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ANALYSIS OF WASTE WATER SAMPLES & INHALATION REPRODUCTION RANGE-FINDING STUDY IN MATED RATS WITH C9 AROMATIC HYDROCARBONS WITH COVER LETTER DATED 051089		
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Chemical Category		
1,1,1-TRICHLOROETHANE (71-55-6)		

CONTAINS NO CBI

Shell Oil Company



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Houston, Texas 77210

EPA-OTS



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86-890000228

May 10, 1989

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U.S. Environmental Protection Agency
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SUBJECT: TSCA SECTION 8(D), HEALTH AND SAFETY DATA REPORTING

As required by Toxic Substances Control Act, Section 8(d), Shell is submitting copies of health and safety studies as outlined in 40 CFR 716. Two reports are enclosed (Report Numbers 382 and 383). Each individual study is accompanied by a cover page as requested under 716.7(b). An index is also enclosed which includes the study title, date, specific chemical substance, CAS number, and Shell's report reference number for ease of future identification. Report Number 382 contains confidential information, and, therefore, two copies are enclosed and marked as required by 716.55.

Sincerely,

J. C. Willett
Manager, Product Safety and Compliance
Oil and Chemical Products
Health, Safety and Environment

JWH/j11

Enclosures

INDEX OF HEALTH AND SAFETY STUDIES SUBMITTED TO EPA
SORTED BY SUBMISSION NUMBER

SUB. NO.	TITLE	RPT. DATE	CHEMICAL	CAS NUMBER	SMELL REF.
382	ANALYSIS OF WASTE WATER SAMPLES. I - DEER PARK MANUFACTURING C COMPLEX CHEMICAL PLANT.	770900	TRICHLOROETHANE, 1,1,1-	000071-88-6	MRC T9R 108-77
			CHLOROFORM	000087-88-2	
			DICHLORO-1,2-ETHANE	000107-08-2	
			ACETONITRILE	000078-08-8	
			TOLUENE	000108-88-3	
			ISOBUTYL ALCOHOL	000078-88-1	
			ETHYLBENZENE	000109-41-4	
			XYLENE	001920-20-7	
			EPICHLOROHYDRIN	000108-88-8	
			CUMENE	000098-82-8	
			XYLENE, O-	000098-47-6	HSE-88-0128
383	INHALATION REPRODUCTION RANGE-FINDING STUDY IN MATED RATS WITH CO AROMATIC HYDROCARBONS (FINAL REPORT).	880400	CUMENE	000098-82-8	
			ETHYLTOLUENE, 4-	000822-96-8	
			ETHYLTOLUENE, 2-	000811-14-3	
			TRIME THYLBENZENE, 1,3,5-	000108-87-8	
			TRIME THYLBENZENE, 1,2,4-	000098-82-6	
			TRIME THYLBENZENE, 1,2,3-	070828-79-8	
			ETHYLTOLUENE, 3-	000820-14-4	

ITEMS RETRIEVED 2

REPORT NO. 382

SUBMISSION OF LISTS AND COPIES OF HEALTH AND SAFETY STUDIES

NAMES OF REQUESTED SUBSTANCES(S)

CAS NUMBER(S)

TRICHLOROETHANE, 1,1,1-
CHLOROFORM
DICHLORO-1,2-ETHANE
ACETONITRILE
TOLUENE
ISOBUTYL ALCOHOL
ETHYLBENZENE
XYLENE
EPICHLOROHYDRIN
CUMENE

000071-85-6
000067-75-3
000107-06-2
000075-05-8
000108-88-2
000078-83-1
000100-41-4
001330-20-7
000106-89-8
000088-82-8

OWRS
OWRS

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REPORT NO. 389

SUBMISSION OF LISTS AND COPIES OF HEALTH AND SAFETY STUDIES

NAMES OF REQUESTED SUBSTANCES(S)

CAS NUMBER(S)

XYLENE, O-
CUMENE
ETHYLTOLUENE, 4-
ETHYLTOLUENE, 2-
TRIMETHYLBENZENE, 1,3,5-
TRIMETHYLBENZENE, 1,2,4-
TRIMETHYLBENZENE, 1,2,3-
ETHYLTOLUENE, 3-

000088-47-6
000088-82-8
000822-86-8
000811-14-3
000108-67-8
000088-63-6
000826-73-8
000820-14-4

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Analysis of Waste Water Samples. I
Deer Park Manufacturing Complex Chemical Plant

Technical Progress Report WRC 105-77

Project No. 52722, 60444
Waste Water Treatment

Printed: September 1977

Author: G. H. Stanko
Reviewer: M. A. Muhs
Approval: M. J. O'Neal
**Participants: P. A. Wadsworth, E. M. Fredericks, J. C. Rapean, H. A. Petersen,
J. L. Havel, R. W. Coffey**
References: Based on work through February 1977
LH 12517, pp. 107-185

Analysis of Waste Water Samples. I

AL

Deer Park Manufacturing Complex
Chemical Plant

G. H. Stanko

COMPANY SANITIZED

CONTAINS MICRO

Technical Progress Report WRC 105-77

Project No. 52722, 60444



SHELL DEVELOPMENT COMPANY

A DIVISION OF SHELL OIL COMPANY

Westhollow Research Center
Houston, Texas

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ABSTRACT

Influent and effluent water samples were collected from the DPMC Chemical Plant during October, 1976. The identification and quantification of the trace organics present in the water were of prime interest. Details for the method of sampling, the sampling schedule, analytical methods used, and the resulting data are presented. Data for biotreater efficiency with respect to reduction of TOC, BOD, and COD across the treatment system as well as fish-toxicity data are also included.

Court ordered timetable and with a final completion date of December 31, 1979. With respect to the petroleum refining category, the EPA initiated a very ambitious program in an effort to meet the deadline. Twelve refineries across the country were selected for sampling of intake water influent to the biotreaters, and plant effluents to determine the presence or absence of organic pollutants based on a 1 ug/liter (ppb) concentration level. The original list of "sixty-five toxic pollutants" was subsequently better defined and resulted in a list of 108 specific organic compounds. The data for the refinery survey would be used to reduce (or increase) the 108 list for the petroleum refining category. The program has been delayed a number of times due to serious problems with the Sampling Protocol and Analysis Protocol. Drafts of both Protocols were made available to industry for comment through American Petroleum Institute activities. Constructive criticism from knowledgeable scientists brought to light many problems which the EPA are trying to resolve at the present time (February 1977).

