooling water from olefins, cellosize, butanol and ethylbutyraldehyde production.

Sampling results [Table 10] show that this discharge meets NPDES limitations. The maximum net TOC concentration during the survey,

7 mg/l, was only 58% of the limitation (12 mg/l). The average net TKN concentration of 0.5 mg/l was the same as the permit limit. October 1977 through March 1978 DMR self-monitoring data [Appendix A, Table 6] show that TOC, TKN and NH_3 daily maximum limitations were violated 5, 1, and 1 months respectively.

Fourteen organic compounds were identified and confirmed by neutral extractable organic analysis. All except one were quantified with concentrations ranging from <1 to 300 $\mu\text{g/l}$. In addition, 13 other organic compounds were identified but could not be confirmed because pure standards were unavailable. Of these 27 compounds, m-dichlorobenzene, o- and p-dichlorobenzene, isophorone and naphthalene are priority pollutants.

Volatile organic samples contained chloroform and toluene at 5 and 100 $\mu\text{g/l}$ respectively. The intake water contained chloroform at a higher concentration (45 $\mu\text{g/l}$) than the discharge (5 $\mu\text{g/l}$). Again, this is probably due to the discharge from Outfall 005 which is located upstream of the water intake. (See Outfall 005 discussion.) These two compounds are also priority pollutants.

Outfall 005 (Station 35)

Cooling water from SEVIN[®] (including synthetic gas unit, tetralone production, 1-naphthol refining, phosgene processing, naphthychloroformate production, methylisocyanate production and SEVIN[®] processes), alkyl benzene, toluene diisocyanate, toluene diamine, POLYOX[®] UCARE[®] polymer Jr, and rigid polyols production is discharged into the Kanawha River through submerged Outfall 005.

During the survey, this discharge contained maximum net TOC, TKN and NH_3 concentrations of 3, 0.6 and 0 mg/l, respectively [Table 10]. DMR data October 1977 through March 1978 were higher with maximum

daily TOC, TKN and NH_3 NPDES permit limitations being violated 3, 1 and 2 months respectively.

Twenty-one organic compounds were identified in this discharge by neutral extractable organic analysis [Table 7]. Of the 21 compounds, 9 could not be confirmed and one, although confirmed, could not be quantified due to either interfering compounds or difficulties in correlation to the flame ionization chromatogram. The other eleven compounds were present in concentrations ranging from 2 to 950 $\mu\text{g/l}$. Four of the compounds (m-dichlorobenzene, o- and p-dichlorobenzene, 2,6-dinitrotoluene and naphthalene) are priority pollutants.

VOA analysis [Table 8] showed this discharge contained the priority pollutants benzene (43 to 260 $\mu\text{g/l}$), bromodichloromethane (8 $\mu\text{g/l}$), carbon tetrachloride (2 to 42 $\mu\text{g/l}$), chloroform (31 to 550 $\mu\text{g/l}$), chlorodibromomethane (2 $\mu\text{g/l}$), ethylbenzene (3 to 30 $\mu\text{g/l}$) and toluene (230 to 910 $\mu\text{g/l}$).

On August 15, Company officials reported a chloroform spill into Outfall 005 cooling water.* VOA data show that chloroform which was detected in the first sample at 51 $\mu\text{g/l}$, increased to 550 $\mu\text{g/l}$ and then decreased to 31 $\mu\text{g/l}$.

Outfall 005 discharges wastewater upstream of the water intake. The volatile organic data [Table 8] show that wastewater from this outfall is being recycled back into the cooling water. For example, 550 $\mu\text{g/l}$ of chloroform was discharged through this outfall August 16, 1978 which resulted in an intake water concentration of 45 $\mu\text{g/l}$. Toluene results are similar with discharge and intake concentrations of 350 and 49 $\mu\text{g/l}$, respectively.

* Company personnel did not identify the source of the spill.

Outfall 008 (Station 36)

Cooling water from Boilerhouse No. 2 is discharged through Outfall 008 into Goff Branch, a tributary to the Kanawha River. During the survey, TOC, TKN and NH_3 concentrations ranged from 6 to 8, 0.3 to 0.4 and $<0.2^*$ mg/l respectively. These concentrations are similar to those observed in the intake water (4 to 7 mg/l TOC, <0.2 to 0.4 mg/l TKN, and $<0.2^*$ mg/l NH_3). DMR data from October 1977 through March 1978 show significantly higher concentrations. Maximum TOC concentrations ranged from 3 to 101 mg/l, TKN from 0.2 to 1.85, and NH_3 0.04 to 0.27 mg/l. TOC permit limitations were in violation in February and March and TKN limitations in February [Appendix A].

Neutral extractable organic data [Table 7] show that the Outfall 008 discharge contained 9 compounds with concentrations ranging from <1 to 87 $\mu\text{g/l}$. Four additional compounds were identified. Of these 4, 1 was confirmed but could not be quantified. The other 3 could not be confirmed due to unavailability of pure standards. Four of the 9 neutral organic compounds are priority pollutants.

VOA data [Table 8] show that the cooling water discharged through Outfall 008 contained 6 compounds, all of which are priority pollutants. Concentrations ranged from 2 to 110 $\mu\text{g/l}$. All of these compounds were identified in the Outfall 005 discharge and four were also identified in the water intake. As this outfall only contains boilerhouse cooling water, the organics are probably a direct result of Outfall 005 wastewater being recycled back into the water intake.

On August 9, NEIC personnel observed a brownish, turbid discharge from Outfall 008. Company personnel immediately investigated the problem and determined that a control valve on the #1 hot line water softener malfunctioned, causing an overflow of lime mud. As the ash

* All values were the same.

system was temporarily out of service, the lime mud was discharged into the cooling water instead of the ash bin. Company personnel repaired the control valve and put the ash system back into service.

Water Intake (Station 37)

Approximately 720,000 m³/day (190 mgd) of Kanawha River water are used for cooling and process water at the UCI plant. Sample data [Table 10] showed that the intake contained small TOC and TKN concentrations (average 5 and 0.3 mg/l, respectively). The intake also contained 10 neutral extractable organic compounds ranging in concentrations from <1 to 25 µg/l [Table 7]. VOA analyses identified 4 compounds with concentrations of 1 to 49 µg/l. Of these 14 organic compounds identified by neutral organic and VOA analyses, 8 are priority pollutants.

As previously noted, the water intake is downstream from the Outfall 005 discharge. VOA data [Table 8] show that Outfall 005 wastewater is being recycled back into the cooling water through the water intake. Outfall 005 wastewater contained very high concentrations of benzene, chloroform and toluene (260, 550 and 910 µg/l, respectively). Water intake concentrations for these compounds were also high (17 µg/l benzene, 45 µg/l chloroform and 49 µg/l toluene) undoubtedly due to recycling of Outfall 005 wastewater. The intake concentrations of these compounds were higher than those observed in cooling water Outfalls 002, 003 and 004.

TOXICITY EVALUATION

A total of 53 organic compounds were identified in the UCI samples. Fifteen of these 53 compounds could not be confirmed by GC/MS. The 53 compounds were searched in the Registry of Toxic Effects of Chemical

Substances (RTECS)* and the Toxline** database to obtain health effects data [Appendix H].

THE RTECS search yielded toxicity information on 34 of the 53 compounds. The Toxline search located 686 references to human health effects from 35 of 38 confirmed compounds. Information on each compound is summarized in Table 11. Seventeen of the 34 compounds identified in RTECS are listed as priority pollutants.

Of the 53 organic compounds, 49 were being discharged to the Kanawha River in concentrations ranging from <1 to 140,000 µg/l. The other four compounds were identified in the influent to the WWTF. The information presented in Table 11 shows that 23 compounds have demonstrated human effects associated with them. The hazards of injecting 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, 4 of the compounds are known carcinogens, benzene to humans, and carbaryl, carbon tetrachloride and chloroform to animals.

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

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

Table 11
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b					Exposure Limits ^c
				Route of Entry	Species	Type or Dose	Duration	Effects ^e	
Acetone	C ₃ H ₆ O	67-64-1	TLm 96: Over 1000 ppm	Oral-human		LDLo: 50 mg/kg			OSHA std (air): TWA 1000 ppm
				Inhalation-human		TCLo: 500 ppm			
				Inhalation-man		TCLo: 12,000 ppm	4H	Eye Central Nerv. System	
				Oral-rat		LD50: 9,750 mg/kg			
				Inhalation-rat		LCLo: 64,000 ppm	4H		
				Inhalation-mouse		LCLo: 110,000 mg/m ³	62M		
				Intraperitoneal-mouse		LD50: 1,297 mg/kg			
				Oral-dog		LDLo: 24 g/kg			
				Intraperitoneal-dog		LDLo: 8 g/kg			
				Subcutaneous-dog		LDLo: 5 g/kg			
alpha-Terpineol	C ₁₀ H ₁₈ O	98-55-5		Intramuscular-mouse		LD50: 2,000 mg/kg			
Aniline	C ₆ H ₇ N	62-53-3	TLm96: 100-10 ppm	Oral-human		LDLo: 50 mg/kg			OSHA std (air): TWA 5 ppm (skin)
				Unknown-human		LDLo: 357 mg/kg			
				Oral-rat		LD50: 440 mg/kg			
				Inhalation-rat		LCLo: 250 ppm	4H		
				Skin-rat		LD50: 1,400 mg/kg			
				Intraperitoneal-rat		LD50: 1,400 mg/kg			
				Oral-mouse		LD50: 464 mg/kg			
				Intraperitoneal-mouse		LD50: 492 mg/kg			
				Subcutaneous-mouse		LDLo: 480 mg/kg			
				Unknown-mouse		LD50: 572 mg/kg			
				Oral-cat		LDLo: 1,750 mg/kg			
				Inhalation-cat		LCLo: 180 ppm	8H		
				Skin-cat		LD50: 254 mg/kg			
				Intraperitoneal-rabbit		LDLo: 200 mg/kg			
				Subcutaneous-rabbit		LDLo: 1,000 mg/kg			
				Skin-rabbit		LD50: 820 mg/kg			
				Skin-guinea pig		LDLo: 1,750 mg/kg			
				Skin-guinea pig		LD50: 1,290 mg/kg			

Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b					Exposure Limits ^c
				Route of Entry	Species	Type of Dose	Duration	Effects ^e	
Aniline,N,N-dimethyl-	C ₈ H ₁₁ N	121-69-7		Oral-human Oral-rat Skin-rabbit		LDLo: 50 mg/kg LDLo: 1,410 mg/kg LD50: 1,770 mg/kg			OSHA std (air): TWA 5ppm (skin)
Aniline,N-methyl-	C ₇ H ₉ N	100-61-8		Intravenous-cat Oral-rabbit Intravenous-rabbit Oral-guinea pig Subcutaneous-guinea pig		LDLo: 24 mg/kg LDLo: 280 mg/kg LDLo: 24 mg/kg LDLo: 1,200 mg/kg LDLo: 1,200 mg/kg			OSHA std (air): TWA 2ppm (skin)
Benzene	C ₆ H ₆	71-43-2 ^d	TLm96:100-10ppm	Oral-human		LDLo:50 mg/kg			OSHA std (air): TWA 10 ppm; C1 25 Pk 50/10M/8H
				Inhalation-human		LDLo:20,000 ppm	5M		
				Inhalation-human		TCLo:210 ppm			
				Inhalation-man		TCLo:2,100 mg/m ³	4YI	Blood Carcino-genic	
				Oral-rat		LD50:3,800 mg/kg			
				Inhalation-rat		LC50:10,000 ppm	7H		
				Intraperitoneal-rat		LDLo:1,150 mg/kg			
				Oral-mouse		LD50:4,700 mg/kg			
				Inhalation-mouse		LC50:9,980 ppm			
				Skin-mouse		TDLo:1,200 gm/kg	49WI	Neoplas-tic	
				Intraperitoneal-mouse		LD50:468 mg/kg			
				Subcutaneous-mouse		TDLo:2,700 mg/kg	13D (preg)	Terato-genic	
				Oral-dog		LDLo:2,000 mg/kg			
				Inhalation-dog		LCLo:146,000 mg/m ³			
Benzene, Chloro-	C ₆ H ₅ Cl	108-90-7 ^d	TLm96:100-1ppm	Inhalation-cat		LCLo:170,000 mg/m ³			
				Intraperitoneal-guinea pig		LDLo:527 mg/kg			
				Subcutaneous-frog		LDLo:1,400 mg/kg			
				Inhalation-mammal		LCLo:20,000 ppm	5M		
Benzene, o-dichloro-	C ₆ H ₄ Cl ₂	95-50-1 ^d		Oral-rat		LD50:2,910 mg/kg			OSHA std (air): TWA 75 ppm
				Subcutaneous-rat		LDLo:4,000 mg/kg			
				Oral-rabbit		LD50:2,830 mg/kg			
				Intraperitoneal-rat		LDLo:7,400 mg/kg			
				Intraperitoneal-guinea pig		LDLo:4,100 mg/kg			
Benzene, o-dichloro-	C ₆ H ₄ Cl ₂	95-50-1 ^d		Oral-human		LDLo: 500 mg/kg			TLV (air): 50 ppm
				Oral-rat		LD50: 500 mg/kg			
				Inhalation-rat		LCLo: 821 ppm	7H		OSHA std (air): C1 50 ppm
				Intravenous-mouse		LDLo: 400 mg/kg			
				Oral-rabbit		LD50: 500 mg/kg			
				Intravenous-rabbit		LDLo: 250 mg/kg			
				Oral-guinea pig		LDLo: 2,000 mg/kg			
				Inhalation-guinea pig		LCLo: 800 ppm	24H		

Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b					Exposure Limits ^c
				Route of Entry	Species	Type of Dose	Duration	Effects ^e	
Benzene,p-dichloro-	C ₆ H ₄ Cl ₂	106-46-7 ^d		Oral-human		LDLo: 500 mg/kg		Systemic	TLV (air): 75 ppm OSHA std (air): TWA 75 ppm
				Oral-human		TDLo: 300 mg/kg			
				Oral-rat		LD50: 500 mg/kg			
				Intraperitoneal-rat		LD50: 2,562 mg/kg			
				Oral-mouse		LD50: 2,950 mg/kg			
				Oral-guinea pig		LDLo: 2,800 mg/kg			
Benzene, Ethyl-	C ₈ H ₁₀	100-41-4 ^d	TLm96:100-10 ppm	Inhalation-human		TCLo:100 ppm	8H	Irritant	OSHA std (air): TWA 100 ppm (skin)
				Oral-rat		LD50:3,500 mg/kg			
				Inhalation-rat		LCLo:4,000 ppm	4H		
				Skin-rabbit		LD50:5,000 mg/kg			
				Inhalation-guinea pig		LCLo:10,000 ppm			
Biphenyl	C ₁₂ H ₁₀	92-52-4		Inhalation-human		TDLo 4,400 µg/m ³		Irritant	TLV (air): 0.2 ppm
				Oral-rat		LD50: 3,280 mg/kg		Neoplastic	OSHA std (air): TWA 0.2 ppm
				Subcutaneous-mouse		TDLo: 46 mg/kg			
				Oral-rabbit		LD50: 2,410 mg/kg			
Butyl alcohol (n-butanol)	C ₄ H ₁₀ O	71-36-3	TLm96:over 1,000 ppm	Oral-human		LDLo: 500 mg/kg		Irritant	TLV(air):50 ppm (skin) OSHA std (air): TWA 100 ppm
				Inhalation-human		TCLo: 25 ppm			
				Oral-rat		LD50: 790 mg/kg			
				Intraperitoneal-rat		LDLo: 970 mg/kg			
				Oral-mouse		LDLo: 3,000 mg/kg			
				Oral-rabbit		LDLo: 4,250 mg/kg			
				Skin-rabbit		LD50: 4,200 mg/kg			
Carbaryl	C ₁₂ H ₁₁ O ₂ N	63-25-2	TLm96:10-1ppm	Oral-man		TDLo: 2,800 µg/kg		Central Nerv. Syst.	OSHA std (air): TWA 5 mg/m ³
				Oral-human		LDLo: 50 mg/kg		95 WI (9 or 100 preg)	NIOSH recm std (air): TWA 5 mg/m ³
				Oral-rat		LD50: 400 mg/kg			
				Oral-rat		TDLo: 5,700 mg/kg			
				Inhalation-rat		LC50: 721 mg/kg			
				Oral-rat		TDLo: 50 mg/kg			
				Intraperitoneal-rat		LD50: 48 mg/kg		Carcinogenic	
				Implant-rat		TDLo: 80 mg/kg			
				Unknown-rat		LD50: 500 mg/kg			
				Oral-mouse		LD50: 438 mg/kg			
				Intraperitoneal-mouse		LD50: 396 mg/kg			

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Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b				Exposure Limits
				Route of Entry	Species	Type of Dose	Duration	Effects ^e
Carbon tetrachloride CCl ₄ (Tetrachloromethane)	56-23-5 ^d	TLM96:100-10 ppm		Oral-dog		TDLo: 388 mg/kg	(preg)	Teratogenic
				Oral-rabbit		LD50: 710 mg/kg		
				Oral-guinea pig		LD50: 280 mg/kg		
				Oral-guinea pig		TDLo: 300 mg/kg	(preg)	Teratogenic
				Oral-hamster		LDLo: 250 mg/kg		
				Oral-chicken		LD50: 197 mg/kg		
				Oral-wild bird		LD50: 56 mg/kg		
				Oral-human		LDLo: 43 mg/kg		
				Oral-woman		TDLo: 1,800 mg/kg		Systemic
				Inhalation-human		TCLo: 20 ppm		Central Nervous System
				Oral-woman		TDLo: 1,800 mg/kg		Pulmonary
				Oral-man		TDLo: 1,700 mg/kg		Central Nervous System
				Inhalation-human		LCLo: 1,000 ppm		
				Oral-rat		LD50: 2,800 mg/kg		
				Inhalation-rat		LCLo: 4,000 ppm	4H	
				Inhalation-rat		TCLo: 300 ppm	(6-15D preg)	Teratogenic
				Intraperitoneal-rat		LD50: 1,500 mg/kg		
Chloroform CHCl ₃ (Trichloromethane)	67-66-3 ^d	TLM96:100-10 ppm		Subcutaneous-rat		TDLo: 133 mg/kg	25 WI	Neoplastic
				Oral-mouse		LD50: 12,800 mg/kg		
				Oral-mouse		TDLo: 4,800 mg/kg	88DI	Carcinogenic
				Inhalation-mouse		LC50: 9,526 ppm	8H	
				Intraperitoneal-mouse		LD50: 4,675 mg/kg		
				Subcutaneous-mouse		LDLo: 12 gm/kg		
				Oral-human		LDLo: 140 mg/kg		
				Inhalation-human		TDLo: 1,000 mg/m ³	1Y	Systemic
				Inhalation-human		TCLo: 5,000 mg/m ³	7M	Central Nervous System
				Oral-rat		LD50: 800 mg/kg		
				Oral-rat		TDLo: 70 gm/kg	78WI	Neoplastic
				Inhalation-rat		LCLo: 8,000 ppm	4H	
				Inhalation-rat		TCLo: 100 ppm	7H (6-15 D preg)	Teratogenic

OSHA std (air):
TWA 50 ppm

NIOSH recm std
(air): Cl 2 ppm/60M

Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b					Exposure Limits ^c
				Route of Entry	Species	Type of Dose	Duration	Effects ^e	
2-cyclohexen-1-one, 3,5,5-trimethyl- (isophorone)	C ₉ H ₁₄ O	78-59-1 ^d		Oral-mouse		LDLo:2,400 mg/kg	120DI	Carcinogenic	
				Oral-mouse		TDLo:18 gm/kg			
				Inhalation-mouse		LC50:28 gm/m ³			
				Intraperitoneal-mouse		LD50:1,671 mg/kg	4H		
				Subcutaneous-mouse		LD50:704 mg/kg			
				Oral-dog		LDLo:1,000 mg/kg			
				Inhalation-dog		LC50:100 gm/m ³			
				Intraperitoneal-dog		LD50:1,000 mg/kg			
				Intravenous-dog		LDLo:75 mg/kg			
				Inhalation-cat		LCLo:35,000 mg/m ³			
				Oral-rabbit		LDLo:500 mg/kg			
				Inhalation-rabbit		LC50:59 gm/m ³			
				Subcutaneous-rabbit		LDLo:3,000 mg/kg			
				Inhalation-guinea pig		LCLo:20,000 ppm	2H		
				Inhalation-frog		LCLo:6,000 mg/m ³	5M		
				Inhalation-mammal		LCLo:25,000 ppm			
				Inhalation-human		TCLo:25 ppm	4H	Irritant	OSHA std (air): TWA 25 ppm
				Oral-rat		LD50:2,330 mg/kg			
				Inhalation-rat		LDLo:1,840 ppm			
				Skin-rabbit		LD50:1,500 mg/kg			
Ethane, 1,2-Dichloro- (Ethylene Dichloride)	C ₂ H ₄ Cl ₂	107-06-2 ^d	TLm96:1,000-100 ppm	Inhalation-human		TCLo:4,000 ppm	H	Central Nervous System	OSHA std (air): TWA 50 ppm Cl 100; Pk 200/5M/3H
				Oral-human		TDLo:428 mg/kg	4H		
				Oral-man		LDLo:810 mg/kg			
				Oral-human		LDLo:500 mg/kg			
				Oral-rat		LD50:680 mg/kg			
				Inhalation-rat		LCLo:1,000 ppm			
				Intraperitoneal-rat		LDLo:600 mg/kg			
				Subcutaneous-rat		LDLo:500 mg/kg			
				Oral-mouse		LDLo:600 mg/kg	2H	NIOSH recm std (air): TWA 5 ppm; Cl 15	
				Inhalation-mouse		LCLo:5,000 mg/m ³			
				Intraperitoneal-mouse		LDLo:250 mg/kg			
				Subcutaneous-mouse		LDLo:380 mg/kg			
				Oral-dog		LDLo:2,000 mg/kg			

Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b				Exposure Limits ^c
				Route of Entry	Species	Type of Dose	Duration	Effects ^e
Ethane, 1,1,1-Trichloro- (Methyl Chloroform)	C ₂ H ₃ Cl ₃	71-55-6 ^d	TLM96:100-10 ppm	Intravenous-dog		LDLo:175 mg/kg		
				Oral-rabbit		LD50:860 mg/kg		
				Inhalation-rabbit		LCLo:3,000 ppm	7H	
				Subcutaneous-rabbit		LDLo:1,200 mg/kg		
				Inhalation-pig		LCLo:3,000 ppm	7H	
				Inhalation-guinea pig		LCLo:1,500 ppm	7H	
				Intraperitoneal-guinea pig		LDLo:600 mg/kg		
				Oral-human		LDLo:500 mg/kg		
				Inhalation-man		LCLo:27 gm/m ³	10M	
				Inhalation-man		TCLo:350 ppm		Psychotropic Central Nervous System
				Inhalation-human		TCLo:920 ppm	70M	
				Oral-rat		LD50:14,300 mg/kg		
				Inhalation-rat		LCLo:1,000 ppm		
				Inhalation-mouse		LCLo:11,000 ppm	2H	
				Intraperitoneal-mouse		LD50:4,700 mg/kg		
Ether, diphenyl (phenylether)	C ₁₂ H ₁₀ O	101-84-8		Oral-dog		LD50:750 mg/kg		
				Intraperitoneal-dog		LD50:3,100 mg/kg		
				Intravenous-dog		LDLo:95 mg/kg		
				Oral-rabbit		LD50:5,660 mg/kg		
				Subcutaneous-rabbit		LD50:500 mg/kg		
1-hexanol, 2-ethyl-	C ₈ H ₁₈ O	104-76-7		Oral-guinea pig		LD50:9,470 mg/kg		
				Oral-rat		LD50:3,370 mg/kg		
								TLV (air): 1 ppm (vapor) OSHA std (air): TWA 1 ppm
				Oral-mouse		LDLo:3,200 mg/kg		
				Skin-rabbit		LD50:2,380 mg/kg		

Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b					Exposure Limits ^c
				Route of Entry	Species	Type of Dose	Duration	Effects ^e	
Isopropyl Alcohol (Isopropanol)	C ₃ H ₈ O	67-63-0	Tlm 96:1000-100 ppm	Inhalation-human Oral-rat Oral-mouse Intraperitoneal-mouse Subcutaneous-mouse Oral-dog Intravenous-dog Intravenous-cat Oral-rabbit Skin-rabbit Intravenous-rabbit Subcutaneous-mammal		TCLo:400 ppm LD50:5,840 mg/kg LDLo:192 mg/kg LD50:933 mg/kg LDLo:6,000 mg/kg LD50:6,150 mg/kg LDLo:5,120 mg/kg LDLo:1,963 mg/kg LDLo:5,000 mg/kg LD50:16 mg/kg LDLo:8,230 mg/kg LDLo:6 mg/kg		Irritant	TLV (air): 400 ppm (skin) OSHA std (air): TWA 400 ppm
Methane, Dichloro- (Methylene Chloride)	CH ₂ Cl ₂	75-09-2 ^d	Tlm96:1,000-100 ppm	Inhalation-human		TCLo:500 ppm	1YI	Central Nervous System	OSHA std (air): TWA 500 ppm; C1 1,000; Pk 2,000/5M/2H NIOSH recm std (air): TWA 75 ppm; Pk 50/15M
				Oral-human		LDLo:500 mg/kg			
				Inhalation-human		TCLo:500 ppm	8H	Blood	
				Oral-rat		LD50:945 mg/kg			
				Inhalation-mouse		LC50:14,400 ppm	7H		
				Intraperitoneal-mouse		LD50:1,500 mg/kg			
				Subcutaneous-mouse		LD50:6,460 mg/kg			
				Oral-dog		LDLo:3,000 mg/kg			
				Inhalation-dog		LCLo:20,000 ppm	7H		
				Intraperitoneal-dog		LDLo:950 mg/kg			
				Subcutaneous-dog		LDLo:2,700 mg/kg			
				Intravenous-dog		LDLo:200 mg/kg			
				Oral-rabbit		LDLo:1,900 mg/kg			
				Subcutaneous-rabbit		LDLo:2,700 mg/kg			
				Inhalation-guinea pig		LCLo:5,000 ppm	2H		
Methane, Tribromo- (Bromoform)	CHBr ₃	75-25-2 ^d		Subcutaneous-mouse Subcutaneous-rabbit		LD50:1,820 mg/kg LDLo:410 mg/kg			OSHA std (air): TWA 0.5 ppm (skin)
Naphthalene	C ₁₀ H ₈	91-20-3 ^d	Tlm96:10-1 ppm	Oral-child		LDLo:100 mg/kg			OSHA std (air): TWA 10 ppm
				Oral-human		LDLo:50 mg/kg			
				Oral-rat		LD50:1,780 mg/kg			
				Subcutaneous-rat		TDLo:3,500 mg/kg	98DI	Neoplastic	
				Intraperitoneal-mouse		LDLo:150 mg/kg			
Naphthalene, 1-methyl-	C ₁₁ H ₁₀	1321-94-4		Oral-rat		LDLo:5,000 mg/kg			
Naphthalene, 2-methyl-	C ₁₁ H ₁₀	91-57-6		Oral-rat		LDLo:5,000 mg/kg			

Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity ^a	Other Toxicity Data ^b					Exposure Limits ^c
				Route of Entry	Species	Type of Dose	Duration	Effects ^e	
Naphthalene, 1,2,3,4-tetrahydro-	C ₁₀ H ₁₂	119-64-2	TLm96:100-10 ppm	Oral-human Oral-rat Skin-rabbit Inhalation-guinea pig		LDLo:500 mg/kg LD50:2,860 mg/kg LD50:17 gm/kg LCLo:275 ppm	8H/17D		
2-naphthalenol (2-naphthol)	C ₁₀ H ₈ O	135-19-3		Oral-human Subcutaneous-rat Oral-rat Subcutaneous-mouse Oral-rabbit Subcutaneous-guinea pig		LDLo:50 mg/kg LDLo:2,940 mg/kg LD50:2,420 mg/kg LDLo:100 mg/kg LDLo:3,800 mg/kg LDLo:2,670 mg/kg			
1(2H)-Naphthalenone, 3,4-Dihydro-	C ₁₀ H ₁₀ O	529-34-0		Oral-rat		LD50:810 mg/kg			
3-penten-2-one, 4-methyl-	C ₆ H ₁₀ O	141-79-7	TLm96:100-10 ppm	Inhalation-human Oral-rat		TCLo:25 ppm LD50:1,120 mg/kg		Irritant	TLV (air): 25 ppm
(4-methyl-3-pentene-2-one)				Inhalation-rat Intraperitoneal-mouse		LCLo:1,000 ppm LD50:354 mg/kg	4H		OSHA std (air): TWA 25 ppm
				Oral-rabbit Skin-rabbit Subcutaneous-rabbit Subcutaneous-frog		LD50:1,000 mg/kg LD50:5,990 mg/kg LDLo:840 mg/kg LDLo:1,440 mg/kg			
Toluene	C ₇ H ₈	108-88-3 ^d	TLm96:100-10 ppm	Oral-human Inhalation-human		LDLo:50 mg/kg TCLo:200 ppm		Central Nervous System Psychotropic	OSHA std (air): TWA 200 ppm; Cl 300; Pk 500/10M NIOSH recm std (air) TWA 100 ppm; Cl 200/10M
				Inhalation-man		TCLo:100 ppm			
				Oral-rat Inhalation-rat Intraperitoneal-rat		LD50:5,000 mg/kg LCLo:4,000 ppm LDLo:800 mg/kg	4H		
				Inhalation-mouse Skin-rabbit Subcutaneous-frog		LC50:5,320 ppm LD50:14 gm/kg LDLo:920 mg/kg	8H		
Toluene, dinitro-	C ₇ H ₆ NO ₄			Oral-human		LDLo:50 mg/kg			TLV (air): 1.5 mg/m (skin) ⁵
Toluene, 2,6-dinitro-	C ₇ H ₆ N ₂ O ₄	606-20-2 ^d	TLm96:100-10 ppm	Oral-rat Oral-mouse		LD50:177 mg/kg LD50:1,000 mg/kg			OSHA std (air): TWA 1500 µg/m ³ (skin)
Toluene, p-nitro-	C ₇ H ₇ NO ₂	99-99-0		Oral-rat Skin-rat Intraperitoneal-rat Oral-mouse		LD50:2,144 mg/kg LD50:16,000 mg/kg LD50:940 mg/kg LD50:1,231 mg/kg			OSHA std (air): TWA 5 ppm (skin)

Table 11 (Cont'd.)
TOXICITY OF ORGANIC COMPOUNDS
UNION CARBIDE INSTITUTE

Abbreviations
(per Registry of Toxic Effects of Chemical
Substances - NIOSH - 1977 Edition)

-
- a Aquatic Toxicity: TLm96 - 96-hour static or continuous flow standard protocol, in parts per million (ppm).
- b Other Toxicity Data: LD50 - lethal dose 50% kill
 LDLo - lowest published lethal concentration
 LC50 - lethal concentration 50% kill
 LDLo - lowest published lethal dose
 TDLo - lowest published toxic dose
 TCLo - lowest published toxic concentration
 TD - toxic dose
 M - minute; H-hour; D-day; W-week; Y-year
 C - continuous
 I - intermittent
- c Exposure Limits: NR - not reported
 NIOSH - National Institute for Occupational Safety and Health
 OSHA - Occupational Safety and Health Act of 1970
 TWA - time-weighted average concentration
 TLV - threshold limit value
 Cl - ceiling
 Pk - peak concentration
- d This chemical has been selected for priority attention as point source water-effluent discharge toxic pollutant (NRDC vs Train consent decree).
- e Blood - Blood effects; effect on all blood elements, electrolytes, pH, protein, oxygen carrying or releasing capacity.
 Carcinogenic - Carcinogenic effects; producing cancer, a cellular tumor the nature of which is fatal, or is associated with the formation of secondary tumors (metastasis).
 Central Nervous System - Includes effects such as headaches, tremor, drowsiness, convulsions, hypnosis, anesthesia.
 Eye - Irritation, diplopia, cataracts, eye ground, blindness by affecting the eye or the optic nerve.
 Irritant - Any irritant effect on the skin, eye or mucous membrane.
 Neoplastic - The production of tumors not clearly defined as carcinogenic.
 Psychotropic - Exerting an effect upon the mind.
 Pulmonary - Effects on respiration and respiratory pathology.
 Systemic - Effects on the metabolic and excretory function of the liver or kidneys.
 Teratogenic - Nontransmissible changes produced in the offspring.

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APPENDIX A

UNION CARBIDE INSTITUTE
(INSTITUTE)

April 1978

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ATTACHMENTS

- A Organic Chemicals Identification
- B Goff Mountain Chemical Landfill Information
- C Bioassay Evaluation
- D Analytical Procedures Evaluation

I. INTRODUCTON

Union Carbide Corporation, Chemicals and Plastics Division (NPDES Permit No. WV0000086), has a chemical manufacturing facility located at Institute, West Virginia. The plant operates 24 hr/day, year around and employs 1,800 people.

The Environmental Protection Agency, Region III requested that the National Enforcement Investigations Center (NEIC) inspect the Union Carbide Institute 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.

On April 7, 10 and 11, 1978, Dr. Wayne Smith, Mr. James L. Hatheway, Mr. Bruce A. Binkley and Mr. D. David Vietti of NEIC visited the plant to inspect process operations, pollution abatement practices and self-monitoring techniques which include the sampling, flow measurement, analytical and bioassay procedures used. The Company, represented by Mr. R.L. Foster, Mr. John Huddleston and Ms. Jan Ketcham, provided information and assistance.

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

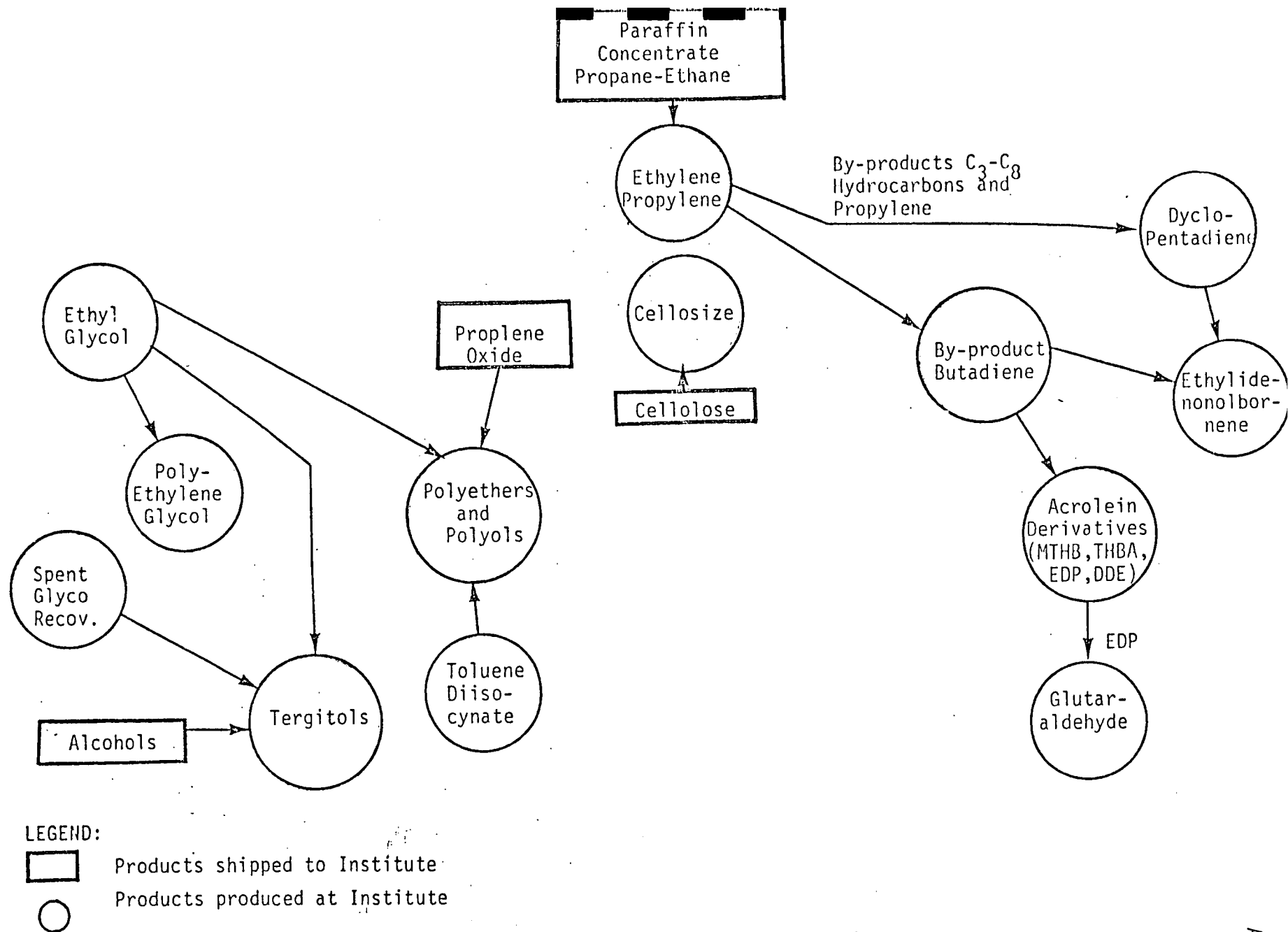


Figure 1. Flow of Materials at Institute

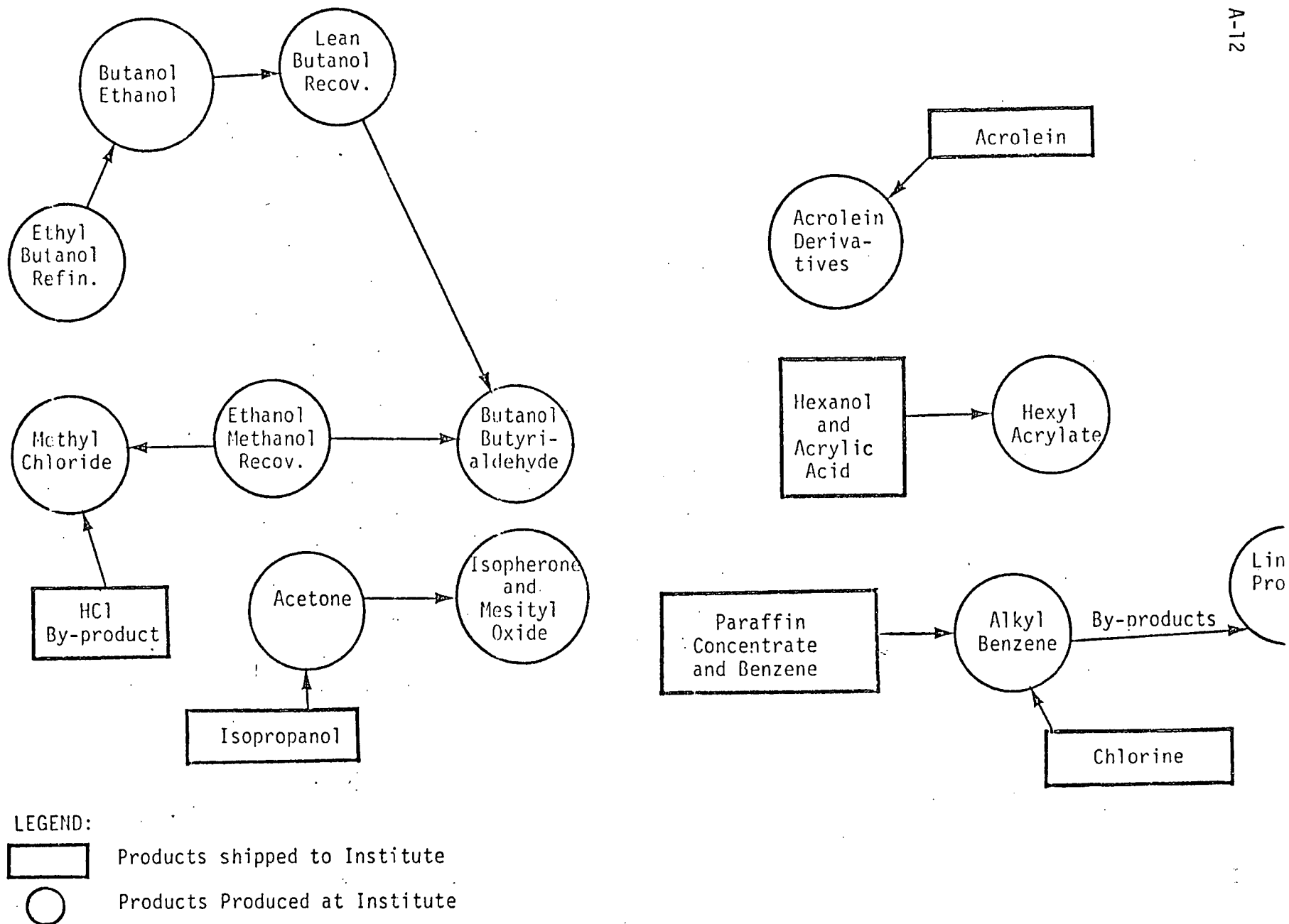


Figure 2. Flow of Materials at Institute

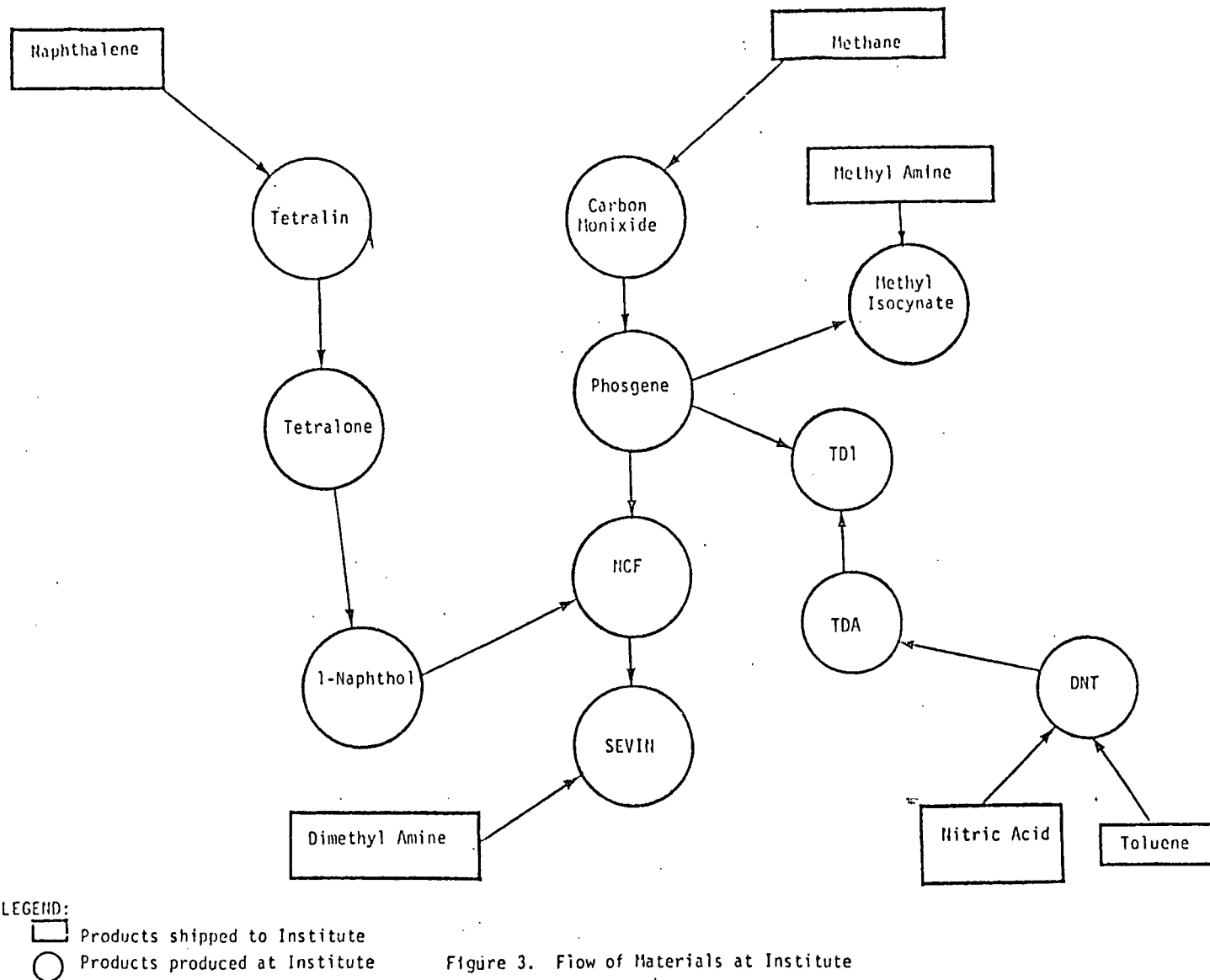


Figure 3. Flow of Materials at Institute

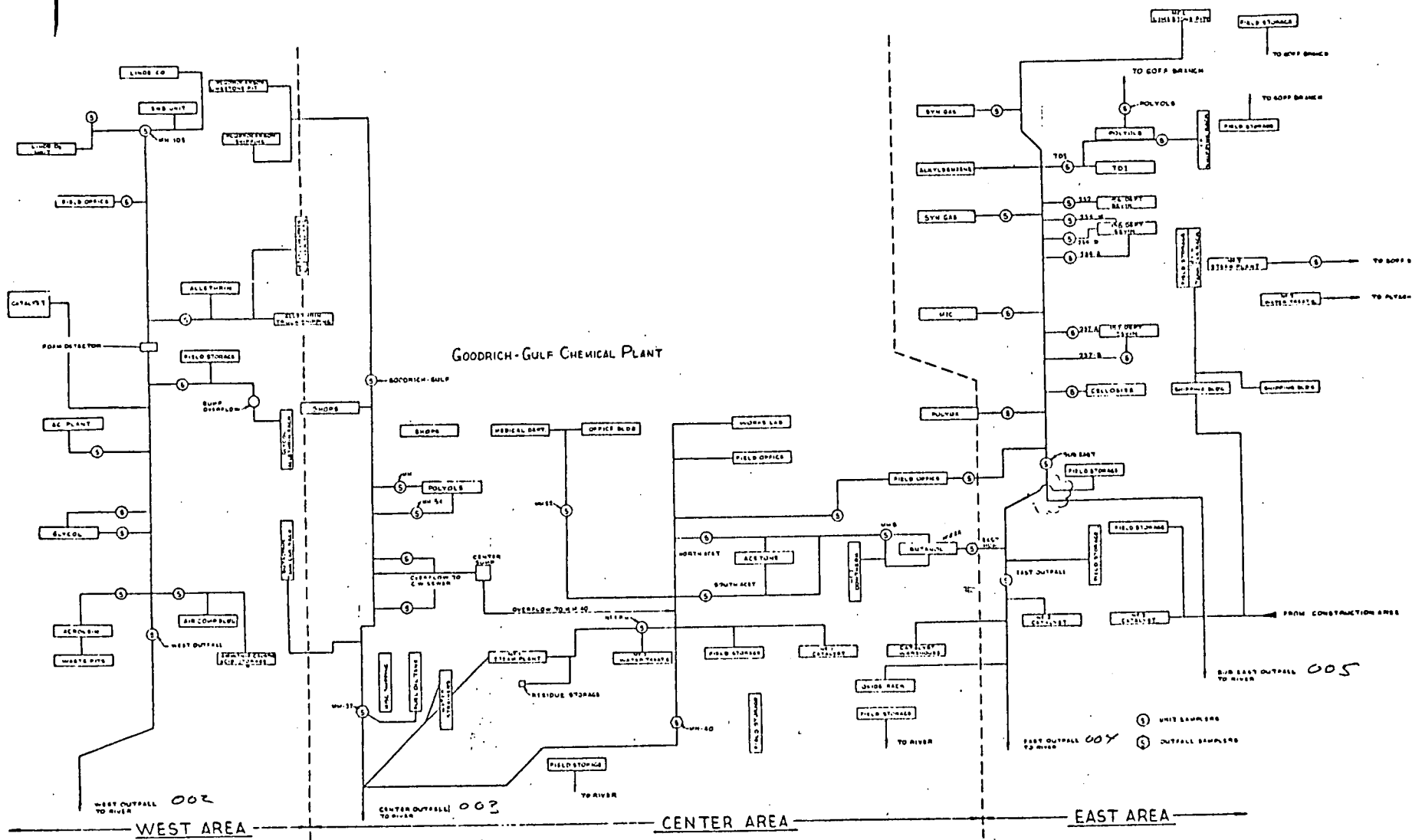


Figure 5. Plant Cooling Water Sewer System

Table 2
NPDES PERMIT LIMITATIONS
OUTFALL 002
UNION CARBIDE COMPANY
INSTITUTE, 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)	N/A				Continuous	Recorded
BOD ₅						
May-Oct.	2,270	(5,000)	5,000	(11,000)	3/week	24 hr. com. ^a
Nov.-Apr.	3,860	(8,500)	8,600	(19,000)	3/week	24 hr. com.
TSS	1,270	(2,800)	1,910	(4,200)	1/day	24 hr. com.
TOC						
May-Oct.	4,540	(10,000)	10,000	(22,000)	1/day	24 hr. com.
Nov.-Apr.	7,720	(17,000)	17,250	(38,000)	1/day	24 hr. com.
TKN	1,500	(3,300)	3,000	(6,600)	3/week	24 hr. com.
NH ₃ N	680	(1,500)	1,360	(3,000)	3/week	24 hr. com.
Chlorides	104,000	(228,000)	136,000	(300,000)	1/week	24 hr. com.
Fecal Coliform	200/100 ml		400/100/ml		1/month	Grab
Threshold Odor No.					1/quarter	24 hr. com.
Temperature			43.3°C(110°F)		1/day	Instantaneous
pH (range)			6.0-9.0		Continuous	Recorded
Other	There shall be no discharge of floating solids or visible foam in other than trace amounts.					

^a Composite

Table 3
NPDES PERMIT LIMITATIONS
OUTFALLS 002, 003, 004, 005, 008
UNION CARBIDE COMPANY
INSTITUTE, 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)		N/A		a
TOC	4 mg/l	12 mg/l	5/week	a
TKN	0.5 mg/l	1.5 mg/l	5/week	a
NH ₃ N	0.2 mg/l	0.6 mg/l	5/week	a
Chlorides	37,000 (302,000)	182,000 (400,000)	1/week	24 hr. com. ^b
Phenolics	32 (70)	82 (180)	1/week	24 hr. com.
Threshold Odor No.	128	256	1/quarter	24 hr. com.
Temperature		43.3°C(110°F)	b	Instantaneous
Sulfide (Outfall 005 only)		N/A	1/week	Grab
pH	The pH shall not be less than 6.0 standard units not greater than 9.0 ^c standard units and shall be monitored once/day, utilizing grab samples. There shall be no discharge of floating solids or visible foam in other than trace amounts.			
Other	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 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 001, 002, 003, 004, and 005. A single test shall be conducted for a flow-weighted aggregate prepared from 24-hour composite samples from Outfall 008.			

b	24-Hour Composite Sample	Parameter
	Individual Outfalls 002, 003, 004 005	TOC, TKN, NH ₃ ^N
	Outfalls 006, 007, 008, 009 010 and 032 combined	TOC, TKN, NH ₃ ^N
b	Composite	

Table 3 (Continued)

<u>24-Hour Composite Sample</u>	<u>Parameter</u>
Outfalls 002, 003, 004, 005 006, 007, 008, 009, 010, and 032 combined	Chlorides, phenolics threshold odor
<p>Sulfides will be determined once per week utilizing grab samples from discharge 005. Deterioration of 24-hour composite samples prevents accurate analysis for sulfides.</p> <p>Temperature measurements will be made daily on Outfalls 001, 002, 003, 004, and 005 during the months of July, August and September. Temperatures will be measured weekly during other times and at other discharge locations.</p>	
<p>c For Outfalls 002, 003, 004 and 005, deviations from the pH range of 6 - 9 which (1) do not exceed 15 minutes for any single instance, (2) do not exceed a total of 30 minutes in any calendar day (3) do not exceed a total of 10 hours in any calendar month, and (4) are within the pH range of 4.0 - 10.5 units, shall not be considered violations of this permit. All such instances of deviation, however, shall be reported quarterly with the discharge monitoring reports.</p>	

ETHYLIDENENORBORNENE AND VINYLNORBORNENE PRODUCTION

Ethylidenenorbornene (ENB) and vinylnorbornene (VNB) are produced [Figure 6] using dicyclopentadiene, butadiene, vinylcyclohexane, vinylnorbornene, bicyclononadiene, gums, ammonia, ethylbenzene, ethylidenenorbornene, noryl phenol, tertbutylcatechol, cyclopentadiene and a patented sodium-potassium-amide catalyst.

The major wastewater sources are the decanters that collect the steam jet condensate and the ammonia scrubber. This wastewater, 190 l/min (50 gpm), is discharged to the WWTF. The non-contact cooling water, 15 m³/min (4,000 gpm) is discharged through Outfall 002. All distillation column residue, 18,200 kg (40,000 lb)/day, is burned in the powerhouse.

There are 16 process emission points and 3 storage tank emission points associated with this processing area. Only three have significant emissions. Point 2e emits 91 kg (200 lb)/hr of cyclopentadiene, point 14e emits a total of 135 kg (300 lb)/hr of cyclopentadiene, ENB and VNB, and point 16e emits cyclopentadiene at 545 kg (1,200 lb)/hr on an intermittent basis (14 minutes per run, 43 runs/yr).*

CELLOSIZE[®] PRODUCTION

CELLOSIZE[®] (hydroxyethyl cellulose) is formed by reacting ethylene oxide, caustic, a viscosity control agent, a pH control agent and cellulose floc [Figures 7 and 8].