In June of 1976, an Analytical Project was initiated to develop the capability of analyzing waste water samples for organic pollutants down to the 1 ug/liter (ppb) level. Analytical laboratories of the EPA located at Athens, Georgia, Cincinnati, Ohio; and Ada, Oklahoma, as well as the Biological Sciences Research Center at Modesto, California, were visited to learn of the necessary requirements and difficulties involved with trace analyses. With the cooperation and advice of numerous individuals, it became possible to develop the capabilities for trace analysis in a relatively short time. Subsequently, we were requested to sample the Deer Park Manufacturing Complex Chemical Plant and to analyze influent and effluent samples for trace organic pollutants. The Chemical Plant was sampled in October 1976 and a very comprehensive analysis was undertaken.

EXPERIMENTAL

Sampling Method

The subject of sampling waste water streams has received a great deal of attention and remains a very controversial issue. The two most common methods of sampling are grab samples and flow rate samples, and either method has its own set of advantages and disadvantages. The grab samples have reported or estimated concentrations based on the weight of the sample collected in the stream. The flow rate samples are based on the weight of the sample collected and the flow rate of the stream.

TECHNICAL PROGRESS REPORT WRC 105-77

ANALYSIS OF WASTE WATER SAMPLES. I
Deer Park Manufacturing Complex Chemical Plant

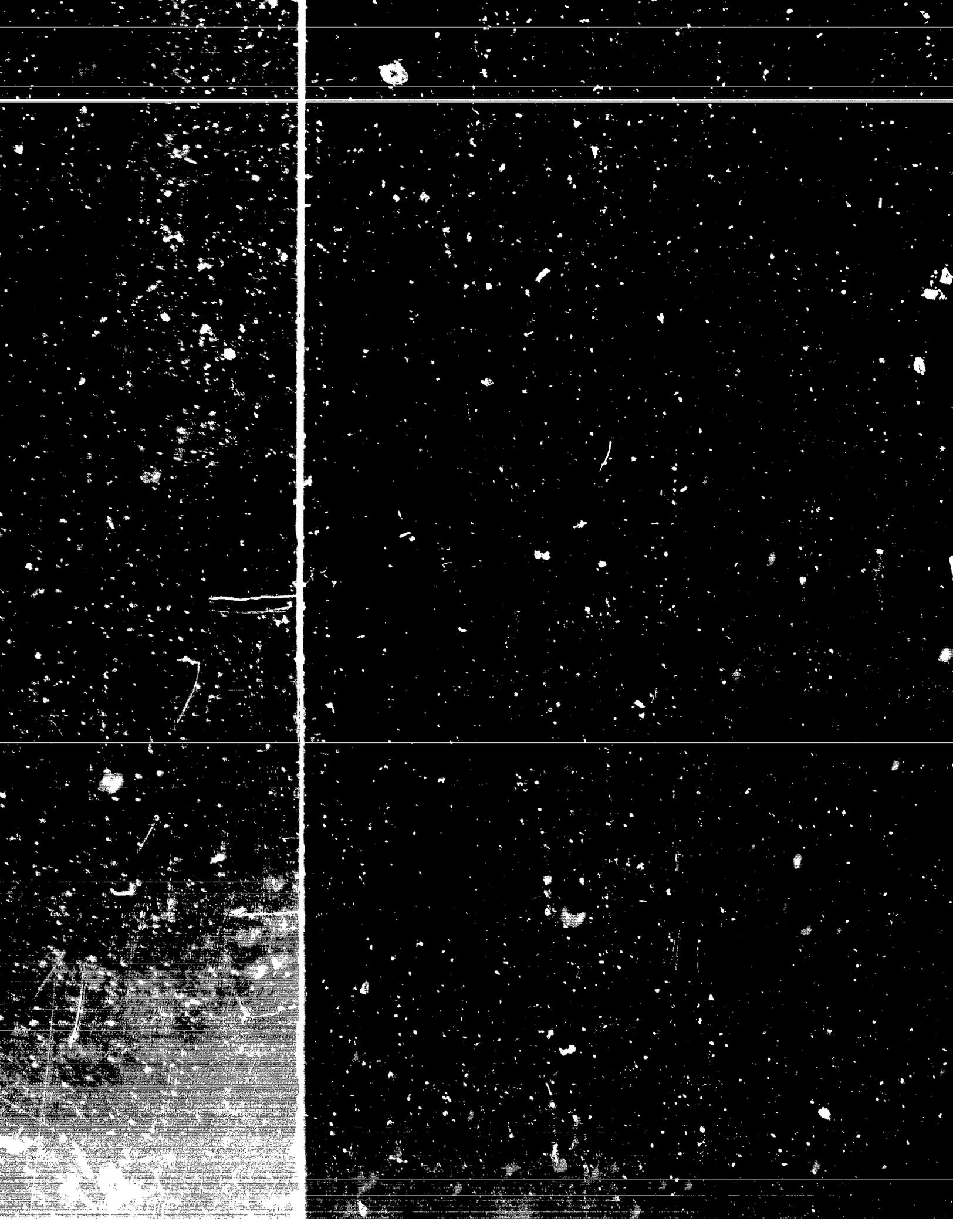
BY

G. H. STANKO

INTRODUCTION

Late in 1974, a nationally televised program focused attention on a study that indicated the presence of a number of alleged toxic organic compounds in the New Orleans, Louisiana, public drinking water. The presence of organohalides such as chloroform caused much public concern and were thought to be formed in some way by the reaction of the chlorine used for disinfection with trace organics or precursors in the raw water. Although there are many possible sources of organics in public water intakes, the TV program strongly suggested chemical and industrial plant effluents and surface runoff from agricultural land where chemical herbicides, pesticides, and fertilizers had been used as the major sources of the organics.

The Federal Water Pollution Control Act Amendments of 1972 (PL 92-500) had been in effect for some time and the Effluent Guidelines Division of the U.S. Environmental Protection Agency was making an effort to implement the many aspects of a very complicated law. However, certain environmentalist groups initiated legal action which resulted in suits filed against Russell E. Train the Administrator (at that time) of the EPA. Essentially, the plaintiffs alleged that the Administrator had not satisfied a statutory duty of timely promulgation of pretreatment standards under Section 307(b) (and other sections) of PL 92-500. In March of 1976, there emerged a proposed settlement agreement between the EPA and the environmentalist groups which would obviate trial in the cases pending in court. On June 9, 1976, the plaintiffs and the EPA filed acceptance of a 307(b) Consent Decree. The EPA now are required to issue effluent limitations and guidelines, new source standards, and pretreatment standards for the "sixty-five toxic pollutants" (Appendix A of Consent Decree) for twenty-one industrial categories (Appendix B of Consent Decree) per the



for sampling and actual sample requirements may be vastly different. The simple fact remains that, for a proposed sampling, one must determine the requirements and devise a procedure that will provide an adequate quantity of a uniformly representative sample and that will keep contamination at a minimum.

Flow calculation for the DPMC Chemical Plant indicated a hold-up time in the treatment plant of approximately 28 hours. Although relatively uniform effluents were expected, it was decided to collect five influent grab samples and three effluent grab samples during 24 hour periods that were approximately 28 hours apart. The actual sampling schedule used for the Chemical Plant is illustrated in Table 1.

Table 1
SAMPLING SCHEDULE
DPMC CHEMICAL PLANT

<u>Date</u>	<u>Influent</u>	<u>Effluent</u>
10/6/76	0900 hrs	-
10/6/76	1700 hrs	-
10/7/76	0100 hrs	-
10/7/76	-	1300 hrs
10/7/76	-	2100 hrs
10/8/76	-	0500 hrs

The amounts of influent and effluent collected and the containers that were used are listed in Table 2.

Due to the large volume of sample required, it was decided to sample with a 5 gal/min pump with Teflon head and internal parts. New Tygon tubing was used for influent and effluent sampling and the total length at each site was approximately 12 feet. The suction line was held approximately 4-6 inches below the stream surface through use of a stainless steel nipple attached to the end of the suction line. At each sampling the pump was operated 10-15 minutes before collection was initiated. Open-top epoxy lined drums (55 gal) were employed at each sampling site for a sample collection vessel.

For influent sampling, 45 gallons were pumped (9 minutes) into the open-top drum. The analytical samples were taken by dipping a 2 liter beaker into the center of the drum and quickly transferring to the proper containers that had been properly labeled and cleaned according to EPA recommendations (Appendix I). The two drum samples were collected by using the sampling pump

Table 2
CONTAINERS USED AND AMOUNTS OF SAMPLE COLLECTED
AT DPMC CHEMICAL PLANT

<u>Sample Container</u>	<u>Number Required for Each Sampling</u>	<u>Purpose</u>
40 ml screw cap glass vials sealed with Teflon faced septa. Vials - Pierce #13074 Septa - Pierce #12722	3	VOA
1 qt polyethylene wide-mouth bottle.	1	Metals
1 qt glass narrow-neck bottle. Cap with Teflon liner.	1	Oil and grease
1 gal glass narrow-neck bottle. Cap with Teflon liner.	1	LLE, TOC, N ₂ , NH ₃ , nitrate, organic chloride
1 gal glass narrow-neck bottle. Cap with Teflon liner.	1	1) COD, BOD, TDS, TSS, phenol, cyanide, pH
1 pt polyethylene wide-mouth bottle	3	2) TOC, SS, VSS, ATP, dehydrogenase activity, sulfide
55 gal epoxy-lined steel drum.	2 ³⁾	4) Biotreating studies
55 gal epoxy-lined steel drum.	2 ³⁾	5) Filtration/Carbon sorption studies
6 gal polyethylene carboys.	3	6) Fish toxicity studies

-
- 1) Sample collected for analyses run at Funa Wood Laboratory.
 - 2) Sample collected for Hydrocarbon Engineering to determine aeration basin condition. One sample was collected from each of the three basins at each sampling.
 - 3) 10 gal of influent or effluent was added to each drum at each sampling. This resulted in drum-size (54 gal) composite samples.
 - 4) Samples of influent collected for Hydrocarbon Engineering.
 - 5) Samples of effluent collected for Chemical Engineering.
 - 6) Samples of effluent collected for Hydrocarbon Engineering.

plus a filter with a 50 micron element. The pump was used to take sample from the collection vessel, filter it, and fill a 6-gal polyethylene carboy. The 6-gal portions of sample were dumped alternately into the two epoxy lined drums until 18 gallons were added at each sampling.

Virtually the same procedure was followed for sampling of the effluent except an additional 18 gallons of sample (total \approx 57 gal) were required. To accomplish this with a 55 gal collection vessel, the analytical samples were collected after 45 gallons of effluent were collected and during the time the rest of the vessel was being filled. The two drum samples were collected in the same manner and were filtered.

A 54-gallon composite sample of effluent (collected at 8-hour intervals in a 24-hour period) was collected in 6-gallon polyethylene carboys for fish toxicity tests. The carboys were stored at $\leq -10^{\circ}\text{C}$ from \approx 10:00 A.M. 10/8/76 to 8:00 A.M. 10/10/76. The carboys were then thawed at room temperature and delivered to Edna Wood Laboratories, Inc., at 8:30 A.M. 10/11/76 for fish toxicity tests.

At each of the six samplings, biotreater (aeration) basin samples (1 pt) were collected from each of the three basins by dipping a metal bucket near one of the aerators.

Sample Preservation

Complete and unequivocal preservation of waste water samples is a practical impossibility. Complete stability for every constituent can never be achieved. At best, preservation techniques, can only retard the chemical and biological changes that inevitably continue after the sample is isolated from its parent source. Methods of preservation are relatively limited and are intended to retard biological action, retard hydrolysis, and reduce volatility of constituents. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing. Methods for preservation of water samples have been published² (Appendix I) by the EPA; however, it is not possible nor practical to comply in many instances. In certain instances, one may be in violation of Department of Transportation regulations when preservatives (HNO_3) are added to samples for air transportation. In some cases, a single sample container may be used to collect sample for as many as six different analyses. The chemical preservatives may be different and not compatible in some instances. The samples taken at the DPMC chemical plant were placed on ice at the sampling site and were kept on ice or under refrigeration until all the analyses were performed. The only exception was

the sample for metals. This sample was on ice until transported to Westhollow then 5 ml (reference 2) of Ultrex nitric acid was added to each container. The samples for metals analysis were not refrigerated beyond this point.

The gallon samples for the various analyses run at Edna Wood Laboratory were delivered in ice per their instruction. Beyond this point we had no further control.

The quart samples for oil and grease analysis were placed in a designated refrigerator in the laboratory at the chemical plant. This was done for each of the six samplings.

Chemical Plant Operation

The sampling schedule for the chemical plant was prepared for a time when operations would be considered normal. The operational parameters and routine test data prior to, during, and after our sampling was obtained for Manufacturing Environmental and Conservation. The plant operational data will appear in a Head Office Report and will not be included as part of this report since the primary thrust of our effort was directed at characterization of the waste water with trace organics of major concern. Only weather conditions and waste water temperatures will be listed. These data are summarized in Table 3.

Table 3
SAMPLING SITE CONDITIONS
FOR CHEMICAL PLANT

Sampling Time/Date	Temp (°F)	Water Temp (°F)	General Weather Conditions
0900 10/6	60	125	Clear and mild.
1700 10/6	74	123	Clear and warm.
0100 10/7	64	124	Clear and mild.
1300 10/7	74	84	Clear and warm.
2100 10/7	64	83	Clear and windy (≈20 mph), weather front moving in.
0500 10/8	54	82	Cloudy, cold with dry slight rain. Had light rain in area from 0300.