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

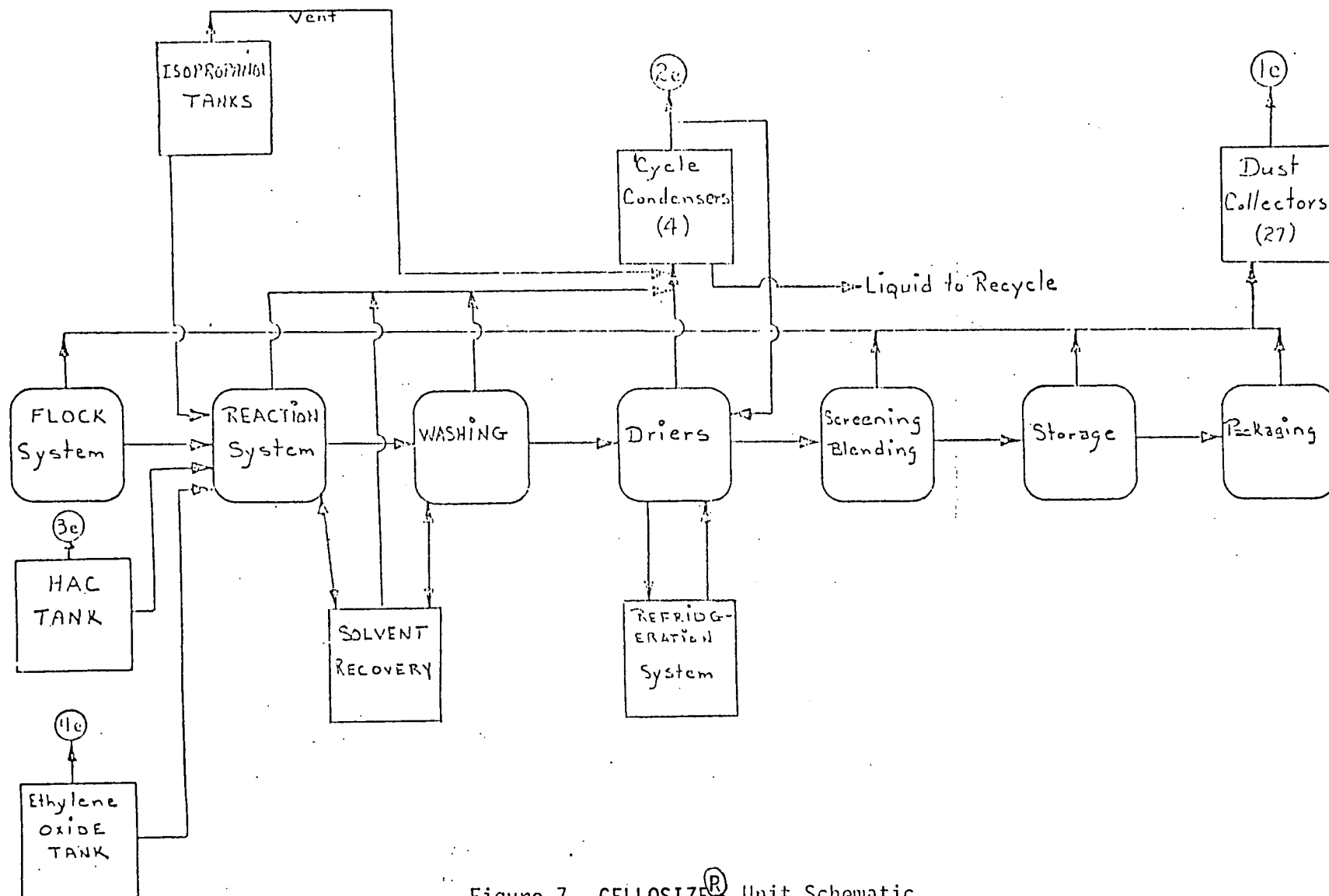


Figure 7. CELLOSIZER® Unit Schematic

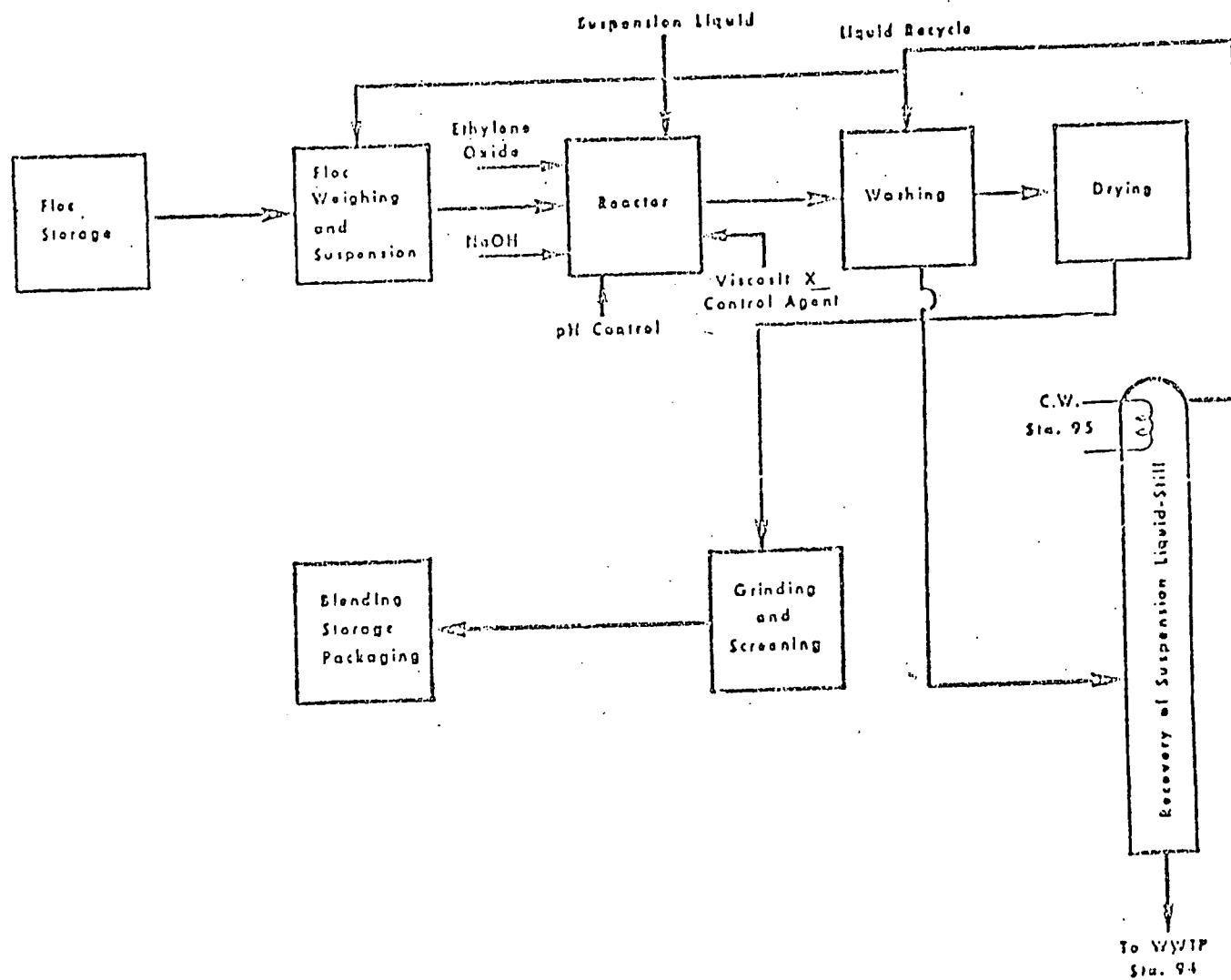


Figure 8. CELLOSIZE® Process Schematic

The major wastewater source is the solvent recovery still. This waste contains isopropanol, benzene and CELLOSIZ[®] and is discharged to the WWTF. The non-contact cooling water, 15 m³/min (4,000 gpm), is discharged through Outfall 004.

There are emissions from 27 baghouses, 4 process vents and 2 storage tanks. However, the emissions are less than 9 kg (20 lb)/hr per emission point.

Solid wastes from the process are collected and either disposed of in the Goff Mountain landfill or sold to a paper company.

ETHYLENE GLYCOL RECOVERY

Spent ethylene glycol containing ethylene glycol, solids, water and methanol is refined in a series of distillation columns to recover the ethylene glycol [Figure 9].

Process wastewaters are the methanol still bottoms and the water from the concentrator. These wastes are combined and discharged to the WWTF. Non-contact cooling water and vacuum jet condensate are discharged through Outfall 002. The solids from the concentrator and methanol are burned in the powerhouse.

Air emission sources include the water, containing some methanol, from the methanol still and the water evaporated from the concentrator. There are no air emissions inventory data available for this unit.

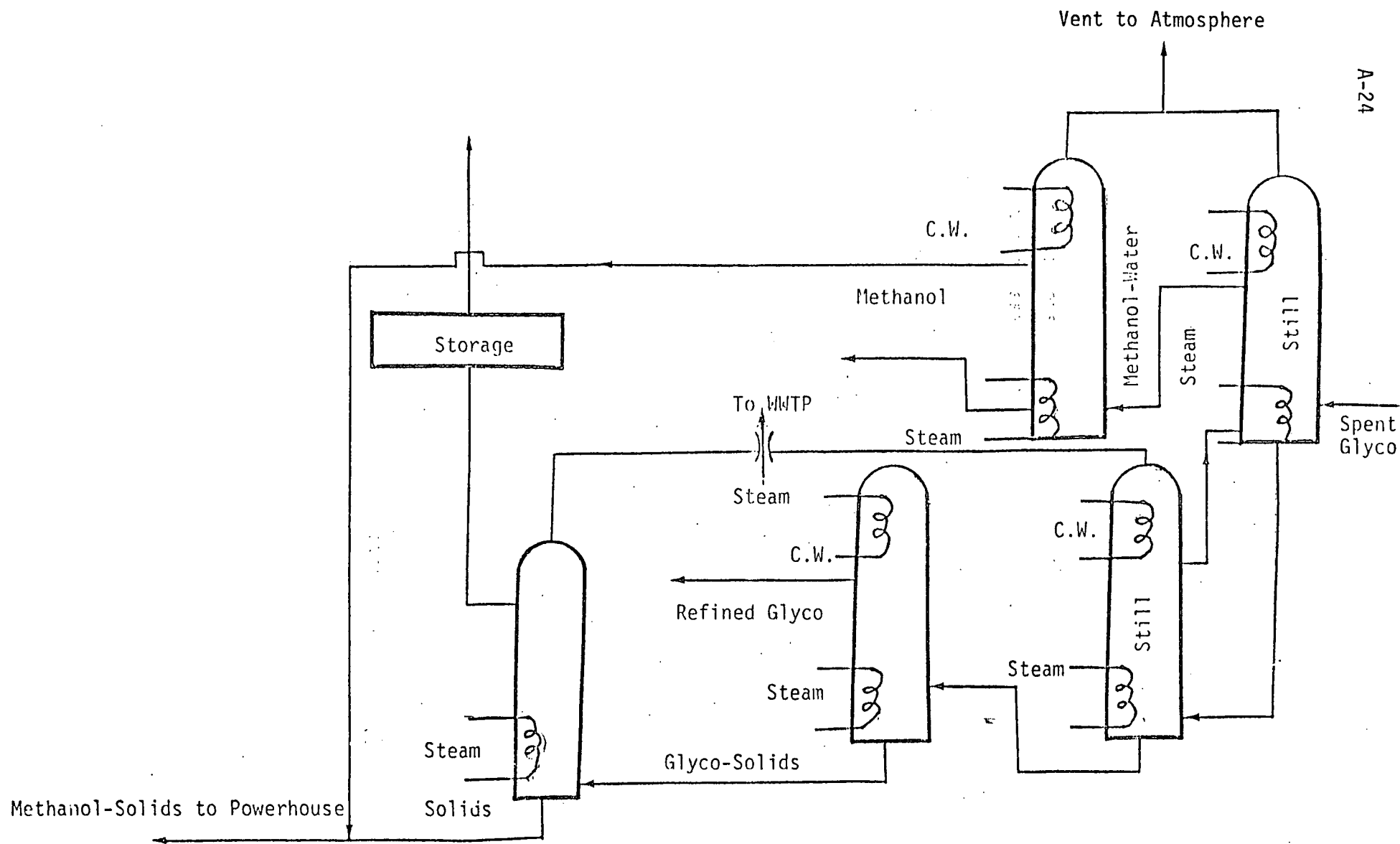


Figure 9. Recovery of Returned Spent Glyco Schematic

POLYETHYLENE GLYCOL (CARBOWAX®) PRODUCTION

Ethylene glycol and a caustic catalyst are reacted to produce several different CARBOWAXES® [Figure 10]. After filtering, the CARBOWAXES® are sold as a liquid or converted to a solid and bagged.

Wastewater sources include the reactor scrubber, cooling filter wash and bin wash waters. This waste is discharged, 152 l/min (40 gpm), to the WWTF for treatment.

The air emission sources include the ethylene oxide storage tanks vents (2), reactor vent and bagging area. The storage tank vapor is condensed to recover the ethylene oxide, reactor off-gases are condensed and water scrubbed and the bagging area emissions are controlled by a baghouse.

Filter solids and spilled solids are disposed of in the Goff Mountain Landfill.

ALLETHRIN UNIT

Other chemicals are produced in the Allethrin unit at different times. These include TERGITOLS® (alkyl phenol type detergents), glutaraldehyde and 100-150 others. Process equipment includes reactors, filters and distillation columns [Figures 11, 12 and 13].

The process wastewaters include filter leaks and washes, condensate from vacuum jets and reactor cleaning. This water, 1,330 l/min (350 gpm), is discharged to the WWTF. Non-contact cooling water (5,200 gpm) is discharged through Outfall 002. Still residues are burned in the powerhouse.

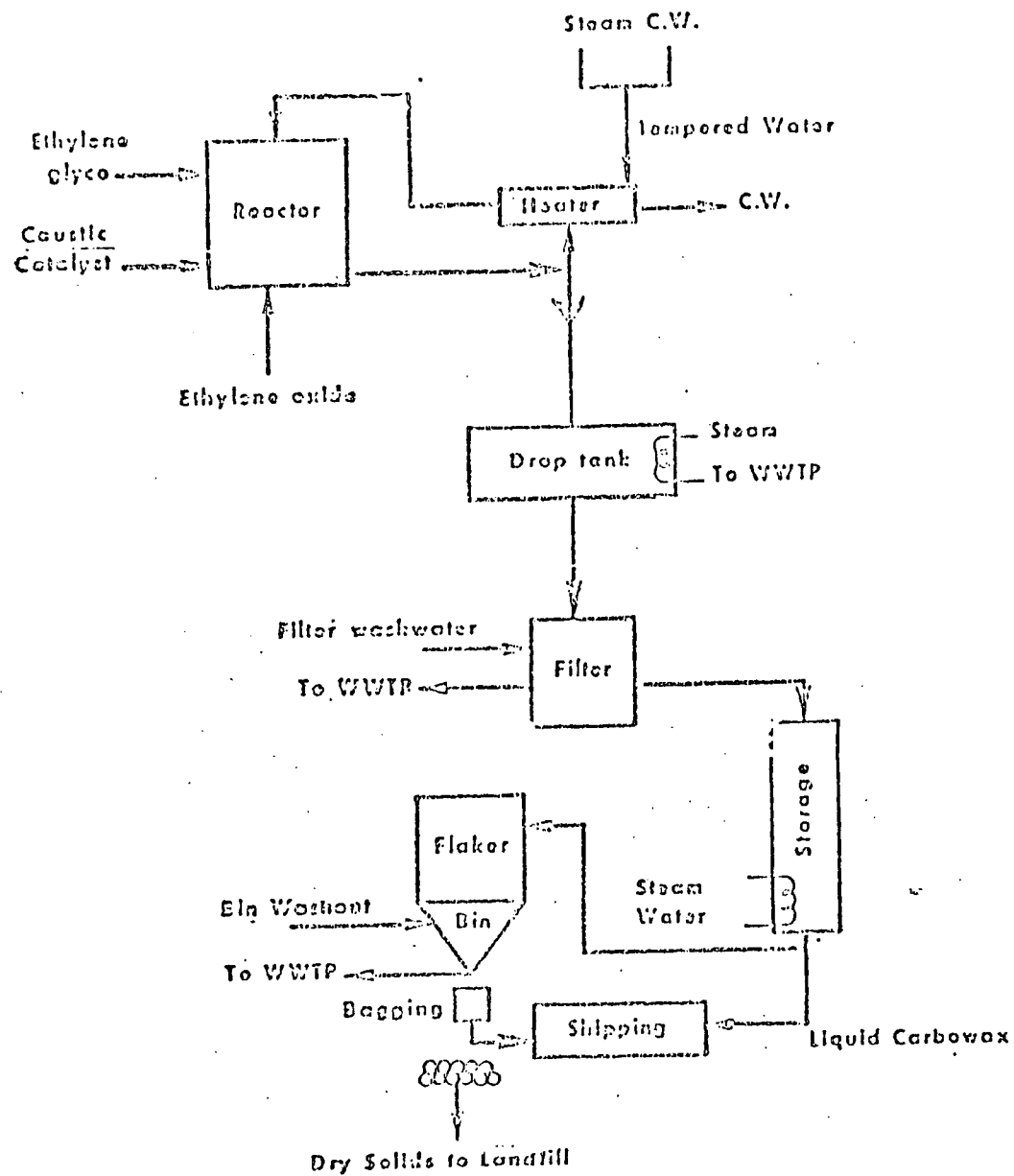


Figure 10. Polyethylene Glycol (CARBOWAX®) Schematic

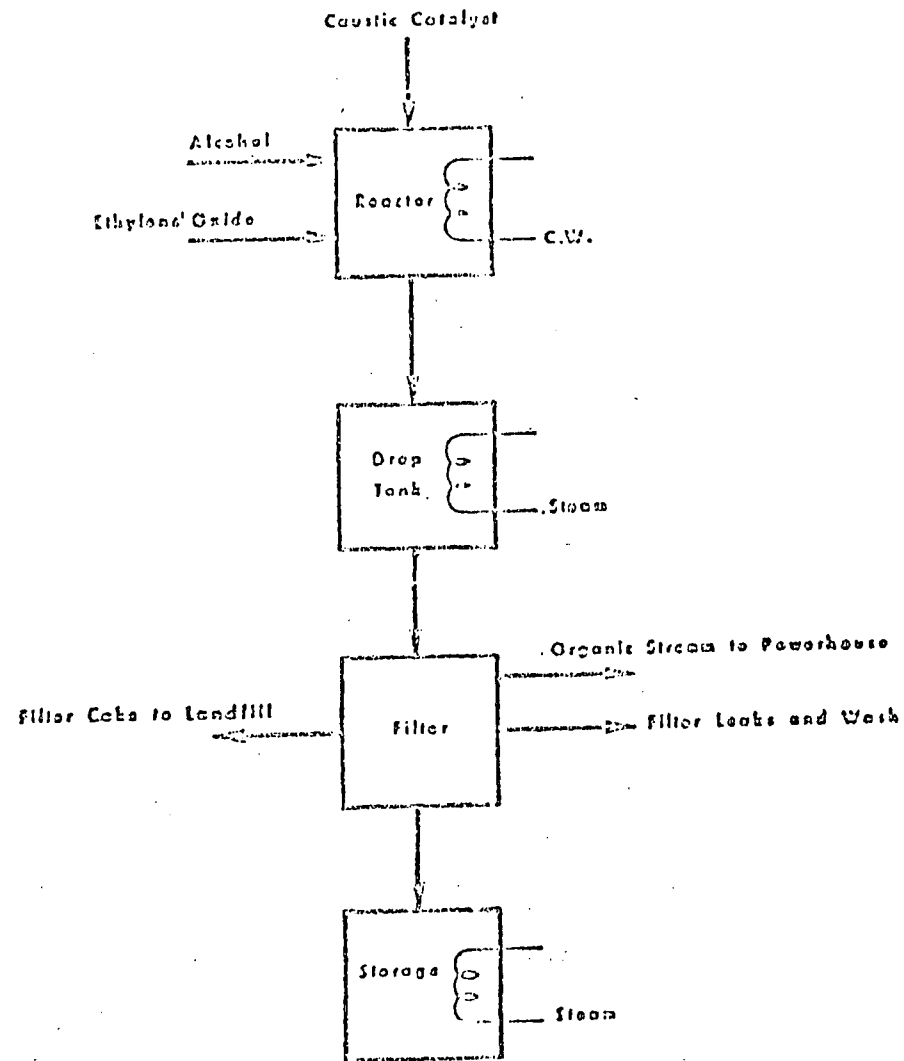


Figure 11. TERGITOL[®] Process Schematic

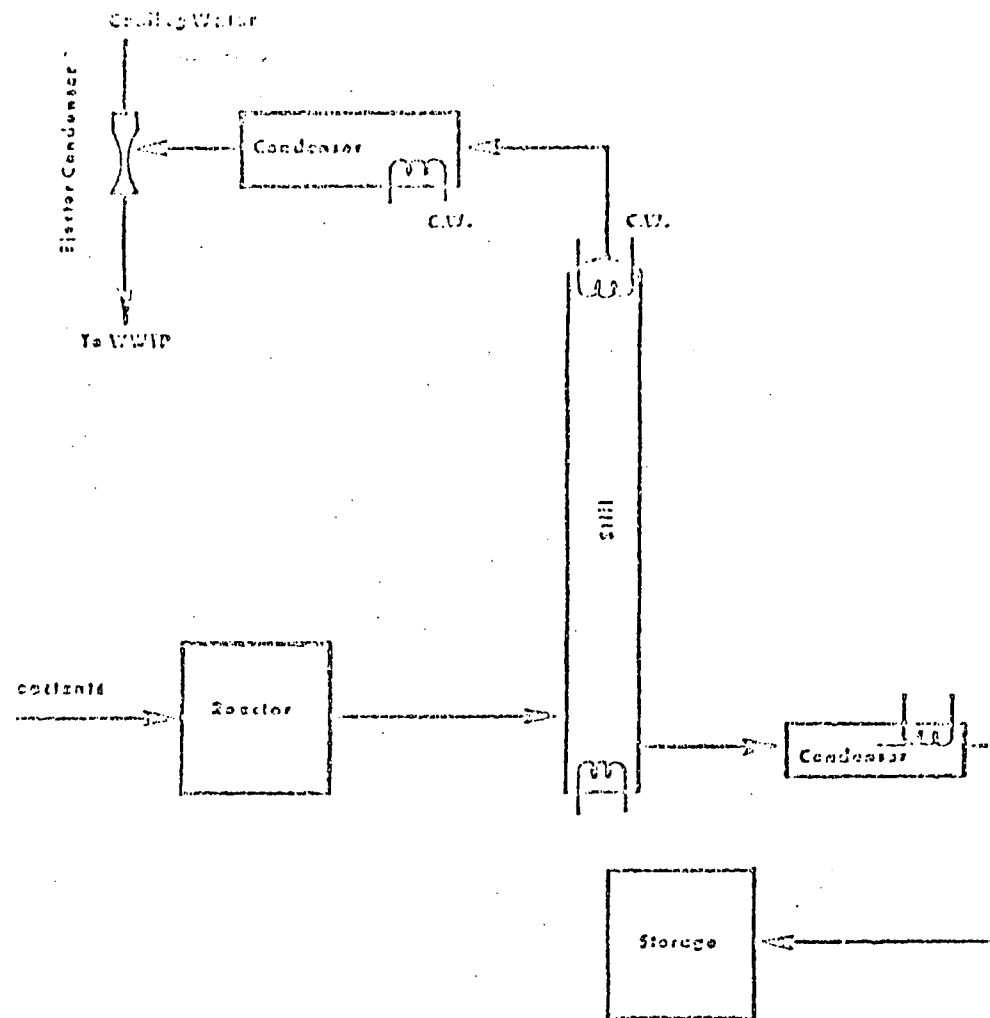


Figure 12. Miscellaneous Chemicals Schematic

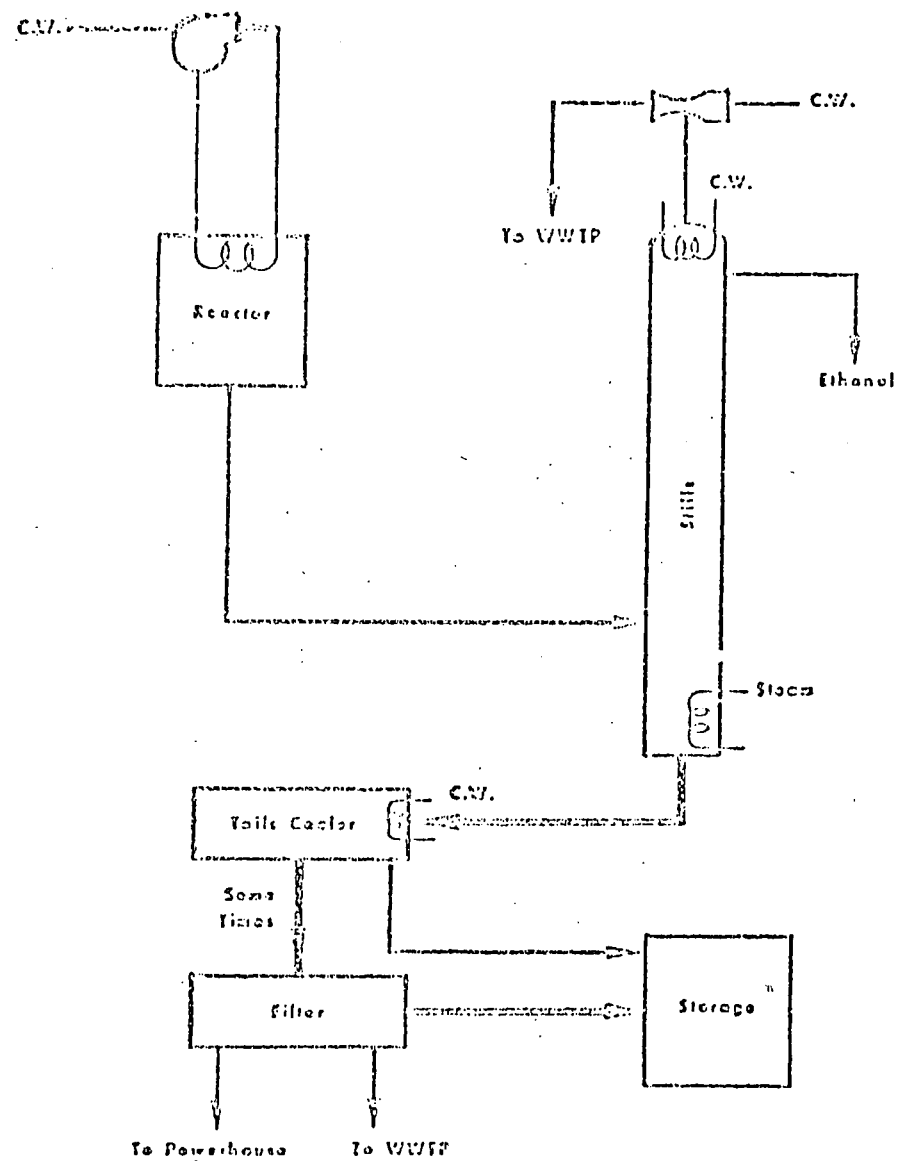


Figure 13. Glutaraldehyde Process Schematic

All air emissions, except the still off-gases, are collected and burned in flares. The still gases are condensed and vented to the atmosphere. Filter solids are disposed of in the chemical landfill.

POLYOL PRODUCTION

Approximately 65 different polyols are produced from 30 raw materials [Figure 14]. Process equipment includes a reactor, ion exchanger, filters, product refiner and solvent recovery still.

Wastewaters include the vacuum jet condensate, still bottoms and ion exchange regeneration. These wastewaters, 950 l/min (250 gpm) are discharged to the WWTF. Non-contact cooling water, 265 l/min (70 gpm), is discharged through Outfall 003.

The air emission points are 6 process vents and 6 storage tank vents; no emission rate exceeds 7 kg (15 lb)/hr. All 12 of these vents are controlled by nitrogen blanketing.

Filter solids and exhausted ion exchange resins are disposed in the Goff Mountain landfill.

BUTANOL AND ETHYLBUTYRALDEHYDE PRODUCTION

Butanol and ethylbutyraldehyde are produced by reacting acetaldehyde, butyraldehyde, hydrogen and caustic as shown in Figure 15.

Wastewaters include tails from the sodium acetate still, the stripping still and the butyraldehyde still. This waste, 190 l/min (50 gpm), is discharged to the WWTF. Non-contact cooling water, 85 m³/min (22,500 gpm), is discharged through Outfalls 003 and 004. Residues from the distillation columns are burned in the powerhouse.

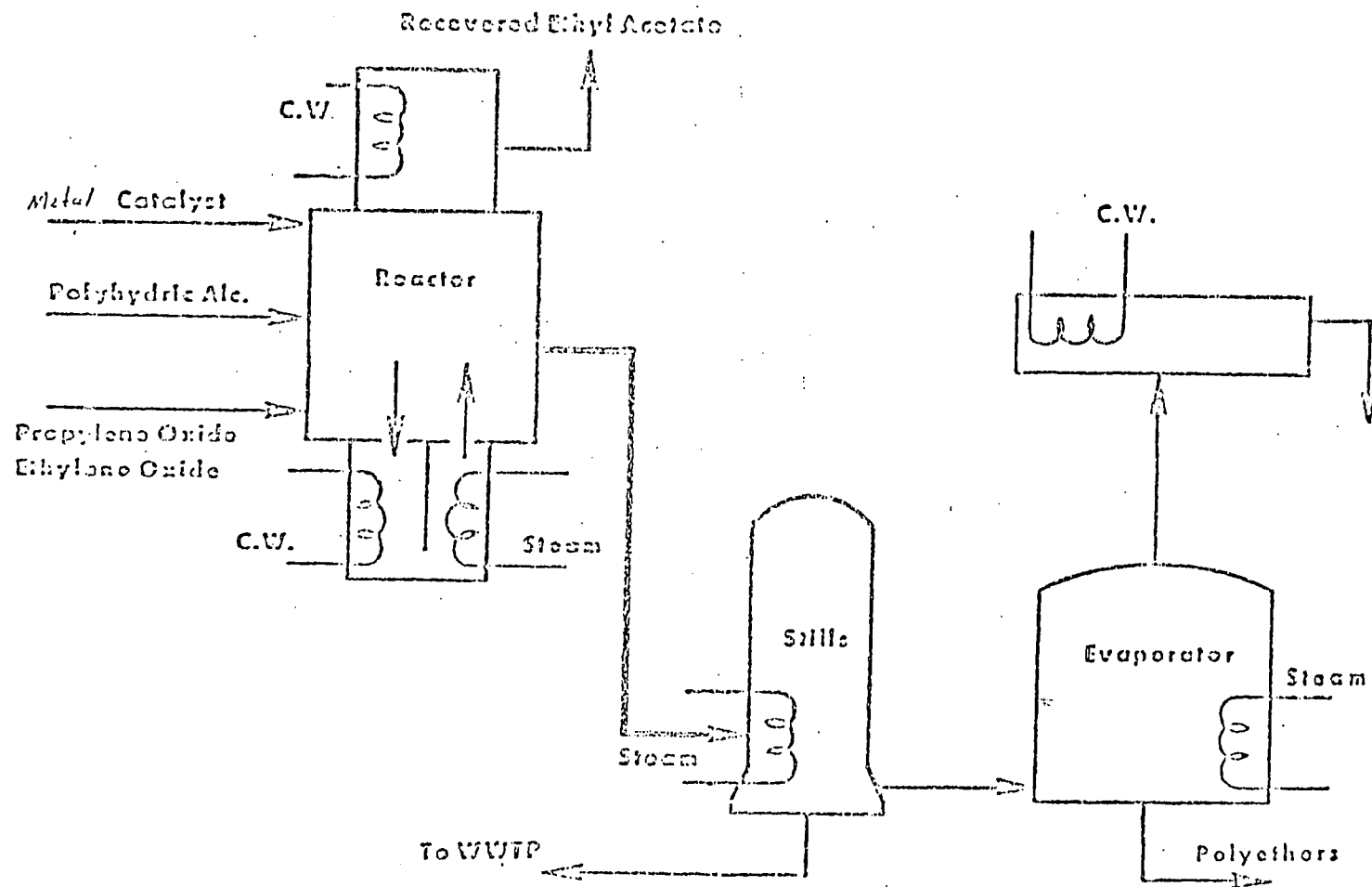


Figure 14. Polyol Process Schematic

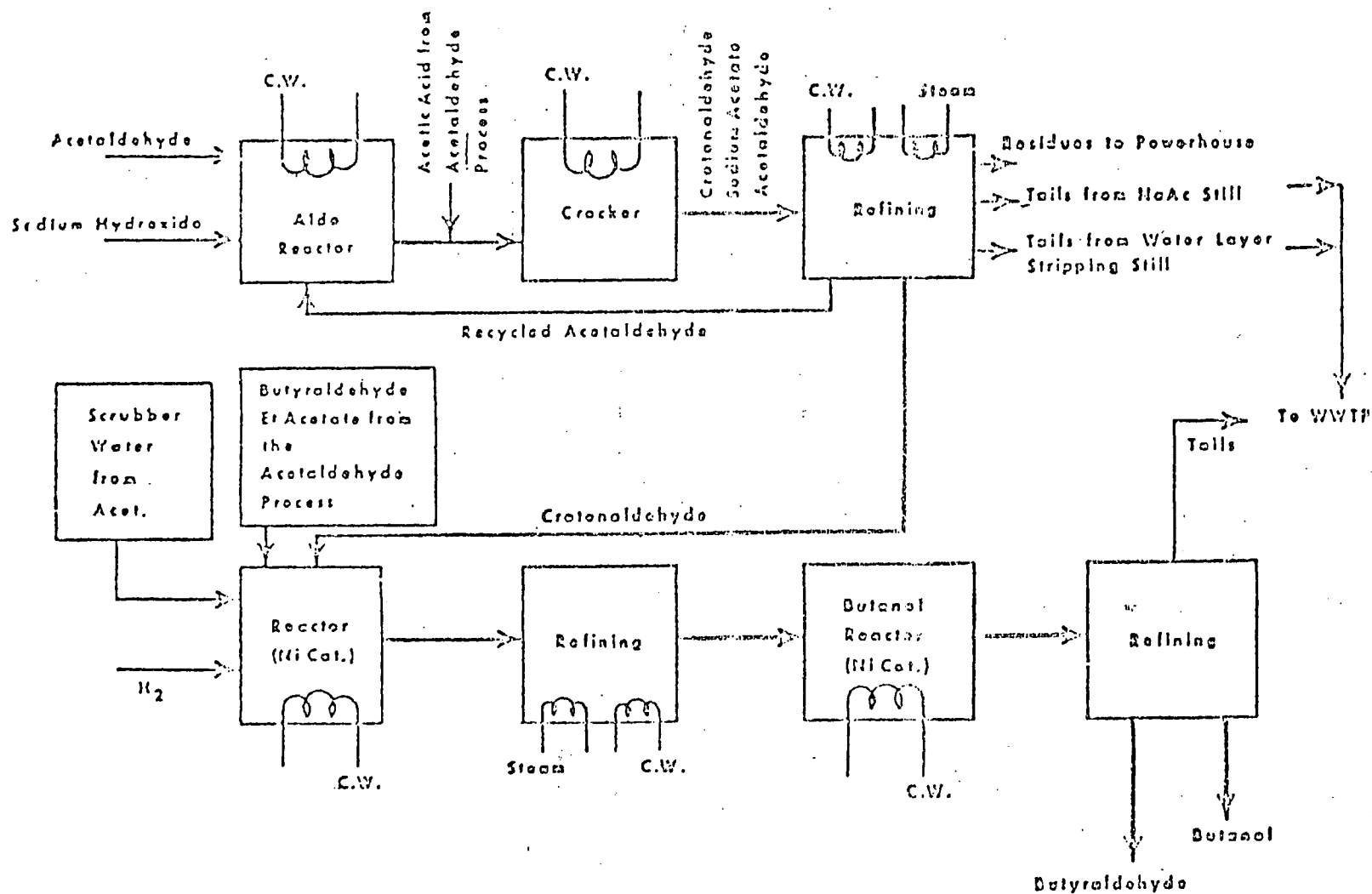


Figure 15. Butanol and Ethylbutyraldehyde Process Schematic.

This entire system operates under pressure; therefore the only air emission sources are pressure relief valves that can open to the atmosphere in case of an emergency.

ACETONE AND MIXED KETONE CRUDE NAPHTHOL AND ISOPHORONE PRODUCTION

Acetone, mixed ketones, crude 1-naphthol, isophorone and mesityl oxide are all produced in this process area.

Acetone and mixed ketones are produced by the catalytic dehydrogenation of isopropyl alcohol [Figure 16]. By using various catalysts the reaction can be controlled to produce only acetone or acetone, alcohols and various ketones. Crude 1-naphthol is produced by converting tetralone to 1-naphthol using a metal catalyst and caustic. Both 1-naphthol and tetralone are intermediates for SEVIN[®] production. Acetone is converted to isophorone and the by-product mesityl oxide by a metal catalyst [Figure 17].

Wastewaters include the isopropanol stripper tails, 1-naphthol conversion unit and the isophorone water layer stripping still. These wastewaters, 1,140 l/min (300 gpm), are discharged to the WWTF for treatment. Non-contact cooling water, 87 m³/min (23,000 gpm), is discharged through Outfall 003. The tails from the several ketone and recovery stills are burned in the powerhouse.

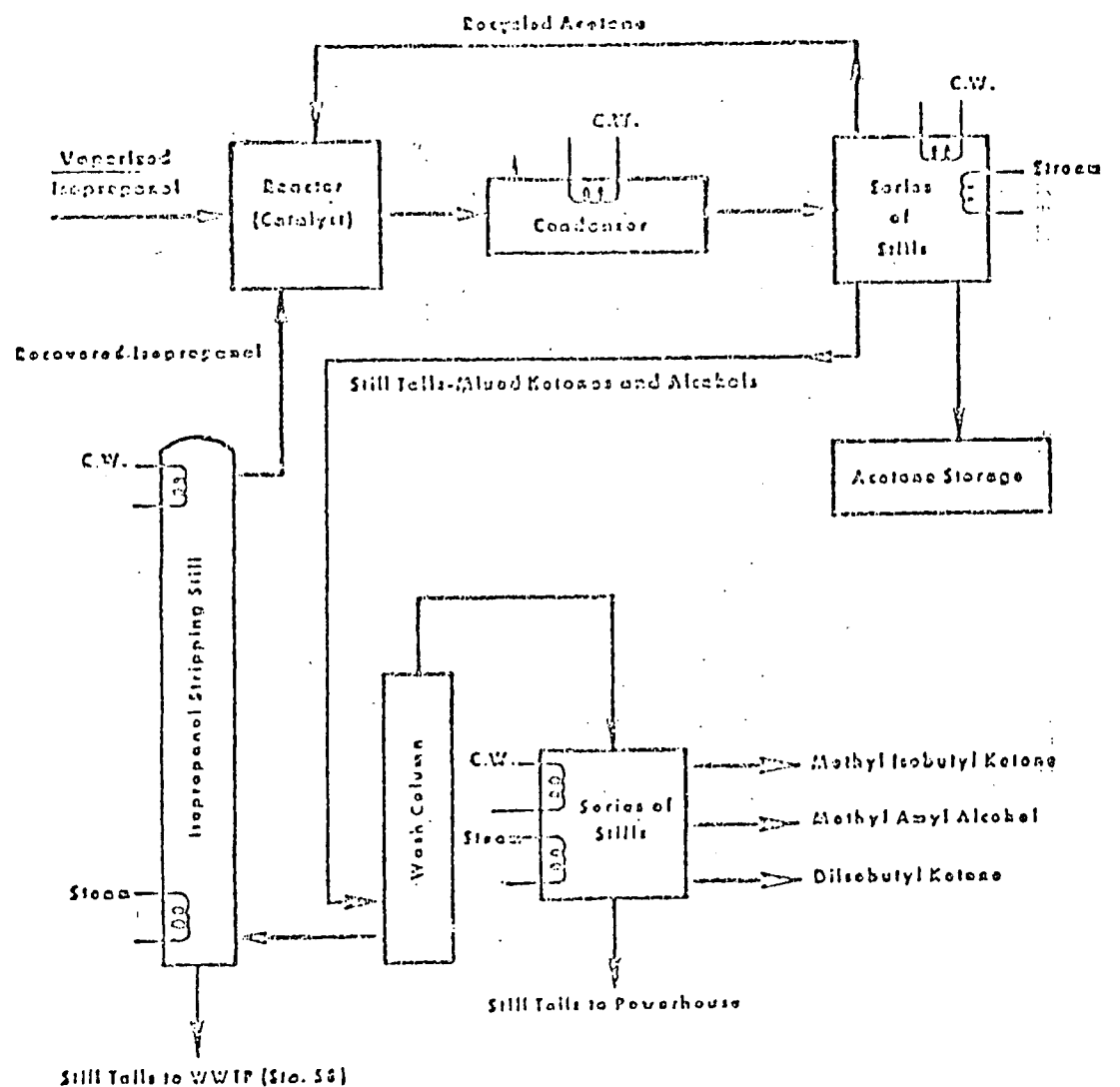


Figure 16. Acetone and Mixed Ketones Schematic

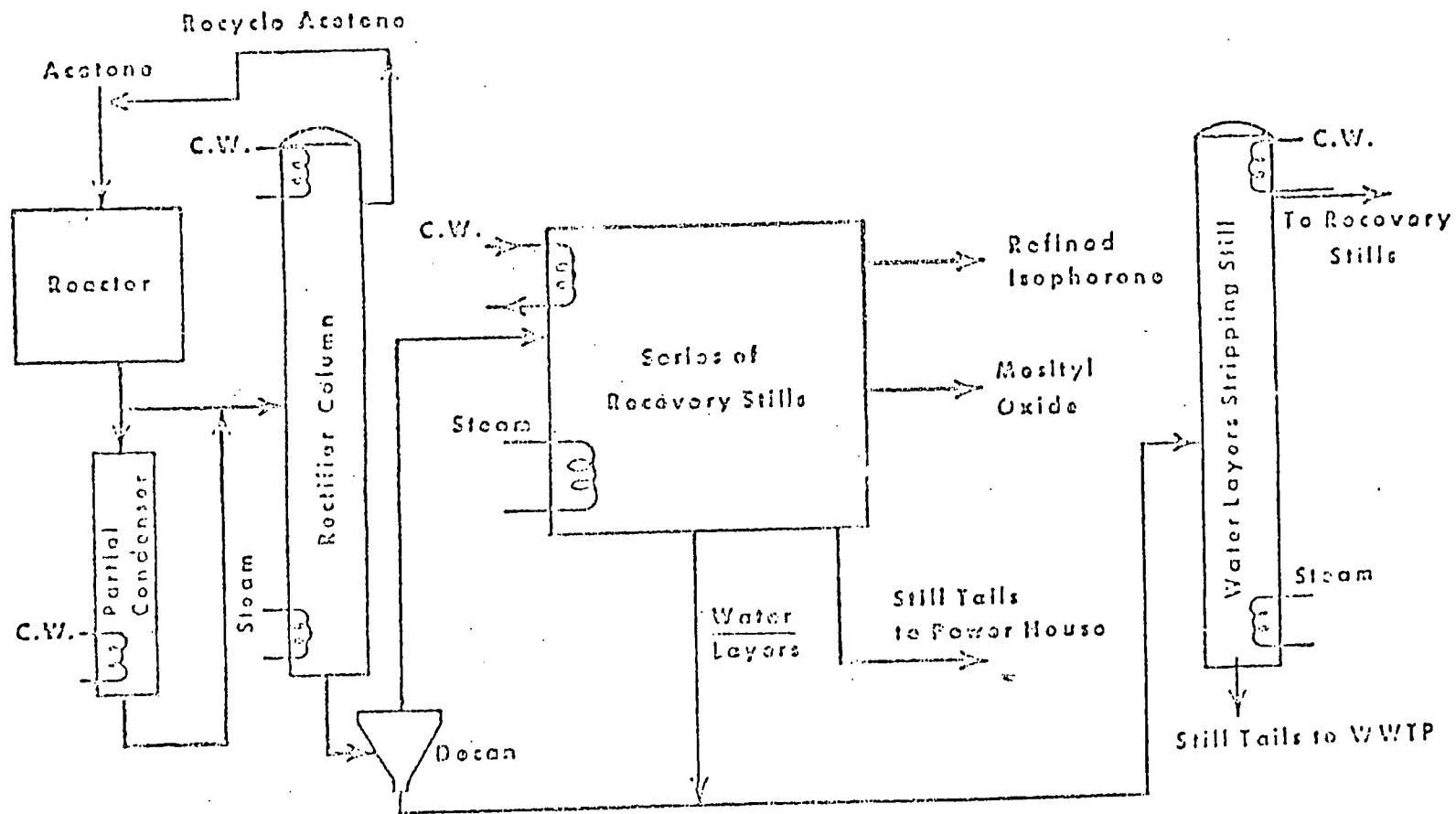


Figure 17. Isophorone Process Schematic

Air emissions from all process vents are collected and burned in the acetone unit petrochem furnace. There are 32 storage tanks and the petro-chem furnace that vent to the atmosphere. Emissions from the tanks are less than 9 kg (20 lb)/hr per tank.

All catalyst is recovered and returned to the vendor for reprocessing.

SEVIN[®] PRODUCTION

The SEVIN[®] manufacturing process includes the synthetic gas, tetralin, tetralone, naphthol, phosgene, 1-naphthol refining, naphthylchloroformate (NCF), methyl isocyanate and SEVIN[®] units. SEVIN[®] is a contact insecticide that, under alkaline conditions, hydrolyzes to 1-naphthol, carbon dioxide and methylamine.

Synthetic Gas Unit

The synthetic gas (syn gas) unit converts natural gas to carbon dioxide (CO₂), carbon monoxide (CO) and hydrogen (H₂), removes the CO₂ from the gas and separates the CO from the H₂ [Figure 18].

The only process wastewater is that from the CO₂ extraction and drying system. It consists of water and monoethanol amine blowdown. This blowdown, 38 l/min (10 gpm), is discharged to the WWTF. Non-contact cooling water, 3,400 l/min (900 gpm), is discharged through Outfall 005.

All air emissions from the syn gas system are collected and flared.

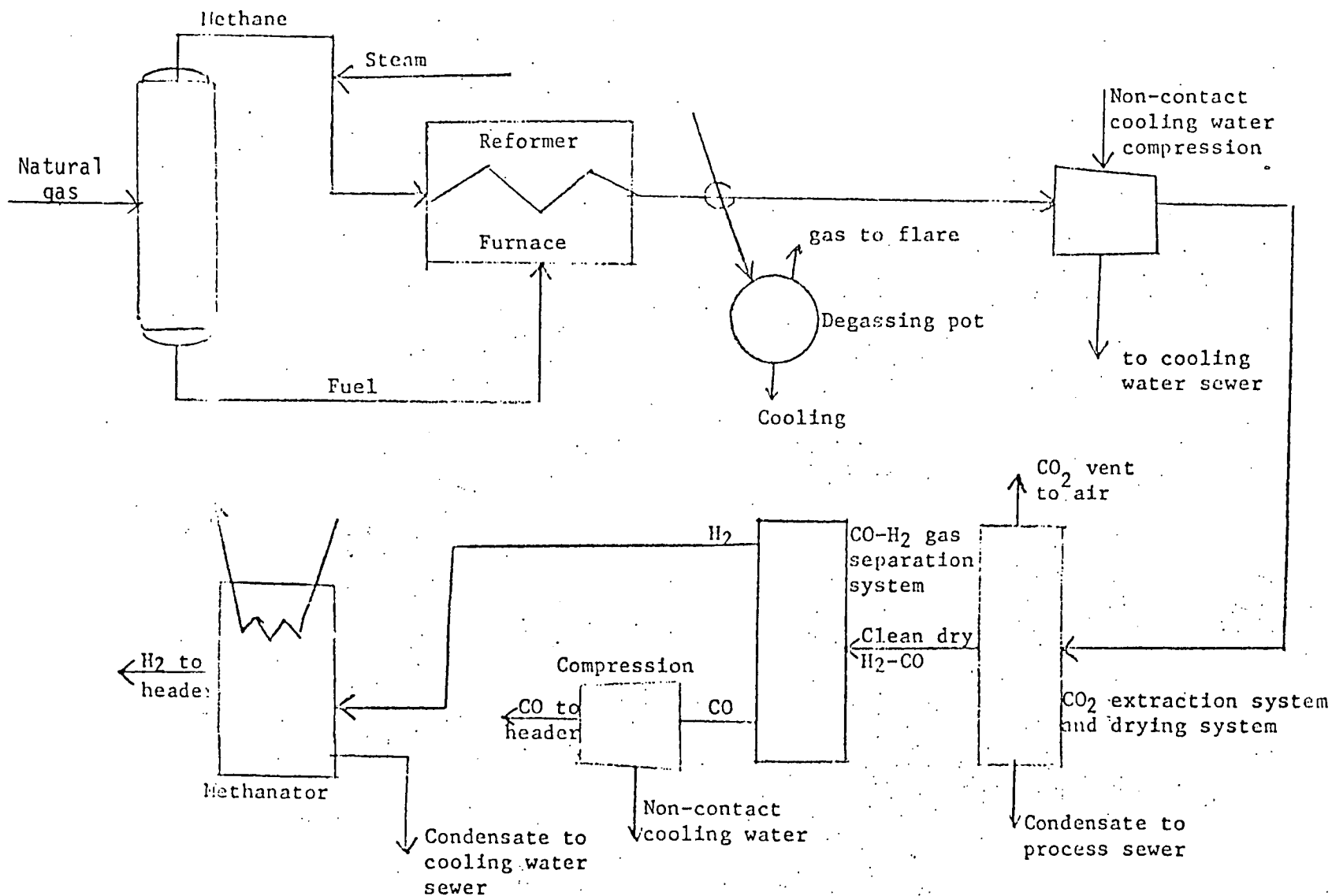


Figure 18. H₂-CO Process Schematic

Tetralin

Tetralin (tetrahydronaphthalene) is produced [Figure 19] by reacting naphthalene, hydrogen and sodium metal. Small amounts of sulfur in the crude naphthalene are introduced into the system and must be removed from the wastewater.

The only wastewater source is the decanter water layer. This water is treated in the wastewater sulfide oxidation system and discharged to the WWTF. Non-contact cooling water is discharged through Outfall 005. The refining still residues are burned at the powerhouse.

The major air emissions from the degasser and the refining system are collected and burned in the petro-chem furnace. Other vents emit less than 0.9 kg (2 lb)/ day per vent to the atmosphere.

Tetralone

Tetralone is produced [Figure 20] by oxidizing tetralin with a catalyst and air. Process equipment includes the oxidizer, 3 decanters and a refining unit with vacuum distillation.

Wastewater discharges include the No. 3 decanter water and the refining tails. The wastewater from the first two decanters flows to the No. 3 decanter before being discharged. In addition, the vacuum jet condensate is collected and decanted prior to being discharged. The majority (80-90%) of the decanter water and still tails are burned in the powerhouse along with the decanter residue. If this waste is not burned it is steam stripped and discharged to the WWTF. Non-contact cooling water is discharged through Outfall 005.

There are 5 process and 4 storage tank air emission points in the process. Vapors from the oxidation process pass through a condenser before being discharged to the atmosphere. The refining system off-gases are condensed and sent to the crude naphthol unit. The maximum emission rate, 11 kg (24 lb)/hr of tetralin, is from the 4 vents on the oxidation system. All still residues are burned at the powerhouse.

The used catalyst is cleaned from the oxidizer on a batch basis and buried in the Goff Mountain Landfill.

1-Naphthol Refining

Crude naphthol is refined [Figure 21] to 1-naphthol in a series of distillation columns. A solvent is used, recovered and recycled in this process. By-product recovery is accomplished by using a series of distillation columns.

The major discharge is the by-product still vacuum jet condensate. This waste is collected and discharged to the WWTF for treatment. All residues from the distillation columns are burned at the powerhouse.

The only air emission sources are the 1-naphthol storage tanks which vent to the atmosphere. Other emissions are collected and burned.

Phosgene Process

Phosgene is produced [Figure 22] by reacting carbon monoxide (CO) with chlorine in the presence of an activated carbon catalyst.