A chemical spill had occurred prior to the first (0900) influent sampling and it was later learned that some of the spill material was collected in the 0900 hour sampling. We actually learned of the spill after most of the sampling was completed and there was some consideration of not analyzing the

collected samples because the 0900 grab sample would not be considered representative of normal operations. However, it was decided to continue with the proposed analyses but not to include the 0900 grab sample in the composite influent sample for trace organics analysis by methylene chloride (LLE) extraction. Also, no volatile organics data (VOA) would be obtained for the 0900 grab sample. However, most other analyses were performed on this grab sample. The resulting data attest to the fact the grab sample was indeed not representative and justified the decision to not include it in the composite influent for trace organics analysis.

The analytical data for the sampling of the DPMC Chemical Plant were obtained by using published standard methods³ when available. The actual methods used for each of the analyses are tabulated in Appendix II.

Two methods used by the EPA and described in "Protocol for the Measurement of Toxic Substances Environmental Monitoring and Support Laboratory - Cincinnati" were employed for the determination of organics. The protocol describes the Bellar purge and trap (VOA) method^b for volatile organics which uses a home-built sparging device connected to a GC with a Hall detector. The volatile organics in the DPMC Chemical Plant samples were determined using a TEOMAR LSC-1 sparging unit coupled directly to a Finnigan 3200F-6103 GC/MS - Data System. The general condition for the VOA analyses are listed in Table 4.

Table 4
GC/MS CONDITIONS FOR VOA SAMPLE METHOD

Mass Range Scanned: 24-230

Transfer and Separator Temperature: 225°C

GC Column: 1/8" x 2', 3% Carbowax 20M on 40/60 Chromasorb T, stainless steel column, 65°C for 20 minutes, then 65°C to 150°C at 4°/minute.

VOA Apparatus Condition: Transfer lines at 24°C

Sample Size and Conditions: 5 ml water sparged with He for 10 minutes at 30 ml/minute. Sample temperature 25°C

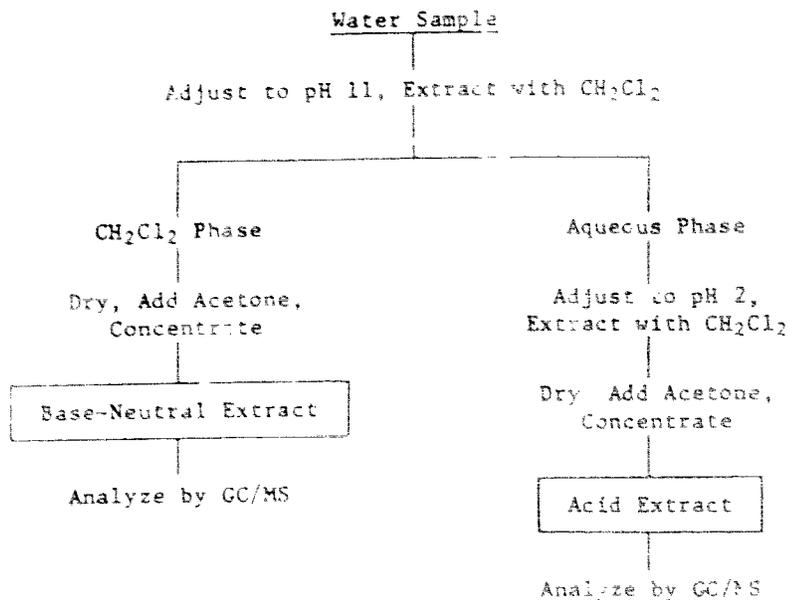
Trap desorbed for 4 minutes at 140°C.

Complete details for modifications made to the LSC-1 and actual procedure that was followed will be reported shortly in a Technical Progress Report.

The Chemical Plant samples were analyzed for volatile organics (VOA) as soon after sampling as possible which was approximately 2 weeks. The LSC-1 unit arrived after the Chemical Plant sampling and became operational within 2 weeks of the actual sampling.

The procedure for liquid/liquid extraction (LLE) of nonvolatile organics with methylene chloride, that was given in the protocol, was followed. The flow diagram for the liquid/liquid extraction was as follows:

METHOD A



The general conditions for the GC/MS analysis of the LLE fractions for nonvolatile organics are listed in Table 5. Complete details for the GC/MS procedure will be reported shortly in a Technical Procedures Report.

The methods used for trace organics in water are constantly being modified or changed as experience is being gained. The procedures used for the analysis of the TSC Chemical Plant were current state-of-the-art at the time of the sampling.

Table 5
GC/MS CONDITIONS FOR LLE FRACTIONS

Mass Range Scanned: 24 to 425
 Transfer and Separator Temperature: 270°C
 Injector Temperature: 275°C
 GC Column: 2 mm x 5' 3% OV 101 on 80/100 Supelcoport,
 Glass Column
 35°C for 20 minutes, then 35°C to 270°C
 at 4°/minute

ANALYTICAL DATA FOR DPMC CHEMICAL PLANT

The data for the general parameters which are part of the NPDES requirements are tabulated in Table 6. The TOC, BOD, and COD data were used to calculate the biotreater efficiency based on the reduction of these three parameters across the treatment system. The calculations indicated reduction of 94.2%, 99.4%, and 92.0% for TOC, BOD, and COD, respectively. The calculation data are listed in Table 7. It appears that the biotreaters were operating very efficiently based on the reduction of the three parameters.

Samples were collected from each of the biotreaters at each of the six sampling times. These samples were analyzed to determine the general biological state of the biobasins. The analytical data obtained are tabulated in Table 8.

It is general practice to use the VSS as a measure of biomass; however, this mass includes both viable and nonviable organisms. Since the DPMC Chemical biotreater is operated with a very long sludge age, it is expected that the percentage of viable organisms in the sludge will be small.

In order to establish the level of bioactivity in the sludge, adenosine triphosphate (ATP) and dehydrogenase activities were measured. Results are given in Table 8.