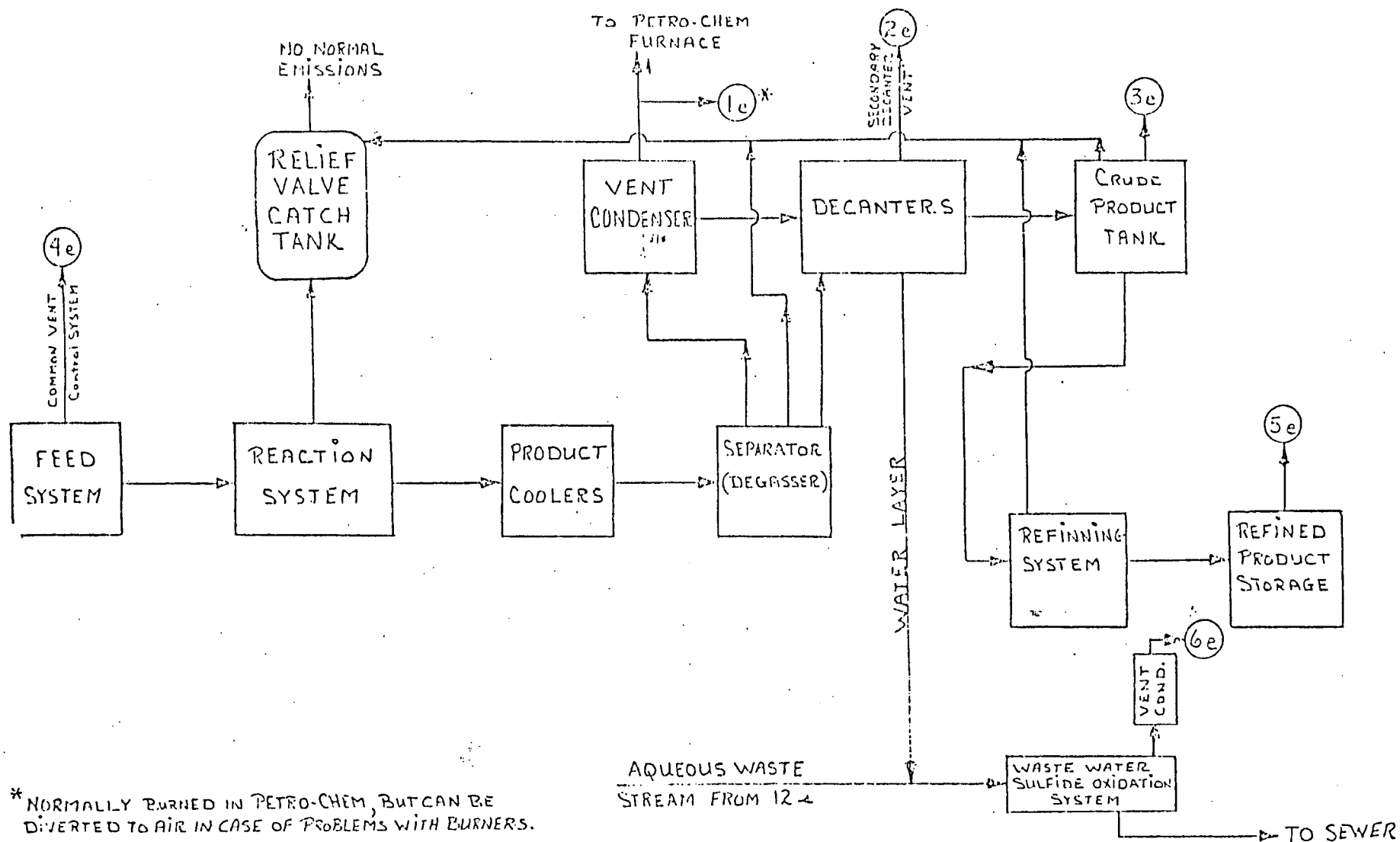


Figure 19. SEVIN® Complex Tetrahydronaphthalene Unit Schematic

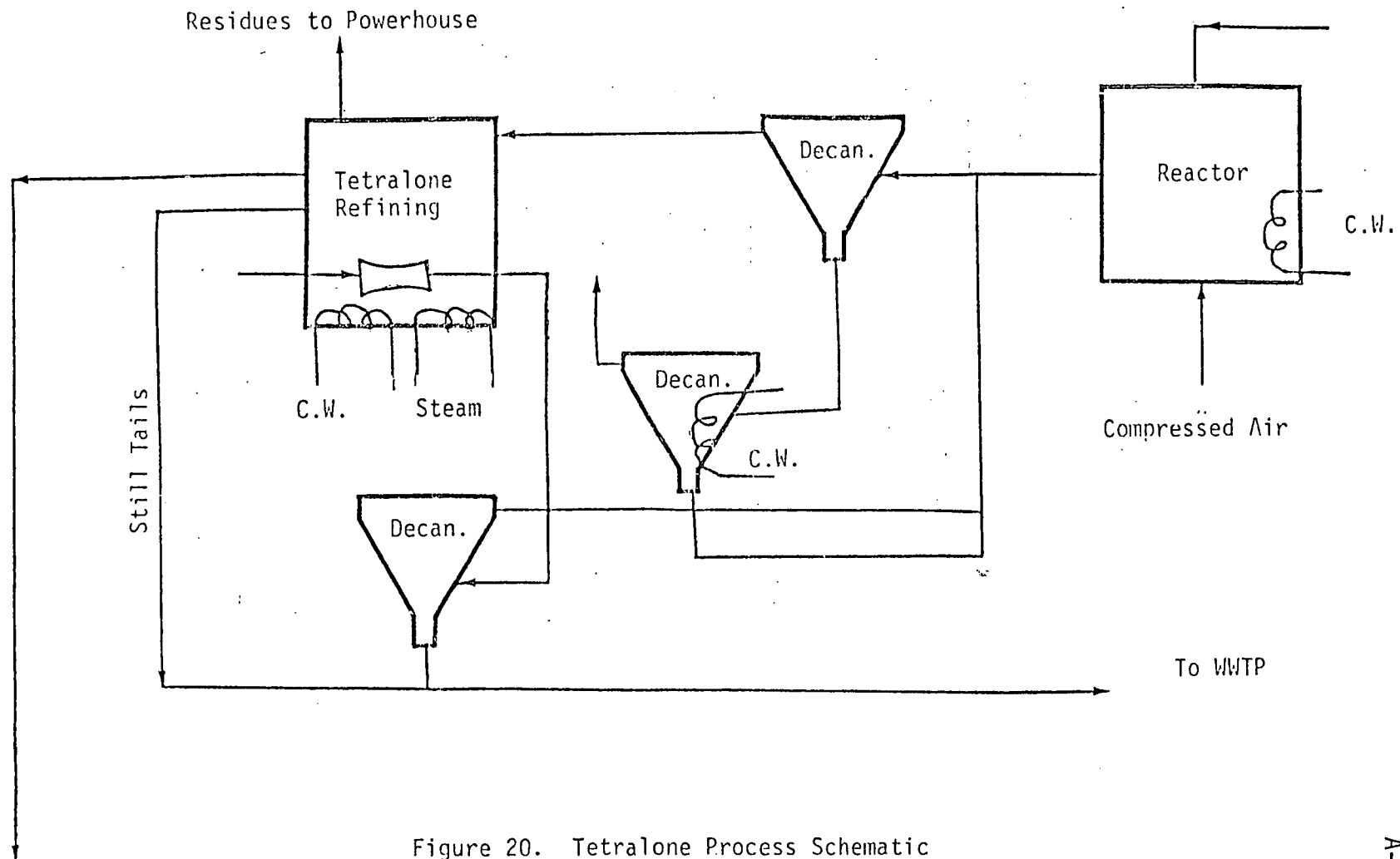


Figure 20. Tetralone Process Schematic

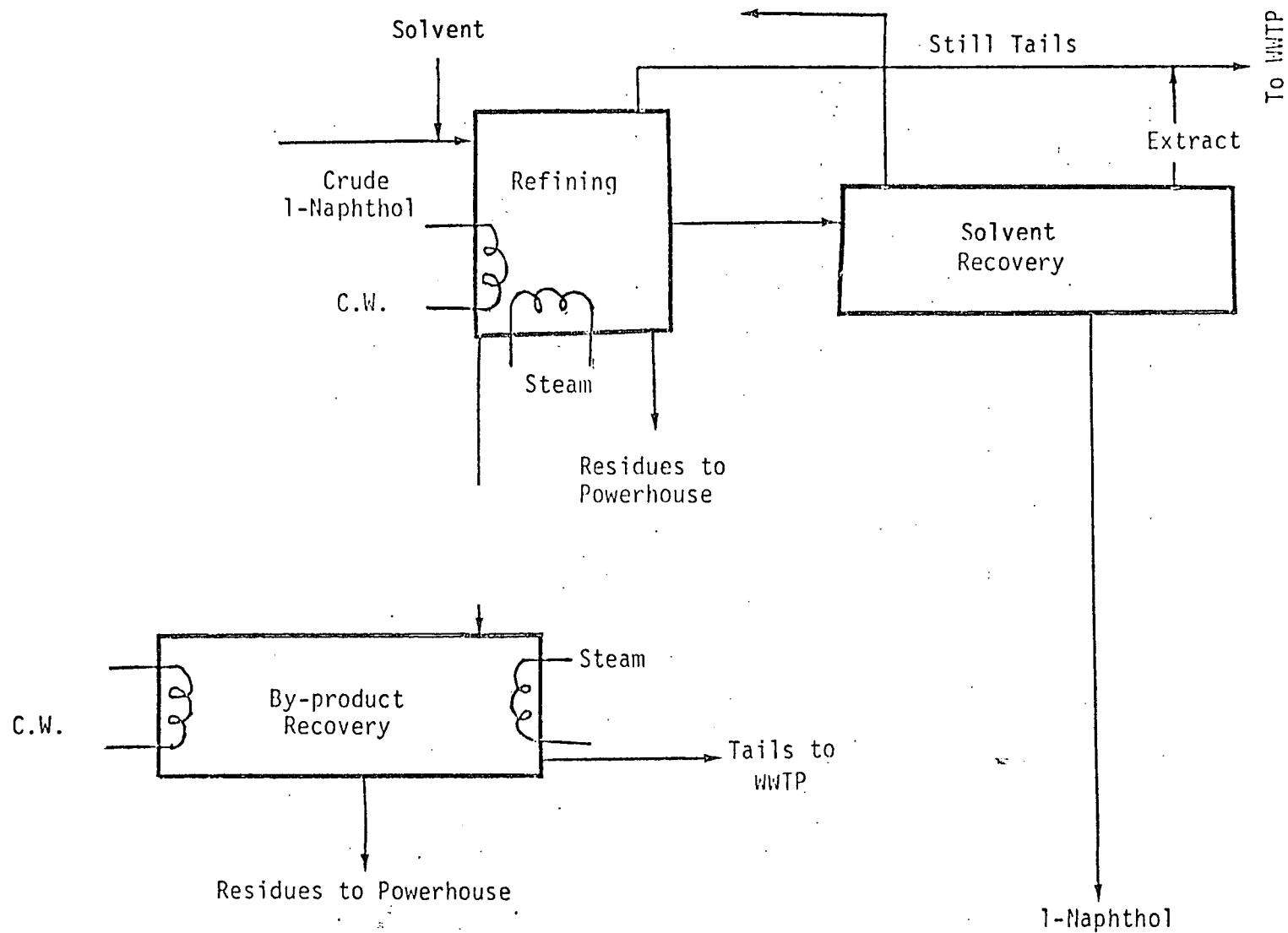


Figure 21. 1-Naphthol Process Schematic

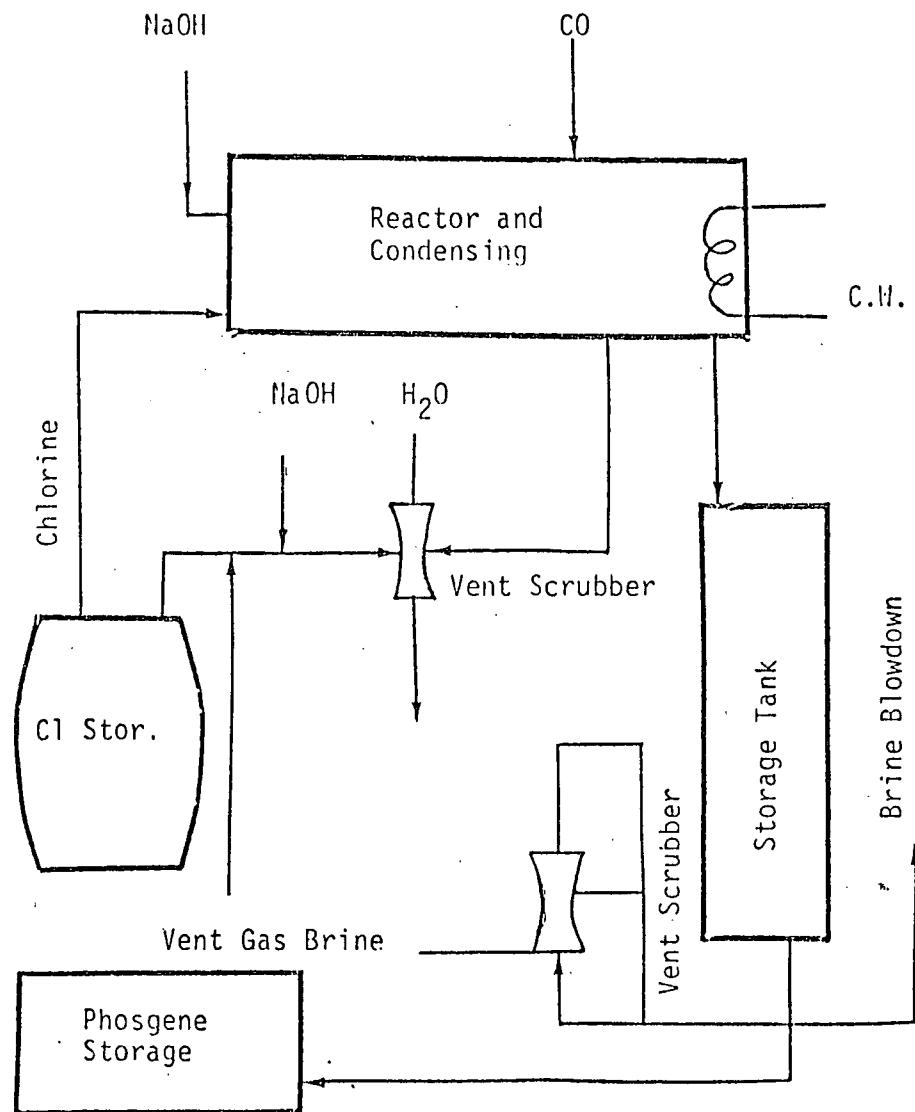


Figure 22. Phosgene Process Schematic

Phosgene is a very toxic gas; therefore contact with air or water is carefully controlled. There are no process wastewater sources, however there are vent scrubbers to neutralize and scrub any chlorine or phosgene that is emitted from the reactor, surge tank or the chlorine storage tank. This wastewater contains sodium chloride and is discharged to the WWTF for treatment. Non-contact cooling water is discharged through Outfall 005.

The spent activated carbon is removed from the reactor and buried in the Goff Mountain landfill.

Naphthylchloroformate and SEVIN[®] Process

Naphthylchloroformate (NCF) is produced by reacting 1-naphthol with phosgene in a solvent mixture of dimethylamine (DMA) and toluene [Figure 23]. Hydrochloric acid is a by-product from the NCF process. SEVIN[®] is then produced by reacting NCF with methyl amine. The DMA and toluene are recovered and recycled to the process.

Wastewater sources include NCF and SEVIN[®] decanters, stripping stills, caustic scrubbers and the vacuum jet system. The decanter water is collected and steam stripped prior to being discharged to the WWTF along with the vacuum jet condensate and the scrubber media. The stripper overheads are returned to the process. The steam condensate is also discharged to the WWTF. The NCF and SEVIN[®] processes are highly corrosive resulting in an unusual number of leaks from condensers, pipes, pumps, etc. This process area is diked so that the contaminated water and pad spills can be recycled to the process. Non-contact cooling water is discharged through Outfall 005.

All air emission vents, except the SEVIN[®] storage bin, dryer and bagging machine vents, are caustic scrubbed and burned in a flare.

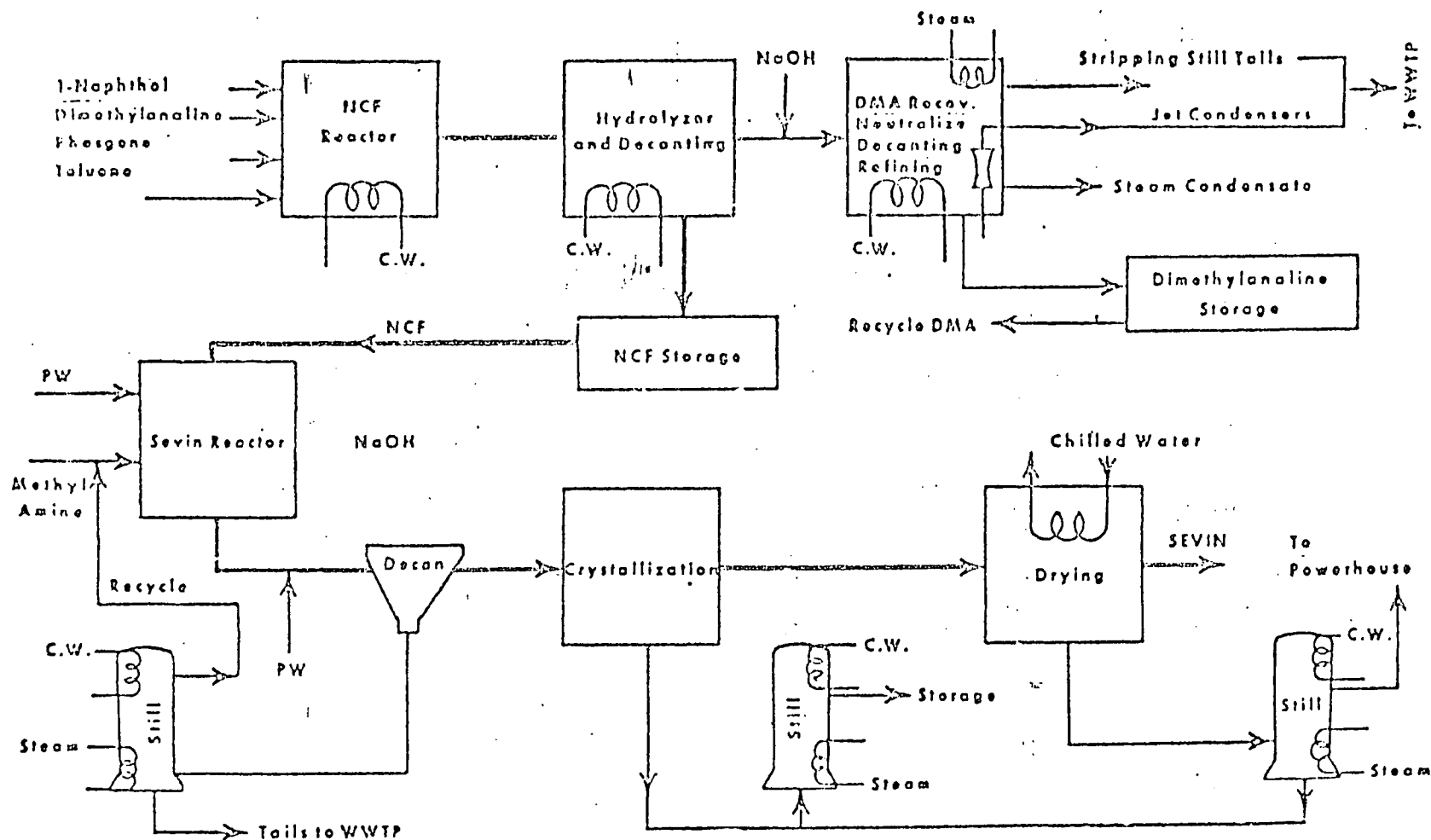


Figure 23. Naphthylchloroformate (NCF) and SEVIN^(R) Processes Schematic

The storage bins and the bagging machine vent to the atmosphere through a baghouse and the drier vapors are condensed to recover the toluene before being discharged to the atmosphere. Only toluene is discharged to the atmosphere (maximum rate 22 kg-(50 lb)/hr). Any SEVIN[®] residue is burned at the powerhouse.

All solid wastes (SEVIN[®], filter cones, bags from the baghouse and area clean-up) from the SEVIN[®] unit are buried in the Goff Mountain landfill.

The NCF to SEVIN[®] process was to be shut down in late 1978, being replaced by the methylisocyanate to SEVIN[®] process and will be shut down in late 1978. SEVIN[®] is the largest volume product at the plant.

Methylisocyanate and SEVIN[®] Processes

Methylisocyanate (MIC) is produced by reacting methylamine with phosgene [Figure 24]. Hydrochloric acid (HCl) is a by-product of this process. Any excess phosgene is hydrolyzed to HCl and the HCl is scrubbed with water.

SEVIN[®] is produced by reacting MIC and methyl chloride in toluene solvent [Figure 25].

The wastewater sources include the HCl scrubber water, the SEVIN[®] and MIC caustic scrubbers, the emergency scrubber and the incinerator scrubber. Currently the HCl scrubber water is used for neutralization at the WWTF. When the new MIC to SEVIN[®] process is in operation the HCl scrubber water will be discharged through limestone pits, neutralized

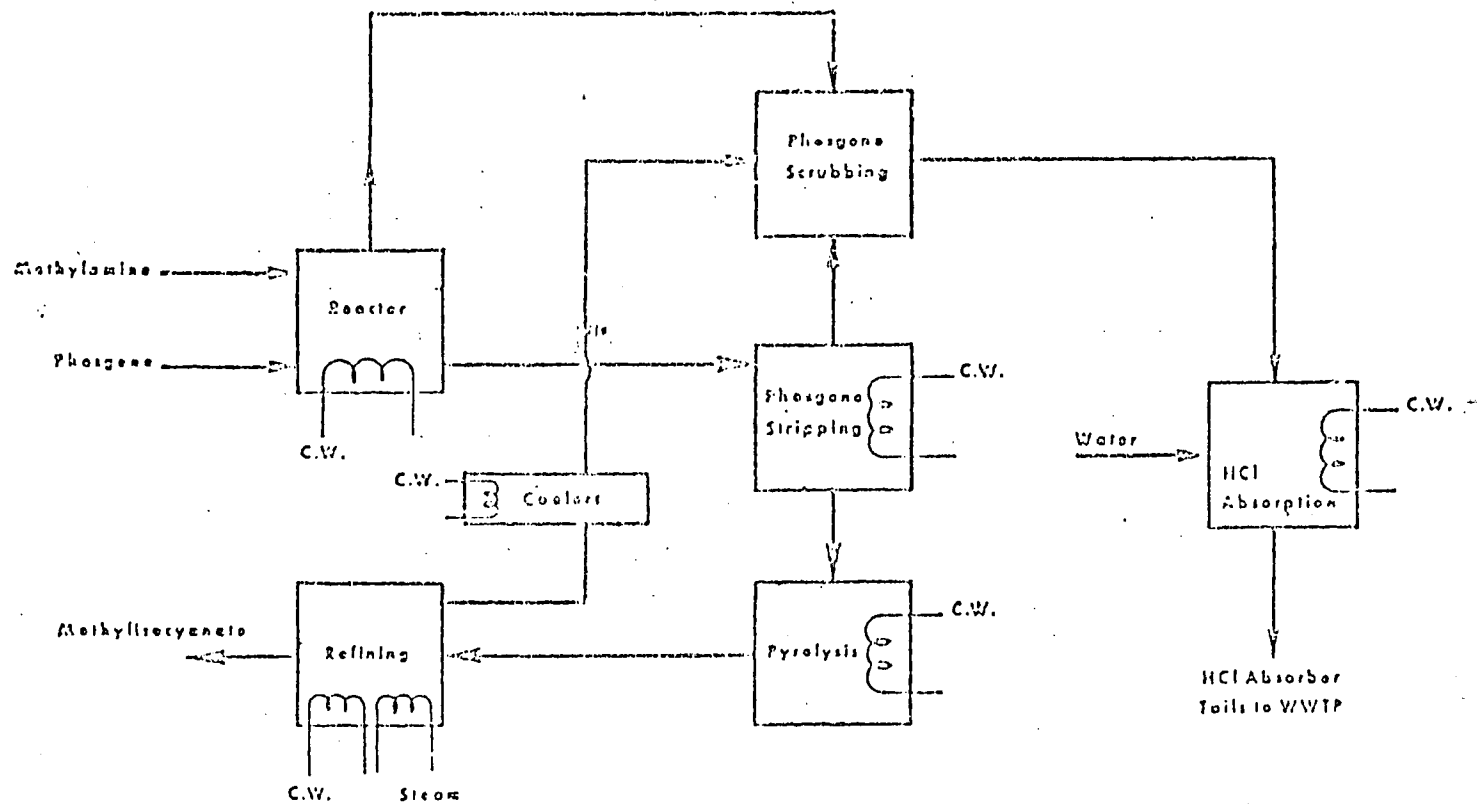
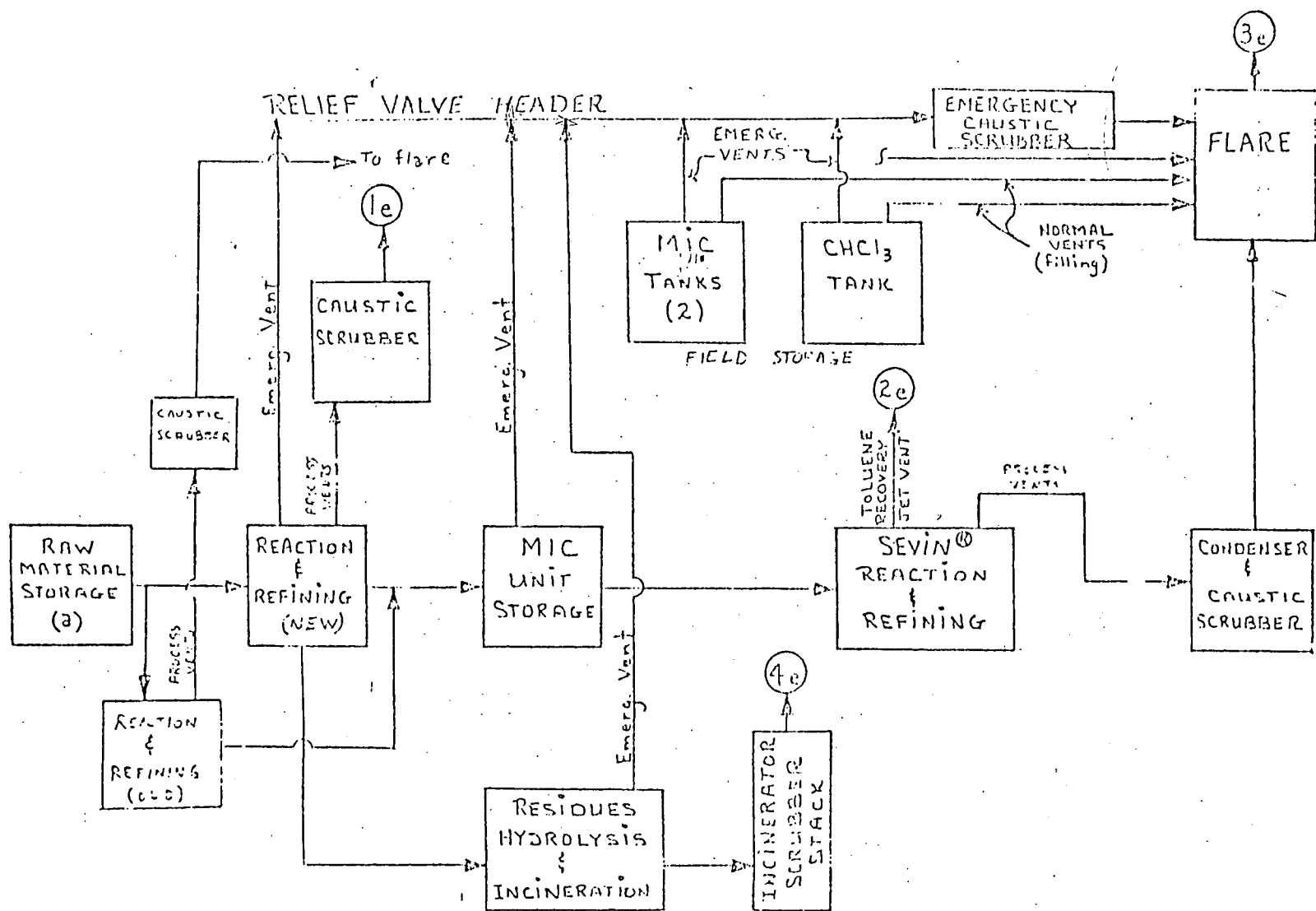


Figure 24. Methylisocyanate Process Schematic



(a) NO NORMAL EMISSIONS
(b) TWO SYSTEMS

Figure 25. SEVIN® Complex MIC to SEVIN® Process Schematic

and discharged through Outfall 005. The caustic scrubber waters are discharged to the WWTF along with the incinerator scrubber water. The emergency scrubber is used only when the flare is inoperable.

All air emissions, except the toluene recovery jet system, are collected and burned in a flare. The MIC and SEVIN[®] reaction and refining emissions are caustic scrubbed prior to burning in the flare. The MIC residues are hydrolyzed and burned in a liquid incinerator and the incinerator gases are caustic scrubbed prior to being discharged to the atmosphere. The toluene recovery vacuum jets discharge 3 kg (7 lb)/hr of toluene to the atmosphere.

Molecular sieves are used in this process and the spent sieves are buried in the Goff Mountain landfill.

ACROLEIN DERIVATIVES PRODUCTION

Several acrolein derivatives are produced by reacting acrolein with other raw materials [Figure 26]. The process equipment includes a reactor, settling pot, decanter and several distillation columns.

Wastewater sources include the settling pot, waste pit, decanter, still and refined tank wash, vacuum jet condenser and tank car wash area. The settling pot water and water from tank car cleaning are discharged to the waste pit to allow solids settling. The waste pit, decanter, still wash and jet condenser waters are discharged to the WWTF. Non-contact cooling water, 3,200 l/min (850 gpm), is discharged through Outfall 002.

Emissions from all equipment are collected and burned in a flare. There are no uncontrolled vents in this system.

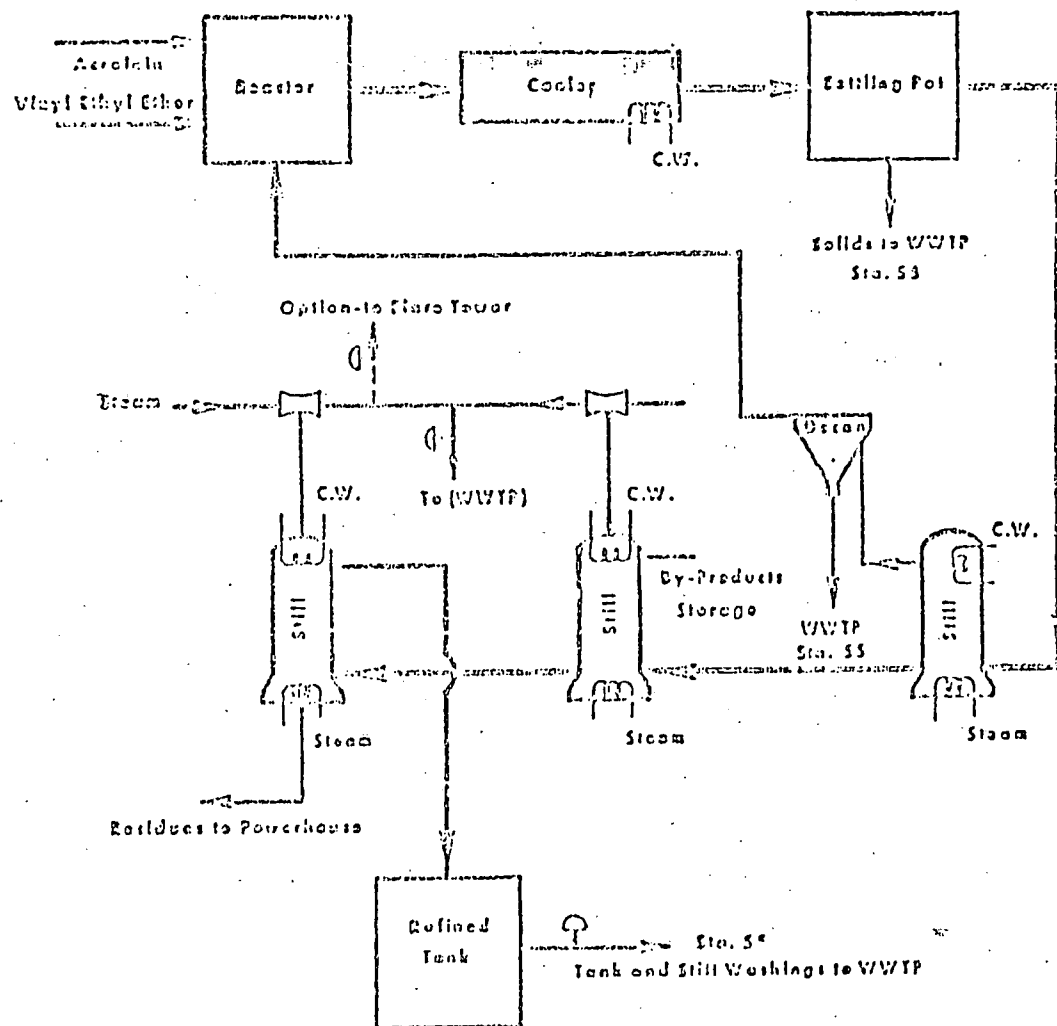


Figure 26. Acrolein Derivatives Process Schematic

Waste pit and still clean-out solids are normally disposed in the Goff Mountain landfill. At times, the solids from the waste pit are discharged to the WWTF. Still and refined tank residues are burned at the powerhouse.

ANHYDROUS ISOPROPANOL RECOVERY

Spent isopropanol is recovered and processed to anhydrous isopropanol in a series of distillation columns [Figure 27].

The only process wastewater source is the vacuum jet condensate. This condensate is discharged to the WWTF. The still water is returned to the ethylene glycol recovery system. Non-contact cooling water is discharged through Outfall 002.

All air emissions, except those from the storage tank, are collected and burned in a flare. The storage tank emissions are less than 0.5 kg (1 lb)/hr.

DINITROTOLUENE PRODUCTION

Dinitrotoluene (DNT) is produced by reacting toluene with nitric acid in the presence of sulfuric acid [Figures 28 and 29].

The wastewater sources include DNT wash, scrubbers water, and cooling tower blowdown. These wastes, 285 l/min (75 gpm), are discharged to a pond for solids settling before being discharged to the WWTF. Cooling water is provided from the unit cooling tower.

The reactor-DNT washer and the spent acid concentrator vents are the major air emission sources. The reactor-DNT washer emissions

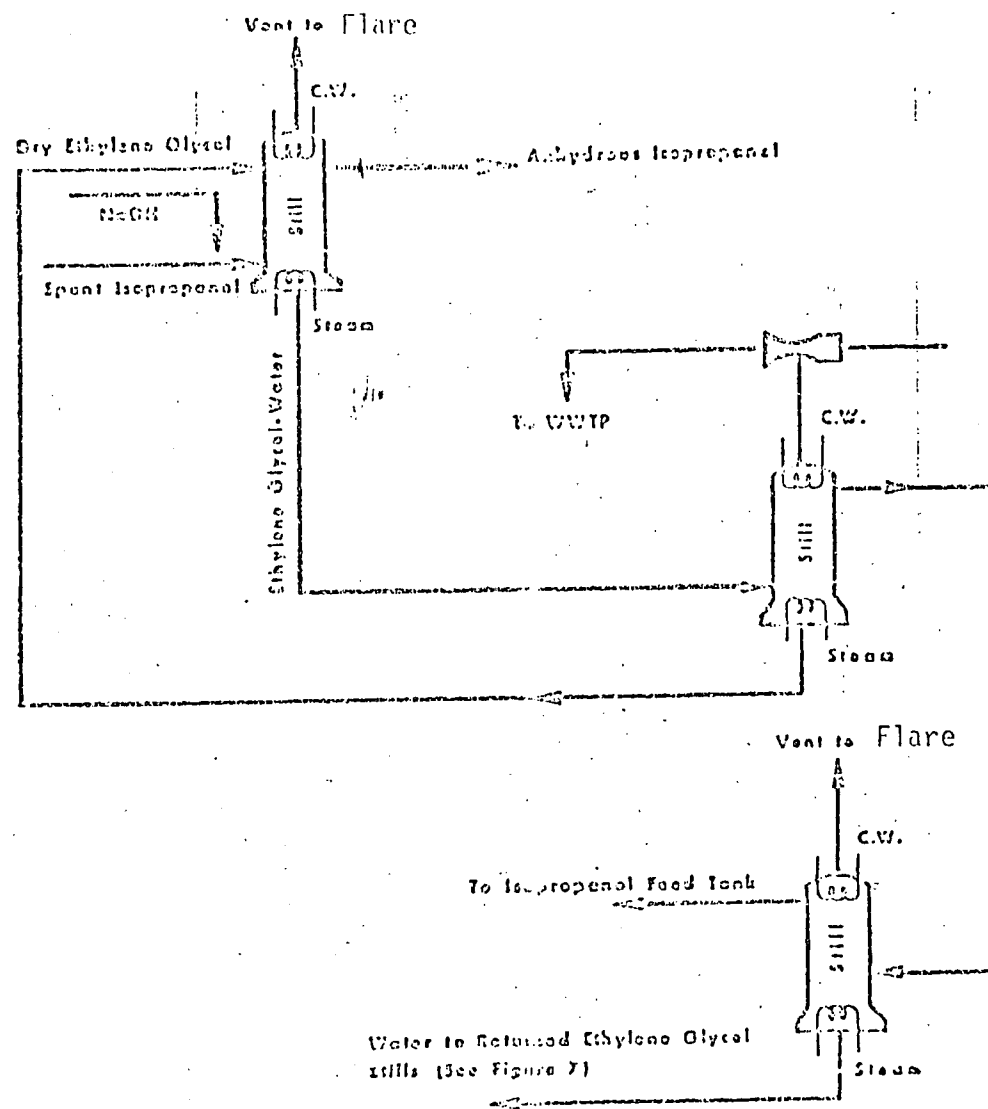


Figure 27. Anhydrous Isopropanol Recovery Process Schematic

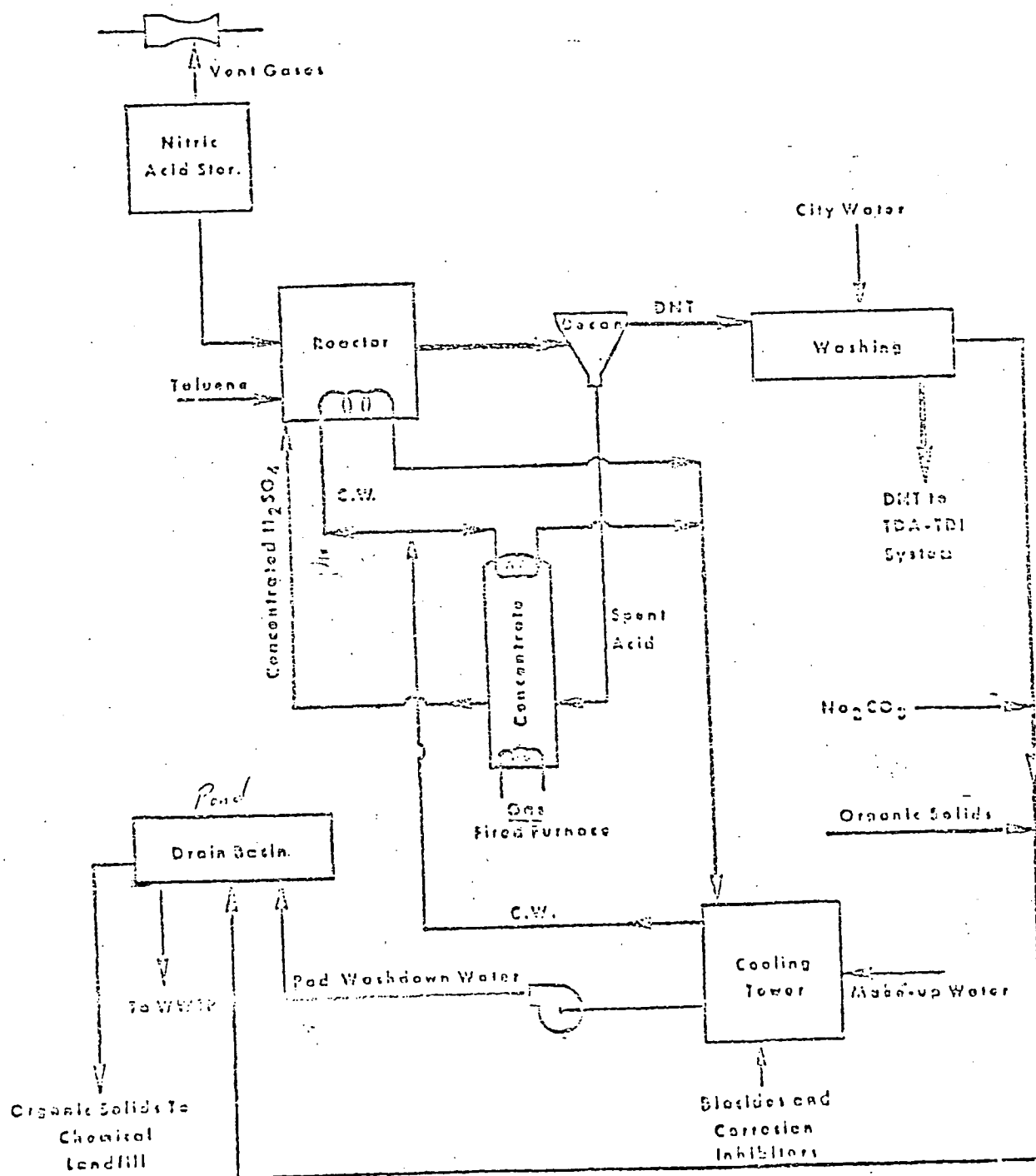


Figure 23. Dinitrotoluene (DNT) Process Schematic

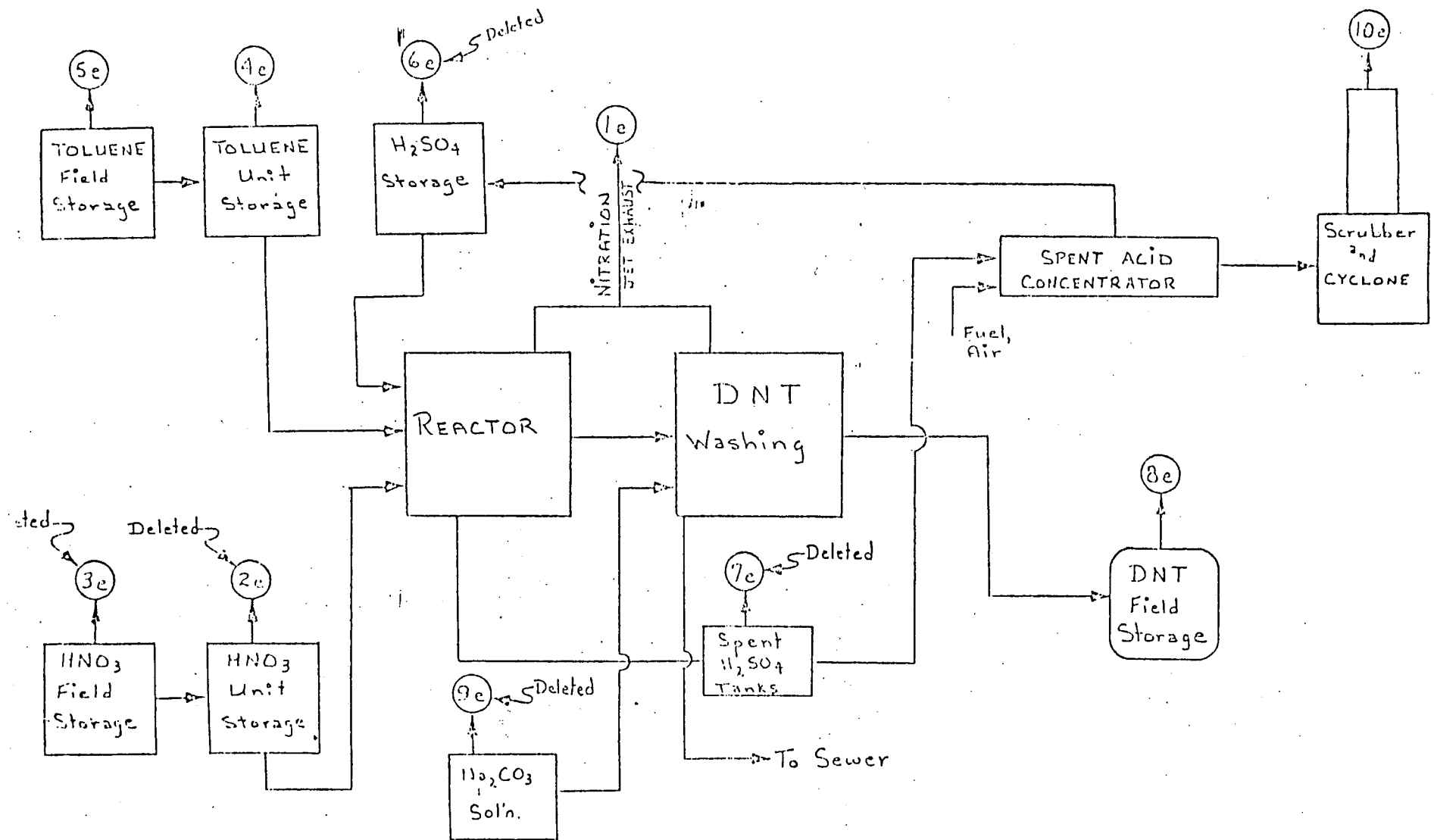


Figure 29. Dinitrotoluene Unit Schematic

are controlled by a water scrubber and the spent acid concentrator emissions are controlled with a venturi scrubber followed by a water scrubber. The spent acid concentrator vents are the major source of NO_x ; the emissions include 450 kg (100 lb)/hr of NO_x and 41 kg (9 lb)/hr of DNT. The DNT field storage vent is submerged in a sump that discharges to the WWTF. The toluene storage tank emissions are controlled by conservation vents.

About once/year, the solids are removed from the settling pond and buried in the Goff Mountain landfill.

TOLUENE DIAMINE PRODUCTION

Toluene diamine (TDA) is produced by reacting DNT in a solvent with hydrogen in the presence of a metal catalyst [Figure 30]. The resulting crude TDA is then refined in a series of stripping stills to obtain refined TDA. The methanol solvent is recycled to the process.

The major source of wastewater is from the secondary solvent removal still. This water containing methanol and TDA is discharged to the WWTF for treatment. Non-contact cooling water is discharged through Outfall 005.

There are 14 air emission points in this process and these emissions are controlled by nitrogen blanketing the process units. No vents discharge more than 5 kg (10 lb)/hr methanol and 0.25 kg (0.5 lb)/hr TDA. Still residues from the stills are burned at the powerhouse.

Normally there are no solid wastes from this process but if product problems occur (about once/year) the solids generated are disposed in the Goff Mountain landfill. The metal catalyst is recovered and reused.

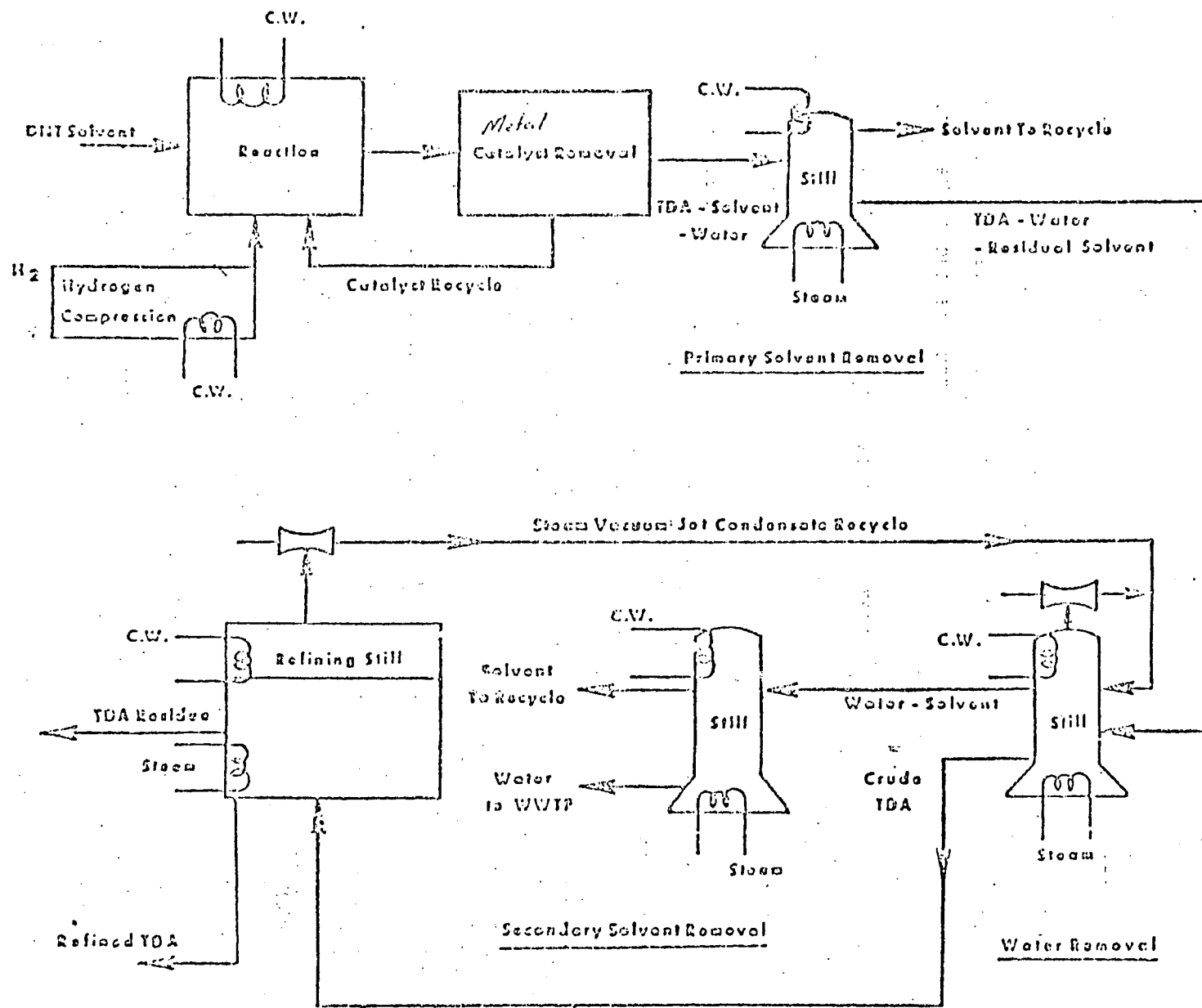


Figure 30. Toluene Diamine (TDA) Process Schematic

TOLUENE DIISOCYANATE PRODUCTION

TDA and phosgene react in a solvent (dichlorobenzene) to form toluene diisocyanate (TDI) [Figure 31]. Phosgene is extremely toxic, therefore all vapors containing phosgene must be collected and the phosgene destroyed.

Wastewater sources include the vent kettle (caustic scrubber to destroy phosgene) and the vacuum jet condensate from the ball mill. These wastes are discharged to the WWTF for treatment. Anhydrous hydrochloric acid (HCl) is produced in the phosgene recovery system. Normally this HCl is used in the methyl chloride plant but, in the event the methyl chloride plant is not operating, HCl is discharged to the neutralization pit, neutralized and discharged through Outfall 005 along with the non-contact cooling water.

All vapors from the reactor, degassers, and TDI refining are collected, caustic treated and burned in a flare. Emissions from all storage tanks are controlled with nitrogen blanketing and conservation vents. There are no emissions greater than 0.5 kg (1 lb)/day from this process.

Recovered TDI ball mill and clean-out solid wastes are buried in the Goff Mountain landfill. Evaporator residue and line flushings are drummed and contract disposed.

ALKYL BENZENE PRODUCTION

Several alkyl benzene (AB) products are made by reacting C_{10} through C_{15} paraffins, chlorine and benzene in the presence of an aluminum chloride catalyst [Figure 32]. Hydrochloric acid (HCl), a by-product, must be washed from the products.