ATP is present in all living cells in approximately constant amount. It was extracted from lysed cells and measured by use of the luciferin-luciferase enzyme system (firefly lantern extract). There was a marked increase in ATP concentration in the sludge of all three basins between 0900 and 1700 hours, which is attributed to the increase in biodegradable carbon in the feed due to a spill. A similar response was seen in the dehydrogenase activity which

Table 6

ANALYTICAL DATA DPMC CHEMICAL PLANT
WASTE WATER

Analysis	Influent				Effluent			
	0900	1700	0100	Composite	1300	2100	0500	Composite
	10/6/76	10/6/76	10/7/76	1700 0100	10/7/76	10/7/76	10/8/76	1300 2100 0500
Total Organic Carbon (TOC), mg/l	1503	795	751	789	41	49	48	46
Nitrite-Nitrogen, mg/l	nil	nil	0.02	0.02	0.01	0.01	0.02	0.018
Ammonia, mg/l	45.8	51.9	51.9	54.0	9.2	7.6	8.5	8.8
Pyridine-Nitrogen, ppm	56	57	53	54	2	2	1	<1
Organic Chloride (ZAL-4/4-OH Method), ppm	65	18	18	23	3	3	3	3
Oil + Grease, mg/l	29.9	21.8	19.9	-	2.2	0.8	0.7	-
Total Suspended Matter, mg/l	9.24	9.4	9.4	-	7.35	7.35	7.35	-
Total Dissolved Solids (by diff.), mg/l	42	54	68	-	7	7	8	-
Biological Oxygen Demand 5-Day 20°C (seeded), mg/l	31,875	23,229	25,311	-	19,514	20,294	20,441	-
Chemical Oxygen Demand (AS ₂ SO ₄ + HgSO ₄ Method, mg/l)	1,300	993	948	-	5	6	6	-
Phenols, as Phenol, mg/l	5,080	2,760	2,300	-	178	5	204	-
Cyanide, CN, mg/l	6.6	-	-	4.2	<0.1	<0.1	<0.1	-
	<0.1	-	-	0.2	<0.1	<0.1	<0.1	-

Table 7
CALCULATIONS FOR BIOTREATER EFFICIENCY

TOC Reduction

Influent Composite (1700/0100)	789 mg/l TOC
Effluent Composite (1300/2100/0500)	46 mg/l TOC

$$\frac{789 - 46}{789} \times 100 = 94.2\% \text{ TOC Reduction}$$

BOD Reduction

Influent Composite (1700/0100)	971 mg/l BOD
Effluent Composite (1300/2100/0500)	6 mg/l BOD

$$\frac{971 - 6}{971} \times 100 = 99.4\% \text{ BOD Reduction}$$

COD Reduction

Influent Composite (1700/0100)	2380 mg/l COD
Effluent Composite (1300/2100/0500)	190 mg/l COD

$$\frac{2380 - 190}{2380} \times 100 = 92.0\% \text{ COD Reduction}$$

Table 8
ANALYTICAL DATA FOR DPMC CHEMICAL PLANT BIOBASINS

	Analysis					
	TOC, mg/l	SS, mg/l	VSS, mg/l	ATP, mg/l	Dehydrogenase Activity, mg/l	Sulfide, mg/l
0900 East Basin	56	5200	4584	1.87	52.0	-
0900 Mid Basin	62	5603	4880	1.81	61.0	-
0900 West Basin	62	4475	4070	2.23	52.5	-
1700 East Basin	68	5055	4400	4.66	80	-
1700 Mid Basin	49	5520	4850	4.37	123	-
1700 West Basin	82	4860	4265	5.13	69	-
0100 East Basin	48	5030	4425	4.18	37	-
0100 Mid Basin	70	5000	5260	4.37	82	-
0100 West Basin	102	4895	4330	3.61	90	-
0900, 1700, 0100 Comp. East Basin	-	-	-	-	-	0.20
0900, 1700, 0100 Comp. Mid Basin	-	-	-	-	-	0.20
0900, 1700, 0100 Comp. West Basin	-	-	-	-	-	0.40
1300, 2100, 0500 Comp. East Basin	50	5000	4700	2.76	119	0.10
1300, 2100, 0500 Comp. Mid Basin	65	4860	4265	2.55	98	0.05
1300, 2100, 0500 Comp. West Basin	47	5175	5090	3.03	139	0.10

was determined by measuring the reduction of a dye (2,3,5-triphenylt: chloride) by the sludge in the absence of oxygen.

Both ATP and dehydrogenase activity levels as well as their response to an increased organic carbon load are indicative of a well operating biotreater

The samples from the DPMC Chemical Plant were analyzed for thirteen metals found on the "122 List". These data are tabulated in Table 9. Each of the six samples were analyzed to determine variations in concentration with time. The greatest variations were observed for chromium, nickel, and zinc. Lesser variations were observed for most other metals. The concentrations of thallium, mercury, lead and beryllium were below the limits of detection which were 5, 1, 4, and 1 $\mu\text{g/l}$, respectively.

The GC/MS data for VOA analyses and analysis of LLE fraction for the DPMC Chemical Plant sampling are listed in Tables 10-15. Tables 10-12 show the data for the final effluent and Tables 13-15 show the data for the biotreater influent. There is an extreme reduction in number and concentration of organics across the treatment system. Only one compound, 1,2-dichloroethane, was found in the effluent at a relatively high concentration ($\approx 170 \mu\text{g/l}$). The rest of the compounds were present at relatively low concentrations. Calculations were made to determine what percent of the TOC resulted from compounds identified in influent and effluent samples. Three values of organic carbon were calculated for each sample, (1) that from the observed compounds on the "122" toxic list; (2) that from all compounds observed whose empirical formula could be determined; and (3) that based on the total concentration of organic compounds observed from the total ion current of the GC/MS determination. In this last case the average percent carbon observed in (2) was used to make the calculation. The results are summarized in Tables 16 and 17.

The amounts observed in the influent are about what was expected. However, with the effluent, concentration of carbon observed by the VOA-LLE/GC-MS technique is extremely low. Obviously a lot of highly polar water soluble, or higher molecular weight materials, are produced in the biotreater relative to the amount of original organic chemicals remaining in the influent.

The bioassay reports for the fish toxicity tests are included as Appendix III. The DPMC Chemical Plant effluent collected on 10/7-8/76 did not exhibit 96 hr TLM to *F. similis* or *Gambusia affinis* fish.

2

5

Table 16
ORGANIC CARBON CONCENTRATION IN EFFLUENT, ppb(ug/l)

<u>Analysis</u>	<u>From Cpds on 122 List</u>	<u>From All Cpds Iden. by GC/MS</u>	<u>From GC/MS Total Ion Current</u>
VOA	44.0	64.5	64.5
LLE-NB	0.0	11.8	16.1
LLE-A	0.0	9.9	14.5
Σ	44.0	86.2	95.1
Measured TOC (mg/l)	46.0	46.0	46.0
% of TOC	0.1	0.2	0.2

Table 17
ORGANIC CARBON CONCENTRATION IN INFLUENT, ppm(mg/l)

<u>Analysis</u>	<u>From Cpds on 122 List</u>	<u>From All Cpds Iden. by GC/MS</u>	<u>From GC/MS Total Ion Current</u>
VOA	5.88	14.63	21.9
LLE-NB	1.06	9.38	21.3
LEE-A	0.84	6.02	20.3
Σ	7.78	30.03	63.5
Measured TOC	789	789	789
% of TOC	1.0	3.8	8.0

DISCUSSION AND CONCLUSIONS

The sampling and analysis of waste water from the DPMC Chemical Plant represent the first of such effort at WRC to comprehensively characterize waste water from a Shell manufacturing location. The data contained in this report are the result of the efforts and cooperation of many individuals and should provide the necessary information which is required to plan to meet forthcoming regulations on plant effluent.

The analysis of a waste water for organics is not a simple task and involves three major steps: (1) representative sampling of a stream, (2) quantitative concentrating of the organics, and (3) GC/MS analysis to identify and quantify the components present. Inadequacies in any one or more of the three steps will seriously affect the quality of the resulting data and, consequently, its value. Invalid environmental data are worse than none at all. In review of the efforts to characterize the water samples from the DPMC Chemical Plant, we feel that the most possible techniques were employed which represent the current state of the art for water analyses.

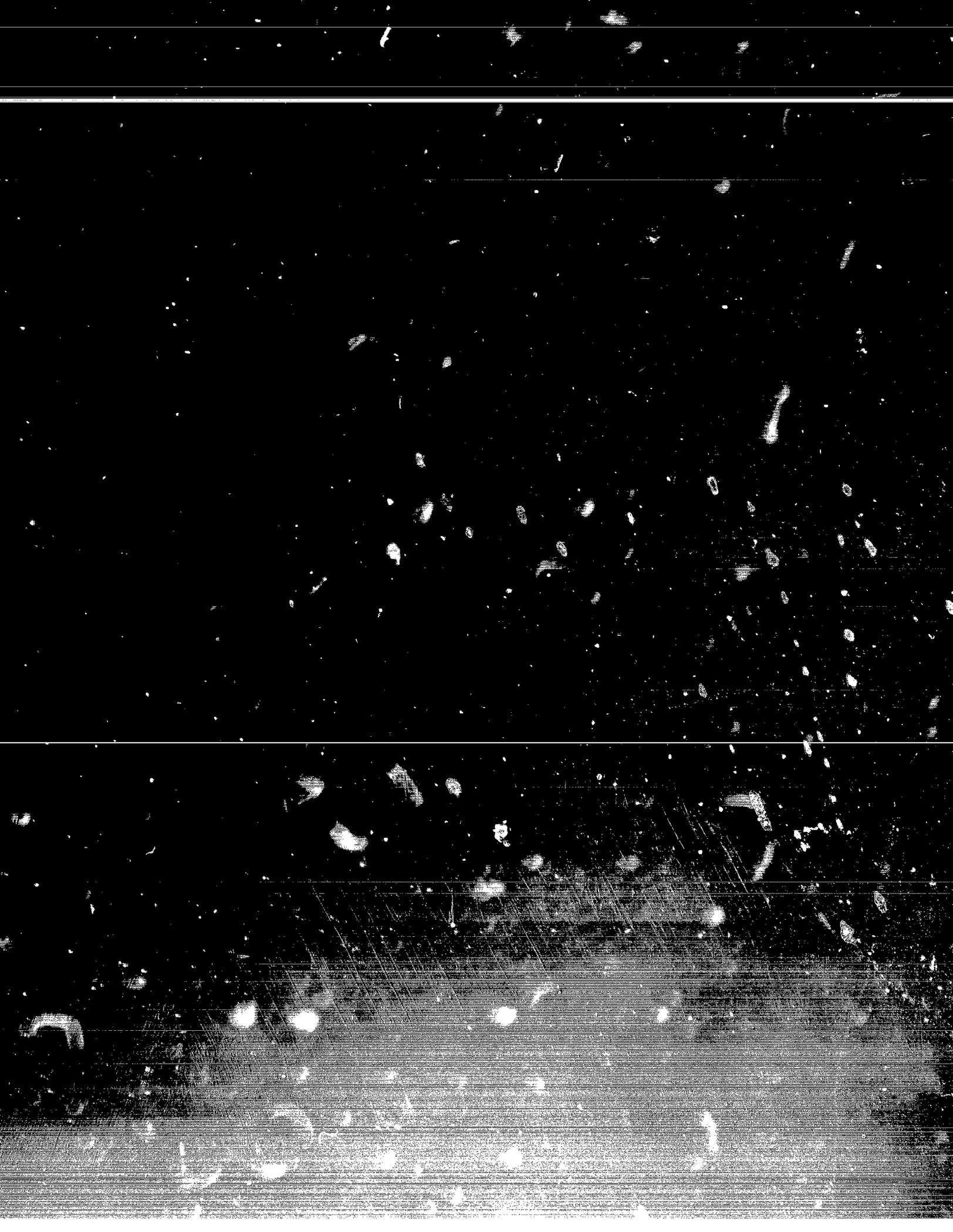
The Consent Decree list of toxics has been changed in the past and will, no doubt, be changed in the future. It is, therefore, necessary to document what was current at a point in time. The EPA list of toxics in effect at the time of the DPMC Chemical Plant sampling was the "122 List" which is included as Appendix IV for future reference.

BIBLIOGRAPHY

1. Handbook for Monitoring Industrial Wastewater, U.S. Environmental Protection Agency Technology Transfer, 1st Edition 1973, Washington, D.C.
2. Manual for Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency Technology Transfer, 1974 Edition, Washington, D.C.
3. Standard Methods for the Examination of Water and Wastewater, Fourteenth Edition 1975, American Public Health Association, 1015 Eighteenth Street NW, Washington, D.C.
4. Bellar, T. A., and J. J. Lichtenberg, "The Determination of Volatile Organic Compounds at the $\mu\text{g/l}$ Level in Water by Gas Chromatography", Environmental Monitoring Series EPA-670/4-74-009, November 1974, U.S. Environmental Protection Agency, Cincinnati, Ohio.