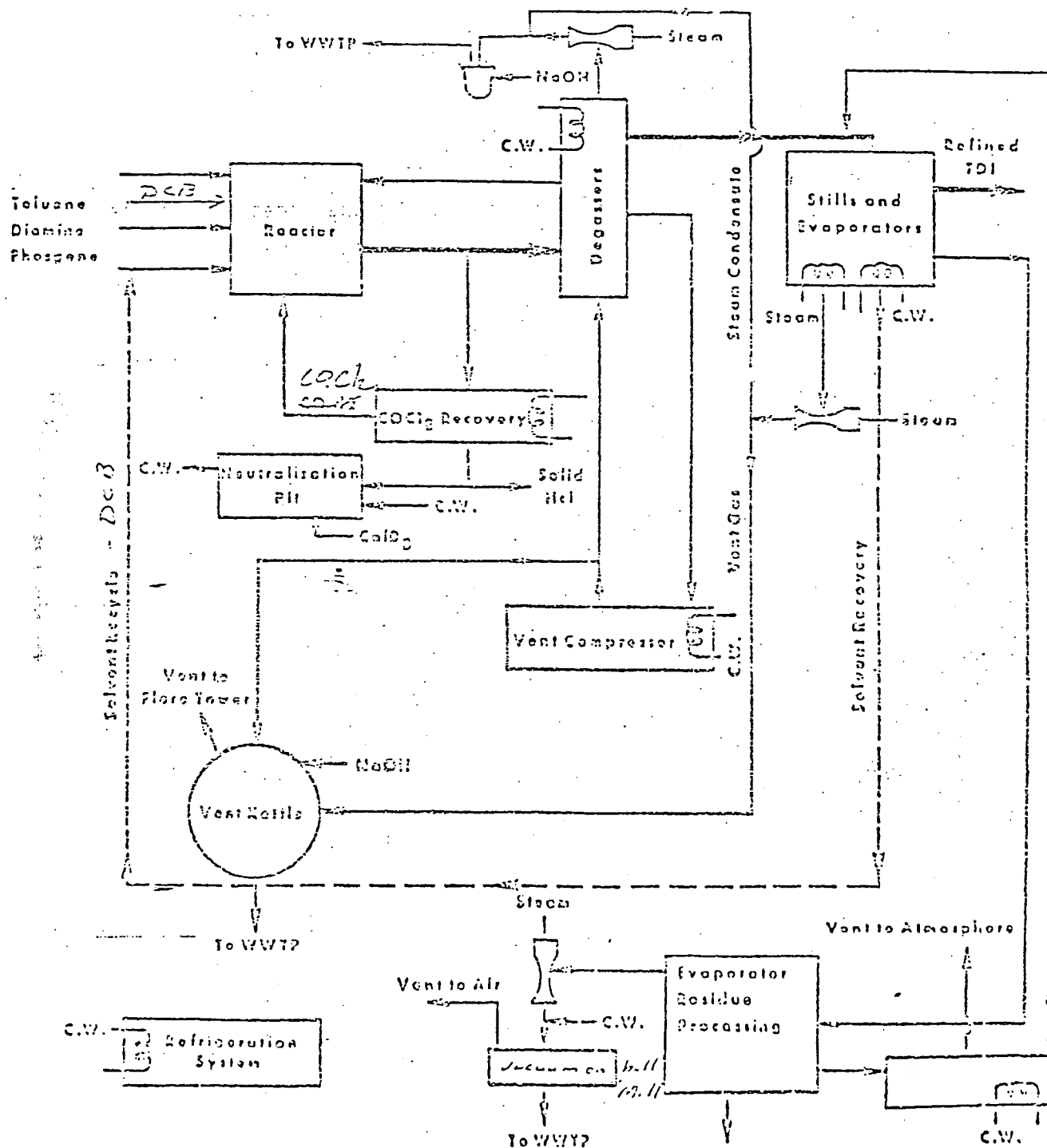


Figure 31. Toluene Diisocyanate (TDI) Process Schematic

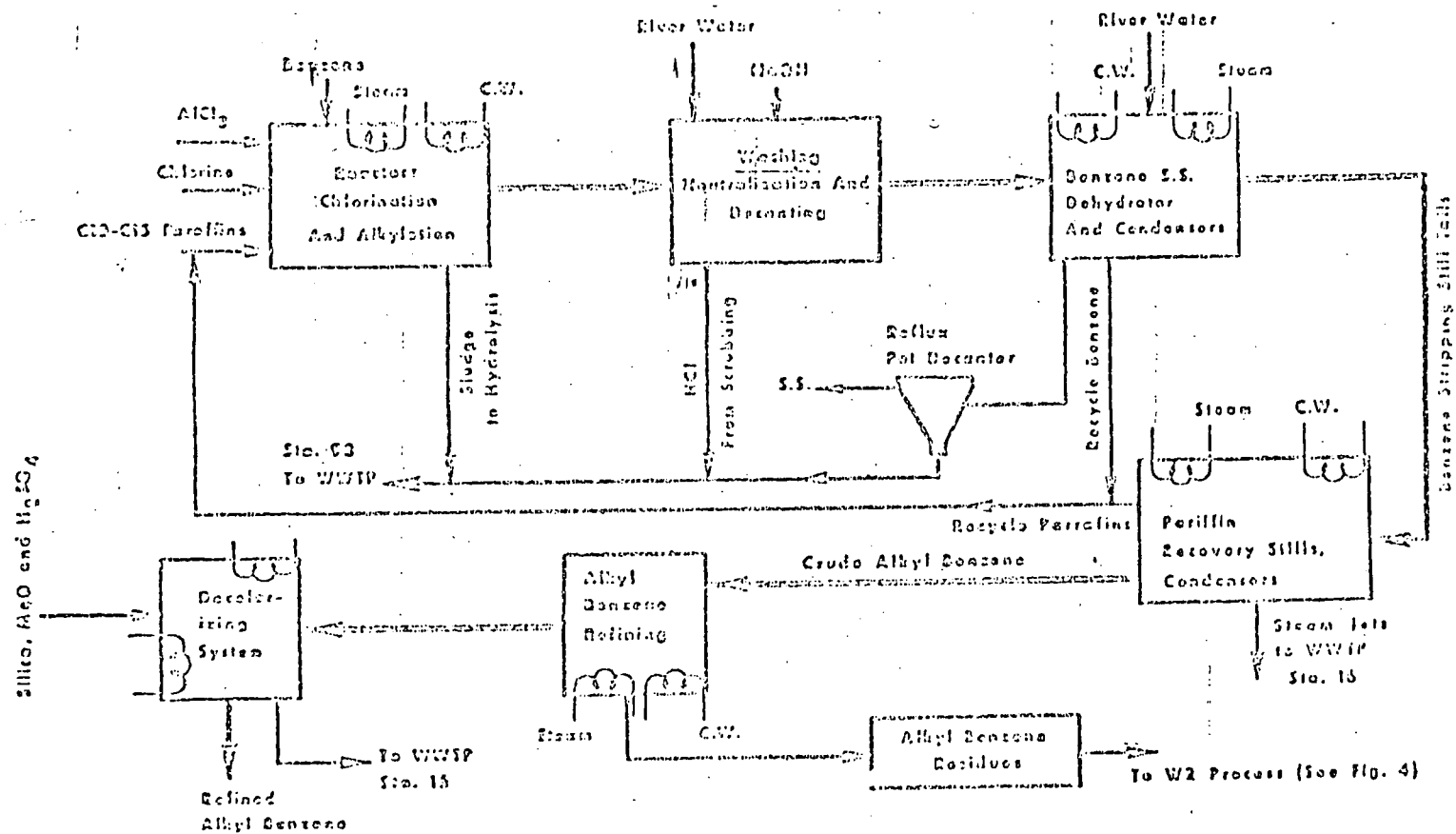


Figure 32. Alkyl Benzene Process Schematic

Wastewater sources include the hydrolysis unit scrubber water, the decanter water, vacuum jet condensate, the decolorizing water and neutralizer wash water. These wastes, 300 l/min (80 gpm), are discharged to the WWTF for treatment. Non-contact cooling water, 14 m³/min (3,700 gpm), is discharged through Outfall 005.

There are 13 air emission points from this unit and all except the sludge hydrolysis vent are controlled by nitrogen blanketing and conservation vents. The sludge hydrolysis emissions are controlled with a water scrubber. Up to 40 kg (90 lb)/hr of benzene is emitted from the storage tanks during tank filling.

Filter cones, drums, hydrolysis sludge and aluminum chloride catalyst are disposed in the Goff Mountain landfill.

LINDE[®] SYSTEM

The LINDE[®] system receives whole residues from the alkyl benzene unit and converts them to heavy and light residues in a distillation column [Figure 33].

The vacuum jet condensate is discharged to a sump where the oil and water are separated. The oil is burned at the powerhouse and the water and solids are discharged to the WWTF. This is the only wastewater source from the unit. Non-contact cooling water is discharged through Outfall 002.

METHYL CHLORIDE PRODUCTION

Methanol and hydrochloric acid react in the presence of an organic catalyst on silica to form crude methyl chloride. The crude product

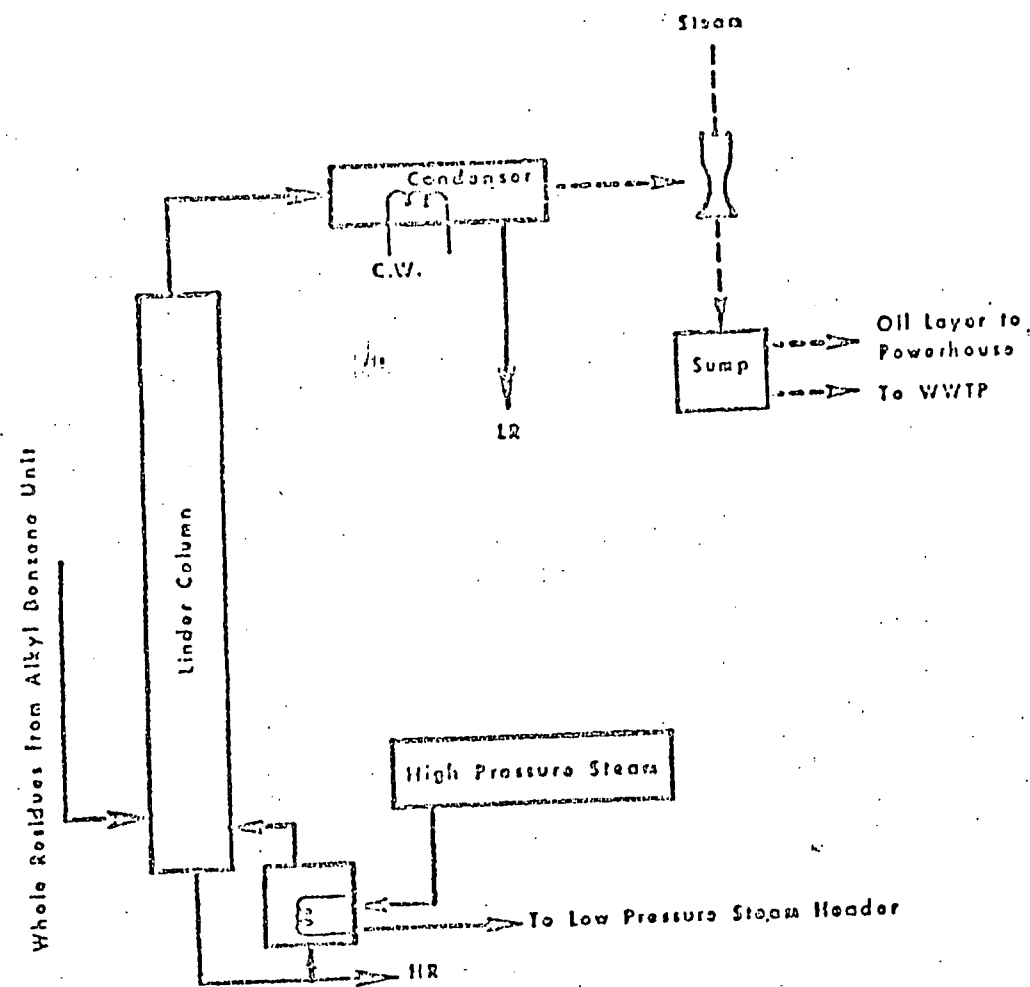


Figure 33. LINDE[®] System - Whole Residues Schematic

is scrubbed with sodium hydroxide, dried with molecular sieves and sulfuric acid (H_2SO_4), compressed and condensed to produce methyl chloride [Figure 34].

The major wastewaters are the methanol recovery still tails, spent H_2SO_4 , regenerator scrubber and the vacuum jet condensate. The still tails, scrubber water and jet condensate are discharged to the WWTF. The spent H_2SO_4 is collected and stored for use at the WWTF. Non-contact cooling water is discharged through Outfall 003.

There are 4 process and 5 storage tank vents in this process. The vapors from the product condenser, dryer and methanol still are water-scrubbed while the spent acid vent discharges to the atmosphere. The storage tank emissions are controlled by nitrogen blanketing. Only 1.8 kg (3.9 lb)/hr of methyl chloride is discharged to the atmosphere.

Spent molecular sieves and catalyst are neutralized and disposed in the Goff Mountain landfill.

UCARE[®] POLYMER JR PRODUCTION

The UCARE[®] Polymer JR System converts hydroxyethyl cellulose (HEC) to several cellulose polymers [Figure 35]. Chemicals used in this process are acetic acid, isopropanol, hydroxyethyl cellulose, and trimethyl amine.

The wastewater consists of the vacuum jet condensate, the flash pot water and the acetic acid scrubber effluent. The vacuum jet condensate and the flash pot water are collected and treated at the WWTF. The acetic acid scrubber media is burned at the powerhouse. Non-contact cooling water is discharged through Outfall 005.

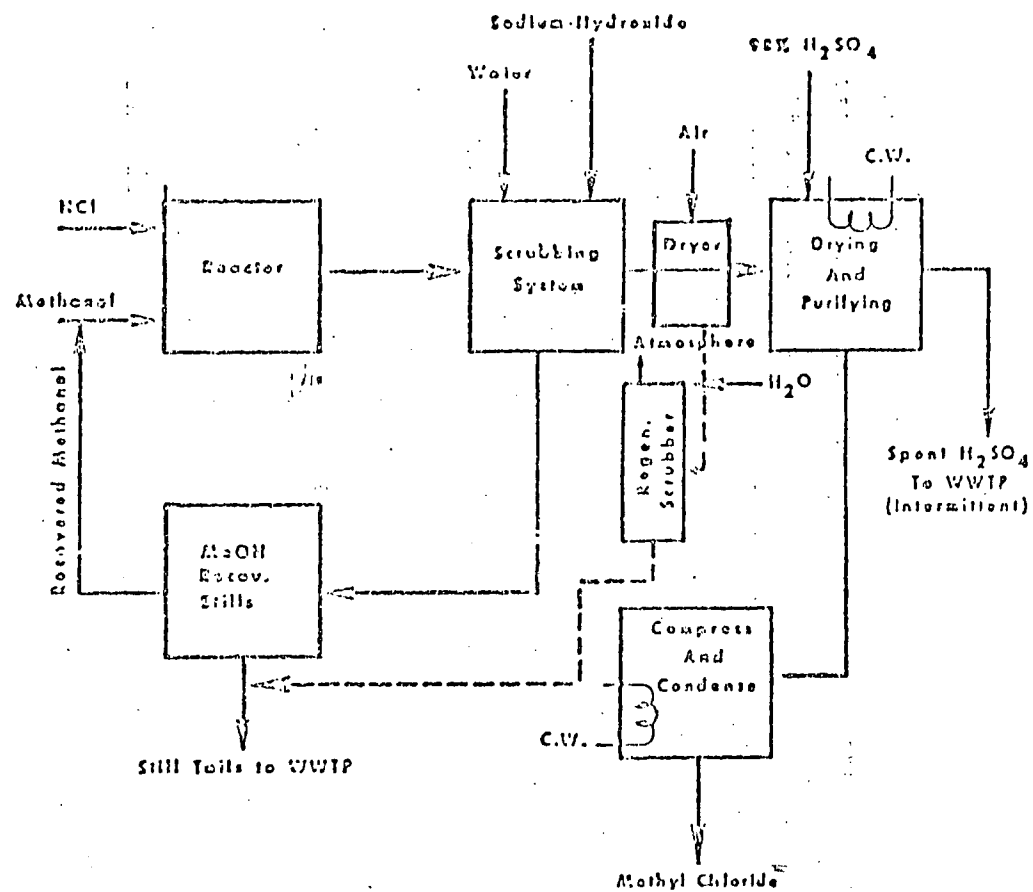


Figure 34. Methyl Chloride Process Schematic

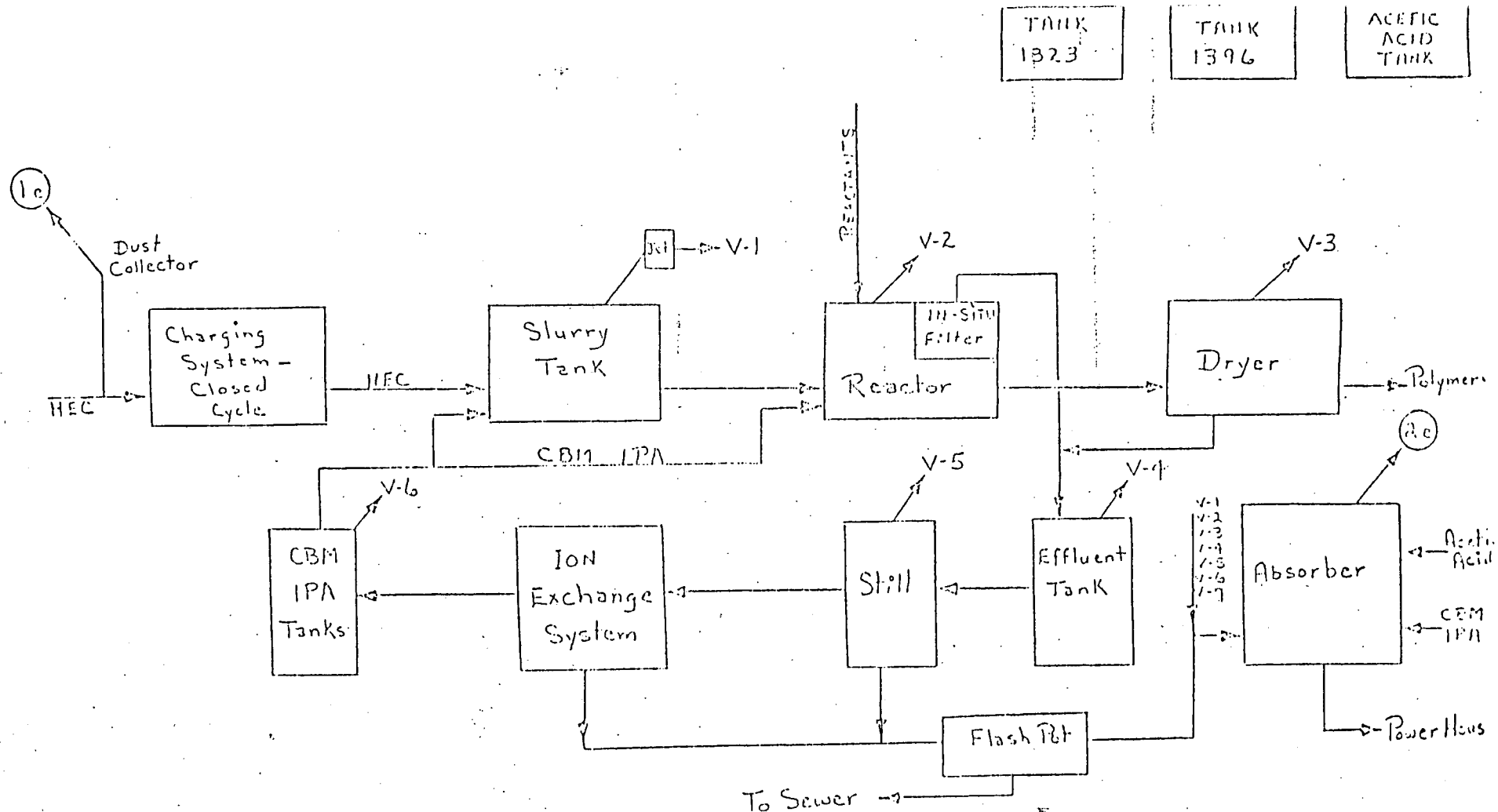


Figure 35. UCARE[®] Polymer JR System Schematic

Air pollution sources include the HEC charging system, process vents, acetic acid scrubber and storage tanks. The HEC charging system gas passes through a baghouse before being discharged to the atmosphere. The vapors from the process vents are collected and acetic acid scrubbed with small amounts discharged to the atmosphere. The emissions from the scrubber are minimized by maintaining a nitrogen blanket on the system. Storage tank emissions are controlled by nitrogen blanketing and nitrogen purging. Less than 2.3 kg (5 lb)/hr of isopropanol is discharged to the atmosphere from this system.

Solid wastes from the ion exchange unit, spilled product and filters are collected and disposed of in the Goff Mountain Landfill.

POLYOX[®] PRODUCTION

The POLYOX[®] unit produces ethylene oxide polymers by reacting ethylene oxide and isopentane in the presence of a rare earth catalyst [Figure 36].

The primary wastewater sources are the vacuum jets from the isopentane recovery system and the caustic scrubber on the drier. This water, 170 l/min (45 gpm), is discharged to the WWTF for treatment. Non-contact cooling water is discharged through Outfall 005. All burnable wastes are collected and burned at the powerhouse.

The drier vent, the solids conveying air process vents, and storage tanks are air pollution emission sources. The emissions from the storage tanks and the isopentane recovery system are controlled by conservation vents and nitrogen blanketing and the reactor vent is controlled with a pressure vent and nitrogen blanketing. The emissions from the drier are caustic scrubbed and vented to the atmosphere. The solids conveying and storage bin air discharge through baghouses before being vented to the atmosphere.

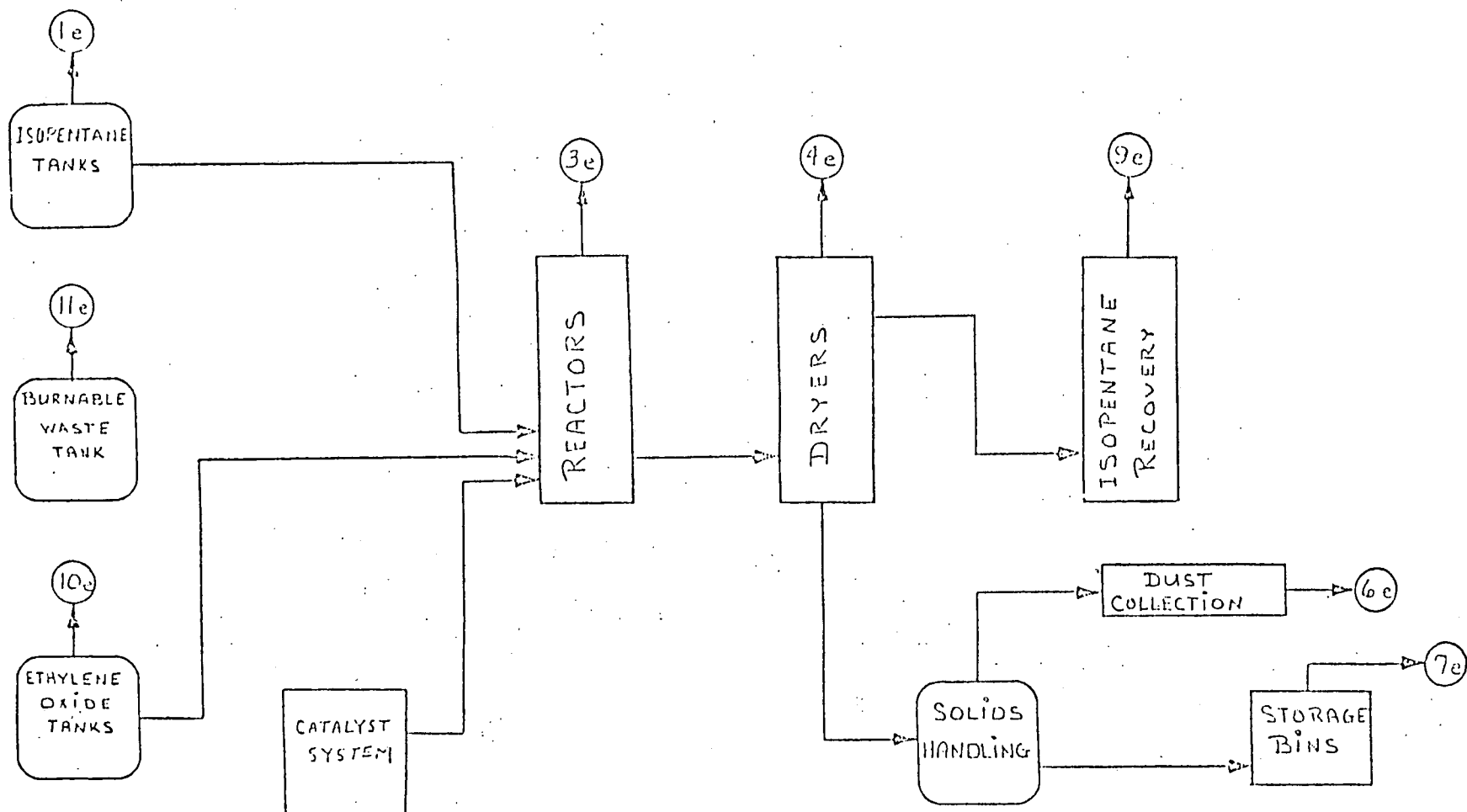


Figure 36. POLOYX® Unit Schematic

Solids from the baghouse and reactor clean-up, including the spent catalyst are disposed of in the Goff Mountain Landfill.

CATALYST PRODUCTION

There are three catalyst producing units and one catalyst metal recovery unit at this plant. The No. 1 catalyst unit produces a metal catalyst by reacting metals and acid with water and other additives [Figure 37]. This is a batch operation.

The only wastewater source is the water from the scrubber on the evaporator. This waste, 380 l/min (100 gpm), is discharged to the WWTF. Non-contact cooling and floor drain waters are discharged through Outfall 003.

There are two air emission sources that discharge NO_x . The vapors from the evaporator are water scrubbed and vented to the atmosphere and roaster gases are discharged through a stack to the atmosphere without control. NO_x [32 kg (70 lb)/hr] is emitted from these sources. During the inspection, emissions of greater than 50% opacity were noted for short periods.

Roaster clean-up, blending and shipping solids are collected and disposed of in the Goff Mountain landfill.

The No. 2 catalyst unit [Figure 38] and the catalyst metal recovery unit [Figure 39] are involved in metal recovery.

Wastewater sources include the roaster, demister, leach tank and precipitator. The leach tank and roaster waters, 1,800 l/min (480 gpm), are discharged to the WWTF for treatment. The demister water is recycled to the process and the precipitator water is used in the No. 3 catalyst unit. Non-contact cooling water and floor drains discharge through Outfall 004. Normally, there is no flow of cooling water.

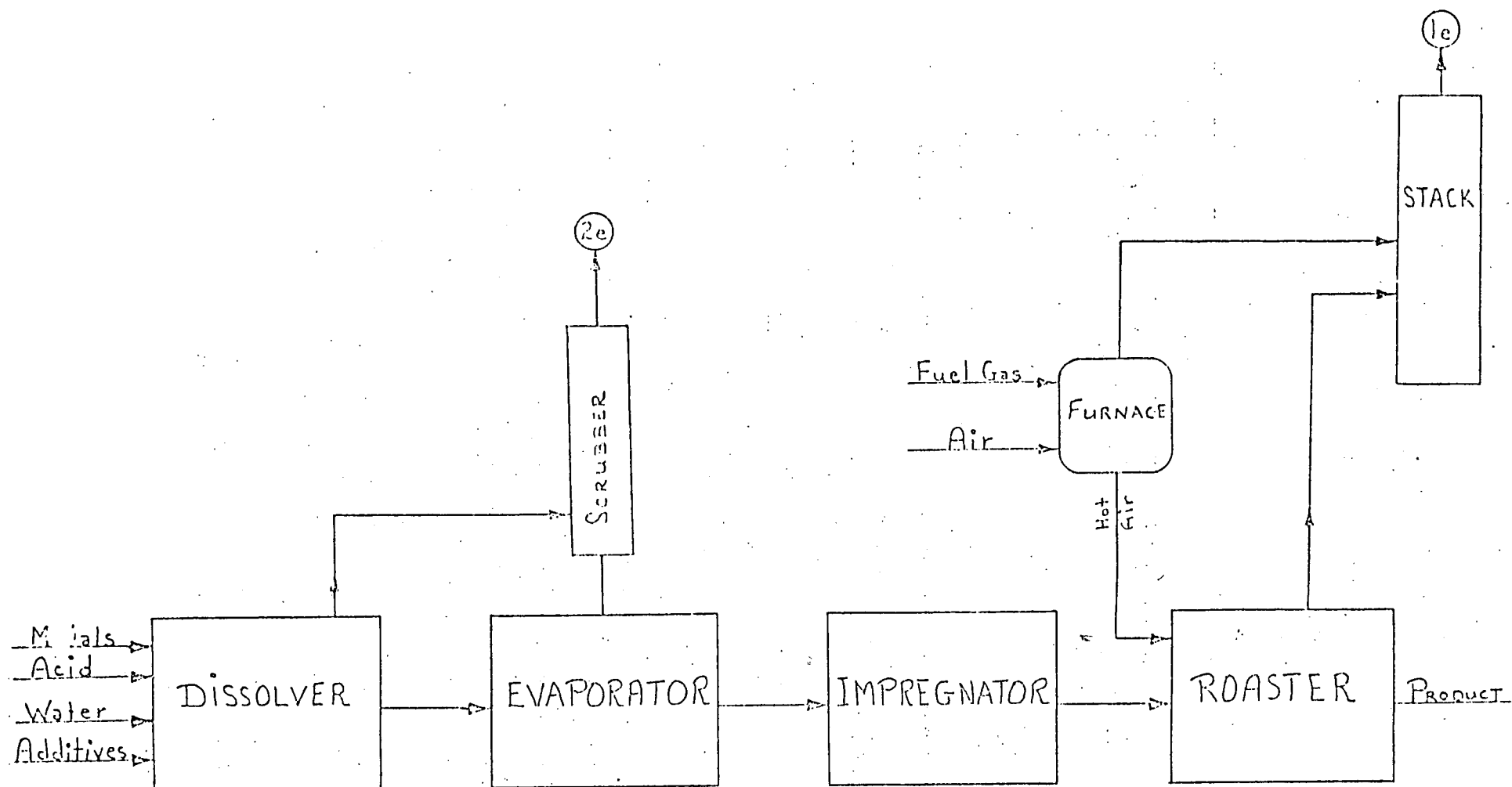


Figure 37. No. 1 Catalyst Unit Schematic

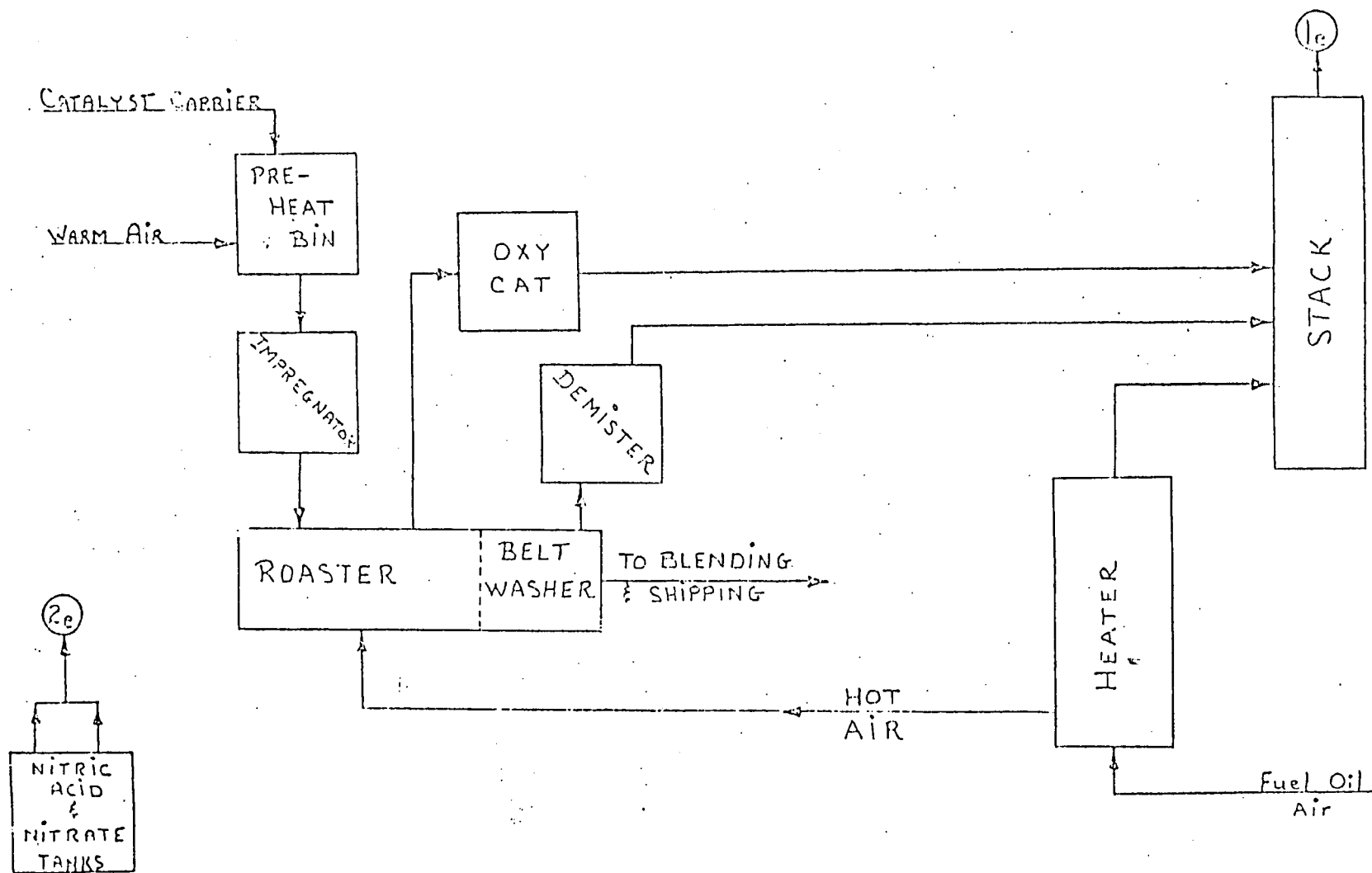


Figure 38. No. 2 Catalyst Unit Schematic.

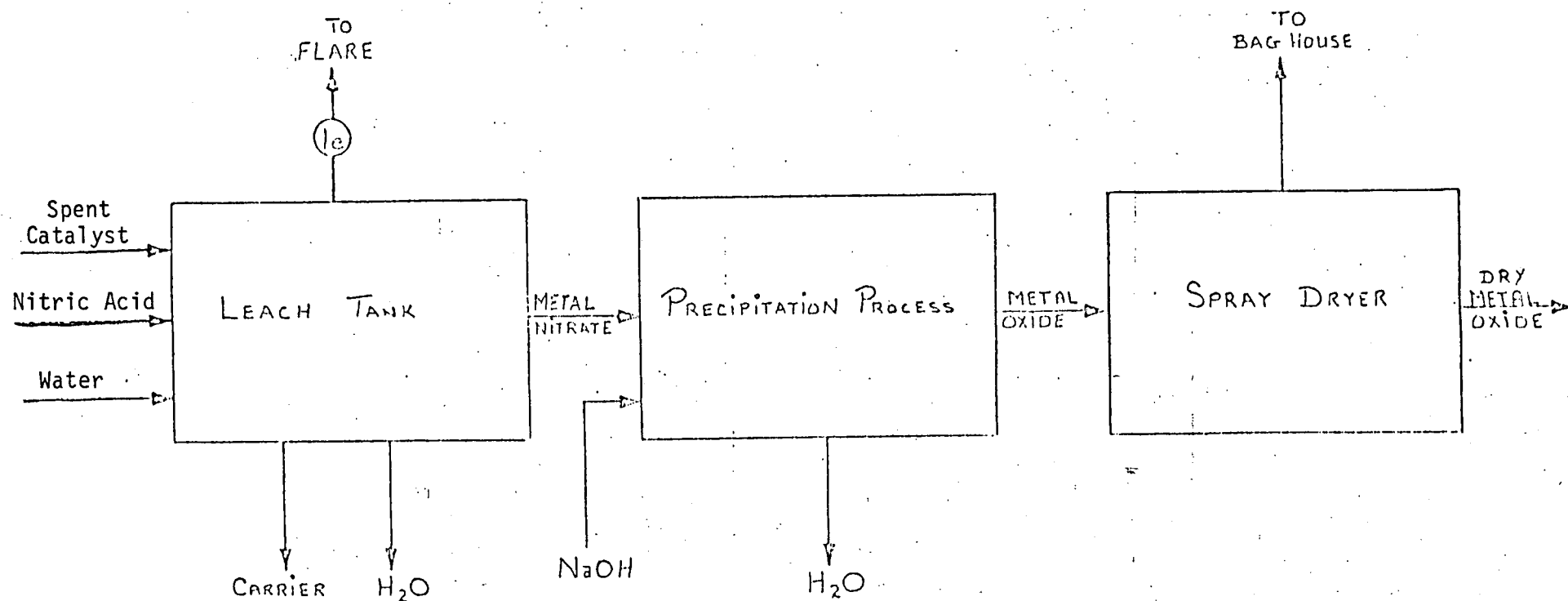


Figure 39. Catalyst Unit - Metal Recovery Schematic

Air emission sources include the No. 2 catalyst stack, the leach tank vent, the drier vent and the nitric acid storage tanks. The stack and leach tank emissions are burned in a flare and the nitric acid storage tanks are nitrogen blanketed. The drier vents to a baghouse and then to the atmosphere.

The No. 3 catalyst unit has no air emissions or solid wastes. The only wastewater source is from the settling tanks. Once per day 19 m^3 (5,000 gal) of water is discharged to the WWTF.

RIGID POLYOLS PRODUCTION

Rigid Polyols are produced by a series of batch reactors and distillation columns [Figure 40]. Raw materials used are ethylene oxide, propylene oxide, caprolactone, methylene chloride, and toluene diisocyanate.

The only wastewater sources are the reactor and refining jets. This water, 230 l/min (60 gpm), is discharged to the WWTF for treatment. Non-contact cooling water, $5 \text{ m}^3/\text{min}$ (1,300 gpm), is discharged through Outfall 005.

There are 12 emission points from this process. Vents 1, 2, 3, 4, 5 and 7 are controlled by vent jets followed by condensers and nitrogen blanketing. Vent 6 discharges through a resin bed to the atmosphere. Vent 8 discharges through a carbon adsorption column to the atmosphere. Vents 9, 10, 11 are nitrogen blanketed; vent 12 is also nitrogen blanketed and has a conservation vent. Methylene chloride is discharged to the atmosphere from the storage tanks during tank filling (30 kg-(65 lb/hr)).

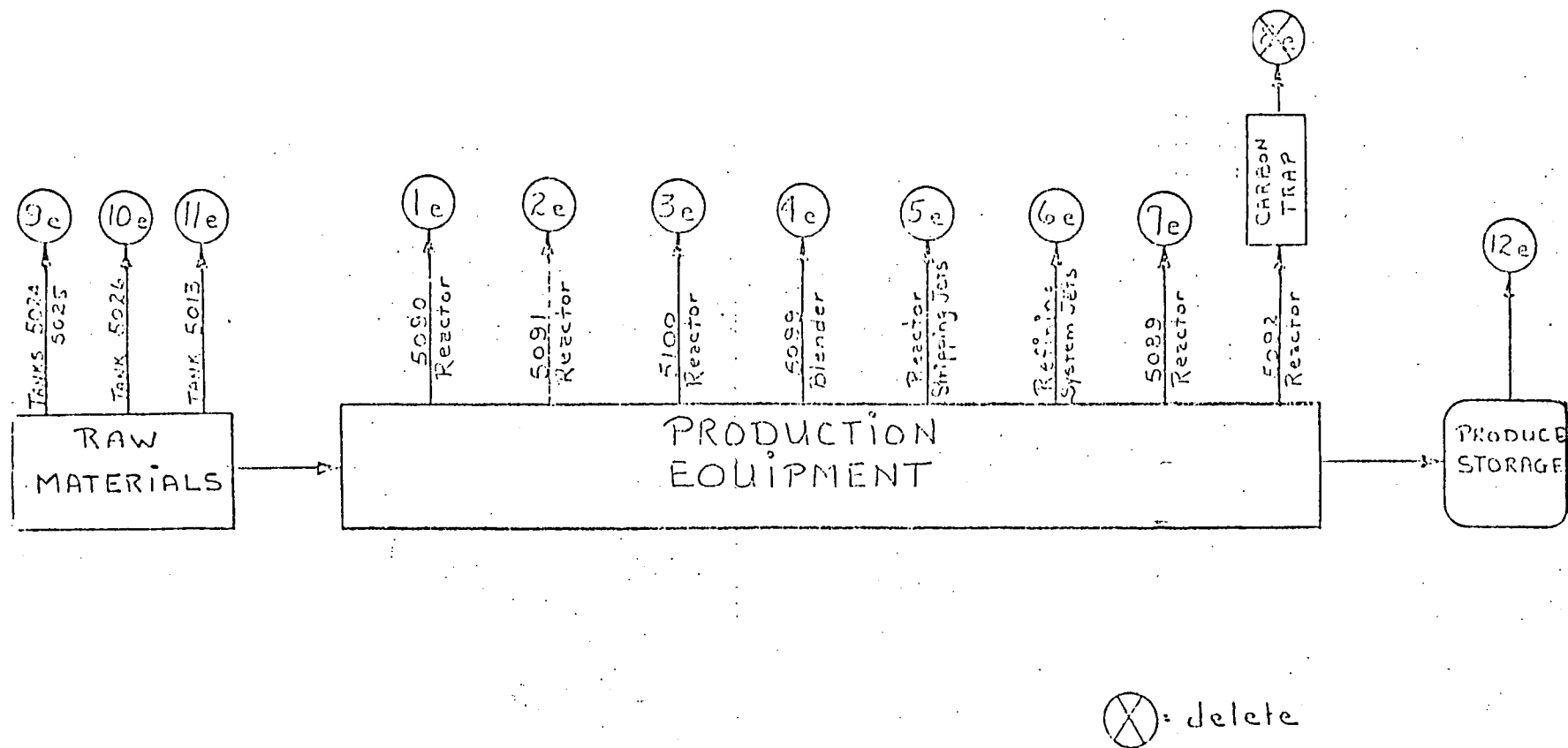


Figure 40. Rigid Polyols Unit Schematic

Solid waste sources include filter waste, used drums, mud slurry from sumps, trenches, carbon traps, resin bed and etc. These wastes are disposed of in the Goff Mountain landfill.

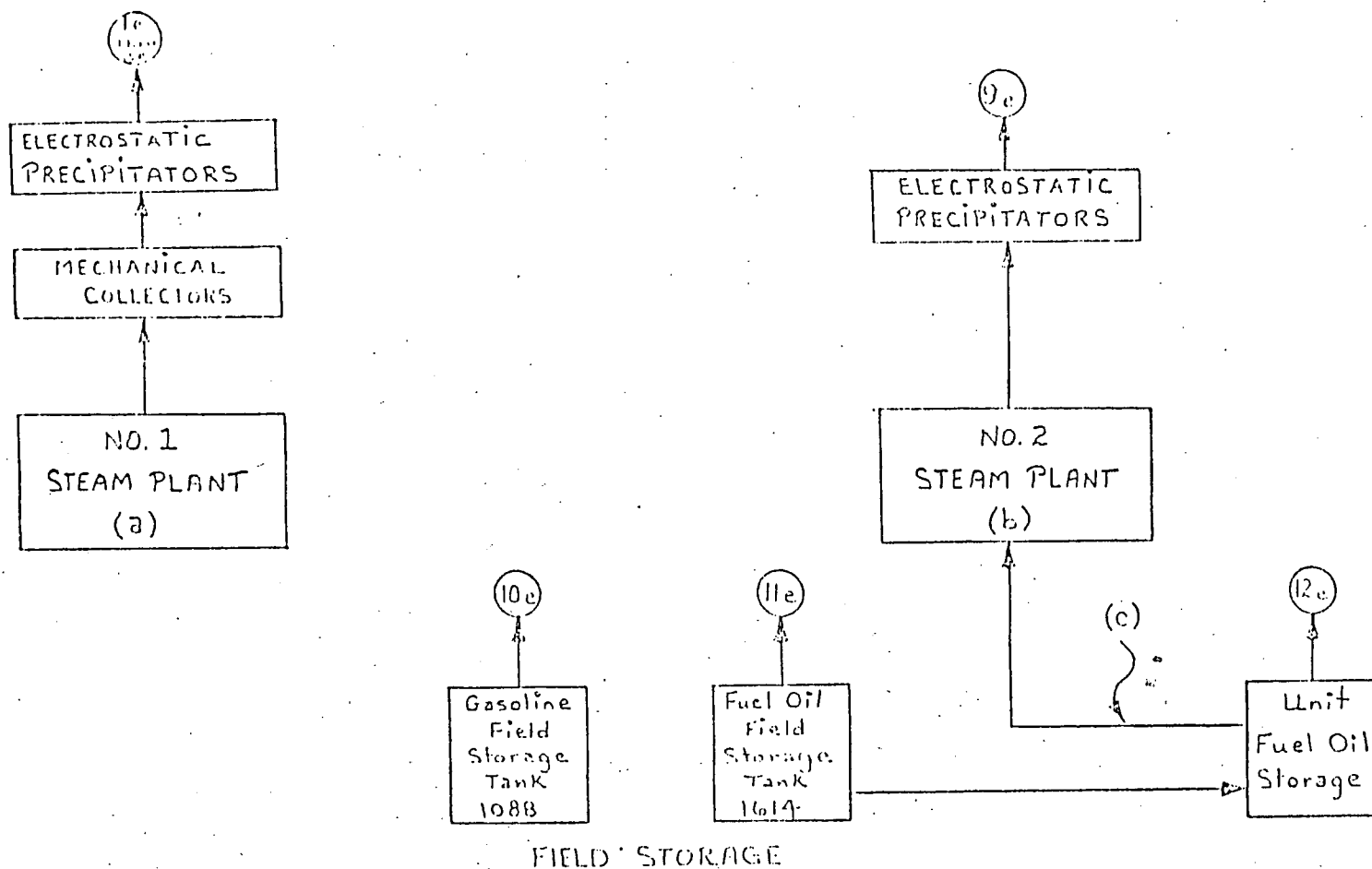
BOILERS

There are two powerhouses at this facility [Figure 41]. The No. 1 powerhouse has eight boilers; emissions are controlled by cyclones and electrostatic precipitators (ESP's). The ESP's were installed between 1968 and 1972 for particulate control. Sulfur dioxide (SO_2) is controlled by burning low sulfur (less than 1%) coal. Source tests, conducted by Union Carbide, indicate the particulate discharges were between 1.8 and 6.8 kg (4 and 15 lb)/hr/stack and the State Regulations allow 8.8 kg (19.3 lb)/hr/stack. The opacity of each of the 8 stacks is monitored by Bailey monitors.

The No. 2 powerhouse has three boilers and one stack. An ESP is currently being installed on each boiler. The Company planned for these ESP's to be operational by August 1978. In addition a Lear Siegler opacity monitor will be installed.

Boiler blowdown and softener backwash from both powerhouses are discharged to the WWTF. Cooling water from No. 1 powerhouse is discharged through Outfall 003 while the cooling water from No. 2 powerhouse is discharged through Outfall 008 (Goff Branch).

Fly ash and bottom ash are disposed of by a contractor (Cunningham Realty). In the event that the contractor cannot take the fly ash, the Company has an emergency fly ash pond. Water overflow from this pond is discharged through Outfall 008 (Goff Branch).



- (a) EIGHT BOILERS VENTED THRU SEPARATE STACKS.
 (b) THREE BOILERS VENTED THRU A SINGLE STACK.
 (c) AS STAND-BY ONLY AFTER COAL CONVERSION IS COMPLETED (AUGUST 1978)

Figure 41. General Systems Schematic

OTHER POLLUTION SOURCES

Domestic, shop and barge dock sump wastes are discharged to the WWTF. In addition, the WWTF receives truck washing waste from a Company-owned truck washing facility. The facility is not managed by UCI personnel.

III. POLLUTION ABATEMENT PRACTICES

The NPDES permit issued to the Union Carbide Institute Plant authorizes the discharge of wastewaters to the Kanawha River through eleven outfalls. The Company has eliminated five of these outfalls. Non-contact cooling water (NCCW) is discharged untreated to the river through Outfalls 002, 003, 004, 005 and 008.

Sanitary wastes, Goff Mountain landfill leachate, and process wastewaters are treated in an activated sludge wastewater treatment facility (WWTF) [Figure 42]. The process wastewaters are neutralized in the plant prior to being pumped to the WWTF. Higher than normal flows and spills are diverted into a $26,500 \text{ m}^3$ (7×10^6 gal) emergency storage pond and then slowly returned to the WWTF for treatment. The combined flow is discharged into two primary clarifiers, 945 m^3 (250,000 gal) each. The clarifier effluent is discharged into an equalization basin ($18,900 \text{ m}^3 - 5 \times 10^6$ gal) equipped with two 50HP aerators to keep the contents mixed. The equalization basin effluent pH is adjusted with either NaOH or H_2SO_4 . H_3PO_4 is added to provide a source of phosphorus as the wastewaters enter the three activated sludge basins ($18,900 \text{ m}^3 - 5 \times 10^6$ gal each). Each basin is equipped with ten aerators, rated at 75 to 100 HP each. The biologically treated wastewater then passes through three 945 m^3 (250,000 gal) final clarifiers and is discharged into the Kanawha River through Outfall 001.

Solids removed from the secondary clarifier are either returned to the activated sludge basins or wasted along with primary solids to a $1,040 \text{ m}^3$ (275,000 gal) thickener. The thickener overflow is returned to the neutralization pit. The thickened sludge (2% solids) is either piped or trucked to Holz Pond which is operated by Union Carbide South

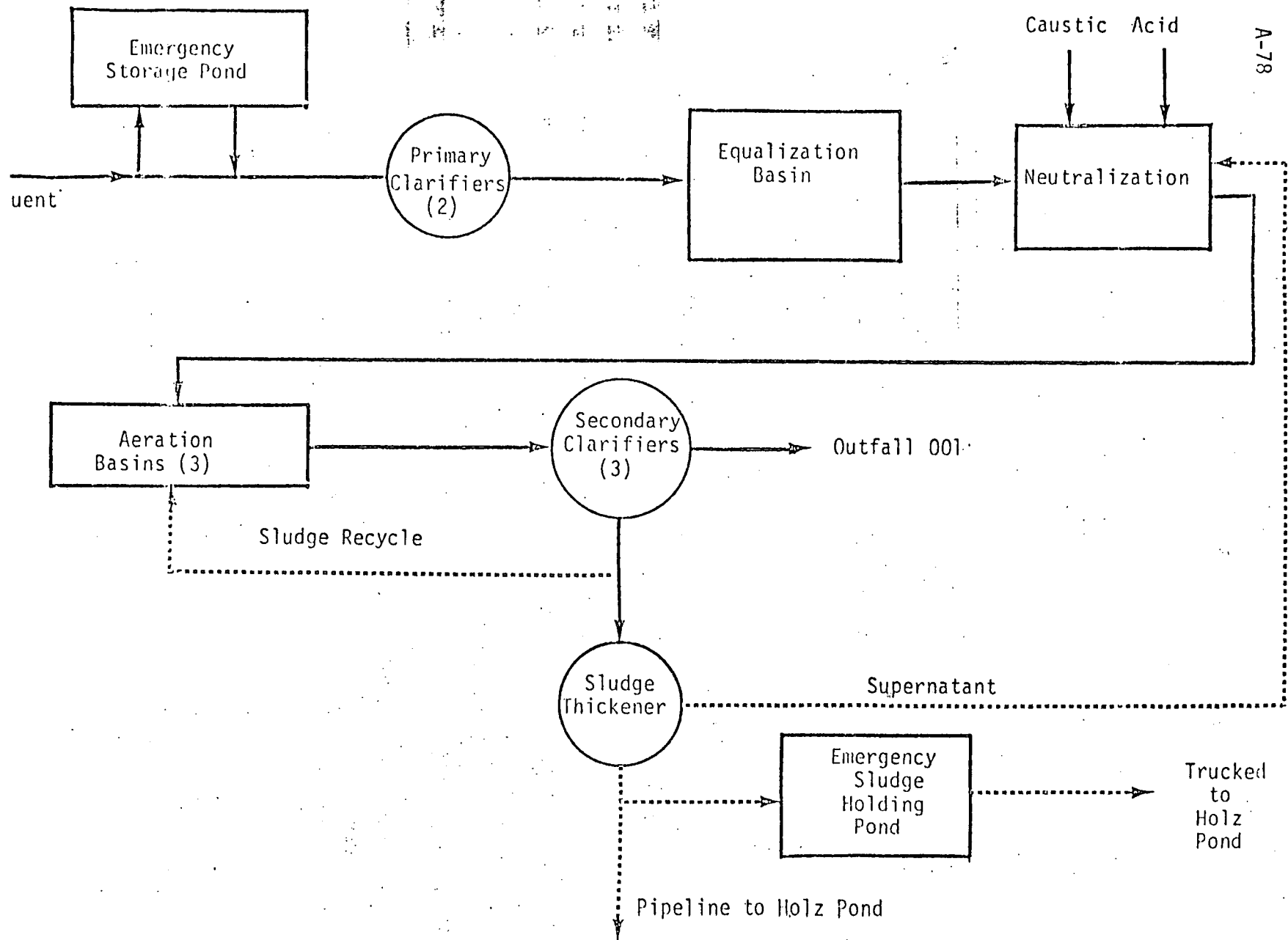


Figure 42. Schematic of Union Carbide Institute Plant Treatment Facilities

Charleston Plant. The supernatant from Holz pond is treated at the South Charleston Sewage Treatment Company.

The Company measures WWTF influent and effluent (Outfall 001) flows by means of a calibrated orifice and Marsh McBirney (Model 250) flow meter, respectively. In addition, the influent (Kennison Flow Nozzles) and effluent (Weir) flow from the aeration basins and the overflow (Kennison Flow Nozzle) from the sludge thickener are also measured. At the time of the NEIC visit, the Marsh McBirney flow meter was not operating. Therefore, effluent flows were calculated based on the influent to the aeration basins less the amount of sludge wasted. Company officials stated that the WWTF discharge normally varies between 17,000 and 18,900 m³/day (4.5 and 5 mgd).

The flow through the Outfalls 002, 003, 004, 005 and 008 is measured periodically by a lithium-tracer technique. Those results are compared to intake meter readings which, in turn, are used to determine daily flow. According to Company officials, these flows remain fairly constant.

Time-weighted composite samples from the cooling water discharges and the emergency ash pond overflow are collected in refrigerated sampling containers. In addition, each outfall is equipped with a total carbon analyzer (TCA) which is telemetered to the WWTF and monitored by the operators. If the TCA indicates a problem, grab samples are collected and analyzed on a GC to identify the source of the discharge.