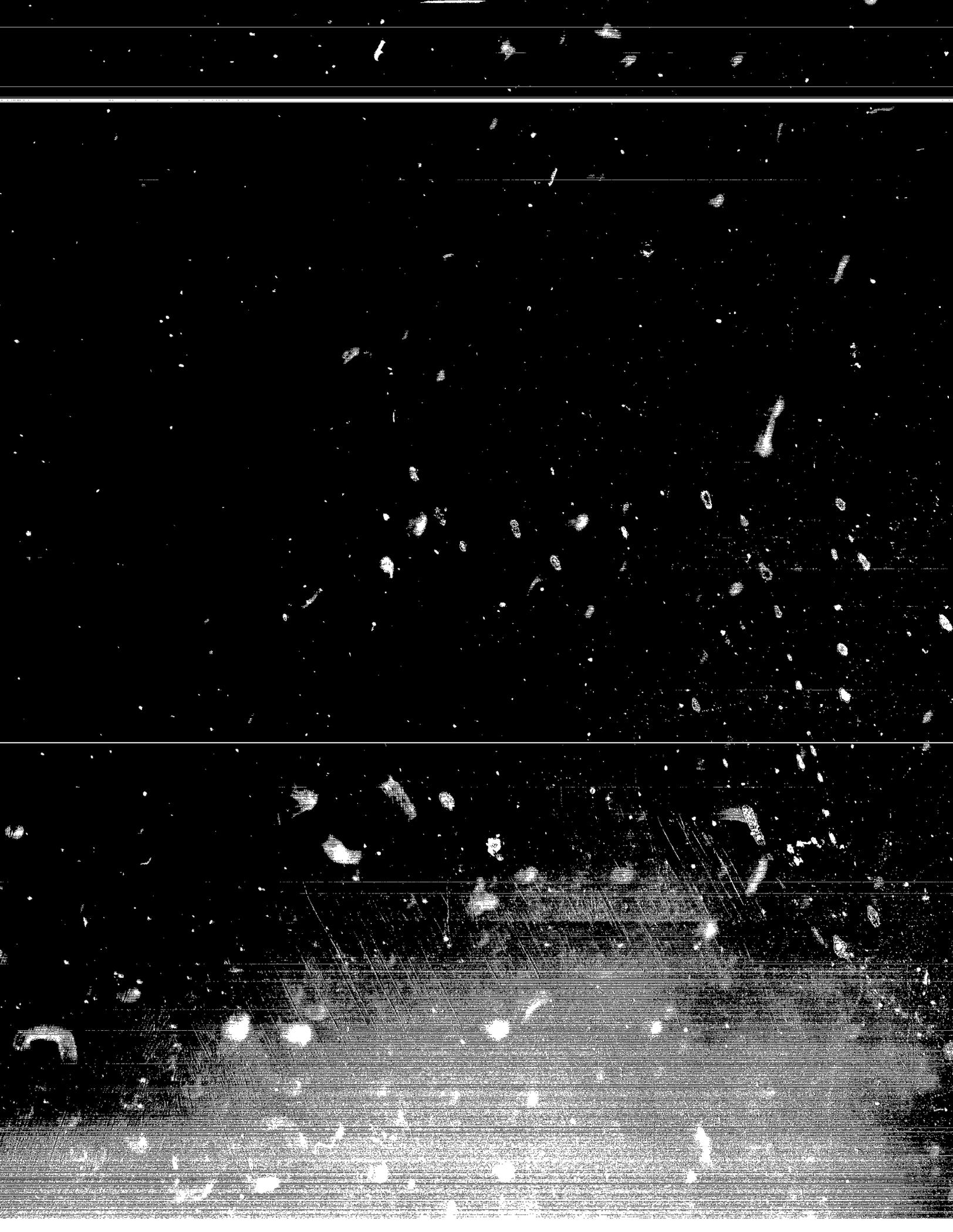
GLOSSARY OF ABBREVIATIONS

ATP	Adenosine Triphosphate
BOD	Biocemical Oxygen Demand
COD	Chemical Oxygen Demand
GC/MS	Gas Chromatographic/Mass Spectrometric
LLE	Liquid-Liquid Extraction
SS	Suspended Solids
TDS	Total Dissolved Solids
TLm	Tolerance Limit
TOC	Total Organic Carbon
TSS	Total Suspended Solids
VOA	Volatile Organics Analysis
VSS	Volatile Suspended Solids



GLOSSARY OF ABBREVIATIONS

ATP	Adenosine Triphosphate
BOD	Biochemical Oxygen Demand
COD	Chemical Oxygen Demand
GC/MS	Gas Chromatographic/Mass Spectrometric
LLE	Liquid-Liquid Extraction
SS	Suspended Solids
TDS	Total Dissolved Solids
TLm	Tolerance Limit
TOC	Total Organic Carbon
TSS	Total Suspended Solids
VOA	Volatile Organics Analysis
VSS	Volatile Suspended Solids



APPENDIX I

SAMPLING SCREENING PROCEDURE
FOR THE MEASUREMENT OF PRIORITY POLLUTANTS

(C O P Y)

SAMPLING SCREENING PROCEDURE
FOR THE MEASUREMENT
OF PRIORITY POLLUTANTS

U.S. ENVIRONMENTAL PROTECTION AGENCY

Effluent Guidelines Division
Washington, D.C. 20460

OCTOBER 1976

Collection of Composites Samples

A 72 hour composite will be collected, maximum time interval between aliquot samples no longer than 30 minutes. The minimum aliquot size shall be 50 ml. The sample must be collected with an automatic sampler using the equipment and methods outlined below. Minimum composite volume must be 2 1/2 gallons.

Automatic Sample Collection

Sampler - A peristaltic pump automatic sampler with timer and a single glass compositing jug is required. The 2 1/2 - 3 gallon compositing jug must be glass and cleaned as outlined below. Sample and pump tubing must be new for each individual outfall or sample location utilized. Vacuum type automatic samplers may be used provided that the sample chambers are glass and that they are cleaned after every use as outlined for the glass compository containers. For raw discharges the sampler sample line velocities should approach 2 feet per second. The sampler or compositing sample container should be insulated and ice (or other means) shall be used to maintain the sample being composited at (4°C) during the compositing procedure. At the completion of the compositing period the 2 1/2 - 3 gallon compositing container shall be sealed with a Teflon lined cap that has been rinsed with methylene chloride. The compositing container shall be shipped without tampering to the analytical lab and shall remain iced at (4°C) during transport and while it is prepared for analysis.

Field Blank Procedure for Automatic Samplers

1. Water Blank must be as organic free as possible. The analytical lab should supply this water in bulk containers for field use. The supplying laboratory shall run analyses of the Water Blank to insure that no organic compounds are present.
2. Procedure - The sampler tubing shall be cut to length but not installed. Two liters of the Water Blank should be pumped through the sampling line and pump tubing into the glass compositing container. The compositing container should be rotated so that the Water Blank rinses all surfaces and discard. Pump 3 liters of Water Blank through the tubing into the compositing container. Again rotate the container and pour blank into a 1 gallon sample bottle that has been prepared as outlined in the non-purgable organics section. Again use a Teflon lined cap that has been

rinsed with methylene chloride. Immediately ice the blank (4°C) and keep on ice at (4°C) during transport and preparation for analysis.