WWTF effluent samples (Outfall 001) are composited by continuously pumping an equal volume aliquot into the glass sampling container. The Company also installed a TCA to continuously monitor the quality of the discharge.

In addition to the sampling and TCA installed to monitor the outfalls, the Company has installed several in-plant samplers, TCA

and GC units to monitor the process wastewaters. Company personnel use these data to identify spills, leaks, poor housekeeping practices, etc.

Union Carbide has done some organic chemicals monitoring in the effluent but the results were preliminary and were not available. During the inspection, NEIC personnel collected a grab sample from Outfall 001 to screen for toxic pollutants and other organics. The analyses showed a total of 30 organic chemicals present in the sample [Attachment A]. Of these, 13 were toxic pollutants and 7 had concentrations of 10 µg/l or greater. These were 1,2 dichlorobenzene (260 µg/l); 1,4 dichlorobenzene (13 µg/l); 2,6-dinitrotoluene (1,000 µg/l); 1,2-diphenylhydrazine (41 µg/l); methylene chloride (20 µg/l); naphthalene (99 µg/l) and bis (2-ethylhexyl) phthalate (25 µg/l). In addition, the insecticide carbaryl was detected at 260 µg/l. No nitrosamines were detected.

The Company has extensive air pollution controls including numerous scrubbers, flares, baghouses, and electrostatic precipitators. In 1977 an air emissions inventory was conducted at the plant. This inventory includes estimates of hydrocarbon, NO_x and SO₂ emission rates, from each vent.

Chemical solid waste, 44,500 kg (98,000 lb)/day is disposed of in the Company owned and operated Goff Mountain landfill along with the chemical solid wastes from the Union Carbide, South Charleston plant. A listing of materials disposed of in this landfill is presented in Attachment C. Trash (paper, wood, etc.) about 3,175mtons (3,500 tons)/yr is disposed in the Regional Development Authority Landfill located at Cross Lanes, West Virginia. WWTF waste sludge is collected and trucked to Holz pond (Company owned) in South Charleston; fly and bottom ash are contract disposed.

The Goff Mountain chemical landfill has been studied by E. D'Appolonia Consulting Engineers. The D'Appolonia Report [Attachment B] states that this landfill has a 0.61m (2 ft) thick selected clay seal to prevent leaching. In addition, the landfill has two extensive drainage systems. The first collects the contaminated waste from the landfill and the second collects the peripheral storm runoff. At the time of the inspection, the storm runoff collection system was in the final construction stages. The contaminated water is treated to remove the oil and retained in a pond. This waste is then discharged slowly to the WWTF for treatment. The peripheral storm runoff will be discharged to Goff Branch. The ground water in the area is sampled and analyzed for pH, total carbon, carbonate carbon and color from 4 wells at least once per quarter.

The landfill standard operating procedure is to surface blend one volume of earth with one volume of waste and then compact these. This earth/waste blending is a continuous operation six out of every eight working hours.

The Company keeps records of all material disposed in the landfill. This landfill is expected to be active until the year 2004.

IV. EVALUATION OF SELF-MONITORING PRACTICES

BIOASSAY PROCEDURES

The bioassay evaluation [Attachment C], conducted on April 11, 1978 shows 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 analyses required by the NPDES permit. Analyses are performed according to EPA-approved methods except for total dissolved solids (TDS) [Attachment D]. TDS is determined by subtracting the difference between total and suspended solids. This results in a value higher than the true value.

Samples collected on weekends are not analyzed until Monday. The TOC, $\text{NH}_3\text{-N}$ and TKN samples are not being cooled but are acidified as required to prevent sample degradation. Lack of proper preservation could result in low TOC and TKN values and higher $\text{NH}_3\text{-N}$ results.

The analytical quality assurance program consists of routine and blend duplicates, blend and spikes, and reference samples for all permit parameters. These procedures are continually being updated by the Union Carbide Technical Center.

SAMPLING PROCEDURES

As previously noted, Company personnel collect time-weighted composite samples from the NCCW and WWTF discharges. Company officials stated that as the discharge flows remain essentially constant, the resulting time-weighted samples should be flow proportional, as required by the NPDES permit.

Samples from the WWTF (Outfall 001) are collected from a manhole at the point where the clarifier discharges join. These samples may not be representative of the combined discharge. Company personnel should either verify that the samples are representative or relocate the sampling station.

Phenolic compound samples are aliquoted from the composite sample and not preserved during collection.

FLOW MONITORING

As previously noted, the flow measurement device on the WWTF discharge (Outfall 001) was not operating. Flows were being calculated based on the amount of wastewater entering the aeration basins, less the amount of sludge wasted. Provided these flow devices are calibrated properly, the resulting calculated value should be acceptable.

SELF-MONITORING DATA

Data from the Discharge Monitoring Reports (DMR's) for October 1977 through March 1978 are summarized in Tables 4 and 5. These data show that the Company has been in violation of permit limitations as shown in Table 6.

Table 4
SUMMARY OF DISCHARGE MONITORING REPORTS
UNION CARBIDE INSTITUTE PLANT
OUTFALL 001

Outfalls	Parameter ^a	Permit Limitations (Daily)		October (Daily)		November (Daily)		December (Daily)		January (Daily)		February (Daily)		April (Daily)	
		Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.
BOD-kg/day	May-Oct	2,270	5,000	397	1,090										
-lb/day		5,000	11,000	875	2,400										
-kg/day	Nov.-April	3,860	8,600			500	1,860	4,860	7,220	8,580	14,200	6,000	8,850	3,360	8,580
-lb/day		8,500	19,000			1,100	4,100	10,700	15,900	18,900	31,200	13,200	19,500	7,400	18,900
TSS-kg/day		1,270	1,910	4,000	46,800	590	3,540	730	1,680	6,580	24,600	4,000	27,100	1,230	12,400
-lb/day		2,800	4,200	8,800	103,000	1,300	7,800	1,600	3,700	14,496	54,200	8,800	59,700	2,700	27,300
TOC-kg/day	May-Oct.	4,450	10,000	1,860	3,900										
-lb/day			10,000	22,000	4,100	8,600									
-kg/day	Nov.-April	7,720	1,729			2,360	4,450	7,200	15,400	12,000	24,400	8,540	16,200	6,270	11,850
-lb/day		17,100	38,000			5,200	9,800	15,900	34,000	26,380	53,800	18,800	35,600	13,800	26,100
TKN-N-kg/day		1,500	3,000	1,040	3,000	860	1,540	1,270	2,000	1,360	1,950	1,140	1,900	730	1,730
-lb/day		3,300	6,600	2,300	6,600	1,900	3,400	2,800	4,400	3,000	4,300	2,500	4,200	1,600	3,800
NH ₃ -N-kg/day		680	1,360	450	1,040	540	1,270	270	730	210	270	185	820	210	680
-lb/day		1,500	3,000	1,000	2,300	1,200	2,800	600	1,600	470	600	406	1,800	460	1,500
Chlorides-kg/day		104,000	136,000	63,600	86,700	55,100	80,100	104,900	128,000	72,200	102,600	59,900	61,700	55,800	78,300
-lb/day		228,000	300,000	140,000	191,000	121,400	176,500	231,000	282,000	159,000	225,900	132,000	136,000	123,000	172,500
Fecal Coliform															
NO./100 ml		200	400	0	0	-	-	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Threshold Odor No.		512	1,024	32	32	128	128	-	-	512	512	-	-	-	-
Temp °C (°F)		N/A	43.3	-	23	-	22	-	23	-	16	-	20	-	21
			(110)		(73)		(72)		(74)		(60)		(68)		(69)
pH range		6.0-9.0		6.8-7.6		7.0-7.9		6.9-7.6		7.0-7.5		6.8-7.4		6.8-7.5	

^a Kg/day and °C calculated by NEIC.

Table 5
SUMMARY OF DISCHARGE MONITORING REPORTS
UNION CARBIDE INSTITUTE PLANT
NCCW Outfalls

Outfalls	Parameters ^a		Permit Limitations (Daily)		October (Daily)		November (Daily)		December (Daily)		January (Daily)		February (Daily)		March (Daily)	
			Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.	Avg.	Max.
002	TOC	mg/l	4	12	0.35	4	-	-	2.2	15	1.7	7	0.8	3	0.7	6
	TKN	mg/l	0.5	1.5	0.05	0.42	-	-	0.14	0.80	0.16	1.2	0.03	0.29	0.09	0.99
	NH ₃ -N	mg/l	0.2	0.6	0.02	0.12	-	-	0	0.03	0.001	0.42	0	0	0	0.06
	pH	range	6.0-9.0				6.9-7.8			5.6-11.5		7.0-10.8	7.3-8.7		6.5-8.8	
	Temp	°C (°F)	N/A	43.3(110)	-	25(77)			-	14(57)	-	9(40)	-	10(50)	-	18(65)
003	TOC	mg/l	4	12	0.5	3	0.65	4	2.4	13	4.8	21	2.6	16	3.1	14
	TKN	mg/l	0.5	1.5	0.06	0.39	0.02	0.09	0.15	1.28	0.14	0.9	0.08	0.94	0.24	1.46
	NH ₃ -N	mg/l	0.2	0.6	0.004	0.03	0	0	0	0.2	0.001	0.01	0	0.05	0	0.05
	pH	range	6.0-9.0			7.2-7.6	6.0-8.2		6.0-7.4		6.0-7.0		6.0-9.0		6.3-9.0	
	Temp	°C(°F)	N/A	43.3(110)	-	22(72)	-	23(74)	-	19(66)	-	14(57)	-	13(55)	-	16(61)
004	TOC	mg/l	4	12	1.3	8	4.6	62	1.9	14	3.6	22	2.3	19	7.2	64
	TKN	mg/l	0.5	1.5	0.05	0.24	0.15	0.36	0.24	1.5	0.34	1.5	0.10	0.32	0.66	3.41
	NH ₃ -N	mg/l	0.2	0.6	0.008	0.06	0.006	0.1	0	0.5	0	0.8	0	0.05	0.03	0.22
	pH	range	6.0-9.0			3.5-7.5	6.0-10.3		7.0-9.2		7.1-8.8		7.2-8.9		6.0-11.0	
	Temp	°C(°F)	N/A	43.3(110)	-	24(75)	-	29(85)	-	26(78)	-	20(68)	-	21(70)	-	24(75)
005	TOC	mg/l	4	12	1.6	29	2.5	16	5.6	28	2.4	10	1.8	8	1.8	10
	TKN	mg/l	0.5	1.5	0.27	1.23	0.34	1.41	0.33	0.79	0.32	1.5	0.36	2.2	0.06	0.27
	NH ₃ -N	mg/l	0.2	0.6	0.21	0.98	0.31	1.92	0.01	0.07	0.03	0.28	0.04	0.33	0.02	0.11
	pH	range	6.0-9.0			6.8-7.8	3.7-11.0		4.5-10.7		3.0-14.3		3.0-10.0		3.0-10.5	
	Temp	°C(°F)	N/A	43.3(110)	-	24(75)	-	23(73)	-	17(60)	-	12(54)	-	12(54)	-	16(60)
008	Sulfide	mg/l	N/A	N/A	0.06	0.24			0.03	0.08	0.03	0.08	0.1	0.4	0.54	1.84
	TOC	mg/l	4	12	0.4	3	0.75	4	1.3	9	3.7	12	3	16	12.8	101
	TKN	mg/l	0.5	1.5	0.06	0.58	0.02	0.2	0.1	0.83	0.42	0.78	0.32	1.85	0.18	1.08
	NH ₃ -N	mg/l	0.2	0.6	0.03	0.17	0	0.04	0.02	0.27	0.03	0.09	0.04	0.16	0.03	0.17
	pH	range	6.0-9.0			6.9-8.5	4.8-10.4		6.8-7.4		7.4-7.9		7.2-10		7.0-9.0	
Combined (002,003, 004,005, 008)	Temp	°C(°F)	N/A	43.3(110)	-	27(80)	-	31(88)	-	20(68)	-	13(56)	-	14(57)	-	27(80)
	Chlorid.	kg/day	137,000	182,000	60,500	94,500	46,400	96,600	82,900	116,300	94,600	111,900	78,600	95,500	81,400	97,200
		lb/day	302,000	400,000	133,155	208,250	102,202	212,875	182,500	256,004	208,384	246,568	173,200	210,365	179,340	214,200
	Phenol.	kg/day	32	82	2.8	4.0	2.7	5.5	5.0	8.4	27.6	138	0.8	3.0	2.6	4.6
		lb/day	70	180	6.2	8.8	5.9	12	11.1	18.4	60.8	305	1.7	6.6	5.8	10.1
	Threshold	Odor No.	128	256	4	4	-	-	-	-	-	-	-	-	-	-

^a kg/day and °C calculated by HEIC.

Table 6
SUMMARY OF PERMIT VIOLATIONS
OCTOBER 1977 THROUGH MARCH 1978

Parameter	No. Months Limitation Exceeded					
	Outfall					
	001	002	003	004	005	008
BOD	3	N/A	N/A	N/A	N/A	N/A
TSS	6	N/A	N/A	N/A	N/A	N/A
TOC	2	1	4	5	3	2
TKN	0	0	0	1	1	1
NH ₃ -N	0	0	0	1	2	0
Chlorides	1	N/A	N/A	N/A	N/A	N/A
pH	0	2	0	2	5	2

In addition, the combined discharges of Outfalls 002, 003, 004 ,005 and 008 also violated the daily maximum phenol limitation in January 1978.

ATTACHMENT A

ORGANIC CHEMICALS IDENTIFICATION

W. Smith
A-91

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.

South Charleston WWTC

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

* Tables II and III not included in this report; available upon request from NEIC.

Table I: Union Carbide Institute; Outfall 001 003-30-A

Compounds Identified and Confirmed

<u>Name</u>	<u>Concentration (ug/l)</u>
Acetone	a
Chloroform	310.
1,2-dichlorobenzene	260. #
Dicyclopentadiene	a
N,N-dimethylaniline	210.
2,6-dinitrotoluene	1000.
2-naphthol	b

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

b-unable to quantitate

Compounds Identified But Not Confirmed

<u>Name</u>
m-aminoacetophenone
1-butoxy-2-propanol
Carbon disulfide
3,4-dihydro-dimethyl-1(2H)-naphthalene isomer
2,5-dimethyl-1H-benzimidazole
2-methyl-1,3-dioxolane
2-methyl-2-pentanol
2-methylpropanenitrile
1,2,6-trimethylpiperidine
1,e,5-trimethyl-2,4,6-trioxohexahydrotriazine

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

TO : Mr. O. John Logsdon
Chemistry Coordinator
Kanawha River Study

DATE: April 25, 1978

FROM : K. E. Nottingham

SUBJECT: Nitrosamine Analysis of Kanawha River Samples

Results

Two samples were submitted for nitrosamine analysis. No nitrosamines were found in either sample. The detection limits are given below.

<u>Station #</u>	<u>Station Location</u>	<u>Date</u>	<u>Time</u>	<u>Sequence</u>	<u>Detection Limit of Dimethylnitrosamine</u>
003-030	U.C. Inst. Outfall 001	4/11/78	0900	A	<0.1 ug/l
003-045	SCWTC Outfall 001	4/12/78	1050	A	<0.05 ug/l

The dimethylnitrosamine detection limit for 003-045 was less than that of 003-030 due to the large amount of interferences in 003-030.

Methodology

One liter of the sample was extracted serially with two 50 ml portions of methylene chloride. The extracts were combined in a 250 ml Kuderna-Danish evaporative-concentrator. 0.7 ml of isooctane was added to the solvent extract and the mixture was concentrated to 1 ml at 60°C.

The extract was analysis by gas chromatography-thermal energy analysis.

Eric Nottingham
K. Eric Nottingham

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

TO : Mr. O. John Logsdon
Chemistry Coordinator
Kanawha River Study

DATE: April 24, 1978

FROM : K. E. Nottingham

SUBJECT: Carbaryl Analysis of Sample No. 003-030A from the Kanawha River Study

Results

Carbaryl was detected on a liquid chromatograph at a concentration of 260 ug/l.

Methodology

The use of liquid chromatography was necessary because Carbaryl can not be detected on a gas chromatograph due to thermal degradation of the compound in a heated injection port.

500 ml of the sample was extracted serially with two 50 ml portions of methylene chloride. The extracts were combined and passed through Na_2SO_4 into a 100 ml round bottom flask. 50 ml of ethyl acetate was added to the flask and the solvents were concentrated to 10 ml in a rotary evaporator at 45°C. The extract was passed through a clean-up column of 3 cm Florisil topped with 1 cm of Na_2SO_4 . The Carbaryl eluted with the 10 ml of ethyl acetate.

The extract was analyzed on a Waters 204 Liquid Chromatograph. A methanol-1% acetic acid gradient was used over 25 minutes at a flow rate of 2.0 ml/min. The gradient was run from 0 to 60% methanol. The dual channel UV detector was operated at wavelengths of 254 nm and 280 nm. The ratio of 254/280 response for both the standards and the Carbaryl peak in the sample was .38.

Eric Nottingham
K. Eric Nottingham

A-96

U.C. INST.

OUTFALL 001

STORET NO

STATION LOCATION: OUTFALL 001

LABORATORY SAMPLE NO: 003-30

DATE & TIME OF SAMPLE COLLECTION: 11APR78 0900 HRS

COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC.
001. ACENAPHTHENE	ND
002. ACROLEIN	NA
003. ACRYLONITRILE	NA
004. BENZENE*	ND
005. BENZIDINE	NA
006. CARBON TETRACHLORIDE (TETRACHLOROMETHANE)*	3.8
007. CHLOROBENZENE*	ND
008. 1,2,4-TRICHLOROBENZENE	ND
009. HEXACHLOROBENZENE	ND
010. 1,2-DICHLOROETHANE*	ND
011. 1,1,1-TRICHLOROETHANE*	ND
012. HEXACHLOROETHANE	ND
013. 1,1-DICHLOROETHANE*	ND
014. 1,1,2-TRICHLOROETHANE*	ND
015. 1,1,2,2-TETRACHLOROETHANE*	ND
016. CHLOROETHANE*	NA
017. BIS(CHLOROMETHYL) ETHER*	NA
018. BIS(2-CHLOROETHYL) ETHER	4.9
019. 2-CHLOROETHYL VINYL ETHER (MIXED)*	ND
020. 2-CHLORONAPHTHALENE	ND
021. 2,4,6-TRICHLOROPHENOL	ND
022. PARACHLOROMETA CRESOL	ND
023. CHLOROFORM (TRICHLOROMETHANE)*	ND
024. 2-CHLOROPHENOL	ND
025. 1,2-DICHLOROBENZENE	260.
026. 1,3-DICHLOROBENZENE	6.6
027. 1,4-DICHLOROBENZENE	13.
028. 3,3'-DICHLOROBENZIDINE	NA
029. 1,1-DICHLOROETHYLENE*	ND
030. 1,2-TRANS-DICHLOROETHYLENE*	ND

* VOA ANALYSES (UNPRESERVED/PRESERVED)

T TRACE

NA NOT ANALYZED FOR

ND NOT DETECTED

NAI NOT ABLE TO ANALYZE DUE TO INTERFERENCE

U.C. INST.
 OUTFALL 001
 STORET NO
 STATION LOCATION: OUTFALL 001
 LABORATORY SAMPLE NO: 003-30
 DATE & TIME OF SAMPLE COLLECTION: 11APR78 0900 HRS
 COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC.
031. 2,4-DICHLOROPHENOL	ND
032. 1,2-DICHLOROPROPANE*	ND
033. 1,3-DICHLOROPROPYLENE*	ND
034. 2,4-DIMETHYLPHENOL	ND
035. 2,4-DINITROTOLUENE	ND
036. 2,6-DINITROTOLUENE	1000.
037. 1,2-DIPHENYLHYDRAZINE	41.
038. ETHYLBENZENE*	ND
039. FLUORANTHENE	ND
040. 4-CHLOROPHENYL PHENYL ETHER	NA
041. 4-BROMOPHENYL PHENYL ETHER	ND
042. BIS(2-CHLOROISOPROPYL) ETHER	ND
043. BIS(2-CHLOROETHOXY) METHANE	ND
044. METHYLENE CHLORIDE (DICHLOROMETHANE)*	20.
045. METHYL CHLORIDE (CHLOROMETHANE)*	NA
046. METHYL BROMIDE (BROMOMETHANE)*	NA
047. BROMOFORM (TRIBROMOMETHANE)*	ND
048. DICHLOROBROMOMETHANE*	ND
049. TRICHLOROFLUOROMETHANE*	ND
050. DICHLORODIFLUOROMETHANE	NA
051. CHLORODIBROMOMETHANE*	ND
052. HEXACHLOROBTADIENE	ND
053. HEXACHLOROCYCLOPENTADIENE	ND
054. ISOPHORONE	2.2
055. NAPHTHALENE	99.
056. NITROBENZENE	ND
057. 2-NITROPHENOL	ND
058. 4-NITROPHENOL	ND
059. 2,4-DINITROPHENOL	ND
060. 4,6-DINITRO-O-CRESOL	ND

* VOC ANALYSES (UNPRESERVED/PRESERVED)

T TRACE

NA NOT ANALYZED FOR

ND NOT DETECTED

NAI NOT ABLE TO ANALYZE DUE TO INTERFERENCE

U.C. INST.
 OUTFALL 001
 STORET NO
 STATION LOCATION: OUTFALL 001
 LABORATORY SAMPLE NO: 003-30
 DATE & TIME OF SAMPLE COLLECTION: 11APR78 0900 HRS
 COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC.
061. N-NITROSODIMETHYLAMINE	ND
062. N-NITROSODIPHENYLAMINE	ND
063. N-NITROSODI-N-PROPYLAMINE	ND
064. PENTACHLOROPHENOL	ND
065. PHENOL (4AAP)	3.5
066. BIS(2-ETHYLHEXYL) PHTHALATE	25.
067. BUTYL BENZYL PHTHALATE	ND
068. DI-N-BUTYL PHTHALATE	ND
069. DI-N-OCTYL PHTHALATE	ND
070. DIEHTYL PHTHALATE	1.2
071. DIMETHYL PHTHALATE	ND
072. BENZO(A)ANTHRACENE (1,2 BENZANTHRACENE)	ND
073. BENZO(A)PYRENE (3,4-BENZOPYRENE)	ND
074. 3,4-BENZOFUORANTHENE	ND
075. BENZO(K)FLUORANTHENE (11,12-BENZOFUORANTHENE)	ND
076. CHRYSENE	ND
077. ACENAPHTHYLENE	ND
078. ANTHRACENE	ND
079. BENZO(G,H,I)PERYLENE (1,12-BENZOPERYLENE)	NA
080. FLUORENE	ND
081. PHENANTHRENE	ND
082. DIBENZO (A,H)ANTHRACENE	NA
083. INDENO (1,2,3-CD)PYRENE	NA
084. PYRENE	ND
085. TETRACHLOROETHYLENE*	ND
086. TOLUENE*	ND
087. TRICHLOROETHYLENE*	ND
088. VINYL CHLORIDE (CHLOROETHYLENE)*	NA
089. ALDRIN	ND
090. DIELDRIN	ND

* VOA ANALYSES (UNPRESERVED/PRESERVED)

T TRACE

NA NOT ANALYZED FOR

ND NOT DETECTED

NAI NOT ABLE TO ANALYZE DUE TO INTERFERENCE

U.C. INST.
 OUTFALL 001
 STORET NO
 STATION LOCATION: OUTFALL 001
 LABORATORY SAMPLE NO: 003-30
 DATE & TIME OF SAMPLE COLLECTION: 11APR78 0900 HRS
 COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC.
091. CHLORANE (TECH, MIXTURE & METABOLITES)	ND
092. 4,4'-DDT	ND
093. 4,4'-DDE	ND
094. 4,4'-DDD (P,P'-TDE)	ND
095. A-ENDOSULFAN-ALPHA	ND
096. B-ENDOSULFAN-BETA	ND
097. ENDOSULFAN SULFATE	ND
098. ENDRIN	ND
099. ENDRIN ALDEHYDE	ND
100. HEPTACHLOR	ND
101. HEPTACHLOR EPOXIDE	ND
102. A-BHC-ALPHA	ND
103. B-BHC-BETA	ND
104. G-BHC-(LINDANE)-GAMMA	ND
105. G-BHC-DELTA	ND
106. PCB-1242 (AROCOR 1242)	ND
107. PCB-1254 (AROCOR 1254)	ND
108. PCB-1221 (AROCOR 1221)	ND
109. PCB-1232 (AROCOR 1232)	ND
110. PCB-1248 (AROCOR 1248)	ND
111. PCB-1260 (AROCOR 1260)	ND
112. PCB-1016 (AROCOR 1016)	ND
113. TOXAPHENE	ND
114. ANTIMONY (TOTAL)	NA
115. ARSENIC (TOTAL)	NA
116. ASBESTOS (FIBROUS)	NA
117. BERYLLIUM (TOTAL)	NA
118. CADMIUM (TOTAL)	NA
119. CHROMIUM (TOTAL)	NA
120. COPPER (TOTAL)	NA

* VOA ANALYSES (UNPRESERVED/PRESERVED)
 T TRACE
 NA NOT ANALYZED FOR
 ND NOT DETECTED
 NAI NOT ABLE TO ANALYZE DUE TO INTERFERENCE

A-100

U.C. INST.
OUTFALL 001

STORET NO

STATION LOCATION: OUTFALL 001

LABORATORY SAMPLE NO: 003-30

DATE & TIME OF SAMPLE COLLECTION: 11APR78 0900 HRS

COMPOSITE TIME: 00 HR

COMPOUND NAME	UG/L CONC.
121. CYANIDE (TOTAL)	NA
122. LEAD (TOTAL)	NA
123. MERCURY (TOTAL)	NA
124. NICKLE (TOTAL)	NA
125. SELENIUM (TOTAL)	NA
126. SILVER (TOTAL)	NA
127. THALLIUM (TOTAL)	NA
128. ZINC (TOTAL)	NA
129. 2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN (TCDD)	NA
65A. PHENOL (BY GC/FID OR GC/MS)	ND

* VOA ANALYSES (UNPRESERVED/PRESERVED)

T TRACE

NA NOT ANALYZED FOR

ND NOT DETECTED

NAI NOT ABLE TO ANALYZE DUE TO INTERFERENCE

ATTACHMENT B

GOFF MOUNTAIN CHEMICAL LANDFILL INFORMATION



UNION CARBIDE CORPORATION

CHEMICALS AND PLASTICS

P. O. BOX 2631, CHARLESTON, W. VA. 25330

E-111

INSTITUTE PLANT

March 29, 1977

MR. JOHN E. NORTHEIMER
Industrial Waste Section
Permits Branch
State of West Virginia
Department of Natural Resources
Water Resources Division
1201 Greenbrier Street
Charleston, West Virginia 25311

Subject: Application I-937-L Requesting Approval for Expansion of the Chemical
Landfill Located at Union Carbide Corporation's Institute, W. Va.,
Organic Chemicals Plant (Reference: W. V. DNR Permit 3141, 1965)

John,

We have completed a plan to upgrade this landfill and extend its
useful life from the year 1989 to 2004.

Permit application I-937-L has been prepared and is forwarded with
this transmittal for your department's approval.

Any questions you may have about the project can be referred to me
for prompt response.

Thank you,

R. L. Foster
Department Head
Environmental Protection

Attachment: (1) Permit Application I-937-L, UCC Institute Plant Chemical Landfill

RLF/ees

APPLICATION NO. I- 937 -L- DATE March 22, 1977

STATE OF WEST VIRGINIA
DEPARTMENT OF NATURAL RESOURCES
DIVISION OF WATER RESOURCES
CHARLESTON 25311

APPLICATION FOR WATER POLLUTION CONTROL PERMIT
DISPOSAL OF INDUSTRIAL WASTE BY LANDFILL OPERATION

In accordance with Sections 5, 6 and 7, Article 5A, Chapter 20 of the Code of West Virginia,
Institute Plant

Union Carbide Corporation - Chemicals and Plastics Division

(Name of Applicant)

P. O. Box 2831

Charleston, West Virginia 25330

(Post Office Address)

hereby makes application for the issuance of a Permit to _____

_____ Construct and/or operate New Facilities

X _____ Modify or extend existing Facilities

Establishment located at or near Institute, Kanawha
(City) (County)

West Virginia.

The applicant hereby certifies that the following information is true and correct to the best of the applicant's knowledge and belief and that the applicant has in operation or can reasonably be expected to place in operation the landfill operation herein described within _____* days after receipt of any permit issued pursuant to this application.

* = Existing licensed chemical landfill (W.V. Permit 3141, DNR) will continue in operation without interruption during the expansion.

Note: For response to questions on this page see appropriate attached pages bearing section numbers corresponding to the question numbers shown here. Supporting documents and reports can be found in the appendix.

A. PLANT AND LANDFILL DESCRIPTION:

1. a. Type of Establishment _____
- b. Principal products _____

- c. Principal raw materials _____

- d. Establishment operation HRS/DAY DAYS/WK WKS/YR
Average _____ _____ _____
Maximum _____ _____ _____
2. Name the streams or waters near which the landfill operation is located

3. Attach a topographic map (7 ½ minute quadrangle) showing the exact location of the landfill (latitude and longitude), and a drawing, on a scale of 1" = 100' indicating the following topographic features including cross-sectional views of: (1) initial contours of the landfill, (2) proposed contours of the landfill upon completion, and (3) the location of diversion ditches that will be used to control surface runoff from outside the landfill area.
4. Discuss pertinent geological information:
 - a. **Soils** — Specify physical characteristics and thickness of soil zone. (Physical characteristics would include color, grain size (sieve analysis), permeability, degree of consolidation and compaction characteristics)
 - b. **Bedrock Units** — Specify the following:
 - (1) Type (lithology)
 - (2) Depth and thickness
 - (3) Dip
 - (4) Fracturing
 - (5) Permeability

Include all logs obtained from coreholes, test pits, test bores or wells. Test bores should have a sufficient overlap of elevation in order that a complete, correlated, stratigraphic section can be obtained.

(Allowances for the dip of strata must be made when determining the location and

Note: For response to questions on this page see appropriate attached pages bearing section numbers corresponding to the question numbers shown here. Supporting documents and reports can be found in the Appendix

c. **Ground Water** — Specify the following:

- (1) Depth to highest ground water table elevation (ft.) (Contour on plane view and/or cross-section)
- (2) Direction of Movement
- (3) Quality (Chemical)

Up-gradient from landfill (Analysis should be made for all potential contaminants in the waste to be disposed of in the landfill)

Indicate sources of information and describe fully any tests used in the determination of properties, physical or chemical, concerning the following:

- (1) Soil and Bedrock
- (2) Ground Water

5. Provide a list of major constituents going to the landfill stating the number of pounds per day of each constituent. List the toxicity of each constituent on the basis of a rat, oral, single dose for LD50. Also provide information concerning the solubility and leachability of the waste.
6. In the case of toxic or noxious materials or materials that could degrade water quality, describe in detail the special provisions to be taken to prevent leaching and percolation of any constituent of the waste into the waters (ground or surface) of the State. Specify the monitoring controls to be maintained to check the quality of the ground and surface waters in the vicinity of the landfill. Indicate sources of information and describe fully any tests used in the determination of properties, physical or chemical, concerning the use of a lining material for the site.
7. Are any putrescible wastes to be disposed of in the landfill? What proportion of the total wasteload is putrescible matter?
8. What precautions are taken to prevent unrestricted disposal by others at this site?
9. Give the expected service life of the landfill.
10. Specify the plan of maintenance for the landfill:
 - a. Type and degree of compaction.
 - b. Application of daily cover.

It is understood that any Water Pollution Control Permit issued pursuant to this application may be revoked or suspended and all of the enforcement procedures set forth in Article 5A, Chapter 20 of the Code of West Virginia invoked in the event that (1) future investigations disclose conditions other than stated in this application; or (2) there is failure to comply with the terms and conditions of such permit issued pursuant to this application, with the plans and specifications submitted herewith, or with the plan of maintenance and method of operation submitted herewith.

By R. L. Foster
(Signature)

Department Head - Environmental Protection
(Title)

Union Carbide Corporation
Chemicals and Plastics Division
Institute, West Virginia

CONSULTING ENGINEER:

Dr. James H. Poellet

(Name)

E. D'Appolonia Consulting Engineers, Inc.

(Company)

10 Duff Road; Pittsburgh, Pa. 15235

(Seal or Number)

Introduction

Section 2

Permit Application No. I-937 for Disposal of Industrial Wastes by Landfill

Union Carbide Corporation's Institute Plant Chemical Landfill

2. Streams and Waters Near the Institute Chemical Landfill Operation

Carbide's Institute Chemical Landfill divides the headwaters of Goff Branch through two peripheral drains. Goff Branch then passes off the landfill area, under W. Va. State Highway 25, across the Institute Plant proper and into a junction with Washington Branch at the East boundary fence of Union Carbide property. The combined streams then pass across the West Virginia Rehabilitation Center and West Virginia State College properties, under the Penn Central Railroad right-of-way and into the Kanawha River as Cabell Branch near railroad mile post 117.

(Note: 1. Surface drainage from the northeast production areas of the Institute Plant enters Goff Branch as the stream crosses the Carbide property.)

Note: 2. Human sewage enters Washington Branch before and after it joins Goff Branch

Note: 3. The headwaters of populated Rocky Fork branch lie Northwest and beyond the ridge topping off the area surrounding the chemical landfill property.)

- End Section 2 -

IntroductionSection 1Permit Application No. I-937 for Disposal of Industrial Wastes by LandfillUnion Carbide Corporation's Institute Plant Chemical Landfill1. a. Type of Establishment

The Institute Plant of Union Carbide is a large organic chemical manufacturing facility. Class I wastes from plant operations are processed in a licensed chemical landfill with peripheral water control, soil/waste blending, leachate collection, and leachate treatment.

b. Principle Products

Chemicals produced at the Institute location include poly-glycols, isocyanates, carbaryls, alcohols, ketones, detergents, polymers, extenders, and refrigerants.

c. Principle Raw Materials

Organic chemicals ranging from methane gas to heavy naphthalene compounds make up the raw material mix. A few proprietary metallic catalysts are used along with limited quantities of hydrogen chloride, ammonia, and sodium hydroxide.

d. Establishment Operation

The production plant and its wastewater treatment unit (See EPA NPDES Permit WV 0000086) operate on a 24-hour day, 7-days/week, 52-weeks/year basis. The chemical landfill described in this permit feeds leachate to the wastewater treatment unit on the same time basis, but waste processing follows an 8-hour day, 5-days/week, 52-weeks/year schedule.

Waste blending occupies approximately six out of every eight operating hours.

Introduction

Section 3

Permit Application No. I-937 for Disposal of Industrial Wastes by Landfill

Union Carbide Corporation's Institute Plant Chemical Landfill

3. a. Topographical Map (7-1/2 minute quadrangle) Showing Exact Location of Fill

An 8 x 11 cut of USGS Quadrangle Map entitled "Saint Albans, W. Va., N3822.9 W8145/7.5 (Revised 1971)" is included with this application.

b. Cross Sectional Views of Initial Contours of Landfill

The embankment profile showing the approximate contour of the original ground surface can be found on sketch 14 in the appended report from the soil consulting firm responsible for the design of the expansion (See D'Appolonia Consulting Engineers, Inc., "Report, Site Investigation and Design for Chemical Waste Landfill", Goff Mountain, Institute, W. Va., Union Carbide Corporation, Chemicals and Plastics Division, Project 75-734, Dwg. 75-734-El3, February, 1976.)

c. Proposed Contours of Landfill Upon Completion

The embankment profile drawing referenced in item 3-b above shows the proposed contours of the landfill upon completion.

d. Location of Diversion Ditches That Will Control Surface Runoff Around Fill

Water runoff from the eastern portion of the landfill property will be removed through the special channel shown on sketch 17 (Dwg. 75-734-El7) in the report cited in 3-b above.

Water removed from the northern peripheral drain area will be controlled through the drain cited in the previous paragraph.

Water removed from the western peripheral drain area will be carried through road drains along Highway 622 and along the haul road into the landfill site (See Figures 12, & 14 - Dwg. 75-734-El2 and 75-734-El3 - in the report cited in 3-b above.)

- End Section 3 -

Introduction

Section 4

Permit Application No. I-937 for Disposal of Industrial Wastes by Landfill

Union Carbide Corporation's Institute Plant Chemical Landfill

4. a. Soils - Physical Characteristics

Physical characteristics of the soils can be found in the consulting engineer's report included with this application (See reference in section 3-b). A narrative discussion can be found on pages 9 through 13 and the soil laboratory test sheets are Figures 9 (Dwg. 75-734-E6), 10 (Dwg. 75-734-E7), and 11 (Dwg. 75-734-E8).

b. Bedrock Units

b-1. Type (lithology) - See the consulting engineer's report cited in Section 3-b. Figures 6 (Dwg. 75-734-E9), 7 (Dwg. 75-734-E10), and 8 (Dwg. 75-734-E11).

b-2. Depth and thickness - ibid

b-3. Dip - ibid (Narrative section and drawings)

b-4. Fracturing - ibid

b-5. Permeability - ibid (Narrative page 11)

b-6. Bore Logs - ibid, Figures 3 (Dwg. 75-734-E3), 4 (Dwg. 75-734-E4), 5 (Dwg. 75-734-E5).

c. Ground Water

c-1. Depth to highest ground water table elevation - ibid, page 8.

c-2. Direction of Movement - ibid, page 8.

c-3. Chemical Quality - ibid, page 14 (Narrative), Table II (See Spring analysis).

Sources of Information

(1) Soil and Bedrock - Standard tests performed by D'Appolonia Consulting Engineers, Inc., are described and reported in their project report 75-734 (See Reference in Section 3-b).

(2) Ground Water - Standard tests, both chemical and physical, were applied by the consulting engineer, D'Appolonia, to evaluate groundwater. Chem-

Constituents Fed to Union Carbide Corporation's Institute Plant Chemical Landfill (Section 5)

A-112

Low value on Mellon Institute		Scale range (LD50 Column Only)		Leachability	++ Base = 260 da/yr oper. Leach Control Measures (Section 6)
Constituent	Pounds/Day ++	Oral LD50 (gm/kg)	Water Solubility (ppm)		
Acrylate Polymer (2-Ethyl-hexylacr.)	14	2.5* (* = As Acrylic Acid)	Under 100*	Nil for Polymer	Force complete polymerization using heat or hydrogen peroxide. Blend.
Aluminum Chloride	70	3.7	Reacts with water to $Al(OH)_3 + HCl$	$Al(OH)_3$ = Insol. HCl = Sol.	Force the reaction with water. $Al(OH)_3$ remains in the fill. HCl reacts with $CaCO_3$ waste in fill to yield innocuous $CaCl_2$ and H_2CO_3 . Blend.
Carbon (Activated)	1197	1.0 +	Insoluble	Nil	Blend with landfill earth
Carbowax + Dirt	1825 1217	10.0 + -	5000 -	High -	Blend immediately with earth in upper reaches of landfill to permit full bio-degradation.
Cardboard From Trade-Name Boxes	1513	10.0 +	Insoluble	Nil	Blend with landfill earth to promote degradation to soil fiber.
Catalyst Carrier (Alumina)	11410	10.0 +	Insoluble	Nil	Blend into landfill as soil stabilizer.
Catalyst - (Limestone)	3615	1.0 +	Insoluble	Nil	Blend into landfill as acid waste neutralant.
CELLOSIZEScrap (Hydroxyethyl Cellulose)	5152	10.0 +	Miscible with water	High	Blend immediately with earth in upper reaches of landfill to permit full biodegradation.

lub.

Nil

Blend widely over fill
rkf as co

IntroductionSection 5Permit Application No. I-937 for Disposal of Industrial Wastes by LandfillUnion Carbide Corporation's Institute Plant Chemical Landfill

The following eight pages document the information requested by the State of West Virginia from landfill applicants seeking to comply with Section 5, Form WRD 1-70.

Included in the tables are:

1. Waste descriptions and principle constituents;
2. Pounds of each constituent charged into the fill daily (basis = 260-day working year);
3. Oral LD50 for each constituent based on single doses fed to rats perorally (source texts: Sax, "Dangerous Properties of Industrial Materials"; Eighth Edition "Merck Index"; Eighth Edition "The Condensed Chemical Dictionary"; Union Carbide, "Material Safety Data Sheets"; Union Carbide, "Reactive and Hazardous Chemicals Manual"; Union Carbide, "Manual of Hazards to Health From Chemicals");
4. Water solubility of principle constituents expressed as parts per million in water where possible (source texts: same as "3" above except for the UCC "Manual of Hazards to Health From Chemicals");
5. Leachability based on chemical and physical properties and the experience of Union Carbide while operating Goff Chemical Landfill under W. Va. DNR-WRD Permit No. 3141.

Note: The sixth column in the tables contains information required in Section 6, Form WRD 1-70. Data is shown here for clarification and quick reference.

- End Section 5 -

tion 5 - Continued

A-114

stituent	Pounds/Day ++	Oral LD ₅₀ (gm/kg)	Water Solubility (ppm)	Leachability	Leach Control Measures (Section 6)
Croton Al- dehyde on Absorbent	1 97	0.1 + -	153 x 10 ³ -	High -	Take up on hydrophobic, solid absorbent and blend thoroughly in upper reaches of fill to give ample bio- degradation time.
Dicyclo- pentadiene Polymer	97	0.1 +	200	Nil for Polymer	Force complete polymeri- zation with hydrogen per- oxide and blend thoroughly into upper reaches of fill.
Dirt-Slag- Trash From a-Naphthol Area	1683 17 (a-Nap.)	- 1.0 +	- Insoluble	- Nil	Blend thoroughly into land- fill work face to promote bio-degradation of a-Naph- thol.
DNT + Dirt	1600 (DNT) 17 (Dirt)	1.0 + -	Insoluble -	Nil (Dinitro- toluene is a solid)	Blend thoroughly into upper reaches of fill to insure thorough bio-degradation.
Drums (Steel)	20	10.0 +	Insoluble	Slight (As FeCl ₃ with chlorides present)	Blend widely over entire landfill workplace to mini- mize contact with chlorides.
Filter Aid (Diatomaceous Earth)	90	10.0 +	Insoluble	Nil	Blend throughout fill as soil conditioner.
Filter Cake (Diatomaceous Earth + Ter- gitol surfactant)	8393 (Earth) 85 (Surf.)	- 1.0 +	Surfactant is Miscible with water.	- High	Blend thoroughly into upper reaches of fill to insure absorption and bio-degrada- tion of surfactant.
Filter Cones (Traces of H ₂ SO ₄)	186 (cones) 2 (acid)	- 0.01 +	- Infinite	- High	Blend the cones with lime- stone wastes to react the acid to CaSO ₄ , an innocuous material.

mit Application No. I-937

tion 5 - Continued

Constituent	Pounds/Day ++	Oral LD ₅₀ (gm/kg)	Water Solubility (ppm)	Leachability	Leach Control Measures (Section 6)
Filtrol-25 + 1 % H ₂ SO ₄ (Georgia Clay with adsorbed acid)	191 (clay) 2 (acid)	- 0.01 +	- Infinite	- High	Blend the waste thoroughly with limestone to react the acid to CaSO ₄ , an innocuous material.
Floor Sweepings (Machine Shop) (Oil + Absorbent)	260 (abs.) 3 (oil)	- 10.0 +	- Insoluble	- High	* Petrol.-base oils degrade poorly & flush out of Goff Fill.
Glass (Broken) (Bottles + Light Tubes with traces of mercury metal)	17 (glass) 0.002 (Hg)	- 0.01 +	- Insoluble	Nil (Hg will not react with the cold HCl or H ₂ SO ₄ in the fill)	Equip dozer operators with breathing air; exclude all other personnel from the site; crush and blend over wide fill area (TLV breath. is 0.05 mg/M air).
Gloves + Plastic (saturated with paraffins)	13 (gloves) 1 (paraffins)	- 10.0 +	- Insoluble	- Nil	Blend waste widely over landfill work face.
Glue (Vinyl Acetate)	39	1.0 +	275 x 10 ³	High	Force the glue to polymer- ize through exposure to air. Spread thinly on fill work face and blend into first 8 inches of soil.
Glutaric Anhydride	25	1.0 +	Reactive with water to form totally sol- uble glutaric acid	High	Wearing proper protection, workers carefully blend the solid anhydride with soil over wide areas in the upper reaches of the fill. Work is done on dry days. Rain reacts the anhydride to a degradable acid form.

tion 5 - Continued

Constituent	Pounds/Day	Oral LD ₅₀ (gm/kg)	Water Solubility (ppm)	Leachability	Leach Control Measures (Section 6)
Hose (Contaminated with Sevin)	80 (hose) 1 (Sevin)	- 0.5	- 40	- Low (Sevin de- grades easily in the ground)	Hose is spread over wide fill area with special effort being taken to bring hose in contact with soil.
Insulation + Trash (Sevin is contaminant)	5451 (insul.) 55 (Sevin)	- "	- "	- "	Treat insulation in same manner as hose.
Isophorone + Slag (SiO ₂)	2 (isoph.) 215 (slag)	1.0 + "	20 x 10 ³	Moderate	Contaminated slag is scattered over a wide area high in the fill to permit long retention time and good bio-degradability.
Lab Waste-512 (Tergitol, Glycol, Carbowax)	8 (glass) 83 (Terg.) 83 (glycol) 83 (Carbo.)	- 1.0+ 1.0+ 10.0+	- Miscible Complete Complete	- High " "	- Blend mixture over a wide area high in the fill to give maximum biodegradation time.
Lab Waste-511 (Hazardous) (Acetone, Isopropanol, Polyols, Pyridine, Styrene-PVA* * = Polyvinyl Acetate)	11 (glass) 103 (acet.) 103 (isopr.) 103 (polyols) 103 (pyridine) 103 (sty-PVA)	- 5.3 5.8 1.0+ 1.0+ 1.0+	- Infinite " 10 Miscible 3 (sty.) 19.6 x 10 ³ (PVA)	- High " Low High Low Moderate	- Workmen in protective gear carefully blend these chem- ical bottles with earth high in the fill after crush- ing under dozer treads. Air polymerizes the styrene-PVA and the other materials are retained in the fill until they are degraded.
Lab Waste-511 (Low Hazard) (Latex,* Polyox adhesive)	36 (glass) 580 (latex) 580 (polyox)	- 1.0+ (EHA) 10.0+ (poly.)	- 1 Complete	- Low High	Materials are crushed into the fill workface over a wide area during dry periods to increase retention time.

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At Application No. I-937

Section 5 - Continued

Situents	Pounds/Day	Oral LD50 (gm/kg)	Water Solubility (ppm)	Leachability	Leach Control Measures (Section 6)
Latex (Reacted Ethylhexyl Acrylate)	308	1.0 + (has EHA)	1	Low	Blend into surface soil of landfill workface; get as much air contact as possible to complete polymerization.
Liners - Drum (Polyethylene)	67	10.0 + (PE)	Insoluble	Nil	Blend into landfill work- face.
Naphthalene Waste	668	1.0 +	Insoluble	Nil	Protect workmen with breath- ing air and blend directly into landfill workface.
α-Naphthol	12	9.0 (rabbit)	Insoluble	Nil	Provide workmen with skin and breathing protection and blend directly into landfill workface.
Paint Waste	79 (latex)	1.0 +	1	Low	Treat as "latex" in item 30 above.
Pipe - Contam- inated (TDI Solids)	73	(Not applicable. Pipe is stainless filled with tolylene diisocyanate insoluble polymer.)			Place pipe in little-used section of fill workface.
Polyols - NIAX	11	1.0 +	10	Low	Crush and blend drum and contents into work face of fill.
Polyox Scrap	2052	10.0 +	Forms gel with water	Low	Blend with earth and spread widely at landfill work face.
Rags (Used) (Oil, Glycol)	69 4 4	- 10.0 + 1.0 +	- Insoluble Complete	- High (petroleum- base oils do not degrade easily)	Treat as "oil" in item 19 above.
Roofing	134	10.0 + (oil)	Insoluble	High (petroleum- base oils do not degrade easily)	Treat as "oil" in item 19 above.

tion 5 - Continued

A-118

stituent	Pounds/Day	Oral LD50 (gm/kg)	Water Solubility (ppm)	Leachability	Leach Control Measures (Section 6)
Residues (Dimethyl- aniline, Sevin, Sand)	148 148 1189	2.0 0.5 -	Insoluble to slight 40 -	Low Low -	Workmen protected against skin penetration and inhalation hazard blend this residue thoroughly with earth for maximum biodegradation.
Resin - Ion Exchange, Cones (Isopropanol)	630 6	1.0 + (Zeol.) 5.8	Insoluble Infinite	Nil High	Cones and zeolite resin blend well into the fill without problem. Isopropanol must be absorbed promptly on soil to speed biodegradation so good blending is a "must".
Sand Blaster Sand (Silica, Ferric Oxide)	30 (silica) 1 (Fe2O3)	10.0 + 10.0 +	Insoluble Insoluble	Nil Nil	Workmen wearing respirators blend the sand into the fill at the work face.
Sevin Floor Sweepings (Oiled Sawdust, Sevin)	857 (oil+wood) 286 (Sevin)	10.0 + 0.5	Insoluble 40	High (See Item 19) Low	Protect workmen against skin penetration and inhalation hazard and proceed as in Item 19 above.
Sevin Product	223	0.5	40	Low	Protect workmen against skin penetration and inhalation hazard and blend the waste over a wide area at the landfill workplace.
Sevin, Speedi-Dri, and Trash (Sevin)	10,838 109	- 0.5	- 40	- Low	Process into landfill as in Item 44 above.

it Application No. I-937

ion 5 - Continued

Constituent	Pounds/Day	Oral LD50 (gm/kg)	Water Solubility (ppm)	Leachability	Leach Control Measures (Section 6)
Sieves, Molecular (Silica, H2SO4)	1167 (silica) 12 (H2SO4)	10.0 + 0.01 +	Insoluble Infinite	Nil High	Process waste as shown in Item 18 above.
Sludges, Limestone (CaCl2, CaCO3)	39 (CaCl2) 3896 (CaCO3)	4.0 10.0 +	Soluble 15	High Slight	Workmen equipped with skin and eye protection spread the sludge on the fill workplace and blend it in.
TDI Ball- Mill Res- idue (Toluene Diisocyan- ate Polymer)	18267 (* = Estimate. TDI is extremely difficult to ingest.)	10.0 + *	Reacts with water to form insoluble solid. TDI-BMR is the product of an intentional reaction to a solid state.	0	Workmen wearing breath- ing air against possible active TDI (TLV = 0.02 ppm in air) blend and react the residue with damp earth.
TDI (Neutral- ized) and Trash (Trash, Urea)	3516 (trash) 390 (urea)	- 1.0 +	- Soluble	- High (readily degra- ded, however, if re- tained in soil. Bac- teria seek the contain- ed nitrogen.)	- Workmen in breathing air against possible active TDI (TLV = 0.02 ppm in air) carefully blend the waste high on the landfill workplace.
TDA-TDI, Trash (Tolylene Di- amine, Tolylene Di- isocyanate)	968 (trash) 161 (TDA) 161 (TDI)	- 0.1 + 10.0 + *	- Soluble Insoluble Solid	- High 0	- Workmen wearing skin pro- tection and breathing air carefully blend the waste on the landfill face.

(* = See Item 48 above)

it Application No. I-937

ion 5 - Continued

tituent	Pounds/Day	Oral LD50 (gm/kg)	Water Solubility (ppm)	Leachability	Leach Control Measures (Section 6)
Sieves, Molecular (Silica, H ₂ SO ₄)	1167 (silica) 12 (H ₂ SO ₄)	10.0 + 0.01 +	Insoluble Infinite	Nil High	Process waste as shown in Item 18 above.
Sludges, Limestone (CaCl ₂ , CaCO ₃)	39 (CaCl ₂) 3896 (CaCO ₃)	4.0 10.0 +	Soluble 15	High Slight	Workmen equipped with skin and eye protection spread the sludge on the fill workface and blend it in.
TDI Ball- Mill Res- idue (Toluene Diisocyan- ate Polymer)	18267 (* = Estimate. TDI is extremely difficult to ingest.)	10.0 + *	Reacts with water to form insoluble solid. TDI-BMR is the product of an intentional reaction to a solid state.	0	Workmen wearing breath- ing air against possible active TDI (TLV = 0.02 ppm in air) blend and react the residue with damp earth.
TDI (Neutral- ized) and Trash (Trash, Urea)	3516 (trash) 390 (urea)	- 1.0 +	- Soluble	- High (readily degra- ded, however, if re- tained in soil. Bac- teria seek the contain- ed nitrogen.)	- Workmen in breathing air against possible active TDI (TLV = 0.02 ppm in air) carefully blend the waste high on the landfill workface.
TDA-TDI, Trash (Tolylene Di- amine, Tolylene Di- isocyanate)	968 (trash) 161 (TDA) 161 (TDI)	- 0.1 + 10.0 + *	- Soluble Insoluble Solid	- High 0	- Workmen wearing skin pro- tection and breathing air carefully blend the waste on the landfill face.

(* = See Item 48 above)

Section 5 - Continued

<u>Constituent</u>	<u>Pounds/Day</u>	<u>Oral LD50 (gm/kg)</u>	<u>Water Solubility (ppm)</u>	<u>Leachability</u>	<u>Leach Control Measures (Section 6)</u>
51. Tetralin	18	2.86	Insoluble	Low	Wearing skin and breathing protection, workmen crush and blend the solid into the landfill work face.
52. Wood, Oil Contaminated (Lube Oil)	103 (wood) 12 (oil)	- 10.0 +	- Insoluble	- High (See Item 19 above)	- See Item 19 above.

- End -

EES/ees

2-21-77

Permit Application No. I-937 - ContinuedSection 6 - Continued

2. (Continued)

- a. Routine surface water sampling and analysis performed according to the sampling schedule diagram included with this application (Control parameters monitored are:

a-1. pH

a-2. Total Carbon

a-3. Carbonate Carbon

a-4. Color (Platinum-Cobalt)

Any deviation from the observed norms for the control parameters is cause for in-depth study of the sample using gas chromatographic, mass spectrometric, atomic absorption, wet chemical or other appropriate standard method approved by the United State Environmental Protection Agency for contaminants in potable water);

- b. Routine ground and leach water sampling and analysis performed according to the sampling schedule diagram for 1974-1978 (included with this application). (The new program to be followed after the landfill upgrading begins will be carried out according to the sample schedule diagram for 1978-2004. Control parameters for leachate water will be those shown in 2a. above. Samples taken from the new borings and the spring will be run for the components described in Table II, "Summary of Water Quality, Goff Mountain Chemical Waste Landfill", D'Appolonia Consulting Engineers, Inc., Report: Site Investigation and Design for Chemical Waste Landfill, Goff Mountain, Institute, W. Va. Project No. 75-734, July, 1976; See 2a. above for control parameters currently being used).

3. Testing of the clay seal included permeability, liquid limit, sieve analysis, hydrometer analysis, water content, compaction, consolidation, pore pressure and shear measurements. Documentation can be found in the design engineering report on the project: D'Appolonia Consulting Engineers, Inc., "Report: Site Investigation and Design for Chemical Waste Landfill, Goff Mountain, Institute, West Virginia", Project No. 75-734, p-11, figures 9, 10, 11, July.

IntroductionSection 6Permit Application No. I-937 for Disposal of Industrial Wastes by LandfillUnion Carbide Corporation's Institute Plant Chemical Landfill

Measures taken at the Goff Chemical Landfill to comply with Section 6 are:

1. Prevention of ground or surface water quality degradation through leaching or percolation of toxic or noxious materials into waters of the State is accomplished through these means:
 - a. An impervious clay seal under all of the working area of the chemical landfill. This seal is illustrated and discussed in the following publications: Hainley, J. D., Slover, E. E., "Union Carbide Chemical Landfill, Institute, West Virginia", Solid Wastes: Origin, Collection, Processing and Disposal (Mantell, et al), Wiley Interscience, New York, pp 1004-1008, 1975; and, D'Appolonia Consulting Engineers, Inc., (Report) "Site Investigation and Design for Chemical Waste Landfill, Goff Mountain, Institute, West Virginia" Project No. 75-734, July, 1976.
 - b. Extensive surface and peripheral drainage systems to keep water from entering the fill (Re: D'Appolonia, *ibid*).
 - c. Collection and bio-treatment of the leachate through a system of internal drains, collection headers, an impervious basin, a process sewer, and a 5-million gallon per day activated sludge wastewater treatment unit. (Re: D'Appolonia, *ibid*; Midwest Technical, Inc. "Goff Mountain Chemical Waste Landfill Project", Jc No. 99102, January, 1977; Mantell, et al, *ibid*, pp-1015-1016).
2. Monitoring controls maintained to check the quality of ground and surface waters in the vicinity of the landfill are:

Introduction

Section 7

Permit Application No. I-937 for Disposal of Industrial Wastes by Landfill

Union Carbide Corporation's Institute Plant Chemical Landfill

No putrescible wastes are charged into the Goff Chemical
Landfill.

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)

IntroductionSection 8Permit Application No. I-937 for Disposal of Industrial Wastes by LandfillUnion Carbide Corporation's Institute Plant Chemical Landfill

The Goff Chemical Landfill is restricted to the processing of selected chemical wastes from three Union Carbide locations in the Kanawha Valley: UCC South Charleston, UCC Technical Center, and UCC Institute. Of the three, the Institute Plant makes greatest use of the facility.

Use of the site is controlled through a special monitoring program built around a 5-step procedure that makes the waste generator consider reprocessing, sale, incineration, or bio-oxidation (liquid phase) before consigning the waste to the chemical landfill (1).

Every disposal requires the completion of a special Order for Waste Removal before the material can even be loaded into its transport container (2). Full agreement must be reached between the waste generator and the landfill technical staff in these areas before the special stamp authorizing waste pickup is applied to the order by the landfill supervisor (3):

1. Health hazard and required personnel protection;
2. Flammability hazard and its minimization;
3. Reactivity hazard and its attenuation;
4. Environmental impact and its elimination;
5. Physical state;
6. Quantity;
7. Location of the waste;
8. Payment for the service;
9. Signature and telephone of the waste generator.

Upon approval by the landfill supervisor, the Order for Waste Removal is presented to the special waste driver under UCC supervision. The driver sees that the order travels with the load as emergency identification and as the "admission ticket" to the landfill (4).

No waste is permitted into the fill work face without a properly approved ticket.

(1) Mantell, et al, "Solid Wastes: Origin, Collection, Processing, and Disposal", Wiley Interscience, John Wiley and Sons, 605 Third Avenue, New York, p-1019, 1964.

(2) Mantell, *ibid*, p-1024.

Introduction

Section 9

Permit Application No. I-937 for Disposal of Industrial Wastes by Landfill

Union Carbide Corporation's Institute Plant Chemical Landfill

The Goff Chemical Landfill is expected to serve
its three Kanawha Valley Union Carbide locations
until the year 2004.

- End Section 9 -

ees 2-22-77

Introduction

Section 10

Permit Application No. I-937 for Disposal of Industrial Wastes by Landfill

Union Carbide Corporation's Institute Plant Chemical Landfill

Surface blending is the primary technique used in working prepared chemical wastes into the Goff Fill. This avoids the heavy gas generation, ponding, and underground fire potential inherent in the pit-and-cover method commonly used in sanitary landfills (1). Keeping this difference in mind, these replies can be made to the specific questions:

1. Type and degree of compaction - An endloader and a bulldozer in combination are used to blend the soil/waste mixture into the fill. Approximately one volume of earth is required for each volume of waste received, and the combination appears to compact at roughly a 2:1 ratio, based on aerial studies made of the site and waste volumes known to have been charged. Compaction greater than this impairs the operation of the internal leachate removal system.
 Note: See Attached Specification Pages 5, 13, 14, 15, and 16.
2. Application of daily cover - Earth/waste blending is a continuous operation six out of every eight working hours, making the need for daily cover as practiced in municipal landfills unnecessary. Under the new program to control surface drainage after 1978, blending will take place in a series of benches with prescribed slopes. This change calls for daily installation of a clay surface seal (equal in quality to the bottom seal of the fill) over every completed portion of the bench being worked. Following the clay seal, grassing will be done as soon as weather permits (1), (2).
 Note: ibid
3. Application of final cover - Final cover (clay seal and grass) will be applied as part of the ongoing daily program (See "2" above). Specifications on the clay and the cover are given in the design engineering firm's report (2).
 Note: ibid

- (1) Mantell, et al, "Solid Wastes: Origin, Collection, Processing, Disposal", Wiley Interscience, John Wiley and Sons, Inc., 605 Third Avenue, New York, pp-1027-101975.
- (2) D'Appolonia Consulting Engineers, Inc., "Report: Site Investigation and Design for Chemical Waste Landfill, Goff Mountain, Institute, W. Va.", Project 75-734, figures 9, 10, 11, 14, pp 20-27, July, 1976.

APPENDIX

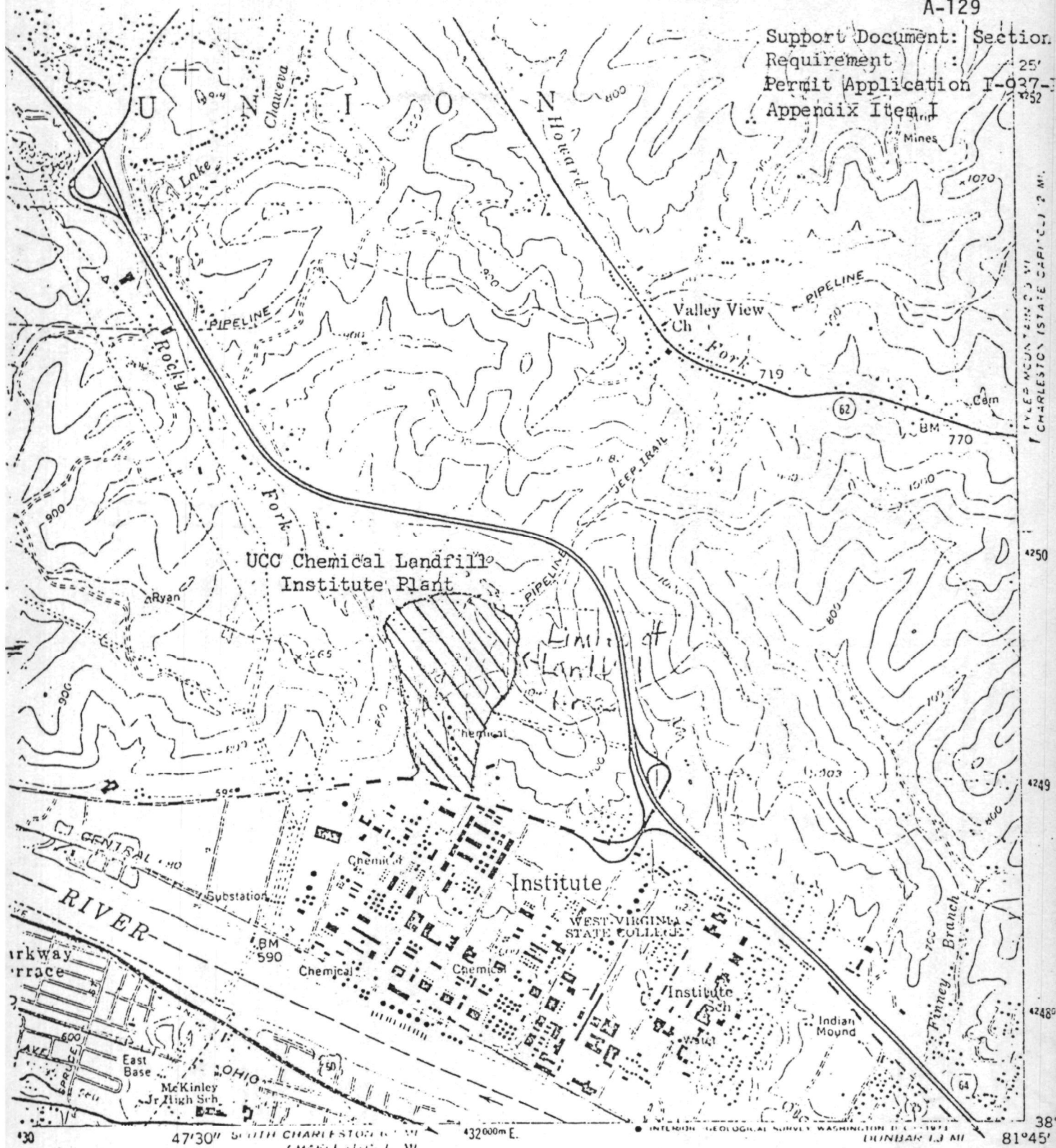
Permit Application No. I-937-L for Disposal of Industrial Wastes by Landfill

Union Carbide Corporation
Chemicals and Plastics Division
Institute Plant
Chemical Landfill

Supporting Documents

ees 3-25-77

Support Document: Section
Requirement : 25
Permit Application I-037-
Appendix Item I



1 MILE
7000 FEET
1 KILOMETER



QUADRANGLE LOCATION

THE P. T. L. CO.
P. O. BOX 150112
CHARLESTON, W. VA.
TELEPHONE (304)

ROAD CLASSIFICATION

Heavy-duty ——— Light-duty ———
Medium-duty ——— Unimproved dirt ———
○ Interstate Route ○ U.S. Route ○ State Route

SAINT ALBANS, W. VA.

1004 PROCESS INDUSTRY WASTES

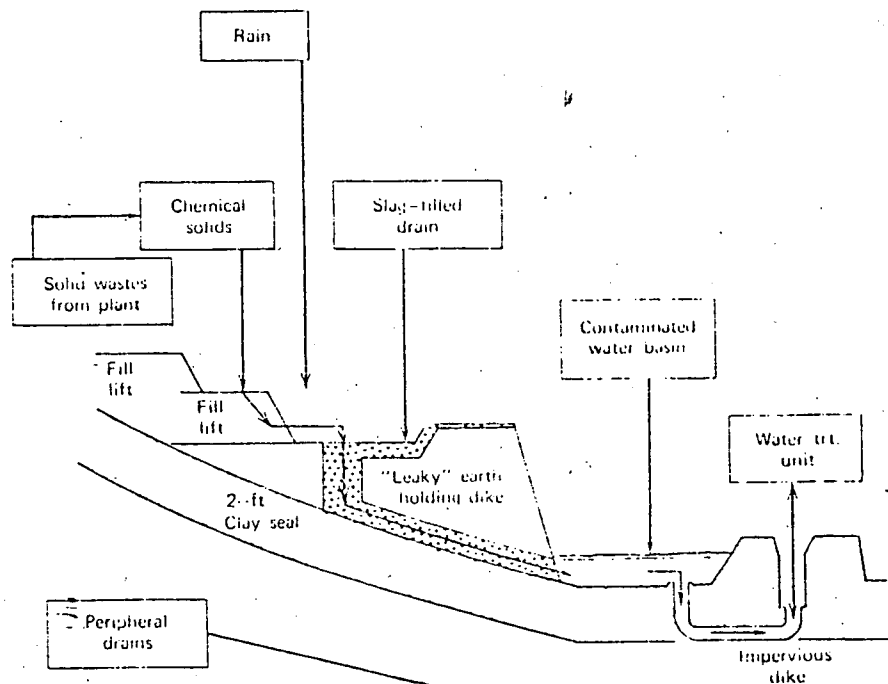


Figure 1. Goff Mountain chemical landfill, Institute plant, Union Carbide Corp. Dynamic wet process biochemical system. (1) No odors allowed; (2) no groundwater pollution; (3) no air pollution allowed (fumes, gas); (4) License required.

Site study is an expensive and time-consuming activity, as Table 2 indicates. However, the success of the landfill is totally dependent on its being done well.

Clay Membrane

A 2-ft-thick, selected-clay seal is the key feature that makes this wet process landfill possible. It has to be placed under dry weather conditions and is rolled at 6-in. intervals to a density and moisture content that ensure impermeability.

The seal is used under the entire landfill (fill lifts, dike keys, contaminated basin, and impervious dike). Field and laboratory tests are run constantly during installation, and compaction layers that do not meet the standards are scraped off and relaid.

Project No. 75-734
Feb. 78
(Revised July 76)

D'AMPTOLONE
CONSULTING ENGINEERS, INC.

Support Document: Section 3
Requirement : b
Permit Application I-937-L
Appendix Item II

Report

Site Investigation & Design for Chemical Waste Landfill

**Goff Mountain
Institute, West Virginia**

Chemicals & Plastics Division
Union Carbide Corporation
South Charleston, West Virginia

1008 PROCESS INDUSTRY WASTES

Table 2 Test Borings and Soil Tests for Subsurface Investigation for Goff Mountain Chemical Waste Landfill

Description	Feasibility Study	Design Phase
Test borings	20	20
Lineal feet of soil sampled with std. split spoon	455	255
Lineal feet of rock coring and pressure testing	416	660
Undisturbed shelly tube samples	26	30
Permanent water sample locations	6	0
Soil gradation and atterberg limits tests	11	22
Unconfined compression tests	29	33
Direct Shear tests	10	16
Laboratory permeability	6	0
Specific gravity	10	0
L. A. abrasion and soundness tests	0	9
Compaction tests		10
Consolidation tests	6	3

Floor seal for the working portion of the landfill is put in place as the fill grows to avoid wasting valuable clay through erosion. An exaggerated cross section of the seal is shown in Figure 5.

Peripheral Drainage

Two types of drainage systems are required for the Goff Chemical Landfill. One handles the contaminated effluent from the fill itself, and the other, discussed here, seeks to divert as much uncontaminated water (both surface and subsurface) around the actual fill as possible.

Figure 6 details the peripheral drain system. The east, west, and north branches have been completed as part of Phase I. The large dam on the south side is part of the third construction phase so drainage in this area is not as shown. The complex lateral drain system on the west side cost roughly \$24,000 and is typical of the approach taken to stabilize slip areas around the fill.

Leachate Control System

Early experience during the sanitary-landfill operating period clearly indicated that Carbide was faced with running a wet process because of the heavy rainfall and high groundwater flow indigenous to the site.

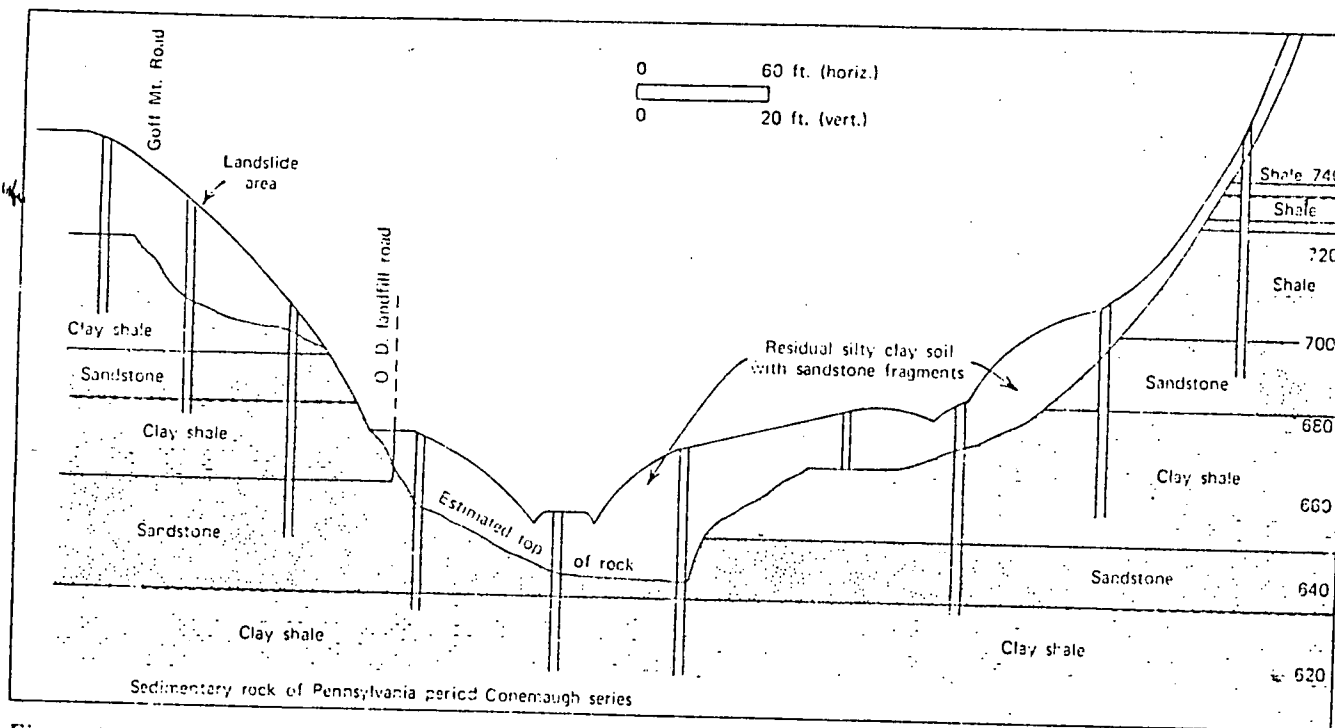


Figure 5. Geologic cross section.

Support Document: Sect:
Requirement :
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Appendix Item VI

Summary of Midwest Technical, Inc., Job No. 99102

"Goff Mountain Chemical Waste Landfill Project"

Leachate from the landfill will be treated in a corrugated-plate
Heil oil separator for removal of the floatable organics and then
retained in a new leachate pond (Re: D'Appolonia, Report, Project
75-734).

It will be metered continuously and monitored periodically before
being routed to the Institute Plant Wastewater Treatment Unit for
bio-oxidation.

AHC/SHW/EES/ees

3-25-77

UNION CARBIDE CHEMICAL LANDFILL INSTITUTE 1015

A vital part of the operation of this continually flushing chemical landfill is pH control. Fortunately, a large quantity of both acidic and basic solid wastes is available and greater or lesser amounts can be blended into the fill lifts as daily readings dictate. Generally, effort is made to keep the leachate around a pH of 6.8-7.2 to meet the needs of the wastewater treatment unit.

There is some indication that the leachate will tend to go^u acid (pH around 5.2) if no effort is made to keep the fill at relatively neutral conditions. Since the operation is basically anaerobic, this is not too surprising.

BOD tests are run occasionally, but the real control is the daily total carbon analysis, which gives results in a matter of seconds. When the landfill is working well the total carbon level is generally around 6000 ppm. When waste loading occurs and insufficient blend earth is being used the figure can climb to 14,000 ppm.

Although it is somewhat difficult to relate total carbon and BOD, there is enough correlation to make a rough comparison with the BOD figures on "sanitary" landfill leachates.

Daily samples are taken from the contaminated collection basin and run for total carbon, pH, color, foam, and carbonate-carbon. The latter is necessary because the total carbon instrument picks up carbon from all sources, including ground minerals. To get a true picture of the organic carbon requiring treatment, the carbonate-carbon is subtracted from the total carbon reading.

Uncontaminated water from the wells and peripheral drains is sampled on a cycle that catches each point about once a week. A total carbon level of 130 ppm is indicative of uncontaminated water at this landfill location. (Naturally occurring organics at other landfill sites would make this figure different.)

A complete air quality control laboratory is available through the Institute Plant's Environmental Protection function. Problems with odors and gases are handled through this group.

Treatment

Oils leaving the fill lifts and collecting on the surface of the water in the contaminated basin are skimmed off and taken as fuel to the plant's steam boilers.

The contaminated water remaining is transported to the institute's activated-sludge wastewater treatment unit for bacterial stabilization. Sludge is removed and collected in an anaerobic basin and the treated water is released to the Kanawha River. Excess sludge is sent back to the chemical landfill to complete the process cycle. Figure 9 is a flow diagram of the treatment unit.¹¹

1016 PROCESS INDUSTRY WASTES

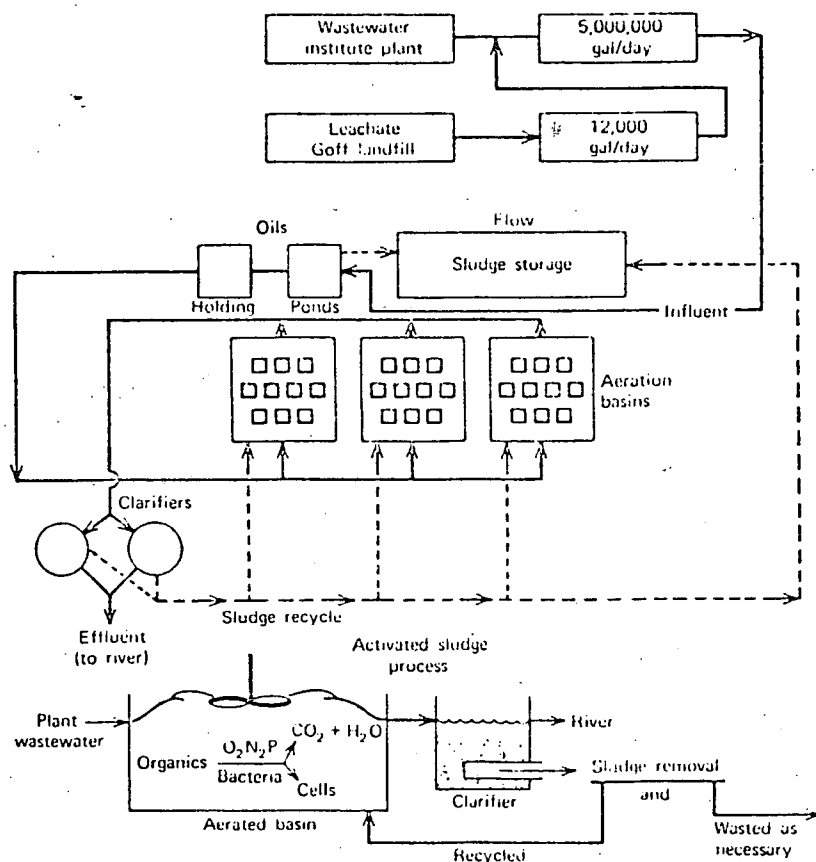


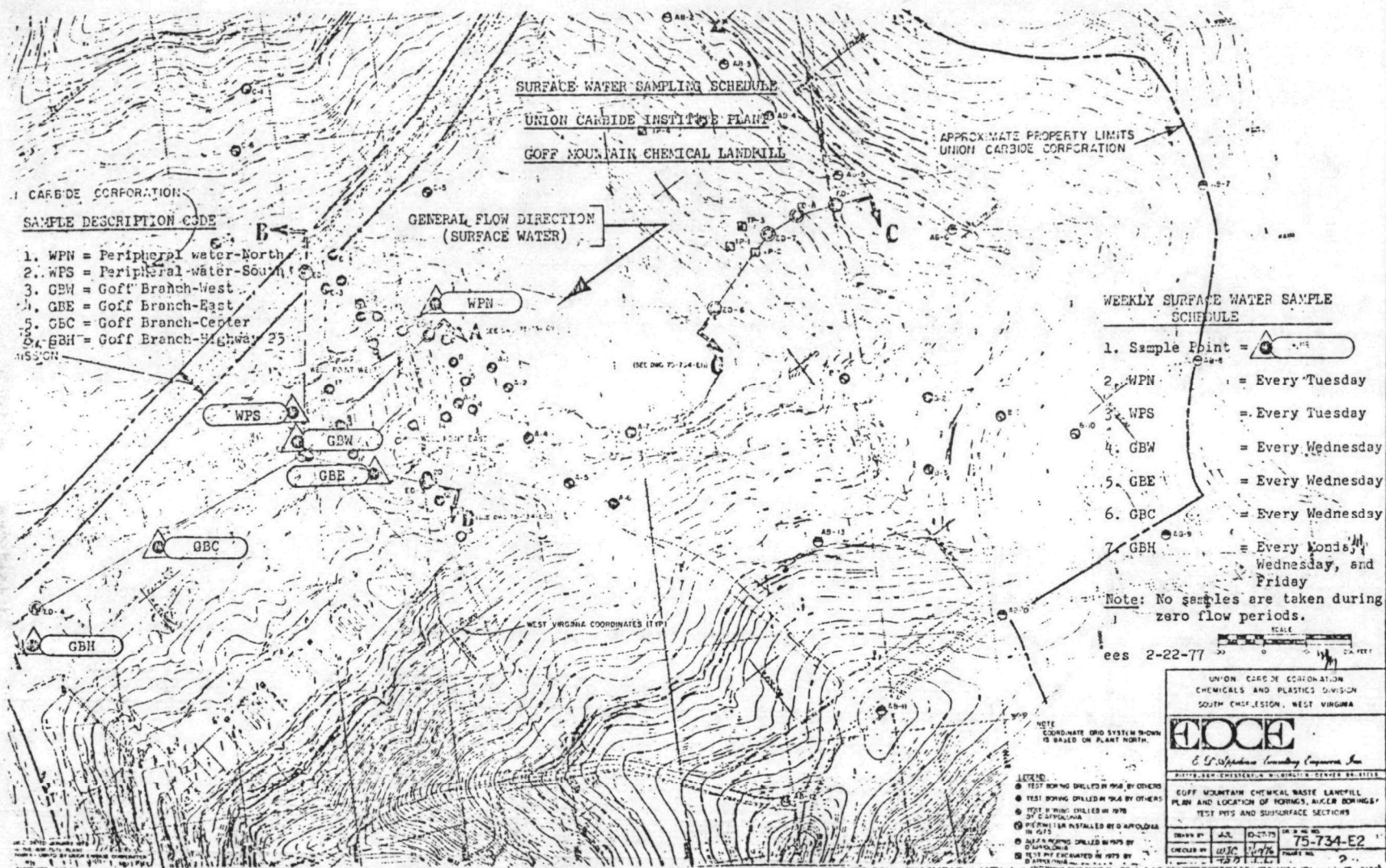
Figure 9. Activated sludge water treatment unit (Union Carbide Institute Plant).

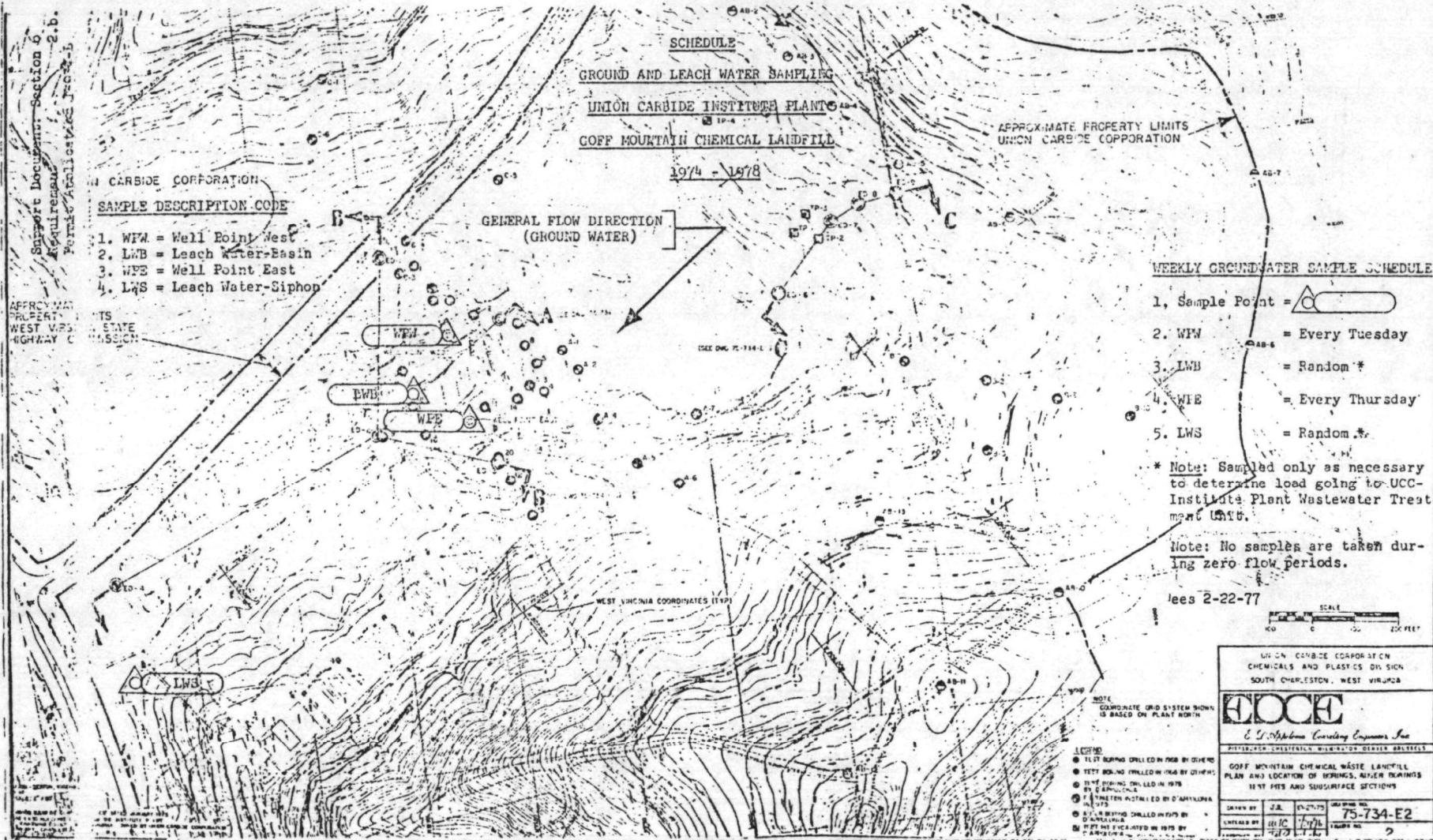
Standards

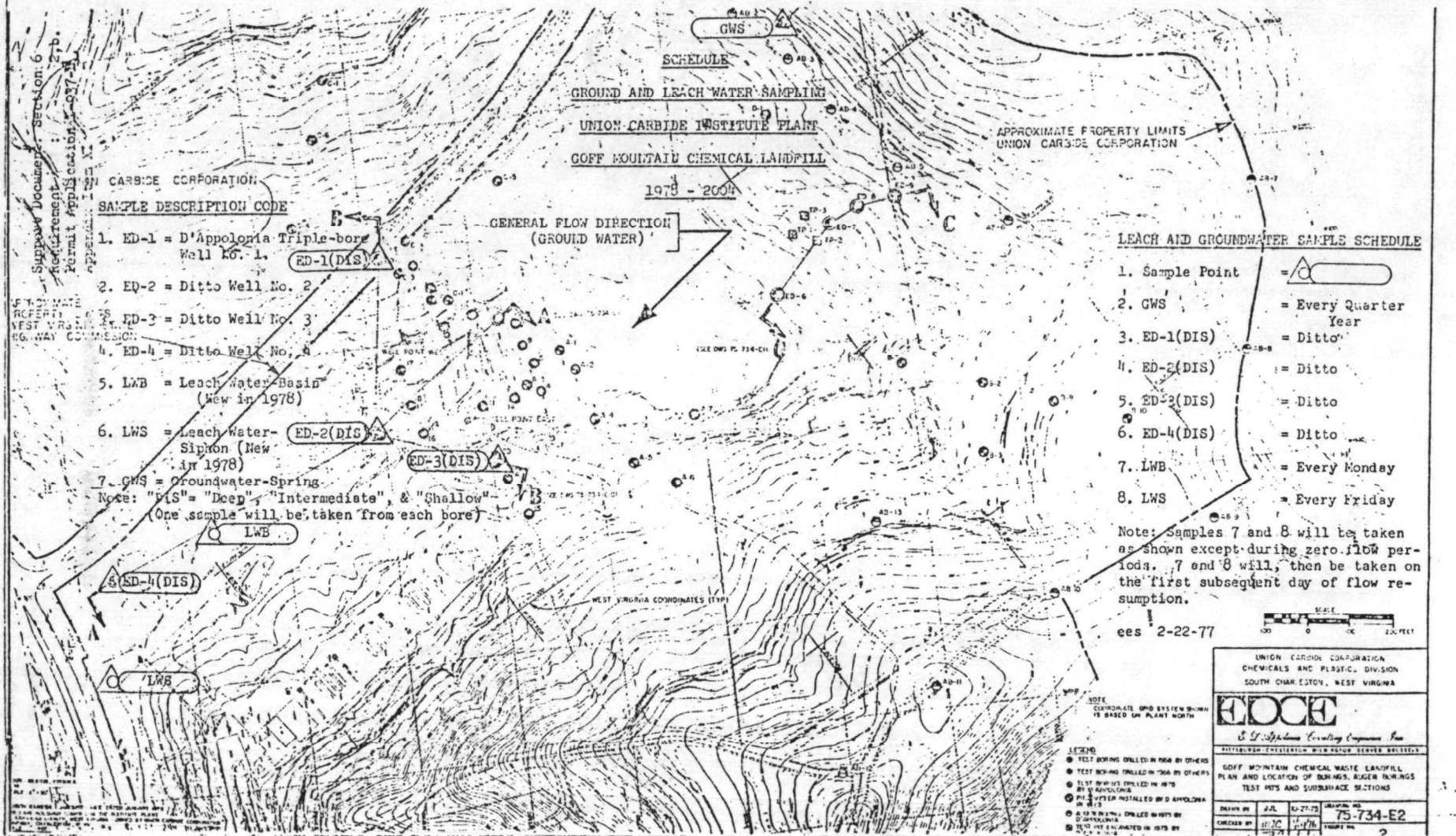
Basically, the Goff chemical landfill is capable of processing any chemical that is acceptable to the bacteria in the wastewater treatment unit. Standard oxygen-uptake tests using treatment unit bacteria are performed in cases of doubt.

Metals (with the exception of iron, sodium, potassium, calcium, and aluminum) are not generally accepted into this landfill because of their toxicity to bacteria both in the fill and at the treatment unit. Recovery is recommended wherever possible and elaborate encasement in concrete is provided in the few cases where reclamation efforts fail.

FRANCIS
ROBERT
WEST VIR
GUYANA









INTERNAL CORRESPONDENCE

CHEMICALS AND PLASTICS

INSTITUTE PLANT P. O. BOX 2831, CHARLESTON, WEST VIRGINIA 25330

Name: Distribution Lists A-1, A-2, A-3
Division: C-1, C-2, C-3, C-6, C-7
Attention: E-1, E-2, E-10, E-11, E-12
F-4, F-5, F-6, F-7

Date: February 17, 1976
Originating Dept.: Environmental Protection

Answering letter date

Copy to: Mr. R. F. Hunt
Mr. R. W. Kiefer
Mr. J. L. Worstell

Subject: General Operating Policy
Goff Landfill

Gentlemen:

The attached General Operating Policy for the Goff Landfill has been approved by the Institute Plant Manager's Department and is being distributed for your use. This policy, which is effective immediately, has been adopted so that wastes can be disposed of economically and within prescribed governmental regulations.

Please note that the Goff Landfill is the fifth and last alternative for waste disposal, and disposal must strictly comply with the attached procedures. Waste generating departments are going to become more involved in developing disposal procedures, record keeping, and transportation of the wastes. The landfill will not be used as a chemical drum storage facility. Wastes and chemicals will be processed as they arrive; therefore, approved disposal procedures must be developed prior to transferring them to the landfill.

Please refer any questions regarding this policy to Mr. R. L. Foster.

Very truly yours,

H. D. Coombs

lh

GOFF LANDFILL OPERATING POLICYI. Purpose of the Chemical Landfill

The purpose of the landfill is to safely and economically dispose of materials or wastes which are contaminated, obsolete, small in volume, or whose physical properties will not allow them to be accepted by other Plant 512 disposal facilities.

II. Criteria for Accepting Wastes at the Chemical Landfill

1. Waste Information - Waste generators are responsible for providing full information (chemical, physical and toxicological) on each waste submitted for disposal (See UCC Reactive and Hazardous Chemicals Manual - Safe Practice Series and 512 Industrial Hygiene Department). The information must be provided on a properly-completed Order for Waste Removal (Form: 512-1747-H) and sent to the 512 Miscellaneous Disposal Coordinator prior to any handling or processing. Information taken from the Order for Waste Removal will serve as the basis for developing the processing procedures.
2. Waste Identification - All waste containers will be marked by the generator prior to shipment for disposal processing. Markings shall include the correct DOT name stencil and label (See DOT Shipping Manual maintained by the 512 Laboratory Service Department), the correct UCC Hazard Signal Code (Engineering Specification FP-211), and the number assigned by the 512 EP Department to the Order for Waste Removal (Form: 512-1747-H) covering the waste.
3. Waste Disposal Method - The 512 E/P Department Staff will determine the disposal alternatives according to the following prioritized criteria:
 - a. Reprocessing Potential - The generating unit will be required to show the reprocessing has been considered and its feasibility evaluated before any other method of disposal will be developed.
 - b. Sales - Should reprocessing be unfeasible, the generator will be requested to submit a Request for Disposal of Excess Chemicals (Form: 512-1724) to the 512 Investment Recovery Coordinator for Materials in Distress. The disposal number assigned by 512 EP to the original Order for Waste Removal (Form: 512-1747-H) will be entered in the appropriate space on Form: 512-1724.
 - c. Thermal Oxidation - Failing to sell the material in distress, the 512 Investment Recovery Coordinator for Materials in Distress will notify the 512 EP Staff and provide the originally-assigned disposal number. 512 EP will reopen consideration of the disposal request and check the possibilities of thermal oxidation for heat recovery at 512 or commercial oxidation by an approved outside contractor.
 - d. Bio-oxidation - If 512 EP finds that thermal oxidation is not possible, the disposal of the waste through bio-oxidation will be considered.

GOFF LANDFILL OPERATING POLICY

Page 2

II. Criteria for Accepting Wastes at the Chemical Landfill (continued)3. Waste Disposal Method (continued)

- a. Chemical Landfill - Where no other viable disposal method can be found, disposal in the chemical landfill will be considered.

Special Note: The red-stamp approval by the 512 EP Department will not be given the waste generator until the appropriate disposal alternative is selected.

4. Waste Acceptance - Waste materials will be accepted subject to the limitations specified below:

- a. Metals - Only those metals found naturally in the local geological environment at Plant 512 will be processed into the chemical landfill. They include: aluminum, calcium, iron, magnesium, potassium, silicone, sodium, and titanium.
Metals that cannot be processed into the landfill are: antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, mercury, nickel, silver, and strontium.
- b. Chemicals - Only those chemicals rendered "safe" for direct blending into the chemical landfill will be processed. Problems of toxicity (particularly those related to human health), flammability (as defined by UCC Engineering Standard FP-210), and reactivity (as defined by the UCC Reactive Chemicals Program) shall be resolved before any waste is classed as "safe" for blending.
- c. Radioactive Waste - Radioactive waste will not be accepted at the chemical landfill.
- d. Biotoxic Waste - Non-biodegradable carcinogenic, mutagenic and teratogenic wastes will not be accepted.
- e. Inorganic Wastes - Biotoxic inorganic wastes will be accepted only at the discretion of the 512 EP Department Staff.
Example: salt, etc.
- f. Laboratory Samples - Samples which meet code 2, 2, 1 (UCC Hazard Signal System, Engineering Standard FP-210) or lower will be accepted into the chemical landfill. (See section on "Record Keeping and Transportation" below for further requirements.)

5. Waste Disposal Processing Procedures

- a. Landfill Operation - Operating activities at the chemical landfill will be consistent with governmental permit and regulatory requirements.
- b. Written Procedures - An approved, written processing procedure will be developed for each waste shipment before it is accepted

GOFF LANDFILL OPERATING POLICYII. Criteria for Accepting Wastes at the Chemical Landfill (continued)5. Waste Disposal Processing Procedures (continued)

- c. Procedure Development - The 512 EP Staff will initiate the development of processing procedures.
- d. Procedure Approval - Each processing procedure must have signature approval from each of the following in the order listed before a waste shipment will be accepted into the chemical landfill:
 - 1. Waste Generating Department Head
 - 2. Safety Department
 - 3. Maintenance Department Head
 - 4. E. P. Department Head

6. Record Keeping and Transportation

- a. Order for Waste Removal - An approved, coded Order for Waste Removal (Form: 512-1747-H) must travel with each waste every time it moves.
- b. Sample and Waste Containers - All shipments of sample and waste containers will conform to the Adjacent Compartment Loading List used by the UCC Distribution Department and to the DOT regulations set by the Federal Government.
- c. Materials Mixing - Mixing of miscellaneous materials in distress will conform to the adjacent compartment loading list used by the UCC Distribution Department.
- d. Waste Vessel Cleaning - Transport tanks will be cleaned to meet the specifications set by the 512 EP Department Staff.
- e. Waste Carriers - All waste carriers will abide by State and Federal transportation regulations.

7. Waste Storage

- a. Storage (General) - Chemicals will not be stored on the chemical landfill. (Waste sent there for disposal will be processed upon arrival. None will be accepted until preparations have been made to carry out processing.)

combining the optimum in environmental acceptability and process economy.

Disposal methods available are shown in Table 6. Figure 10 illustrates the supervisory organization necessary to maintain the program. Notice the variety of experience and the group's position as another plant production unit. This eliminates the idea that environmental protection is a "service function" and emphasizes the point that pollution control is a regular part of doing business and everyone's responsibility.

Help with any environmental problem is available through the

Table 6 Institute Plant Disposal Methods

Method	Advantages	Disadvantages
1. Reprocess the waste	Best return for UCC No environ. problem	Investment cost; technical difficulty; complex waste might result
2. Sell the waste	Return for UCC No environ. problem	Toxicity problems, shipping problems, market problems
3. Burn the waste	Heat recovered as steam	Air, water, land pollution, and transportation problems
4. Biooxidation of waste	Protects the Kanawha River	High cost: (2.5¢ lb. of contained organic chemical) High investment: (\$27/lb of contained, continuously fed organic chemical) Strict control required to avoid air, water, and land pollution
5. Landfill the waste	Ensures final disposal of waste	High cost: (\$14 per yd ³ or \$37 ton on the average) Strict control required to avoid air, water, and land pollution Requires land often difficult to obtain, reuse, and replace
<p><i>Note.</i> Steps 1-3 return some value to the company and add little to the environmental pollution problem.</p> <p>Methods 4 and 5 stabilize the waste but represent a sizeable cost to the company.</p> <p>In the case of each waste, Institute Environmental Protection considers the possibility of following each course (beginning with item 1) before deciding on final disposal.</p> <p>Every possible effort is made to avoid using steps 4 and 5</p>		

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Plant's Environmental Protection Department (Figure 10), but the waste-producing unit bears the cost and responsibility for complete, effective disposal. Round-the-clock availability, variety of talent, and a close working relationship (not limited by particular specialty) make the Institute's environmental team highly effective.

A branch of the plant laboratory is devoted entirely to problems in the environmental area. Although it is not shown in Figure 10, it is an integral part of the team and provides the critical function necessary to insure full environmental control.

Monitor Program

Close monitoring of incoming wastes is the key to successful operation of the Goff Chemical Landfill. Full control of all wastes carried in moving vehicles is retained by the Institute plant's Environmental Protection Department.

The supervisor of the miscellaneous disposal function (Figure 10) permits no vehicle-borne waste (liquid or solid) to enter any Plant disposal facility without a properly completed, signed, and stamped "Order for Waste Removal."

Persons requesting waste disposal are required to submit an order bearing their signature, a complete chemical description of the waste, special instructions, and an account number to the Miscellaneous Disposal Supervisor before waste containers or transportation are supplied.

The disposal supervisor reviews the order with the requester and makes certain the latter has entered proper instructions covering hazards to the disposal personnel (and the environment), method of disposal, chemical composition, protective devices, physical properties, and the account number to receive the costs attending disposal. An important item at this stage in the process is the requester's signature, because each order is treated like a legal contract. Any incident arising from failure to identify the waste or the hazards is made the responsibility of the disposer. All reprimands, costs, investigations, and methods to prevent recurrence become his problems along with the writing of necessary reports. With the full backing of the plant's management in the same line organization, there is no difficulty in getting cooperation of people with waste problems.

When the disposal supervisor is satisfied that an order has been correctly completed, he assigns a special code number, affixes a special red stamp, and notifies the appropriate driver or crew to pick up the order from his office and handle the waste according to the written instructions. Each load must carry an order in transit. No plant driver

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will accept any waste without a red-stamped order. Through and experience, the drivers themselves have become excellent. They have become accustomed to the appearance of the common wastes and refuse to pick up any unfamiliar material. Changes they observe are immediately reported to the disposal supervisor.

Disposal unit operators must receive a ticket for each shipment of waste or they will not accept it. They, also, require the driver to sign and date the order upon arrival at the facility, and they forward signed tickets to the disposal supervisor. A log book and a list of returned tickets is retained by the disposal supervisor as a check on the disposal facilities he uses. In the case of the landfill, the disposal orders are the final records from which the feed mix and volume loading are calculated.

Instructions are provided with each book of blank forms (Table 7) and spaces for all the data needed are provided on the form itself (Figure 11).

Segregation

Solid waste production at the Institute plant is fairly characteristic of all the Goff Chemical Landfill users, so a glance at Table 8 will give some idea of the general breakdown, how it is handled, and (1969 figures) indication of amounts.

Instructions for maintaining the necessary solids-segregation are provided each production department Table 9.

Recovery

Recovery of any waste either as-is or through reprocessing is the most desirable method of disposal whenever it can be practiced. Environmental problems are generally eliminated and the company receives an economic return. Carbide believes in this approach and applies this philosophy in its plants and in its design and development work on new processes.

Each "Order for Waste Removal" and each request for disposal of materials involving wastes at the Institute plant are considered in light of the disposal methods shown in Table 6. Most desirable, of course, is the first (reprocessing), and least is the fifth (landfill).

Selling the waste can often benefit the corporation if a buyer has equipment Carbide does not possess and is willing to follow regulations governing toxicity and reuse. Toward this end the corporation has provided a formal, in-house organization that helps its plants realize value from certain lines of waste. The Institute makes liberal use of

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Table 7. Order for Waste Removal Instructions (Cover Page for Each Pad of Forms)

OBVERSE

Use ball point pen. Read all instructions before completing form. This is NCR 3 part form. Remove 3 sheets or put in backing board. This form is to be filled out in its entirety by person requesting disposal and returned to Waste Disposal Co-ordinator for approval; in duplicate. It is the responsibility of the person filling out this order, to list all personal precautions that must be taken by personnel handling this material in the source production unit. Failure to insert this information, resulting in injury or illness to personnel handling this material, will compel an investigation by the Industrial Hygiene Department. Fill in all components contained in mixtures. Mail completed form to Building S112.

Material--Fill in name of material completely. Do not write chemical terms. The men who handle this material are not chemically trained. If you have a mixture or compound, write in all components that make this mixture, unless it is a trade named item.

Ordered by--Name of person filling out form and who is requesting the removal. This should be one and the same person. No filling out forms for someone else.

Building--Where material is to be picked up.

Date--Date form is filled out.

REVERSE

Shop order number--The shop order number issued to your department for disposal of material. This is important. Form will be returned if number is not listed. If the proper number is not known, find out and write it down somewhere on this cover.

Quantity--How many pounds or gallons of material are to be disposed of? This information is important. Four drums will consume one yard of landfill at a cost of \$14. For every four drums removed from your unit you spend an additional \$36.00 (Container Cost). Total cost: \$50 4 drums.

Description--Indicate the consistency of the material. Is it water thin or thick? Is the material solid? Are polymers or solids present in liquid? Mark appropriate box. Be sure and indicate the type of container you have. Check appropriate box.

Components--If a single material, write it in. If a Mixture of several materials or a brand name Plant Mixture, list all materials that make up this material and their percentages. This is important and order will be returned if not filled in.

Hazard rating--To be filled in by Disposal Supervisor.

Destination of material--To be filled in by Disposal Supervisor.

Unit hazard operating instructions--This must be filled in completely. Common phrases will not be accepted, such as "Normal Precautions", etc. Full precautions required by unit personnel must be listed. If there is no hazard, give authority.

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FORM: 512-1747-G

ORDER FOR WASTE REMOVAL

Disposal No. 1

Material: _____ Phone _____
 Ordered By: _____ Located at _____ Date _____ Power House _____
 Shop Order No. _____ Quantity _____ Yds., #, Gal. No. _____

<input type="checkbox"/> GOFF LANDFILL Cobb <input type="checkbox"/> Burn & Store <input type="checkbox"/> Inert Fill <input type="checkbox"/> Stone & Crush <input type="checkbox"/> Return <input type="checkbox"/> Chain & Scrap <input type="checkbox"/> Atlas	WASTE TREATMENT <input type="checkbox"/> Plant <input type="checkbox"/> Sump <input type="checkbox"/> Sludge Pond <input type="checkbox"/> Dewatering Tank <input type="checkbox"/> Oil Slt. Tank <input type="checkbox"/> Steam Plant	<input type="checkbox"/> Liq. Thin <input type="checkbox"/> Liq. Thick <input type="checkbox"/> Solid <input type="checkbox"/> Polymer Present	No. <input type="checkbox"/> Drum 55 Gal. <input type="checkbox"/> Drum 30 Gal. <input type="checkbox"/> Can 5 Gal. <input type="checkbox"/> Fiber Pak 50 <input type="checkbox"/> Fiber Pak 30	No. <input type="checkbox"/> Dump Pan <input type="checkbox"/> Dump Hopper <input type="checkbox"/> Dump Truck Dump Tank No. _____ Tank Trailer No. _____	M.D.C. FILE																							
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Name	%	H	F	S																								

UNIT HAZARD OPERATING INSTRUCTIONS

ARE AS FOLLOWS. _____

PRECAUTIONS Chemical gloves—coverall goggles—coveralls required.
 Do not allow skin contact with material. Do not breathe in any
 vapors. Follow unit hazard instructions listed.

Disposal crew must sign By: _____
 Date: _____



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Figure 11. Form order for waste removal.

Table 8 Solid Waste Production, Institute Plant, March 27, 1969; Time Base: 260-Day Year

Waste Class	Method of Disposal	Amount (yd ³ /day)	Amount (tons/day)
Burnables	Compaction and incineration in Charleston city incinerator	66	2.0
Chemical	Treatment and burial in licensed chemical landfill—Goff Mountain (UCC owned)	20	10.0
Inerts	Crushed into inert landfill—Cobb property (UCC owned)	106	39.7
Mixed burnables	Crushed into inert landfill—Cobb property (UCC owned)	60	1.8
Metals	Salvage, Sales	6	3.8
Power plant ash	Ponding and landfill at low points around plant and adjacent property	291	367.2
Power plant lime sludge	Sluiced to flyash landfill	2	1.4
Waste treatment plant sludge	Ponding and further biodegradation	33	28.0
Daily solid waste totals		584	453.9 (907,800 lbs)

service by making it the second most desirable disposal route (Table 6).

Heat recovery is a third, and rather marginal, choice as a waste-handling process. Benefit in terms of free Btus is realized, but it can be offset by environmental problems and costs that go with processing certain wastes.

To date the biooxidation and chemical landfill processes in use at the Institute location have not been operated as recovery units (although some work in this area has been done on a limited number of chemicals). Consequently, every effort is made to avoid using these two systems unless it becomes absolutely necessary.

One might say that the plant's attitude is one of fighting to keep wastes out of the treatment plant and the landfill rather than one of "using them because they are available."

Pretreatment

A cost roughly equal to the annual operating expense for the Goff Chemical Landfill is spent each year in transporting and pretreating miscellaneous wastes at their source units.

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Requirement : {

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Table 9 Institute Plant Miscellaneous Wastes and How to Handle

Institute plant miscellaneous wastes fall into the categories shown below, should be handled in the manner indicated:

	Proper Receptacle	Removal Request	Disposal
1. Burnable (Paper, rags, cafeteria waste, food and beverage containers, cardboard under 3 ft in any diagonal dimension)	Closed-top hopper marked "Burnable Waste" Only	Call truck dispatcher at 6264	Charleston incinerator (512 truck)
2. Chemical (Any chemically contaminated medium, foams and plastics, kitchen grease, spent catalysts, contaminated dewatering, used oil, urethanes (except standard insulation sections), earth, raschig rings, limestone, unrecoverable pipe and equipment)	Consult Disposal Coordinator at 6475 or 6573	By "Order for Waste Removal (Form- 512-1737-E)" Only (Submit to: Disposal Coordinator Bldg. 23 Trailer, Plant 512)	As specified in Disposal Manual
3. Inert (Uncontaminated insulation waste, finished urethane insulation sections, building demolition waste)	Open pan marked "Non-Burnable Waste," or dump truck	Call truck dispatcher at 6264	Cobb pr inert 1
4. Mixed burnables (Cardboard over 3 ft in any diagonal dimension, wood, empty fiber paks, crates, wood reels)	Open pan marked "Non-Burnable Waste," or dump truck	Call truck dispatcher at 6264	Cobb pr inert
5. Metals (All types not chemically contaminated beyond recovery)	Ground at unit stations marked "Materials to Salvage"	Call truck dispatcher at 6264	Salvage

Note. The "Disposal Co-ordinator" mentioned above is the Miscellaneous Non-Technical Supervisor (Figure 9).

Good waste preparation in advance of landfilling seems to be an important factor in the Goff facility's performance. Overwet slurries modified with more earthlike wastes from other sources are more easily blended into the lifts. Acid sludges are mixed with ash absorbents, then blended with crushed limestone to give an almost neutral mix that can be safely worked by landfill equipment. Plastic dopes are more easily managed if they can be absorbed on waste filter aids taken from presses handling compatible chemicals. Health hazards, flammability, and reactivity govern the type of pretreatment given any one waste, and processing in both the source unit and at the landfill may be necessary.

Again, the fundamental limits on the degree of pretreatment are the hazards of the specific chemicals involved and the effect on the landfill and treatment unit bacterial populations.

An up-to-the-minute working knowledge of the chemicals in the waste source plants is essential to the landfill supervisor (Figure 10). Carbide's Reactive Chemicals Reference Service provides a large part of the needed information.

Dynamic Wet Process

Carbide views its chemical landfill as a dynamic wet process biochemical system and not a vault for the impoundment of solid wastes. Its flow-through design permits the entire fill mass to be treated as a chemical process where many of the principles of solution chemistry and biological stabilization may be applied.

Earth-Waste Blending Technique

Each chemical waste charged to the Goff landfill must be worked into the lifts according to the hazards it presents, its physical state, and its degradability. This calls for a variety of techniques from reagent-filled treatment pits to simple surface mixing with the landfill's blend soil. On the average, some form of surface blending will be employed at some point in each disposal, so it is discussed here as the primary landfill process.

The procedure currently used evolved as experience with both the older impoundment-type landfill and the newer wet process was obtained.

Waste-cell construction, commonly used in sanitary landfill, was abandoned after only a few weeks of operation in 1965. Rain water collecting in the cells before coverage and on the surface after applying the necessary clay seal caused a swampy condition that mired our largest bulldozers and made it impossible to get good contact between the soil and the waste.

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Appendix Item XIVg

A primitive form of the method Carbide uses today was the revolutionary step. To avoid the miring problem and get the coverage without too much movement, a crawler crane with bucket was employed. Waste was dropped into pits (sometimes deep) from the transport trucks and the crane clammed a amount of earth in on top. Then, a mixing process with bucket was conducted. It was basically a lift-drop-lift process gradually gave a mixture that resembled soil. When the mixture much like the soil being added, it was lifted from the pit and over as wide an arc as the crane boom would permit and simply with a bulldozer during the next stretch of dry weather. The blending method worked, but it was extremely slow and Ponding after every rain finally forced the company to search for a better way to operate the fill.

Work on the design for permanent impoundment diked away at this time, so the problem of overcoming the ponding was included. In 1969 the resulting wet process was installed and the difficulties disappeared.

Today, waste is either spread on the lift surface in depth less than 6 in. or it is placed in trenches with depths limited to width of the spread is limited to that of the machine doing the (generally an end-loader with a 1 1/2-yd bucket). Blend earth at one of the narrow ends of the spread then bulldozed over to a depth of 6-in. This gives a "sandwich" of earth and waste approximately half one and half the other.

Up to this point in the process, the operator has been to maintain the 6-in. layer of earth between his machine tread and waste to avoid picking up unblended material and clogging. Next he begins to walk his machine over the sandwich. The end-loader forces the track-cleats down into the blend earth and the latter into the waste. By observation, the operator decides when the earth-waste mixture seems most homogeneous. At that time he proceeds to spread the entire mixture over as wide an area as possible on the lift.

Rubber-tired vehicles have been tried in this work, but were unable to make good blends (particularly on wet days). Motor vehicles not only do the blending well, but they also overcome the objection to placing rubber in contact with organic-chemical waste. Bulldozers and end-loaders with tracks have been used on this work. Operators, when given a choice, prefer the end-loader because of superior ability to place earth exactly where they want it. Given the same sizes, the end-loader seems to do this type of landfill work more rapidly.

Support Document: Section 1
Requirement :
Permit Application I-937-1
Appendix Item XIVh

FILL-SOIL REUSE AND SUPPLY

Ackenheil's study indicates that there will be enough overburden and "borrow" earth to sustain this landfill's blending requirements for the full 20-year life span. Supplies of this earth are removed as they are needed rather than all at once in order to keep as much natural cover on the ground as possible.

As a hedge against unforeseen problems in obtaining blendearth at the site, the landfill supervisor has taken option on all borrow removed from in-plant construction projects. What is not used at the chemical landfill is taken to the company's nearby inert landfill.

Operation has continued long enough to permit some estimation of the time required for stabilizing the waste mix being fed to the fill. Conversion to the wet process required excavation of some of the older waste-soil mixtures, and it was found that some could be reused as blend soil after as little as 18 months. It may be possible to use less than the predicted 14.7 acres over the 20-year life of the fill.

Gas Generation Control

Gas generation within the landfill has not been a problem since the wet process has been installed. The combination of blending, water flush, and no seal on the lifts seems to minimize formation and vent off dangerous concentrations uniformly over the surface and through the internal drains. Explosimeter readings and checks for hydrogen sulfide have given nil readings.

Ponding Control

Ponding is a surface phenomenon that results when rain water collects on poorly drained, clay-sealed, landfill surfaces. Seals made with semipermeable clay can produce a variation of the same problem even with proper drainage. In the latter case the semipermeable clay absorbs just enough water to become spongy. Generally, the affected area appears to be the same as the rest of the fill surface, and the condition does not come to light until some unsuspecting heavy equipment operator runs his machine into it and finds he cannot get out.

Ponding problems at the Goff property disappeared when the wet process was put into use. Without the surface seal, pools cannot form, and the internal drainage removes water from any semipermeable fill zones. Regular addition of wastes containing limestone and alumina with a particle-size of $\frac{1}{2}$ in. keeps the fill surface open and provides the added benefit of keeping the ground from freezing in the winter time. This is particularly important to the continued operation of the waste-blending operation.

Support Document: Section
Requirement :
Permit Application I-937-
Appendix Item XIVi

Drainage

Much of the necessary drainage protection has been designed for this landfill (Figure 6). There are day-to-day problems, however, which must be resolved by the operating crew.

Most of the difficulties relate to drainage immediately adjacent to the fill-lift area. This is inside the peripheral drain shown in the plan, and the trenches change position as the size of the fill lift changes. Minor local slides and siltation cause blockage in the trenches. These must be controlled by the landfill equipment operator. Periodic removal of suspected slippage zones and regular scraping of the bottoms is a regular part of his job.

Slag drains in the "leaky" dike have shown a tendency to plug when watery sludge wastes are processed. Some removal and replacement of the slag has been necessary as a consequence. (A better pre-treatment of sludge wastes seems to be correcting the problem.)

Maintenance of drain planes across the top of the fill lifts has been given close attention. A 2% slope was recommended in the original process design, and this seems to be adequate as long as the drains are kept free of slight high spots.

Slippage

Slippage is a problem around the Goff fill site because of the clay deposits and rather steep slopes on the sides of the hollow. A shear, similar to that experienced in West Virginia highway construction, occurs after long periods of high rainfall.

Landfill supervision has to be ever alert to the first signs of bulges on hillsides) of slip so action can be taken to prevent a slide. Dikes within the fill have been no problem because they are keyed to the top of the bed rock, but this technique is difficult to use on the hollow's walls.

Most of the time it is necessary to determine the full area of the slip, strip it off, remove soil to the top of underlying rock, then re-lay interlocking riprap as a base. Concrete is sometimes required for the repair. When the repair is complete, grass and trees are planted to hold it in place.

Equipment

Because of its ability to lift and place earth easily, the Carbide track-equipped end-loader is its basic piece of chemical processing equipment. It is assigned continually to waste-earth on an 8-hr/day, 5-day week basis. Through arrangement with an equipment rental agency, a backup can be obtained in the event of failure.

Support Document: Section
Requirement :
Permit Application I-93
Appendix Item XIVj

State landfill regulations specify that coverage equipment be available at every approved landfill at all times. The end-loader is left at the chemical landfill site to meet this requirement. A 30-ton crawler crane rigged with a special perforating weight for drum crushing also remains at the site. If coverage or fire problems arise, this machine can be quickly rigged with an available clam bucket. Some of the drainage and lift-surface work is done by rental bulldozers on a part-time basis. This keeps the end-loader on the blending operation and speeds disposal generally.

Gradalls, scrapers, and similar personnel are hired when necessary to dig some of the special pits needed in pretreatment, maintain the peripheral drains, and repair slips.

Dump trucks and special pan-trucks are provided by the plants that use the landfill service. The Institute keeps three pan-trucks, one dump truck, and two flat-beds busy full-time in solid waste transportation.

Waste Containers

The practice of grabbing any old can and jamming solid waste into it for treatment at the chemical landfill cannot be tolerated. Drums do not deteriorate readily (one removed from this fill after 5 years' burial still looked new and had active chemical in it), they are costly in themselves, and they take up fill volume without using it efficiently. Closing the spaces between drums placed in landfill cannot be done without considerable expense. The unsealed voids represent landfill volume not devoted to waste degradation, and they provide air passages that promote underground fires.

The Institute follows an active program of drum elimination at the waste source. Lined, slope-sided garbage cans with lids are used wherever possible. The waste-filled liners slip easily out of the cans onto the chemical fill, and the cans are taken back to the source. Plastic liners break easily under the end-loaders's weight so the waste processes satisfactorily.

Dump trucks and dump pans (both lined with plastic and unlined) deliver the bulk of the chemical waste. Problems with these vehicles center around proper loading and cleaning processes to avoid littering roads and emitting odors or material along the transport route. Haulage practices that do not meet ICC regulations give the disposal supervisor the right to refuse landfill use to the disposer. "Orders for Waste Removal" are withheld pending correction of questionable waste-handling plans.

Tank trailer rigs are currently used to move the contaminated water from the landfill. An inexpensive pipeline connecting to the sewer

Support Document: Section 8
Requirement : 8
Permit Application I-937-L
Appendix Item XIVk

Support Document: Section 10
Requirement : 1, 2, 3
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Appendix Ite

about El. 890, and proceed down the slope. Initial excavation in each borrow area should begin at the natural drainageways and continue as a bench across the hillside. By sloping the borrow area bench about 5 percent to the rear of the cut, a drainage channel will be formed. This channel should be sloped to provide drainage to the natural drainageways.

At the completion of the first cut across a borrow area, a permanent diversion channel should be constructed to divert water around the area (Figure 6). This diversion has two purposes: (1) to reduce the runoff entering the working area and creating difficult, wet working conditions, and (2) to reduce the potential for erosion in the borrow area and the resulting sediment discharge to the stream below the landfill.

4.0 CLAY SEAL AND BLANKET

A clay seal is required under the chemical landfill to minimize potential contamination of the groundwater by the leachate. This seal must be constructed prior to placing chemical wastes in the valley bottom or on the hillsides. In areas where the natural soil cover is a clay more than one foot thick after stripping the organic material, the seal may be formed by compacting the in-place soils. In areas where rock is exposed, a one-foot-thick compacted clay seal is to be placed in accordance with Specification 2.08. In all areas, this seal should extend upslope 3 to 5 feet above the surface of the landfill to insure continuity of the seal as the landfill rises.

To reduce infiltration of rainfall and to insure that runoff from the completed areas of the landfill is not contaminated by chemical wastes, the completed or inactive areas of the landfill are to be covered and sealed with a one-foot-thick compacted clay blanket (Specification 2.08). This blanket should be placed as the completed portions of the landfill are shaped and graded for drainage. The clay blanket should be constructed concurrently with the landfill bench slopes so that slope runoff is not contaminated by chemical wastes. The blanket should be topsoiled, fertilized

Support Document: Section 10
Requirement : 1, 2, 3
Permit Application I-937-L
Appendix Item XVIa 13.

- c. Earth Backfill - Structures: Earth materials used for filling and backfilling around concrete structures shall be of a quality approved by the Engineer. Large stones, clods, refuse, or debris of any kind shall not be included in the backfill. All earth backfill shall be placed in horizontal layers not over nine (9) inches thick measured loose and compacted using an approved compactor to 100 percent of Standard Proctor maximum density for cohesive soils, and 75 percent relative density for granular soils.
- d. Measurement and Payment: Backfill around concrete structures and vitrified clay pipe will not be measured for payment, and the cost thereof shall be included in the price bid for the structure or vitrified clay pipe.

2.08 EMBANKMENTS

- a. Scope: The Work to be performed under this article consists of constructing the earth fill portion of the toe embankment, the leachate dike, the channel dikes, the clay seal and blanket, and the earth fill to support the concrete channels to the lines and grades shown on the Plans or as directed by the Engineer.
- b. Preparation for Work: Before placement of any embankment or blanket material, the existing surface shall be prepared as specified under Sections 2.03, "Clearing and Grubbing," 2.06, "Foundation Preparation" and 2.04, "Stripping."

When the embankment is to be placed and compacted on sloping ground, the slopes that are steeper than six (6) horizontal to one (1) vertical shall be continuously benched over those areas. Benching shall be of sufficient width to permit operations of placing and compacting equipment. Material thus cut out shall be recomacted along with the new material.

- c. Construction: Compacted embankments shall be constructed of suitable materials at acceptable water contents, as approved by the Engineer. No embankment shall be placed upon frozen earth, and no frozen material shall be placed in any embankments. All stones or rocks larger than six (6) inches in diameter and all roots, debris, and other undesirable material shall be removed from the embankment material either prior to placement or prior to compaction.

The embankment shall be constructed in uniform layers not to exceed nine (9) inches thick, measured loose, and compacted by a sheepsfoot roller. Each lift shall extend the full width of the area being filled before starting the next lift. Each layer shall be uniform in thickness and shall be spread and

Support Document: Section 10
Requirement : 1, 2, 3
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Appendix Item XVIIb

14.

- d. Compaction: Complete control of the compaction operation shall be exercised, under the direction of the Engineer, by developing regional moisture-density diagrams covering the range of soils available. Suitable criteria for judging the adequacy of compaction attained shall be developed by making an adequate number of field density determinations and by making check compaction tests on the embankment and fill material. If there is doubt as to whether the required degree of compaction has been attained, check density and compaction tests will be immediately performed by the Engineer to provide a basis for determination. Further placing of fill material in the embankments shall cease until these checks have been made.

When each layer of material has been conditioned to the best practicable moisture content for compaction purposes, it shall be compacted by an approved tamping roller. For uniformity of compaction, a minimum of six (6) passes of the roller shall be made for each layer. A complete roller pass is defined as the passing of a specified roller over the entire surface of the layer with a minimum overlap of one (1) foot between successive trips of the roller. Successive layers shall not be placed until the layer under construction has been thoroughly compacted.

Care shall be taken to prevent over-rolling, weaving of equipment, and slickensides in the embankment material. If any portion of the embankment is overcompacted, that portion shall be worked with harrow, scarifier, or other suitable equipment to a sufficient depth, and the material shall be recompacted properly to achieve the desired density. If excess weaving of the embankment material being placed occurs, further filling shall be halted, the material disced and dried until the compaction operation does not cause excess weaving.

The distribution and gradation of the materials throughout the earth fill shall be as directed by the Engineer and shall be such that the fill will be free from lenses, pockets, streaks or layers of material differing substantially in texture or gradation from the surrounding material. The combined excavation and placing operations shall be such that the compacted impervious fill materials, when compacted in the embankment, will be blended sufficiently to secure the best practicable degree of distribution, compaction and stability, subject to the approval and direction of the Engineer.

As soon as practicable after beginning any section of embankment, the central portion shall be raised or crowned with grades of not less than two (2) percent nor more than four (4) percent so that the surface of the fill will drain freely toward the slopes.

Support Document: Section 10
Requirement : 1, 2, 3
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15.

At the close of each day's Work, or where Work is to be stopped for a period of time, the entire surface of the compacted fill shall be sealed by a method approved by the Engineer. If after a prolonged rainfall the top surface of the embankment is too wet and plastic to work properly, the top material shall be removed to expose firm soil. The top material shall be removed to expose firm soil. The top material so removed shall be disposed of at the Contractor's expense. The entire surface of any section of the embankment under construction shall be maintained in such condition that construction equipment can travel on any part of the section. Ruts in the surface of any layer shall be suitably filled or eliminated by grading before compaction. Care shall be taken to prevent excess travel over any section of the embankment to avoid over-rolling of that section.

Where compaction by means of the roller specified for use on the embankment is impracticable, or undesirable, and in locations where reasonable maneuverability of the specified roller is not possible, the Contractor will be required to use other equipment for providing compaction equivalent to that which would be obtained by the specified roller.

Moisture Control: Prior to and during the compacting operation, the material in each layer of fill shall have the best practicable moisture content for compaction. However, the water content shall not be less than five (5) percent below or more than two (2) percent above the optimum water content as determined by the moisture density relationship. The moisture content shall be uniform throughout the layer. No compaction will be permitted when moisture conditions are such that the required density cannot be obtained. Material too dry for proper compaction in the embankment must have water added prior to compaction, and material too wet for compaction in the embankment must be aerated or otherwise dried.

Moisture content-density testing shall be carried out in general accordance with the provisions of ASTM D-698 (Standard Proctor) Method C. The acceptable compaction for the embankments shall be 100 percent of the optimum dry density as determined by this testing.

Partial Embankment Construction: Due to the staged construction of the landfill, the toe embankment will be built in two sections. Where the Stage III toe embankment is tied to the Stage II toe embankment, care shall be taken to insure an adequate bond is formed between the two (2) sections of embankment. This shall be done by removing material on the face of the existing embankment and tying the new fill into this face with properly constructed

Support Document: Section 10
 Requirement : 1, 2, 3
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 Appendix Item XVII
 16.

- g. Compaction Equipment: The embankments shall be compacted with a water- or sand-ballasted sheepsfoot tamper-type roller having tamping feet uniformly staggered over its cylindrical surface and equipped with cleaners. Each tamping foot shall project not less than nine (9) inches from the roller cylindrical surface and shall have a cross-sectional area of not more than ten (10) square inches at a plane normal to the axis of the shank six (6) inches from the drum surface. The spacing shall be such as to provide not less than one tamping foot for each 100 square inches of cylindrical drum surface. The total weight of the roller in pounds divided by the total area of the tamping feet in one row parallel to the axis of the roller shall not be less than 250 pounds per square inch. The size of the roller shall be such that this unit pressure may be increased to a maximum of 750 pounds per square inch by ballasting, if required. Self-propelled tamper-type rollers which do not meet the above specifications may be used if the Contractor can demonstrate that the roller will result in the required degree of compaction without excessive passes.
- The Contractor's equipment will be calibrated and suitable adjustments made as the Work progresses.
- h. Measurement: The fill portion of the embankments will not be cross-sectioned in the field, but will be measured for payment by plan quantities based on volume placed above the actual stripping or excavation line. The stripping or excavation line shall be determined by cross sections taken in the field after completion of the stripping operations, as set forth in these Specifications. Topsoil will not be included in embankment measurement. Riprap, filter blankets, drains, and other items specified elsewhere in the Specifications will not be included in embankment measurement.
- i. Payment: Payment for construction of the fill portion of the embankments will be made at the Contract unit price bid for "Embankment," measured as specified above. This price and payment shall constitute full compensation for borrow area treatment, excavation, hauling, placing, manipulating, compacting, and for all material, equipment, labor, water, supplies and all other incidentals necessary to complete the Work.

2.09 FILTER BLANKETS

- a. Scope: Granular filter blankets as shown on the Plans shall be constructed under the downstream slopes of the toe embankment, leachate dike and beneath the concrete channels.
- b. Material: The material for the filter blankets shall be clean.

ATTACHMENT C

BIOASSAY 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 WV0000073.

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 Union Carbide Corp.

Location Tech. Center Charleston, West Virginia

Date 11 April 1978

Investigator Bruce A. Binkley

Company Representative Jack Dawson (Asst. to Gene Wiggly)

Test Method 96-hour Static A.S.T.M. and Standard
Methods 14th Edition - Just received EPA 600/4-78-012
January 1978

Dilution Water

Source D-Charleston City tap water (Chemical filtered)
Should use Reverse osmosis water

Chemical Analyses Performed None

Pretreatment Carbon filtration

Loading rate Aug 24 per fish can 1.2g → 12 grams / 15 liter

Test temperature-average and range 18-22° ± 2°C Advice use of Constant temperature water bath.

Chemical parameters monitored and frequency Dissolved Oxygen Concentration pH temperature Monitored daily - Expected to include total NH₃-N 1st half of test concentrations at beginning and end of test.

Duration and frequency of test Doubtful 96-hour static

Definition of adverse effect Death

Frequency of observations 24-hour intervals

Method of calculating EC₅₀ Stewart Line Graphical Interpolation - Process of going to Computerized program.

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

Methods used for all chemical analyses EPA approved Standard Methods

Other relevant information All test chambers are aerated by glass bubblers - Dissolved oxygen levels BED and on C12 are excessively high. Concentrations and volumetric dilutions reframed then fish randomly added. Washing test chambers - Hot water detergent, where Rinse - will require Acid Rinse - Should include 17 solvent Rinse (Acetone acceptable)

Effluent Water

Source Union Carbide Dischargers C23, C25, C32, C35, C72, C74
(C09, C14, C15, C16, C17, C24, C27, C28, C31, C36, C39) Appropriate
 Retention time None

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

Holding time and conditions Never exceeds 24-hours Occasionally
exceeds 8-hours.

Pretreatment None

Chemical Analyses Performed pH, Dissolved Oxygen Concentration,
Temperature

Test Organisms

Species fathead Minnow

Life stage Vermont 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 (Bulbair jar type)

Volume 15 liter

Volumetric exchange rate N/A

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

Number of organisms per concentration 10 fish per Concentration

ATTACHMENT D

ANALYTICAL PROCEDURES EVALUATION

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

o : 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
Institute, West Virginia

April 11, 1978

Inspection Attendees

Jack Rittenhouse
L. J. Cockran
David Vietti

Affiliation

Chief Chemist, Union Carbide
Chemist, Union Carbide
Chemist, EPA-NEIC

Introduction

The laboratory was evaluated for its ability to generate and report reliable NPDES self-monitoring data. The inspection consisted of a review of the analytical methods in use, laboratory techniques, instrument calibration and maintenance, sample preservation and holding times, data handling and record keeping, and quality control practices and documentation.

The chief chemist and his assistant were interviewed concerning chemical and bacteriological laboratory procedures. The chief chemist has approximately 19 years of analytical laboratory experience and his assistant, 24. The technical analysts have background training in analytical chemistry ranging from 1 to 25 years.

Standard reference samples for BOD, TOC, suspended solids, chlorides, total Kjeldahl nitrogen, pH, TDS, total alkalinity, ammonia, sulfate, fluoride, and orthophosphorous were 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 inspection, the data and information obtained, the evaluation form presented on the following pages was completed and conclusions, summary, and recommendations were prepared.

Conclusions and Summary

Samples collected during weekends were iced, but not analyzed until the following Monday. These samples were not properly preserved. Samples for TOC, $\text{NH}_3\text{-N}$, and TKN require acid as well as cooling to assure stability. Samples preserved only by cooling may have low TOC + TKN results compared to properly preserved samples. $\text{NH}_3\text{-N}$ results would tend to be higher if organic matter is present.

Total dissolved solids are being "analyzed" by subtracting the difference between total and suspended solids. This is not consistent with the approved procedure. TDS results obtained in this manner would be higher than true values.

An excellent analytical quality assurance program consisting of routine and blind duplicates as well as blind and routine spikes and reference samples is in use for all permit parameters. Technical guidance and continuing updating of quality control procedures is being provided by the Union Carbide Technical Center.

Recommendations

1. Recommended preservation techniques should be employed when the samples are not being analyzed as soon as compositing is completed. Acid should be added to the sample for proper preservation when the parameter to be analyzed is TOC, TKN, or $\text{NH}_3\text{-N}$.
2. Sample holding times prior to analyses in conformance with 40 CFR 136.3 regulations must be adhered to.
3. Total dissolved (filterable) residue must be analyzed according to approved EPA analytical testing procedures (40 CFR 136.3).
4. The use of the 5-tube MPN procedure for NPDES self-monitoring should be initiated immediately.
5. In order to improve their already excellent analytical quality assurance program, thermometers calibrated with a National Bureau of Standards thermometer should be purchased. Control charts could be constructed to further document their analytical quality control program.
6. Generally, the results of the reference samples indicated that the laboratory facility was doing an adequate to good job for most parameters in generating data for the NPDES self-monitoring program. However, TDS, TOC, and low level TKN results were not within acceptance limits. The results of these parameters would place serious doubt upon the validity of past reported data. Further participation in a performance evaluation program on a regular basis may establish the validity of the past self-monitoring data.

- ☒ 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:

9. and 11. Consistently proper preservation techniques are not being employed. The samples collected on Saturday and Sunday are not being analyzed until Monday.

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.

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 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. Good workable recording and recordkeeping system are being utilized. All the original documents and bench cards 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 his own green record notebook and they are reviewed periodically by the supervisory chemists.

13. An excellent company internal auditing program in existence and in use.

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

General Comments on Laboratory Procedures:

1. Dissolved solids were being "analyzed" by subtracting the difference between total and suspended solids.
3. Additional parameters are being analyzed to meet state permit requirements.
5. The state of West Virginia does not have a state certification program
- 7-9. The laboratory quality control program consists of analysis of routine and blind duplicate samples as well as known and unknown spiked and reference samples. The program is administered internally with assistance and guidance provided by the Union Carbide Technical Center. The results looked excellent.

Results of NEIC Quality Control Check Samples Analyzed by Laboratory

Parameter	Union Carbide at Institute, mg/l	True, mg/l
TOC	1) 20*	1) 44.8
	2) 121*	2) 165
BOD	1) 16	1) 28.7
	2) 160	2) 264
TSS	AA) 25.8	AA) 30
	AC) 900	AC) 950
Chlorides	1) 35.8	1) 28.1
	2) 100.1	2) 85.4
TKN	1) 1.17*	1) 0.34
	2) 5.35	2) 4.80
NH ₃	1) 0.19	1) 0.15
	2) 1.66	2) 1.72
pH	1) 7.3	1) 7.9
	2) 8.1	2) 8.4
TDS	1) 131*	1) 71.7
	2) 460*	2) 318.3
Total Alkalinity	1) 17.6	1) 10.4
	2) 37.1	2) 35.7
Sulfate	1) 9.9	1) 12.0
	2) 106.4	2) 102.4
Fluoride	1) 0.16	1) 0.2
	2) 1.00	2) 1.1
Orthophos	1) 0.01	1) 0.017
	2) 0.51	2) 0.513

*Not within acceptance limits.

APPENDIX B
LITHIUM FLOW VERIFICATION PROCEDURES
AND
SAMPLING PROCEDURES

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

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

SAMPLING PROCEDURES

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 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 ₂ SO ₄ & Ice 4. Oil & Grease/H ₂ SO ₄ & Ice 5. Phenolics/H ₃ PO 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: right;">☆GPO 777-941</div>

reverse

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

CHAIN OF CUSTODY RECORD

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
Building 53, Box 25227, Denver Federal Center
Denver, Colorado 80225

[illegible]

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

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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, Organics, and Trace Metals. 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 - BOD, TSS, 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 I below.

Parameter	Technique	Detectn. Limit mg/l	Reference
BOD	Multiple bottle dilution	2	Std. Methods ^a pg 543
TSS	Glass fiber filter filtration	1	Std. Methods pg 94
TOC	Combustion-Infrared	1	Std. Methods 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
Chloride	Mercuric nitrate	1	Std. Methods pg 616
			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.

Written methods prepared from "Standard Methods" for BOD and TSS are included as Attachments I & II. Additional precautions taken during the analysis of the samples are discussed below by parameter.

BOD

The dissolved oxygen meter was calibrated by the azide modification of the Winkler method ("Standard Methods", 14th edition, 1975, pg. 443) to assure accurate D.O. measurements. Samples were seeded with seed material that was acclimated to the specific waste being studied. The D.O. depletions were normal for all dilutions of all samples.

Quality control consisted of duplicate analysis of seven samples and analysis of EPA reference sample #276-2 on six different days. Additional quality control procedures are described in Attachment I. Since two duplicate samples did not have valid dilutions, the precision was calculated from five sets of data. The relative standard deviation of the duplicate results is 25%. One reference sample result was invalid because of improper preparation. The mean accuracy of the five valid reference sample results is 94.5%.

TSS

The analytical and quality control procedures described in Attachment II were closely followed. The relative standard deviation of five duplicate determinations is 3%. The mean accuracy of analysis of a standard reference sample on four different days was 105%.

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

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.

METALS

The samples from this study were analyzed for the following metals: Al, As, Cd, Cr, Cu, Ni, Pb, Sn, and Zn. The samples consisted of water samples. Methods approved by the EPA for the NPDES program (40 CFR 136, Federal Register, December 1, 1976) were used in the analysis of all water samples. The references to the methods used in the analysis of the water samples for each metal and the detection limits for each metal are listed in Table I. The detection limits in Table I for the water samples are reported in units of milligrams per liter.

The methods listed in Table I for each element were closely followed. There were no significant deviations from the approved method. As an added precaution, all analyses, with the exception of mercury, were performed using background correction procedures in order to preclude extraneous signals from the sample matrix.

Water Samples

Aluminum: Sample replicates and spikes were analyzed for aluminum. Only one sample replicate contained a detectable quantity for aluminum. This replicate agreed with the original sample within 17%. The recoveries for the sample spikes ranged from 80% to 100% with an average recovery of 87%. This represents a slight negative bias in the aluminum results. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.9 mg/l while the true value was 0.904 mg/l aluminum.

Arsenic: Sample replicates and spikes were analyzed for arsenic. Only one sample replicate contained a detectable quantity of arsenic. This replicate agreed with the original sample within 12%. The recoveries for the sample spikes ranged from 110% to 150%, with an average recovery of 130%. This represents a positive bias in the arsenic results. The EPA reference standards #2 and #3, lot 575, were analyzed. The experimental values were 0.11 and 0.16 mg/L, while the true values were 0.109 and 0.154 mg/L arsenic respectively.

Cadmium: Sample replicates and spikes were analyzed for cadmium. None of the sample replicates contained detectable quantities of cadmium. The recoveries for the sample spikes ranged from 104 to 110%, with an average recovery of 106%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.06 mg/L, while the true value was 0.073 mg/L cadmium.

Chromium: Sample replicates and spikes were analyzed for chromium. None of the sample replicates contained detectable quantities of chromium. The recoveries for the sample spikes ranged from 102% to 104% with an average recovery of 103%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.2 mg/l, while the true value was 0.204 mg/l chromium.

Copper: Sample replicates and spikes were analyzed for copper. Only one sample replicate contained a detectable quantity of copper. This replicate agreed with the original sample within 26%. This represents a difference in concentration of only 0.03 mg/l. The recoveries for the sample spikes ranged from 96% to 104% with an average recovery of 99%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.1 mg/l, while the true value was 0.102 mg/l copper.

Nickel: Sample replicates and spikes were analyzed for nickel. The replicate results varied from 3% to 35% relative percent difference. The 35% difference represents a concentration difference of only 0.03 mg/l. The recoveries for the sample spikes ranged from 102 to 110% with an average recovery of 107%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.21 mg/l, while the true value was 0.152 mg/l nickel.

Lead: Sample replicates and spikes were analyzed for lead. None of these sample replicates contained detectable quantities of lead. The recoveries for the sample spikes ranged from 92% to 134% with an average recovery of 113%. This represents a slight positive bias in the lead results. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.45 mg/l, while the true value was 0.352 mg/l lead.

Tin: Sample replicates and spikes were analyzed for tin. None of the sample replicates contained detectable quantities of tin. The recoveries for the sample spikes ranged from 58 to 90% with a mean recovery of 74%. This represents a negative bias in the determination of tin. This is not surprising since tin is known to be unstable in solution. The EPA reference standard #3, lot 575, does not contain tin. Therefore, no AQC data is available for tin.

Zinc: Sample replicates and spikes were analyzed for zinc. The relative percent difference for the zinc replicates ranged from 0% to 29%. The 20% relative percent difference represents a concentration difference on only 0.015 mg/l. The recoveries for the sample spikes ranged from 152% to 168% with an average recovery of 159%. This represents a positive bias in the zinc results. Laboratory contamination of the zinc spikes was investigated by determining the zinc concentrations of laboratory reagent blanks using the same acid that was used to preserve the samples in the field. The laboratory reagent blanks were found to contain no zinc. The EPA reference standard #3, lot

575, was analyzed for zinc. The experimental value was 0.17 mg/l, while the true value was 0.174 mg/l zinc. The fact that the experimental results for EPA reference standard #3, log 575, were in good agreement with the true value provided by the Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, together with the fact that the reagent blank contained no zinc, indicates that the water samples were inadvertently spiked at a higher level than that which was expected. The average value of the field blanks was 0.04 mg/l zinc. Sample results having this approximate concentration are questionable.

Table 1
ANALYTICAL METHODS AND DETECTION LIMITS - WATER SAMPLES

Metal	Technique	Detection Limit, mg/l	Reference ¹
Al	Flame Atomic Absorption	0.3	A, p. 92
As	Flameless Atomic Absorption	0.002	B
Cd	Flame Atomic Absorption	0.03	A, p. 101
Cr	Flame Atomic Absorption	0.04	A, p. 105
Cu	Flame Atomic Absorption	0.04	A, p. 108
Ni	Flame Atomic Absorption	0.06	A, p. 141
Pb	Flame Atomic Absorption	0.2	A, p. 112
Sn	Flame Atomic Absorption	1.0±	A, p. 150
Zn	Flame Atomic Absorption	0.01	A, p. 155

¹A = Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, (1974).

B = Atomic Absorption Newsletter, 14 109 (1975).

ORGANICS

Several techniques for the analysis of organic compounds were utilized for the waste source evaluation, Union Carbide facilities and South Charleston WWTP Survey. 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, volatiles,

and selected samples were analyzed for priority pollutants. Other samples, notably nonpurgeables, were analyzed by direct aqueous injection analysis (DAI). Carbaryl was analyzed by high pressure liquid chromatography (HPLC).

NEUTRAL EXTRACTABLE ANALYSIS

GC/MS Identification: Methylene chloride extracts of the water, and acetone extracts of the sediment samples were concentrated to small volumes and exchanged with isooctane and analyzed by GC/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 of the complete computer library and a follow up 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.

Pack 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 V to X).

DIRECT AQUEOUS INJECTION ANALYSIS (DAI)

Selected samples were analyzed by DAI gas chromatography/mass spectrometry (GC/MS). An aliquot of a sample is injected directly into the inlet system of a gas chromatograph interfaced to a mass spectrometer equipped with a computerized data system. Generally, low boiling semi-volatile compounds that purge poorly are analyzed by this method.

Quality Control: Blanks, duplicate and spiked samples were analyzed concurrently with the survey samples. None of the thirteen selected DAI compounds were found in any of the three blank samples.

Five spiked samples representing eleven compounds were analyzed. (One sample contained as many as three spiked compounds. Some compounds, such as acetone were spiked into more than one sample.) Of the eleven discrete spikes the mean recovery was 116% with a Relative Standard Deviation of 19%.

Two sets of replicates were analyzed with four compounds detected. The average percent Relative Standard Deviation (% RSD) was 15. The average percent difference of all the sets of replicates was 22.

VOLATILES 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 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 Kanawha 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.

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 $\mu\text{g/l}$.

Compound	<u>Summary of blank results ($\mu\text{g/l}$)</u>		
	Times Detnd. (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 Detnd. (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%
Tetrachloroethane	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 µg/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 number 1276 WS.

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

PRIORITY POLLUTANTS ANALYSIS

GC/MS Identification: Selected samples were analyzed for priority pollutants by GC/MS using the recommended EPA procedure (1). The volatiles were measured using the same technique described previously for the volatiles analysis, because both techniques are the same. The extractable organics were analyzed for both acids, and neutrals, and bases combined as recommended.

REFERENCES

1. "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S. EPA, EMSL, Cincinnati, Ohio, March 1977, revised April 1977.

ATTACHMENT I
BIOCHEMICAL OXYGEN DEMAND - DO PROBE PROCEDURE

(5 Days, 20°C)

STORET NO. 00310

1. Scope and Application

- 1.1 The biochemical oxygen demand test is a laboratory bioassay procedure used to estimate the quantity of oxygen that is required to stabilize the biodegradable matter in a wastewater.
- 1.2 The test was originally designed for and works most reliably on raw and treated domestic wastes. The test can be applied to industrial wastes with careful attention to interferences and correct choice of biological seed.

2. Summary of Method

- 2.1 An appropriate number of dilutions of each sample are prepared using dilution water with added nutrients so that at least one dilution has a depletion of at least 2 mg/l and a residual DO of at least 1 mg/l after incubation for 5 days in the dark at 20°C.
- 2.2 Dissolved oxygen is measured by a DO probe based on the polarographic principle. The probe is calibrated with air saturated water at known temperature and atmospheric pressure.

3. Sample Handling and Preservation

- 3.1 Samples should be stored in ice or in a refrigerator at 4°C and analyzed as soon as possible but no later than 24 hours after collection.

4. Apparatus

- 4.1 Glass or tin-lined still to produce distilled water.
- 4.2 Five gallon glass bottles wrapped with nylon tape to store dilution water.
- 4.3 Incubation bottles, approximately 300 ml, with standard ground glass tops and plastic caps to maintain water seals. The exact volume of each bottle is measured using water at 20°C with class A volumetric glassware and any that are not 300 ± 5 ml are discarded.
- 4.4 An incubator with a continuous temperature recorder controlled at $20 \pm 1^\circ\text{C}$. A calibrated mercury thermometer is placed in the incubator in a water-containing flask and the temperature is checked daily.
- 4.5 A dissolved oxygen meter, automatically temperature compensated, if possible, with a self-stirring probe.
- 4.6 A Tekmar SDT Tissuemizer with variable speed control to homogenize samples.
- 4.7 Barometer

5. Reagents

- 5.1 Distilled water, free of organic contaminants as indicated by the Permanganate Test as follows: Determine the consumption of potassium permanganate by adding 0.20 ml of KMnO_4 solution (0.316 g/l) to 500 ml of the distilled water and 1 ml of conc. H_2SO_4 in a stoppered glass bottle. The water has passed the test if the permanganate color does not disappear in less than 10 minutes upon standing at room temperature. Ideally, the color should be retained for 30 minutes.
- 5.2 Phosphate buffer solution: Dissolve 8.5 g potassium di-hydrogen phosphate, KH_2PO_4 , 21.75 g dipotassium hydrogen phosphate, K_2HPO_4 , 33.4 g disodium hydrogen phosphate heptahydrate, $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$ and 1.79 g ammonium chloride NH_4Cl in about 500 ml distilled water and dilute to one liter. The pH of this buffer should be 7.2. Store in the refrigerator and discard (including any of the following reagents) if there is any sign of biological growth in the bottle.
- 5.3 Magnesium sulfate solution: Dissolve 22.5 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in distilled water and dilute to one liter.
- 5.4 Calcium chloride solution: Dissolve 27.5 g anhydrous CaCl_2 in distilled water and dilute to one liter.
- 5.5 Ferric chloride solution: Dissolve 0.25 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in distilled water and dilute to one liter.
- 5.6 1 N H_2SO_4 and 1 N NaOH solutions.
- 5.7 Sodium sulfite solution, 0.028 N: Dissolve 1.77 g anhydrous Na_2SO_3 in one liter distilled water. Prepare daily.
- 5.8 Reagent grade potassium iodide.
- 5.9 Starch indicator solution: Add a cold water suspension of 59 g soluble starch to 800 ml of boiling water, with stirring and boil for a few minutes. Cool, dilute to approximately 1 liter and let settle overnight. Use supernate and preserve with 5 ml of chloroform.
- 5.10 Glucose-glutamic acid solutions: A) Dissolve 150 mg of each in distilled water and dilute to 1 liter. B) Dissolve 100 mg of each in distilled water and dilute to 1 liter. Split up each solution into 25 ml bottles or tube, autoclave at 121°C for 1/2 hour, and store at 4°C or prepare fresh daily.
- 5.11 Biological seed.

6. Glassware and Dilution Water Preparation

- 6.1 All dilution water and reagent storage bottles, BOD incubation bottles, and other glassware must be free of organic contaminants and toxic metals. Clean all glassware with hot soapy water, rinse with 3 N HCl , rinse three times with hot tap water and twice with distilled water. Any glassware with a film should not be used.
- 6.2 The distilled water should be cooled to 20°C , saturated with oxygen by bubbling air through the water and then stored at 20°C until use. Just prior to using the dilution water, add 1 ml each of the magnesium sulfate, calcium chloride, ferric chloride, and phosphate buffer solution solutions for each liter of water. The biological seed should be added (5 ml seed/l of dilution water) to the dilution water just before use.

7. Selection of Seed

7.1 All chlorinated domestic wastes and most industrial wastes require seeding because of low microbial populations. The standard seed material is primary treated sewage that has been stored at 20°C for 24 hours. However, it is important that, if possible, the seed to be used has been exposed to the waste that is being measured. Therefore, an effluent from a treatment process or a receiving water collected below the outfall will sometimes be used as seed material.

8. Interferences and Pretreatment of Samples

- 8.1 Blend samples containing non-homogeneous particulate matter with the Tekmar SDT Tissuemizer. Thirty seconds is usually adequate.
- 8.2 Neutralize samples with a pH outside of the range 5-10 using the 1 N acid or base. Most samples do not require neutralization because the buffering capacity of the dilution water and dilution of the samples.
- 8.3 Residual chlorine kills the seed organisms. All samples except those known not to contain residual chlorine should be checked as follows: Add 5 ml of 1 N H_2SO_4 , 2 g KI crystals and 1 ml of starch solution to 100 ml of sample. Add the 0.028 N sodium sulfite solution in 0.1 ml increments until the purple color disappears. Each 0.1 ml increment corresponds to 1 mg/l Cl_2 . Add a proportional volume of 0.028 N sodium sulfite to an aliquot of sample for testing. If there is any uncertainty, add an extra increment of sulfite. An excess of sulfite solution of 1 ml/l sample causes a BOD of less than 0.5 mg/l, which is insignificant.
- 8.4 Many organic compounds and trace metals are toxic to the seed organisms. Sometimes this interference can be eliminated by sample dilution. Higher BOD values from the more dilute aliquots is evidence of sample toxicity. These results should be carefully evaluated before being reported.
- 8.5 Samples containing more than 9 mg/l DO at 20°C may be encountered during winter months or in localities where algae are growing actively. To prevent loss of oxygen during incubation of these samples, reduce the DO to saturation by bringing the sample to about 20°C in a partly filled bottle and agitating it by vigorous shaking.

9. Calibration of Dissolved Oxygen Meter

- 9.1 Carefully fill 3 BOD bottles by use of a siphon with dilution water (containing nutrients but not seed) that has been saturated with air at 20°C. Using the table in the DO meter manual, find the DO concentration at the ambient atmospheric pressure and 20°C. Set the temperature dial on the meter if necessary to 20°C and adjust the calibration knob until the meter reads the value determined from the table. Save the other two bottles for checking the meter during the analysis.
- 9.2 Drifting of the meter response or a very slow response to DO changes is usually caused by a coated or torn electrode membrane.

10. Sample Analysis Procedure

- 10.1 Since most samples require more than 7 mg/l of O_2 for stabilization, dilutions are required before incubation. Prepare a sufficient number of dilutions so that at least one aliquot depletes at least 2 mg/l and has a residual DO of at least 1 mg/l after incubation. Usually three and sometimes four dilutions are required. Dilutions up to 1% are made directly in the BOD bottles. A guide to sample size selection follows:

<u>Measurable BOD Range</u>	<u>Sample Size, ml</u>	<u>Factor</u>	<u>% Dilution</u>
4 - 12	150	2	50
8 - 24	75	4	25
12 - 36	50	6	16.67
20 - 60	30	10	10
40 - 120	15	20	5
60 - 180	10	30	3.33
120 - 360	5	60	1.67
200 - 600	3	100	1

For dilutions less than 1%, the sample is first diluted 1/10 or 1/100 with dilution water and then the dilutions are completed in the BOD bottles. The samples should be homogenized and shaken just before aliquots are taken. A graduate cylinder is used to measure volumes of 15 ml or larger. Large bore pipets are used for smaller volumes. One bottle per dilution is prepared. Exercise care in filling the bottles with dilution water so as not to have the water into the neck of the bottle more than 1/8".

- 10.2 Prepare two bottles with seeded dilution water. Depletion of these samples should be about 0.6 mg/l if domestic sewage is used for seed. Blank values over 1.0 mg/l indicates contaminated dilution water or incubation bottles.
- 10.3 Prepare one bottle with 5 ml of glucose-glutamic acid standard A and one bottle with 10 ml of standard B and fill with seeded dilution water. The results for standards A and B should be about 200 and 160 mg/l, respectively.
- 10.4 Measure the initial DO of all samples, being careful not to displace any of the dilution water. At the same time the DO is measured, the probe mixes the samples. Wash the probe with distilled water between each sample. After determining the DO it may be necessary to add a small amount of dilution water to prevent trapping bubbles in the bottle when stoppering. Place a water seal in the neck of the bottle and place a cap over the neck to maintain the water seal.
- 10.5 It is helpful to measure the DO of the samples after two days in order to judge the adequacy of the dilutions selected. Pour off the water seal before measuring the DO. Calibrate the DO meter according to the directions given in Section 9. Measure the DO of the most concentrated dilution of each sample. If there is less than 2 mg/l residual DO, increase the dilution factors on subsequent days and measure the DO in the next most dilute sample. If the DO on the second sample is less than 4 mg/l, re-aerate with an air stone attached to an air pump being careful not to displace any of the water. Record the initial residual and re-aerated DO values. Discard any sample with a residual DO below 1 mg/l. If there is less than a 2 mg/l depletion, increase the strength of the dilutions on subsequent days.
- 10.6 The final DO measurements are made within 4 hours of 5 days of when the samples were set up. Calibrate the DO meter by the method given in section 9. Any dilutions resulting in residual DO's that are 1 mg/l or greater and depletions that are 2 mg/l or greater are valid. Calculate the BOD values by the following formula:

$$BOD_5 = F[(D_i - D_f) - f(B)]$$

where D_i = initial DO of sample, mg/l

D_f = final DO of sample, mg/l

B = the mean depletion of the two seeded dilution water blanks, mg/l

f = decimal fraction of dilution water in sample bottle

F = whole number dilution factor of sample

For example, 30 ml of sample was used, the initial DO was 8.2 mg/l and the final DO was 1.7 mg/l. The initial DO of both of the seeded blanks was 8.1 mg/l and the final DO was 7.3 mg/l

$$\begin{aligned} BOD_5 &= 10[(8.2 - 1.7) - 0.9(8.1 - 7.3)] \\ &= 10[6.5 - 0.9(0.8)] \\ &= 10[6.5 - 0.7] \\ &= 10[5.8] \\ &= 58 \text{ mg/l} \end{aligned}$$

- 10.7 Report the average value of all of the valid dilutions to the nearest whole number with at most two significant figures. If the DO depletions increase with increasing dilution, toxicity is indicated and the results should be carefully evaluated before being reported.
- 10.8 The results of the A&B glucose-glutamic acid standards should be between 160-240 and 130-190 mg/l, respectively. High results indicate a very efficient seed or contaminated samples. Low results indicate a poor seed or blank values that were too high.
- 10.9 The mean of the seeded dilution water blank depletions should be below 1 mg/l, ideally 0.6 mg/l. High values indicate contaminated nutrients and minerals, dilution water or glassware. Correct any problems before proceeding.
- 10.10 Report the BOD values from different dilutions as duplicates on the AOC sheets.
- 10.11 Attach the incubator temperature recorder chart to the BOD Data/Calculation Sheet (attached).

Prepared by H. Carter 6/9/78

BOD Data/Calculation Sheet, Rev. 6/9/78

Analyst _____ Study _____

Date/Time In _____
Date/Time Out _____

B-18

Sample No.													
Sample pH													
Sample vol, ml													
Initial DO, mg/l													
2-day DO, mg/l													
Re-aerate DO, mg/l													
Final DO, mg/l													
Gross O ₂ dep., mg/l													
Blank Corr., mg/l													
Net O ₂ depl., mg/l													
Factor													
BOD ₅ , mg/l													
Mean BOD ₅ , mg/l													
Sample No.													
Sample pH													
Sample vol, ml													
Initial DO, mg/l													
2-day DO, mg/l													
Re-aerate DO, mg/l													
Final DO, mg/l													
Gross O ₂ depl., mg/l													
Blank Corr., mg/l													
Net O ₂ depl., mg/l													
Factor													
BOD ₅ , mg/l													
Mean BOD ₅ , mg/l													

DO Probe Calibration

	temp	horr.	press.	DO, mg/l
initial				
2-day				
5-day				

TOTAL SUSPENDED SOLIDS

STORET NO. 00530

1. Scope and Application
 - 1.1 The method is applicable to drinking, surface and saline waters, and to domestic and industrial wastes.
 - 1.2 The detection limit of the method is 1 mg/l.
2. Summary of Method
 - 2.1 A homogenized sample is filtered through a pre-washed glass fiber filter. The residue retained on the filter is washed and then dried to constant weight at 105°C and weighed to the nearest 0.1 milligram. The TSS is calculated from the amount of residue per unit volume of sample.
 - 2.2 The filtrate from this method may be used to determine the total dissolved solids.
3. Sample Handling and Preservation
 - 3.1 Samples should be stored at 4°C and analyzed as soon as possible, but no later than 7 days after collection.
4. Apparatus
 - 4.1 Whatman GF/C glass fiber filter discs, 43 mm.
 - 4.2 Millipore membrane filtering apparatus with reservoir and a coarse fritted disc as a filter support.
 - 4.3 Aluminum drying pans, 50 mm and metal tray.
 - 4.4 Tekmar SDT Tissuemizer.
 - 4.5 Drying oven, 103°-105°C.
 - 4.6 Desiccator, with Drierite indicating desiccant.
 - 4.7 Analytical balance, 160 g capacity or larger, sensitive to 0.1 mg and one weight equivalent to the optical range of the balance.
 - 4.8 Graduate cylinder and wide bore pipets.
5. Balance Calibration
 - 5.1 Using a balance with an optical range of 1.0 g, place a 1.0 g (15%) weight on the balance pan, set the weight control knob to 1.0 g, release the balance and set the zero point with the optical zero knob. With the balance released, slowly turn the weight control knob back to zero. The optical scale should come to rest exactly at 1.0 g. If the reading is more or less than 1.0 g, arrest the balance, remove the top housing cover and adjust the sensitivity weight. Repeat the calibration check.
6. Procedure
 - 6.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus with wrinkled surface up. While vacuum is applied, wash the disc with 100 ml of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus, place in aluminum pan, and dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Weigh immediately before use. After weighing, handle the filter with forceps only.

- 6.2 Homogenize all non-uniform samples with blender and shake the bottles before withdrawing an aliquot to assure taking a representative sample.
- 6.3 Choose a maximum sample volume that will filter in 5 minutes or less. Measure volumes smaller than 15 ml with wide bore pipets and larger volumes with graduate cylinders. Discard any sample which does not filter in 5 minutes and filter a smaller sample volume.
- 6.4 Wash the graduated cylinder or pipet and with the suction on, wash the filter funnel wall, filter and residue with two twenty-five ml portions of distilled water allowing complete drainage between washings. Remove all traces of water by continuing to apply vacuum after water has passed through.
- 6.5 Carefully remove the filter from the filter support. Place in an aluminum pan and dry at least one hour at 103-105°C. Cool and weigh immediately or place in a desiccator for later weighing. Re-dry and re-weigh 10% or at least one filter per set of samples. If the incremental weight loss is less than 0.5 mg, calculate the results based on the original weights. If the weight loss exceeds 0.5 mg, re-dry and re-weigh all of the filters and re-check 10% of the filters.
- 6.6 Analyze two blanks per set of samples by filtering 100 ml of distilled water through two prepared filters. The amount of additional weight loss after the filters have been prepared is nearly independent of the volume of water filtered. Therefore, add the mean blank weight loss to the residue weight for each sample.
- 6.7 Analyze 10% or at least one sample per set in duplicate.
- 6.8 Analyze a standard sample with each sample set.
- 6.9 Calculate the results as follows:

$$TSS = \frac{(W_G - W_T) + B}{V_S}$$

W_G = Gross weight of filter and residue, mg

W_T = Tare weight of filter, mg

B = The mean of the two blank results, mg

$$\text{Where } B = \frac{B_1 + B_2}{2}$$

$$B_1 = B_T - B_G$$

B_T = Tare weight of filter, mg

B_G = Gross weight of filtering

V_S = Volume of sample filtered, l

Analyst _____ Study _____ Date/Time Filters in Oven _____
 Date/Time Out _____

Sample No.										
Sample Vol., l										
Re-check Wt., mg										
Gross Wt., mg										
Tare Wt., mg										
Residue Wt., mg										
Blank Corr., mg										
Corr. Res. Wt., mg										
TSS, mg/l										
Sample No.										
Sample Vol., l										
Re-check Wt., mg										
Gross Wt., mg										
Tare Wt., mg										
Residue Wt., mg										
Blank Corr., mg										
Corr. Res. Wt., mg										
TSS, mg/l										
Sample No.										
Sample Vol., l										
Re-check Wt., mg										
Gross Wt., mg										
Tare Wt., mg										
Residue Wt., mg										
Blank Corr., mg										
Corr. Res. Wt., mg										
TSS, mg/l										

Balance Calibration

	Reading on 100 mg weight, mg
Tare	
Gross	
Re-check	

Analysis of Organic Pollutants in Water by Direct
Aqueous Injection, Gas Chromatography-Mass Spectrometry

NEIC - September 1978

1.0 Introduction

- 1.1 Many volatile organic compounds are soluble in water at concentrations exceeding 1 mg/l. However, they are not suitable for Volatile Organics Analysis (V.O.A.) due to their low purgeability. This method is suitable for GC/MS identification, confirmation, and quantitation of the previously mentioned types of compounds.

2.0 Summary of Method

- 2.1 A sample is injected into the inlet system of a gas chromatograph. After vaporization, the aqueous sample is carried through a column by an inert carrier gas. The sample components are partitioned between the carrier gas and a stationary liquid phase on an inert solid support. The column effluent is introduced into a quadrupole mass spectrometer by means of a glass jet separator. From the interface, the sample is passed into an electron impact ionization source. The various ion fragments are filtered by a quadrupole mass filter and detected by a continuous dynode electron multiplier. The signal is then fed to a computer controlled data system for processing. Compounds are matched with standard spectra stored in a library and identified based upon their spectral similarity and relative retention times. Concentrations are calculated for each identified compound based upon its relative response to an internal standard.

3.0 Interferences

- 3.1 Particulate matter - Particulate or suspended matter should be removed to prevent both plugging of syringes and formation of condensation nuclei. Allowing particulates to settle before analysis is acceptable.
- 3.2 Stability - Aqueous solutions of D-chloroform (CDCl_3) are unstable. The CDCl_3 can exchange to CHCl_3 .

Stock standards of deuteriochloroform (7500 ng/ μl) that are prepared 24 hours prior to dosing and analysis, display large losses in response.

Even stock standards prepared 8 hours prior to dosing and analysis, exhibit some loss of response. Stock standards of D-chloroform are prepared in vials that have approximately two ml. head space. Volatility losses occur in this head space. No losses are observable if the stock standard solution is refrigerated and used within four hours of preparation.

- 3.3 Identical Retention Times - It is possible with any given column and operating conditions, to have two compounds that elute at identical retention times. It is especially important to choose an internal standard that does not coelute with another compound of interest. This problem is minimized by using GC/MS.

4.0 Apparatus

- 4.1 Finnigan 3200 Gas Chromatograph/Mass Spectrometer System with a Finnigan INCOS data system and Revision 3.1 software (1).

5.0 Reagents and Materials

- 5.1 Purity of Reagents - All chemicals used for standards and internal standards shall be of the highest purity available.

- 5.2 Purity of Water - All water shall be of sufficient purity such that no background is observed above the detection limit of the compounds of interest. Filtration through activated carbon will eliminate any interferences.

- 5.3 Carrier Gas - Only high purity helium shall be used.

5.4 Column

- 5.4.1 Column Tubing - Stainless steel, oil free. Dimensions 1/8" OD x 20'.

- 5.4.2 Solid Support - Chromosorb W acid washed 80/100 mesh.

- 5.4.3 Liquid Phase - Carbowax 20m - 5% loading.

- 5.5 Internal Standard - Dilute 50 μ l of deuteriochloroform to 10 ml with water. Shake well to assure all D-chloroform is in solution. The concentration of this solution is 7500 ng/ μ l.

- 5.5.1 Prepare this solution fresh every four hours and keep refrigerated.

5.6 Standards

- 5.6.1 Concentrated Standards - Prepare stock standards of each compound of interest by weighing out 50 mg of pure compound and diluting this with water to a volume of 50 ml. Stability of stock solutions is enhanced by keeping the solutions refrigerated. Stock solutions should be prepared fresh every two weeks.

- 5.6.2 Analytical Standards for GC/MS - Dilute the concentrated standards by adding 0.5 ml of each concentrate to a 12 ml vial and bringing the volume to 10 ml. This working standard should be prepared each day. Each μ l of working standard is equal to 50 ng (50 ng/ μ l).

- 5.7 Mass Spectrometer Performance Standard - Prepare a 150 ng/ul aqueous solution of Pentafluorobromobenzene and refrigerate until ready for use. This solution is stable for one month.

6.0 Samples and Sampling Procedure

- 6.1 Sample Collection - Samples should be collected so that no air remains in the bottle as a head space once the vial cap is tightened.
- 6.2 Sample Containers - 1 oz. glass bottles equipped with Teflon lined silicone septa and screw caps (Pierce #13074 and #12722 or equivalent). Before sampling, wash used bottles with soap (Alconox or equivalent) and tap water, rinse with tap water. New bottles require only washing with tap water. Bake bottles at 200°C and septa at 80°C for 30 minutes. Allow to cool in a desiccator with charcoal adsorbant to maintain an organics free atmosphere. Then cap the bottles and hold for sampling.
- 6.3 Sample Size for Analyses - The sample size must be small to prevent overloading of the column. For aqueous analysis, a sample size of 5 ul is optimum.
- 6.4 Sample Storage - Storage time of samples should be kept to a minimum. If storage cannot be avoided, the bacterial action, as well as volatility losses, should be minimized by refrigeration (2).

7.0 Procedure

7.1 Mass Spectrometer Calibration

- 7.1.1 Adjust and calibrate the mass spectrometer according to the manufacturers specifications.
- 7.1.2 Analyze a sample of pentafluorobromobenzene (PFBB).
- 7.1.3 Determine if the PFBB spectrum meets the performance criteria (3) (Attachment 1). Proceed to analyses if it does or retune the instrument to meet the performance criteria.
- 7.1.4 Analyze a standard mix of the compounds of interest and determine if the response is within an acceptable range of the previously established response factors. If not, determine the cause of the problem, make the necessary corrections and reanalyze the standard.

7.2 Sample Analysis

- 7.2.1 Equilibrate the sample bottles to ambient temperature and pipette 1 ml of sample into a 12 ml vial. Composite samples may be prepared by pipetting one ml volumes of each sample into a 12 ml vial. Dose the sample (composite) with 10 ul of internal standard solution for each one ml of sample to yield an internal standard concentration of 75 ng/ul.

7.2.3 Equilibrate the GC oven temperature to 70°C.

7.2.4 Inject 5 µl of the dosed sample, turn the vacuum diverter off and immediately start collecting M.S. data using the following conditions:

Mass Range 33 - 130 AMU

Scan Time - 3 seconds

After four minutes start the G.C. oven program
(60/min) oven max = 180°C

7.2.5 Collect data until the last components have eluted from the G.C. column. Typically this would be 320 scans or about 16 minutes.

7.3 Data Evaluation

7.3.1 After each analysis, collected data is analyzed by the procedure - Computer Assisted Evaluation of Direct Aqueous Injection GC/MS Data (4).

References

- (1) Finnigan INCOS Data System Operators Manual, Revision 3; Finnigan 3200 GC/MS Systems Manual, Finnigan Corporation, Sunnyvale, California
- (2) Standard Recommended Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Gas Chromatography, ASTM D-2908-74, p 480-487
- (3) Memo of J. Eichelberger and W. Budde, March 10, 1978, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, Subject - Perfluorobromobenzene Reference Compound for Use with Typical Purge and Trap Columns that Do Not Transmit DFTPP Readily.
- (4) Computer Assisted Evaluation of Direct Aqueous Injection GC/MS Data - Procedure Developed by the Chemistry Branch of the EPA, National Enforcement Investigations Center, Denver, Colorado, September 1978

Computer Assisted Evaluation of
Direct Aqueous Injection GC/MS Data

NEIC - September 1978

1.0 Introduction

- 1.1 This procedure is a slightly modified version of the priority pollutant data evaluation procedure (1). Minor modifications were made to enhance the handling of direct aqueous injection (DAI) analyses data for the Kanawha River Valley project (August 1978).

2.0 Summary of Method

- 2.1 GC/MS data files are processed by location of an internal standard that is used for response reference. Compounds of interest in a user library are reverse searched using an absolute retention time window. If a compound is located and passes the match criteria, it is quantitated and the spectrum printed. Printed results are manually audited and the data verified or rejected.

3.0 Summary of Modifications

- 3.1 The compound detection routine (Detect) was changed to use absolute retention times for location of the retention time window. Only masses 41 through 125 were used in locating compounds due to the Argon background (m/e 40) in the system.
- 3.2 The required spectrum match parameter limit (fit) in the compound identification routine (Detec 2) was set to 450. This lower limit was necessary due to the poor character of the spectra of the DAI compounds. Poor character here means that the spectra contain few ions and their response (sensitivity) is poor.
- 3.3 The names of procedures used in both the DAI data evaluation and the priority pollutant evaluation were changed to allow independent operation of the two procedures.

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 set up this procedure, the user must understand and be proficient in the use of MSDA (2).

6.0 Procedure

6.1 Procedure Set Up

- 6.1.1 Create the procedures from the trace of EVDAI in Appendix I.

6.2 Library Set Up

- 6.2.1 Build a user library containing each compound of interest. Appendix II is a library list of the DI library. The first entry must always be the internal standard and each entry must include the quantitation parameters and retention times.
- 6.2.2 Execute EVDAI, 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 Appendix II.

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:

EVDAI 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 printout of the spectra at a peak that was identified by the procedure.

6.3.4 Appendix III is an example of PPEVAL output. The "No" 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) "Computer Assisted Evaluation of Organic Priority Pollutant GC/MS Data", US EPA, National Enforcement Investigations Center, September 1978.
- (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.

APPENDIX Ia.

TRACE OF PROCEDURE EVAL

```

* 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 NAMELIST WITH THE NAMES OF THE FILES TO BE ]
* ;[PROCESSED. EXECUTE THE PROCEDURE AS FOLLOWS: ]
* ;[ PPEVAL LIBRARYNAME, NAMELIST E.G. PPEVAL VO.SAMPLE ]
* ;[C REVISED 30AUG73 O.J.LOGSDON II EPA-NEIC 303-234-4651 ]
* ;SETS PPSCAN;EDLL YES(-;S;W;E);EDLL NO(-;W;E)
* ;SETN S2;SET4 S1;PPEV1;FEED;BEEP;BEEP;BEEP
*
ERASE
SETS PPSCAN
EDLL YES (-;S;W;E)
EDLL NO (-;W;E)
SETN S2
SET4 S1
PPEV1
* ERASE
* ;[PART OF PROCEDURE PPEVAL ]
* ;[GET THE NEXT NAMELIST ENTRY AND CONTINUE PROCESSING ]
* ;[AT PPEV2 ]
* ;GETN;PPEV2;LOOP
*
ERASE
GETN
PPEV2
* ERASE
* ;[PART OF PPEVAL. THIS PROCEDURE SETS THE LIBRARY ENTRY ]
* ;[POINTER TO THE FIRST ENTRY, WHICH MUST ALWAYS BE THE INTERNAL ]
* ;[STANDARD. LOCIS IS THEN CALLED AND THE INTERNAL FOUND ]
* ;[THE SPECTRUM NUMBER OF THE INTERNAL STANDARD IS ]
* ;[STORED IN !10 FOR FUTURE REFERENCE. THE LIBRARY POINTER ]
* ;[IS THEN RESET TO THE BEGINNING. THE QUANTITATION LIST SET TO ]
* ;[THE FILE NAME AND EMPTIED OUT. DETECT IS CALLED TO LOCATE EACH ]
* ;[COMPOUND (IF PRESENT). QUAN IS THEN CALLED TO CALCULATE ]
* ;[THE RESULTS AND THE PROCEDURE RETURNS TO PPEV1 TO GET THE ]
* ;[NEXT FILE TO PROCESS. ]
* ;FILE(K PRIN.99/N;E)
* ;EDLL PPLIST(-;W;E)
* ;SETJ *1;CHRO(I;H1.900.300;E);SET4 *1;LOCIS;SET10 !14;SET4 *0
* ;SETO S1;EDOL(-;W;E);EDSL(-;W;E);SETL S3;DETECT;QUAN(I;H;E)
* ;EDLL PPLIST(0!1;E)
* ;P.LIN(OPP)
* ;FILE(C PRIN.99/N,M;E)
* ;FEED
* ;BEEP
*
ERASE

```

APPENDIX Ib.

```

* ;[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;N2,10,400;E;D-60,60;E)
* ;LOCIS1
*
ERASE
SET14
SEAR (I;S;V2500000;N2,10,400;E;D-60,60;E)/V
LOCIS1
* IF LOCIS1 .!14
* ;[PART OF PPEVAL ]
* ;[NO INTERNAL STANDARD FOUND]
* ;PRIN(QIS)
* ;RETU PPEV2
*
IF LOCIS1.!14
PRIN (QIS)
RETU PPEV2
SET10 !14
SET4
SET0 S1
EDQL (-;U;E)
EDSL (-;U;E)
SETL S3
DETECT
* ;[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 !4,.*1
* ;IF !24.*1,14
* ;SET14 #0
* ;SET1 !10
* ;EDLL PPLIST(S;U;E)
* ;SEAR/V(I;S;V2500000;M41,125;N1,10,10;D-20,20;E)
* ;PRIN/KX(!4,2;!14,6;!15,6;!16,7;C;E)
* ;DETECI
* ;LOOP
*
SET4 !4,.*1
IF #!124,14
SET14
SET1 !10
EDLL PPLIST (S;U;E)
SEAR (I;S;V2500000;M41,125;N1,10,10;D-20,20;E)/V
PRIN (!4,2;!14,6;!15,6;!16,7;C;E)/KX
DETECI
* ;[PART OF PPEVAL]
* ;[IF THE FIT IS LESS THAN OR EQUAL TO 750 ]

```

APPENDIX Ic.

```

* :[DATA IN THE QUANLIST ASSIGNED EARLIER. ]
* :[ALSO CHECK AND PASS ONLY PEAKS WITH ]
* :[A FIT OF 750 OR GREATER ]
* :IF DETEC2 !16,DETEC2 +450
* :SET1 !14
* :CHRO(I;R;S;+;N1,3;A>5,3;G-4,4;D-5,5;E)
* :DETEC3
* :RETU DETEC1
*
IF DETEC2!16,DETEC2+450
SET1 !14
CHRO (I;R;S;+;N1,3;A>5,3;G-4,4;D-5,5;E)
DETEC3
* IF !26 DETEC3,DETEC3
* :SPEC(*;N;H;E)
*
IF DETEC3!26,DETEC3
SPEC (*;N;H;E)
RETU DETEC1
EDQL (-;N;+;A;E)
LOOP
QUAN (I;H;E)
EDLL PPLIST (B!1;E)
PRIN (OPP)
FILE (C PRIN.99/H,M;E)
FEED
BEEP
LOOP
FEED
BEEP
BEEP
BEEP

```


APPENDIX IIa.

NAM	NUM	NAME	WT	FORMULA	REL.RET.TIME/CAS*	RET TIME	BASE	AREA	U.P.*1	U.P.*2
						MASS AMT.	REF. PEAK	RESP. FILE	RESP. FACTOR	
DI	1:	D-CHLOROFORM								
119										
			1.000			84.000 75.00	7:27 DI 1	91648. :S	0.000 1.000	0.000
DI	2:	ETHYL ETHER								
74										
			0.358			45.000 50.00	2:27 DI 1	37312. :S	0.000 0.353	0.000
DI	3:	ACETONE								
58										
			0.504			43.000 50.00	3:27 DI 1	175372. :S	0.000 1.201	0.000
DI	4:	METHYL ETHYL KETONE								
72										
			0.659			43.000 50.00	4:27 DI 1	202240. :S	0.000 1.602	0.000
DI	5:	ACRYLONITRILE								
53										
			0.933			53.000 50.00	6:57 DI 1	26209. :S	0.000 0.446	0.000
DI	6:	STYRENE								
104										
			1.978			104.000 50.00	13:33 DI 1	63809. :S	0.000 0.951	0.000
DI	7:	1,1 DIMETHOXYETHANE								
90										
			0.470			75.000 50.00	3:30 DI 1	134144. :S	0.000 0.391	0.000
DI	8:	ISOPROPANOL								
60										
			0.705			45.000 50.00	5:15 DI 1	84736. :S	0.000 1.094	0.000
DI	9:	DIETHYL KETONE								
86										
			0.886			86.000 50.00	6:35 DI 1	84992. :S	0.000 0.313	0.000
DI	10:	ISOBUTRONITRILE								
69										
			0.980			42.000 50.00	7:10 DI 1	59904. :S	0.000 0.928	0.000
DI	11:	N-BUTANOL								
74										
			1.490			56.000 50.00	11:05 DI 1	14080. :S	0.000 0.162	0.000
DI	12:	PROPANE,2,2'-OXYBIS								
102										
			0.362			45.000 50.00	2:42 DI 1	134912. :S	0.000 0.857	0.000
DI	13:	1,3-DIOXOLANE,2-METHYL								
			0.805			73.000 50.00	5:20 DI 1	65350. :S	0.000 0.774	0.000
DI	14:	1,1-DICHLOROETHANE								
92										
			0.544			56.000 50.00	4:03 DI 1	15230. :S	0.000 0.211	0.000

APPENDIX Iib.

NAM NUM: NAME		RET TIME		BASE	AREA	U.P.*1	U.P.*2
WT FORMULA		MASS	AMT.	REF. PEAK	RESP. FILE	RESP. FACTOR	
REL. RET. TIME/CAS#							
DC	1: DCHLOROFORM						
119			7:15	84	163348.	0.000	0.000
	1.000	84.000	75.00	DC	1	:S	1.000
DC	2: ETHANOL						
46	C2.H6.O		5:18	45	106624.	0.000	0.000
	0.731	45.000	200.00	DC	1	:S	0.235
DC	3: MESITYL OXIDE						
98	C6.H10.O		10:35	83	93568.	0.000	0.000
	1.462	83.000	220.00	DC	1	:S	0.219
DC	4: ETHANOL, 2-METHOXY-, ACETATE						
118	C5.H10.O3		13:57	43	565609.	0.000	0.000
	1.924	58.000	200.00	DC	1	:S	0.731

APPENDIX IIIa.

QUANTITATION REPORT

FILE: D90578E

DATA: D90578E.T1
 09/05/78 9:28:00
 SAMPLE: MIX D 50NG/UL +IS
 CONDS.: 70-180
 FORMULA: Q6/MIN
 SUBMITTED BY: JJS

INSTRUMENT: 3200E1
 ANALYST: JJS

WEIGHT: 0.000
 ACCT. NO.: J240

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

NO NAME
 1 D-CHLOROFORM
 2 ACRYLONITRILE
 3 1,1 DIMETHOXYETHANE
 4 ISOPROPANOL
 5 DIETHYL KETONE
 6 ISOBUTRONITRILE
 7 N-BUTANOL
 8 PROPANE,2,2'-OXYBIS
 9 1,3-DIOXOLANE,2-METHYL
 10 BUTANE,1-CHLORO

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	%TOT
1	84	147	7:21	1	1.000	A 88	652404.	75.000 NG/UL	14.29
2	53	137	6:51	1	0.932	A 88	222688.	50.000 NG/UL	9.52
3	75	69	3:27	1	0.459	A 88	208334.	50.000 NG/UL	9.52
4	45	101	5:03	1	0.687	A 88	552015.	50.000 NG/UL	9.52
5	86	130	6:30	1	0.894	A 88	145855.	50.000 NG/UL	9.52
6	42	144	7:12	1	0.980	A 88	369055.	50.000 NG/UL	9.52
7	55	222	11:06	1	1.510	A 88	72404.	50.000 NG/UL	9.52
8	45	53	2:39	1	0.351	A 88	512704.	50.000 NG/UL	9.52
9	73	118	5:54	1	0.803	A 88	409852.	50.000 NG/UL	9.52
10	55	79	3:57	1	0.537	A 88	122668.	50.000 NG/UL	9.52

APPENDIX IIIb.

NAM	NUM:	WT FORMULA
DI	1:	119
DI	2:	74 C4.H10.O
DI	3:	58 C3.H5.O
DI	4:	72 C4.H8.O
DI	5:	53 C3.H3.N
DI	6:	104 C8.H8
DI	7:	90 C4.H10.O2
DI	8:	60 C3.H8.O
DI	9:	86 C5.H10.O
DI	10:	69 C4.H7.N
DI	11:	74 C4.H10.O
DI	12:	102 C6.H14.O
DI	13:	88 C4.H8.O2
DI	14:	92 C4.H9.CL

NAME

D-CHLOROFORM
 ETHYL ETHER
 ACETONE
 METHYL ETHYL KETONE
 ACRYLONITRILE
 STYRENE
 1,1 DIMETHOXYETHANE
 ISOPROPANOL
 DIETHYL KETONE
 ISOBUTRONITRILE
 N-BUTANOL
 PROPANE, 2,2'-OXYBIS
 1,3-DIOXOLANE, 2-METHYL
 BUTANE, 1-CHLORO

IDENTIFICATION REPORT

FILE: D:E90578E.TI

NO	SCAN	PURITY	FIT
1	148	378	996
2	49	351	728
3	73	344	661
4	97	410	740
5	0	0	0
6	277	268	928
7	73	73	207
8	97	123	260
9	148	43	323
10	137	37	133
11	217	110	533
12	49	105	459
13	106	42	360
14	97	164	329

ATTACHMENT V

Methodology: Carbaryl Analysis

A liter of the sample was extracted serially with three 50 ml portions of methylene chloride. The extracts were combined and passed through Na_2SO_4 into a 250 ml round bottom flask. 50 ml of ethyl acetate was added to the flask and the solvents were concentrated to 10 ml in a rotary evaporator at 45°C . The extract was passed through a clean-up column of 3 cm Florisil topped with 1 cm of Na_2SO_4 . The Carbaryl was eluted with 20 ml of ethyl acetate. The 30 ml of ethyl acetate was concentrated to 10 ml on a hot plate under a gentle stream of carbon filtered air.

The extract was analyzed on a Waters 204 Liquid Chromatograph with a M Bondapak C_{18} column. A methanol - 1% acetic acid gradient was used over 25 minutes at a flow rate of 2.0 ml/min. The gradient was run from 0 to 80% methanol. The dual channel UV detector was operated at wave lengths of 254 nm and 280 nm.

Quality Control: A blank and a spike were analyzed along with the samples. The blank did not contain any interferences at the retention time of Carbaryl. The spike was at a concentration of 250 ug/l of Carbaryl and the recovery was 117%.

The presence of Carbaryl in the samples was established by the coincidence of retention time and confirmed by the ratio of the 254 to 280 response.

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: 21 and 41 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 VII
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 ^{liter} 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 liter 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.

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

and acrylonitrile were prepared in a separate standard mix.

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.

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 Carboxpack.

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

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.

Computer Assisted Evaluation of
Organic Priority Pollutant GS/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.

.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)

.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

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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 NAMELIST WITH THE NAMES OF THE FILES TO BE ]
* ;[PROCESSED. EXECUTE THE PROCEDURE AS FOLLOWS: ]
* ;[ PPEVAL LIBRARYNAME, NAMELIST ,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;U;E);EDLL NO(-;U;E)
* ;SETN $2;SET4 $1;PPEVA;FEED;BEEP;BEEP;BEEP
*
ERASE
SETS PPSCAN
EDLL YES (-;S;U;E)
EDLL NO (-;U;E)
SETN $2
SET4 $1
PPEVA
* ERASE
* ;[PART OF PROCEDURE PPEVAL ]
* ;[GET THE NEXT NAMELIST ENTRY AND CONTINUE PROCESSING ]
* ;[AT 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(-;U;E)
* ;SETI *1;PARA(I;H;E);CHRO(I;H1,1050.350;E);SET4 *1;PPEVC;SETI0 I14;SET4 *0
* ;SET0 $1;EDQL(-;U;E);EDSL(-;U;E);SETL $3;PPEVE;QUAN(I;H;E)
* ;EDLL PPLIST(B;1;E)
* ;PRIN(@P1)
* ;FILE(C PRIN.99/N.M.;E)
* ;FEED
* ;BEEP
*
ERASE
FILE (K PRIN.99/N;E)
EDLL PPLIST (-;U;E)
SETI *1
PARA (I;H;E)

```

APPENDIX IIB.

```

CHRO (1;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(1;$;V2500000;N2,10,600;E;D-60,60;E)
  * ;PPEVD
  *
ERASE
SET14
SEAR (1;$;V2500000;N2,10,600;E;D-60,60;E)/V
PPEVD
  * IF PPEVD,114
  * ;[PART OF PPEVAL ]
  * ;[NO INTERNAL STANDARD FOUND]
  * ;PRIN(@P2)
  * ;RETU PPEVB
  *
  IF PPEVD,114
  PRIN (@P2)
  RETU PPEVB
SET10 114
SET4
SETQ $1
EDQL (-;W;E)
EDSL (-;W;E)
SETL $3
PPEVE
  * ;[PART OF PPEVAL ]
  * ;[THIS ROUTINE LOCATES COMPOUNDS IN THE ]
  * ;[SAMPLE FILE BY COMPARING THE SPECTRA IN THE LIBRARY ]
  * ;[WITH THE SAMPLE. RELATIVE RETENTION TIMES ARE USED ]
  * ;[AND REFERENCED TO THE INTERNAL STANDARD FOUND EARLIER.]
  * ;[THE LIBRARY POINTER IS BUMPED AND TESTED TO ]
  * ;[SEE IF THE LAST LIBRARY ENTRY HAS BEEN PROCESSED. ]
  * ;[THEN THE CURRENT SCAN NUMBER IS SET TO THE INTERNAL ]
  * ;[STANDARD LOCATION BY RECALLING THE CONTENTS OF 110. ]
  * ;[STORE THE SCAN NUMBER OF ]
  * ;[THE BEST MATCH IN VARIABLE 14 AND ALLOW INTEGRATION ]
  * ;[AT THAT SPECTRUM NUMBER ONLY ]
  * ;[IF THE COMPOUND IS NOT FOUND, PLACE A NOT FOUND ]
  * ;[ENTRY INTO THE QUANTITATION LIST FOR LATER REFERENCE ]
  * ;SET4 14,,#1
  * ;IF 124#1,14
  * ;SET14 #0
  * ;SET1 110
  * ;EDLL PPLIST($;W;E)
  * ;SEAR/V(1;$;V2500000;N1,10,10;D-20,20;E)
  * ;PRIN/KX(14,2;114,6;115,6;116,7;C;E)
  * ;PPEVF
  * ;LOOP
  *
SET4 14,,#1
IF #1124,14
SET14
SET1 110
EDLL PPLIST ($;W;E)
SEAR (1;$;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
  * ;EDQL(-;N;#;A;E)
  *

```

APPENDIX IIC.

```

PPEVG
* [PART OF PPEVAL ]
* ;[ACCESS ANY SCANS IDENTIFIED IN DETECT ]
* ;[AND INTEGRATE THEIR AREAS. RECORD THE ]
* ;[DATA IN THE QUANLIST ASSIGNED EARLIER. ]
* ;[ALSO CHECK AND PASS ONLY PEAKS WITH ]
* ;[A FIT OF 750 OR GREATER ]
* ;IF PPEVG !16,PPEVG *700
* ;SETI 114
* ;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 114
CHRO (I;R;S;*:N1,3;A>5.3;G-4.4;D-5.5;E)
PPEVH
* IF 126 PPEVH,PPEVH
* ;SPEC(*;N;H;E)
*
IF PPEVH!26,PPEVH
SPEC (*;N;H;E)
RETU PPEVF
EDQL (-;N;*:A;E)
LOOP
QUAN (I;H;E)
EDLL PPLIST (B11;E)
PRIN (@PI)
FILE (C PRIN.99/N,M;:E)
FEED
BEEP
LOOP
FEED
BEEP
BEEP
BEEP

```

APPENDIX IID.

PRINP2.ME = C20;T;
C;T;
C;T;
;E

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:

NAME
 FORMULA
 RET.TIME/CAS#

RET TIME	BASE	AREA	U.P.#1	U.P.#2
MASS AMT.	REF. PEAK	RESP. FILE	RESP. FACTOR	
1: 1,4-DICHLOROBUTANE (INTERNAL STANDARD)				
6 C4.H8.CL2	3:30	55	0.	0.000
1.000	55.000200.00	VI 1	C	1.000
2: BROMOCHLOROMETHANE (INTERNAL STANDARD)				
3 C.H2.CL.BR	0:44	138	0.	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	56.000200.00	VO 1	VS	1.000
4: 03 ACRYLONITRILE				
3 C3.H3.N	0:00	53	0.	0.000
0.000	53.000200.00	VO 1	VS	1.000
5: 04 BENZENE				
3 C6.H6	2:19	78	0.	0.000
0.000	78.000200.00	VO 1	VS	1.000
6: 06 CARBONTETRACHLORIDE				
2 C.CL4	1:45	117	0.	0.000
0.000	117.000200.00	VO 1	VS	1.000
7: 06 CHLOROBENZENE				
2 C6.H5.CL	3:50	112	0.	0.000
0.000	112.000200.00	VO 1	VS	1.000
8: 10 1,2-DICHLOROETHANE				
9 C2.H4.CL2	1:26	62	0.	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	97.000200.00	VO 1	VS	1.000
10: 13 1,1-DICHLOROETHANE				
9 C2.H4.CL2	0:52	63	0.	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	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	83.000200.00	VO 1	VS	1.000
13: 23 CHLOROFORM				
8 C.H.CL3	1:20	83	0.	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	96.000200.00	VO 1	VS	1.000
15: 30 1,2-TRANS-DICHLOROETHENE				
5 C2.H2.CL2	1:01	96	0.	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	63.000200.00	VO 1	VS	1.000
17: 33A 1,3-CIS-DICHLORO-1-PROPENE				

APPENDIX IIIA.

18: 33B 1,2-TRANS-DICHLORO-1-PROPENE				
110 C3.H4.CL2	2:34	75	0.	0.000
0.000	75.000200.00	VO 1	VS	1.000
19: 38 ETHYLBENZENE				
106 C8.H10	0:00	91	0.	0.000
0.000	91.000200.00	VO 1	VS	1.000
20: 44 METHYLENECHLORIDE				
84 C.H2.CL2	0:04	84	0.	0.000
0.000	84.000200.00	VO 1	VS	1.000
21: 47 BROMOFORM				
250 C.H.BR3	3:02	173	0.	0.000
0.000	173.000200.00	VO 1	VS	1.000
22: 48 BROMODICHLOROMETHANE				
162 C.H.CL2.BR	1:59	83	0.	0.000
0.000	83.000200.00	VO 1	VS	1.000
23: 49 TRICHLOROFLUOROMETHANE				
136 C.CL3.F	0:19	101	0.	0.000
0.000	101.000200.00	VO 1	VS	1.000
24: 51 DIBROMOCHLOROMETHANE				
206 C.H.CL.BR2	2:32	129	0.	0.000
0.000	129.000200.00	VO 1	VS	1.000
25: 85 TETRACHLOROETHENE				
164 C2.CL4	3:22	166	0.	0.000
0.000	129.000200.00	VO 1	VS	1.000
26: 86 TOLUENE				
92 C7.H8	3:29	91	0.	0.000
0.000	91.000200.00	VO 1	VS	1.000
27: 87 TRICHLOROETHENE				
130 C2.H.CL3	2:20	130	0.	0.000
0.000	95.000200.00	VO 1	VS	1.000

APPENDIX IIIB.

RET. TIME/CAS#	MASS	AMT.	RET. TIME	BASE REF. PEAK	AREA	U.P.#1	U.P.#2
FORMULA					RESP. FILE	RESP. FACTOR	
1: D10-ANTHRACENE (INTERNAL STANDARD)			3:44	188	44864.	0.000	0.000
1.000	188.000	20.00	BN	1	:S	1.000	
2: 01 ACENAPHTHENE			2:39	154	0.	0.000	0.000
C12.H10	0.710	154.000	BN	1	:S	0.506	
3: 05 BENZIDINE			5:00	184	0.	0.000	0.000
C12.H12.N2	1.345	184.000	BN	1	:S	0.047	
4: 08 1,2,4-TRICHLOROBENZENE			1:18	188	0.	0.000	0.000
C6.H3.CL3	0.349	74.000	BN	1	:S	0.182	
5: 09 HEXACHLOROBENZENE			3:28	284	0.	0.000	0.000
C6.CL6	0.893	284.000	BN	1	:S	0.264	
6: 12 HEXACHLOROETHANE			0:43	201	0.	0.000	0.000
C2.CL6	0.192	117.000	BN	1	:S	0.398	
7: 18 BIS(2-CHLOROETHYL)ETHER			0:37	93	0.	0.000	0.000
C4.H8.O.CL2	0.165	93.000	BN	1	:S	0.205	
8: 20 2-CHLORONAPHTHALENE			2:12	162	0.	0.000	0.000
C10.H7.CL	0.589	162.000	BN	1	:S	0.612	
9: 25 1,2-DICHLOROBENZENE			0:41	146	0.	0.000	0.000
C6.H4.CL2	0.183	146.000	BN	1	:S	0.786	
10: 26 1,3-DICHLOROBENZENE			0:31	146	0.	0.000	0.000
C6.H4.CL2	0.138	146.000	BN	1	:S	0.519	
11: 27 1,4-DICHLOROBENZENE			0:34	146	0.	0.000	0.000
C6.H4.CL2	0.152	146.000	BN	1	:S	0.895	
12: 35 2,4-DINITROTOLUENE			2:59	165	0.	0.000	0.000
C7.H6.O4.N2	0.803	165.000	BN	1	:S	0.191	
13: 36 2,6-DINITROTOLUENE			2:46	165	0.	0.000	0.000
C7.H6.O4.N2	0.744	165.000	BN	1	:S	0.184	
14: 37 1,2-DIPHENYLHYDRAZINE (MEAS. AS AZOBENZENE)			3:06	77	0.	0.000	0.000
C12.H10.N2	0.834	77.000	BN	1	:S	1.066	
15: 39 FLUORANTHENE			4:34	202	0.	0.000	0.000
C16.H10	1.223	202.000	BN	1	:S	0.714	
16: 40 4-CHLOROPHENYL PHENYL ETHER			2:59	204	0.	0.000	0.000
C12.H9.O.CL	0.799	204.000	BN	1	:S	0.288	
17: 41 4-BROMOPHENYL PHENYL ETHER							

APPENDIX IVA.

248	C12.H9.O.0R	0.893	248.000	20.00	BN	1	0.	0.000	0.000
BN	18: 42 BIS(2-CHLOROISOPROPYL)ETHER					45	0.	0.000	0.000
170	C6.H12.O.CL2	0.193	45.000	50.00	BN	1	:S	0.664	
BN	19: 43 BIS(2-CHLOROETHOXY)METHANE					93	0.	0.000	0.000
172	C5.H10.O2.CL2	0.359	93.000	50.00	BN	1	:S	0.586	
BN	20: 53 HEXACHLOROCYCLOPENTADIENE					237	0.	0.000	0.000
270	C5.CL6	0.000	237.000	20.00	BN	1	:S	1.000	
BN	21: 54 ISOPHORONE					82	0.	0.000	0.000
130	C9.H14.O	0.309	82.000	50.00	BN	1	:S	0.984	
BN	22: 55 NAPHTHALENE					120	0.	0.000	0.000
120	C10.H8	0.379	120.000	20.00	BN	1	:S	1.287	
BN	23: 56 NITROBENZENE					77	0.	0.000	0.000
123	C6.H5.O2.N	0.300	77.000	50.00	BN	1	:S	0.457	
BN	24: 62 N-NITROSODIPHENYLAMINE (MEAS AS DIPHENYLAMINE)					169	0.	0.000	0.000
169	C12.H11.N	0.857	169.000	20.00	BN	1	:S	0.145	
BN	25: 63 N-NITROSODIPROPYLAMINE					70	0.	0.000	0.000
130	C6.H14.O.N2	0.247	130.000	50.00	BN	1	:S	0.058	
BN	26: 66 DI-(2-ETHYLHEXYL)PHTHALATE					149	0.	0.000	0.000
390	C24.H38.O4	1.536	149.000	20.00	BN	1	:S	0.841	
BN	27: 67 BUTYLBENZYLPHTHALATE					149	0.	0.000	0.000
312	C19.H20.O4	1.460	149.000	20.00	BN	1	:S	0.581	
BN	28: 68 DI-N-BUTYLPHTHALATE					149	0.	0.000	0.000
270	C16.H22.O4	1.098	149.000	20.00	BN	1	:S	1.732	
BN	29: 69 DI-OCTYLPHTHALATE					149	0.	0.000	0.000
390	C24.H38.O4	1.866	149.000	20.00	BN	1	:S	0.580	
BN	30: 70 DIETHYLPHTHALATE					149	0.	0.000	0.000
222	C12.H14.O4	0.821	149.000	20.00	BN	1	:S	0.953	
BN	31: 71 DIMETHYLPHTHALATE					163	0.	0.000	0.000
194	C10.H10.O4	0.714	163.000	20.00	BN	1	:S	0.817	
BN	32: 72 BENZO(A)ANTHRACENE					228	0.	0.000	0.000
228	C18.H12	1.670	228.000	20.00	BN	1	:S	0.120	
BN	33: 76 CHRYSENE					228	0.	0.000	0.000
228	C18.H12	1.670	228.000	20.00	BN	1	:S	0.120	
BN	34: 77 ACENAPHTHYLENE					152	0.	0.000	0.000
152	C12.H8	0.683	154.000	20.00	BN	1	:S	0.883	

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

APPENDIX IVC.

PH	1:	D10-ANTHRACENE (INTERNAL STANDARD)	2:43	188	44864.	0.000	0.000
188	1.000	188.000 50.00	PH	1	:S	1.000	
PH	2: 21	2,4,6-TRICHLOROPHENOL	1:46	196	0.	0.000	0.000
196	C6.H3.O.CL3	0.000 196.000100.00	PH	1	:S	0.461	
PH	3: 22	4-CHLORO-3-METHYLPHENOL	2:06	142	0.	0.000	0.000
142	C7.H7.O.CL	0.000 142.000100.00	PH	1	:S	0.524	
PH	4: 24	2-CHLOROPHENOL	0:27	128	0.	0.000	0.000
128	C6.H5.O.CL	0.000 128.000100.00	PH	1	:S	1.014	
PH	5: 31	2,4-DICHLOROPHENOL	1:13	162	0.	0.000	0.000
162	C6.H4.O.CL2	0.000 162.000100.00	PH	1	:S	0.714	
PH	6: 34	2,4-DIMETHYLPHENOL	1:11	122	0.	0.000	0.000
122	C8.H10.O	0.000 122.000100.00	PH	1	:S	0.617	
PH	7: 57	2-NITROPHENOL	0:37	139	0.	0.000	0.000
139	C6.H5.O3.N	0.000 139.000100.00	PH	1	:S	0.534	
PH	8: 58	4-NITROPHENOL	5:01	139	0.	0.000	0.000
139	C6.H5.O3.N	0.000 65.000100.00	PH	1	:S	0.000	
PH	9: 59	2,4-DINITROPHENOL	2:53	184	543744.	0.000	0.000
184	C6.H4.O5.N2	1.000 184.000*1000.	PH	1	:S	0.219	
PH	10: 60	4,6-DINITRO-O-CRESOL	2:57	198	781312.	0.000	0.000
198	C7.H6.O5.N2	1.000 198.000*1000.	PH	1	:S	0.319	
PH	11: 64	PENTACHLOROPHENOL	3:12	266	0.	0.000	0.000
264	C6.H.O.CL5	0.000 266.000100.00	PH	1	:S	0.242	
PH	12: 65A	PHENOL	0:52	94	0.	0.000	0.000
94	C6.H6.O	0.000 94.000100.00	PH	1	:S	1.025	

APPENDIX VIA.

QUANTITATION REPORT

FILE: SMA3A

DATA: SMA3A.MI

0:00:00

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

CONDS.:

FORMULA:

INSTRUMENT: SYSIND

WEIGHT: 0.000

SUBMITTED BY:

ANALYST:

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 ETHYLENE CHLORIDE
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	1191050.	200.000 PPB	4.55
2	49	75	1:15	1	0.299	A 88	1120880.	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	886280.	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	119882.	200.000 UG/L	4.55
13	83	103	1:48	1	0.430	A 88	1612750.	200.000 UG/L	4.55
14	96	88	1:28	1	0.351	A 88	774512.	200.000 UG/L	4.55
15	63	167	2:47	1	0.665	A 88	1088560.	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	568556.	200.000 UG/L	4.55
18	173	221	3:41	1	0.880	A 88	1054980.	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	0.969	A 88	1099630.	200.000 UG/L	4.55
22	91	251	4:11	1	1.000	A 88	1879520.	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	55884.	200.000 UG/L	4.55

QUANTITATION FOR THIS
COMPOUND MANUALLY ADDED
on

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.

D-62 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 ul 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. (Pufuorotri-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.

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-Biller 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

APPENDIX II. a.

```

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 MNP BE SEARCHED AGAINST THE
* :MNP 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      OJLOGSDON11 ]
* :SET4 11
* :EDLL YES(-;$:W:E);EDLL NO(-;W:E)
* :SETN OCTEMP;EDNL(-;$1:$2;W:E)
* :SET11 #0
* :OCEVO
* :BEEP;BEEP;BEEP
* :ERASE
* :[PROCEDURE OCEVAL IS COMPLETE]
*
SET4 11
EDLL YES (-;$:W:E)
EDLL NO (-;W:E)
SETN OCTEMP
EDNL (-;$1:$2;W:E)
SET11
OCEVO
* SETN OCTEMP;SETN #0;GETH;SET4 S1
* :GETH;GETH S1;SETN 111;SET11 111#1;GETN
* :OCEV1
* :SETL OCTEMP
* :EDLL(-;W:E)
* :FILE(K PRIN.99/11:E)
* :OCEV2
* :SET12 #0
* :SETS OCEV2;SETS #0
* :EDSL(-112;W:E)
* :OCEV3
* :OCEV5
* :BEEP
* :LOOP
*
SETN OCTEMP
SETN
GETH
SET4 S1
GETN
SETN S1
SETN 111
SET11 #111
GETN
OCEV1
* PARA(1:H:E)

```

APPENDIX II. b.

```

* ;SETS OCEV2:EDSL(-;W:E)
* ;SETS OCEV1:EDSL(-;W:E)
* ;MAP(I;F1;U100;V250000;33,300;N>2.5,7;H1,2000,500;E)
*
PARA (I;H:E)
SETS OCEV2
EDSL (-;W:E)
SETS OCEV1
EDSL (-;W:E)
MAP (I;F1;U100;V250000;33,300;N>2.5,7;H1,2000,500;E)
SETL OCTEMP
EDLL (-;W:E)
FILE (K PRIN.99;N;E)
OCEV2
* IF OCEV2 #25000,OCEV2 !24
* ;OCEV2A
* ;PRIN (001)
* ;EDLL (B!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#1!24,OCEV2A!4
OCEV2B
* EDLL(S;W:E)
* ;SEAR/V(I;S;0;V250000;N1,200,750;D-25,25;E)
* ;PRIN/KX(!4,6;!14,5;!15,8;!16,6;C;E)
* ;SETS OCEV2:EDSL(!14;W:E)
* ;SETS OCEV1:EDSL(-!14;W:E)
*
EDLL (S;W:E)
SEAR (I;S;0;V250000;N1,200,750;D-25,25;E)/V
PRIN (!4,6;!14,5;!15,8;!16,6;C;E)/KX
SETS OCEV2
EDSL (!14;W:E)
SETS OCEV1
EDSL (-!14;W:E)
LOOP
PRIN (001)
EDLL (B!1;E)
PRIN (002)
FILE (C PRIN.99,M;N;E)
FEED
SET!2
SETS OCEV2
SETS
EDSL (-!12;W:E)
OCEV3
* GETS
* ;SPEC(I;';T;H30,350;E)
* ;LOOP
*
GETS
SPEC (I;';T;H30,350;E)
LOOP
OCEV5
* SETL S3
* ;OCEV6
* ;SET4 N8
* ;SETS OCEV1;SETS #0
* ;OCEV7

```

APPENDIX II. c.

```

* ;FEED
*
SETL S3
OCEV6
  * IF OCEV6 *25000,OCEV6 124
  * ;IF OCEV5 126,OCEV5
  * ;RETU OCEV6
  *
  IF OCEV6*25000,OCEV6 124
  IF OCEV5 126,OCEV5
  RETU OCEV6
SET4 NB
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.

PRIN01.ME = C:D:T: ORGANICS CHARACTERIZATION REPORT FILE:
;S1:C2:T:
;D:C2:E

PRIN02.ME = C2:T: NUM SPEC: PURITY FIT
;C:E

APPENDIX VIB.

NAM	NUM:	WT FORMULA	NAME
VI	1:	126 C4.H8.CL2	1,4-DICHLOROBUTANE (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:	106 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.H10	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:	130 C2.H.CL3	87 TRICHLOROETHENE
VI	25:	62 C2.H3.CL	89 VINYL CHLORIDE

IDENTIFICATION REPORT

FILE: D:SMAS.A.MI

NO	SCAN	PURITY	FIT
1	251	420	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	63 251	799	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.

APPENDIX III.

0/00/00 0:00:00

ORGANICS CHARACTERIZATION REPORT

FILE: 0:0114593N.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:	145	C6.H4.CL2	DICHLOROBENZENE ISOMER (NC)
39	4:	130	C8.H18.0	2-ETHYL-1-HEXANOL (NC)
39	5:	133	C9.H14.0	ISOPHORONE (NC)
39	6:	162	C8.H18.03	BUTYL CARBITOL (NC)
39	7:	0		POLY GLYCOL ETHER (NC UNKNOWN)
39	8:	134	C9.H18.0	1-PHENYL-1-PROPANONE (NC)
39	9:	154	C12.H10	BIPHENYL (NC)
39	10:	170	C12.H18.0	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-TERT-BUTYL-P-CRESOL (NC)
39	14:	96	C4.H4.O.N2	4(IH)-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	C8.H18.03	BIS-(2-ETHOXYETHYL)ETHER (NC)
39	27:	222	C10.H22.05	2,5,8,11,14-PENTAOKAPENTADECANE (NC)

NUM	SPEC	PURITY	FIT
1	0	0	0
2	0	0	0
3	0	0	0
4	221	446	856
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	854
16	696	395	856
17	928	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	830
27	0	0	0

APPENDIX E

Bacteriological Methods

Bacteriological Methods

Bacteriological analyses of fecal coliform bacteria densities were performed according to standard procedures using the Most Probable Number technique*. Using aseptic techniques, all samples were collected in sterile bottles prepared by the accepted procedure. Replicate analyses was performed for quality control purposes; these data showed very good control and are available in the NEIC laboratory files.

* Rand, M. et al, 1975. Standard Methods for the Examination of Water and Wastewater. 14th Ed. APHA - AWWA - WPCF, 1193 pp.

APPENDIX F

MUTAGEN ASSAY METHODS

Mutagen Assay Methods

I. Sample Extraction

A 4:1 (80% benzene, 20% isopropanol) mixture of solvents was placed in a clean, 1 gallon amber solvent bottle and continuously stirred during the extraction procedure to assure adequate mixing.

For basic extractions, 1-liter portions of sample were adjusted above pH 12 with NaOH. Each 1 liter aliquot was extracted three times (5 minutes each) with 35 ml of fresh solvent. The solvent fraction was then separated, mixed with anhydrous sodium sulfite to remove any emulsion and filtered into a one-liter round bottom flask. The aqueous fraction was retained for acidic extraction.

The combined solvent fractions (35 ml x liters of sample extracted) were evaporated to dryness at 50°C in a rotoevaporator. The residue was resuspended into 15 ml sterile dimethylsulfoxide (DMSO), labeled and stored at 4°C until assayed by the Ames Procedure.

II. Bacterial Mutagenicity Assay

The Standard Ames Bacterial Assay was performed using the plate incorporation assay as described by Ames, et al*. Acid and basic sample extracts were screened with standard Salmonella typhimurium tester strains TA 98, TA 100, TA 1535 and TA 1537. Samples were first tested individually; if the sample extract demonstrated an elevated reversion rate, a dose-response relationship between concentration of sample extract and number of revertant colonies was determined for each responsive tester strain.

* Ames, B.N., McCann, J., and Yamasaki, E., *Methods for Detecting Carcinogens and Mutagens with the Salmonella/Mammalian-Microsome Mutagenicity Test*. Mutation Research, 31 (1975) 347-364.

Samples exhibiting a negative mutagenic response were subjected to metabolic activation by addition of S-9 mix (supernatant from 9000 xg centrifugation of rat liver homogenate). The Bacterial Assay was then repeated as described above.

III. Quality Control

A three-liter volume of sterile distilled water was added to a 1-gallon amber glass bottle and treated as a sample. This served as a blank on the sample bottles, distilled water, extracting solvents, emulsion removal, and the concentration process. A DMSO blank was tested to ensure that the material did not interfere with test results.

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

Rat liver homogenate was tested with 2-aminofluorene against strains TA 98 and TA 100 to confirm the metabolic activation process. Sterility checks were performed on solvents, extracts, liver preparation, and all culture media.

APPENDIX G

BIOASSAY METHODS

BIOASSAY METHODS

Toxicity tests consisted of 96-hour bioassays performed according to EPA standardized methods (EPA-600/43-78-012). A continuous flow proportional diluter was used which provided a series of six effluent concentrations and a 100% dilution water control. Test chambers were of all glass construction and of 8 liter capacity. Flow rates were regulated to provide a minimum of nine volumetric exchanges of test solution for each chamber for each 24-hour period. All concentrations were done in duplicate with all test chambers containing ten fish.

The test fish used were young of the year fathead minnows (*Pimephales Promelas Rafinesque*) obtained from the Newtown Toxicology Laboratory located at Cincinnati, Ohio. The fish, averaging approximately 4 cm in length, were acclimated for 96-hours prior to testing in Kanawha River water and given prophylactic treatment (25 mg/l Oxytetracycline HCl) to prevent bacterial infection.

Dilution water was obtained from the Kanawha River at a point approximately 3 km (2 river miles) upstream of the mouth of the Elk River. The dilution water was stored in 1100 liter (300 gallon) epoxy-coated wooden reservoirs and was replenished every 24-hours.

Bioassay test water from Union Carbide Institute was collected by continuous flow compositing at outfall 001. A 180 liter (47 gallon) composite sample was transported to the diluter every six hours for the duration of the test.

All test chambers were monitored daily for pH, temperature and dissolved oxygen concentration (Table G-1). In addition the high,

middle and low concentrations were analyzed for total alkalinity. Water temperature in the test chambers was maintained at $23.5^{\circ}\text{C} \pm 1^{\circ}\text{C}$ by use of a constant temperature recirculating water bath.

Mortalities in each test chamber were recorded at 24-hour intervals. The 96-hour LC_{50} value was calculated by computerized tape program based on the Spearman-Kärber probit technique.

TABLE G-1
Physical-Chemical Characteristics
Union Carbide Institute Effluent
August, 1978

Parameter	Effluent Concentration(%)						
	Control (Kanawha River Water)	10	18	32	56	75	100
24-hour							
DO mg/l	6.5	6.2	6.0	6.1	5.9	5.8	5.5
Temp. °C	23.0	23.2	22.8	22.7	23.0	22.5	22.7
pH	7.2	7.2	7.3	7.2	7.2	7.2	7.2
Total alkalinity	34				66		92
48-hour							
DO mg/l	6.5	6.0	6.0	6.0	5.8	5.0	5.0
Temp. °C	23.3	23.4	23.2	23.1	23.4	22.8	23.0
pH	7.2	7.2	7.3	7.2	7.2	7.2	7.2
Total Alkalinity	29				63		84
72-hour							
DO mg/l	7.0	6.5	6.5	7.5 6.5	5.5	5.2	5.0
Temp. °C	23.2	23.4	23.2	23.0	23.2	22.7	22.8
pH	7.3	7.3	7.3	7.3	7.3	7.2	7.2
Total Alkalinity	28				56		77
96-hour							
DO mg/l	6.9	6.0	6.0	5.0	4.5	4.0	3.5
Temp. °C	23.2	23.5	23.0	23.1	23.3	23.0	23.2
pH	7.2	7.0	7.1	7.0	7.0	6.9	6.9
Total Alkalinity	29				62		88

APPENDIX H

TECHNICAL INFORMATION

DATA BASE DESCRIPTION