Composites Container

Containers - Prepare narrow-mouth 3 gallons glass/sample bottles for use by washing with detergent and rinsing with tap water followed by distilled water. Heat the bottles at 400°C in a muffle-furnace or dry heat sterilizer for 30 minutes or alternatively, rinse with methylene chloride and air dry at room temperature. Caps for the bottles must be lined with Teflon.

Collection of Grab Samples

Grab samples (a minimum of one per day) will be taken for the analysis of phenol, cyanide, mercury and volatile organics (purgable). Samples will be taken of the raw process discharge, the treated effluent, and the treated effluent after chlorination, when chlorination is practiced.

Sample Location

It is recommended that the samples be collected from mid-channel at mid-depth. Samples should be collected at a turbulent, well mixed section of the channel.

Cyanide

Volume of sample - one liter.

1. Container: one liter plastic bottle.
2. Use only new bottles.
3. Keep the sample stored in amber bottles or out of the light.

Preservation: Add NaOH to pH 12 at 4°C.

Mercury

Sample volume - one pint.

1. Container: one pint narrow mouth plastic bottle.
2. Use only new bottles.
3. Rinse with 10 percent nitric acid
4. Rinse three times with distilled water.

Preservation: Add 40% NaOH to pH 12 at 4°C.

MS: 11-11

Phenols

Sample volume one pint.

1. Container: one pint narrow mouth bottles - glass only (amber).
2. Use new bottles only.

Preservation: Add H_3PO_4 to pH 4 and add 1.0 g of $CuSO_4$ per liter at 4°C.

VOA (Purgables)

1. Container: Samples are to be collected in 125 ml screw cap vials* or crimp top vials sealed with Teflon faced silicone septa*.
Vial - Pierce #13074 or equivalent
Septa* - Pierce #12722 or equivalent
2. Sample bottles (use only new bottles) should be thoroughly washed with detergent and rinsed with tap water followed by distilled water. The bottles and septa should be heated to 105°C for one hour and cooled to room temperature in an area known to be free of organic vapors. When cool, the bottles are sealed using the septum (Teflon side down) and screw cap. The bottles should remain sealed until just before filling with samples or blank solution.

Sample Method

Fill each container to overflowing. Carefully set the container on a level surface. Place the septum (Teflon side down) on the convex sample meniscus.

Seal the sample with the screw cap. To insure that the sample has been properly sealed, invert the sample and lightly tap the lid on a solid surface. The absence of entrapped air bubbles indicates a proper seal. If air bubbles are present, the sampler should open the bottle and add a few additional drops of sample, then reseal. The sample must remain hermetically sealed until it is analyzed.

Preservation: Cool sample to (4°C) and ship.

Sample Labeling - The sample bottle shall be labeled as to sample number, date and time of sampling, source, point of sampling, any preservative added.

Shipment

The samples and blanks should be carefully identified with water proof labels and ink. Each sample should be wrapped with shipping plastic then placed in a water proof insulated shipping carton containing sufficient ice or an equivalent to cool the sample to 4°C and maintain that temperature until arrival at EPA contract laboratory for GC/MS analysis.

APPENDIX II

ANALYTICAL METHODS USED FOR ANALYSES
OF DPMC CHEMICAL PLANT

WRC 105-77

0042

APPENDIX IIANALYTICAL METHODS USED FOR ANALYSES
OF DPMC CHEMICAL PLANT

Phenols	- Standard Methods, 14th Edition, 510 A Distillation Step for Methods B and C. 510 C Direct Photometric Method, pp. 576-581.
Cyanide	- Standard Methods, 14th Edition, 413 B Total Cyanide after Distillation. 413 C Titrimetric Method, pp. 367-370.
pH	- Standard Methods, 14th Edition, 424 pH Value, pp. 460-465.
Total Suspended Solids (TSS)	- Standard Methods, 14th Edition, 208 D Total Nonfilterable Residue Dried at 103-105°C, p. 94, or EPA STORET No. 00530.
Total Dissolved Solids (TDS)	- Standard Methods, 14th Edition, 208 A Total Residue Dried at 103-105°C, 208 D Total Nonfilterable Residue Dried at 103-105°C, pp. 91, 92, 94, or EPA STORET No. 70300.
Biochemical Oxygen Demand (BOD)	- Standard Methods, 14th Edition, 507 Oxygen Demand (Biochemical), pp. 543-550.
Chemical Oxygen Demand (COD)	- Standard Methods, 14th Edition, 508 Oxygen Demand (Chemical), Procedure 4,a), pp. 550-554.
Oil and Grease	- Standard Methods, 14th Edition, 502 A Partition - Gravimetric Method, pp. 515, 516.
Nitrogen (Ammonia)	- Standard Methods, 13th Edition, 132 B Nesslerization Method, pp. 226-231.
Sulfide	- Standard Methods, 13th Edition, 228 B Methylene Blue Visual Color-Matching Method, pp. 555-558.
Total Organic Carbon (TOC)	- A.I.M. D 7839-74.
Kjeldahl Nitrogen	EMS-10170.
Mercury	- Cold Vapor Technique, EPA STORET No. Total 71900.

- Metals (Total)** - Sample Preparation 4.1.3 (P.82-83) Manual of Methods for Chemical Analysis of Water and Wastes, EPA-625-/6-74-003a (1976 Edition) was used to prepare samples for antimony, beryllium, and zinc. The rest of metals were prepared as an industrial effluent as described in "Atomic Absorption Newsletter", 14, page 111 (1975). All the metals were done using the furnace technique except zinc which was done by the flame technique.
- Organic Chloride** - Method described in TIR WRC-120, "Characterization of Organic Chlorides in Chemical Plant Effluents".

APPENDIX III

BIOASSAYS FROM EDNA WOOD LABORATORIES, INC.

Fish sizes: Largest 2.25 g 60 mm
 Smallest 1.5 g 55 mm
 Average 1.9 g 58 mm
 EDNA WOOD LABORATORIES
 4820 Old Spanish Trail Houston, Texas 77021
 Bioassay Work Sheet
 Physiological Observations
 Shell Development
 Bioassay No. 4539-2

Sample Marked DPHC Effluent 10/8/76
 Received 10/11/76
 No 96 hr TLm but fish show some distress in 100% sample
 Sample Concentrations, % by volume
P. similis
 species fish used
 5 fish/10 liters

	10%		25%		40%		63%		100%	
	1 ₁	1 ₂	2 ₁	2 ₂	3 ₁	3 ₂	4 ₁	4 ₂	5 ₁	5 ₂
<u>11 Oct 76</u> <u>1445</u> START										
<u>12 Oct 76</u> <u>1445</u>	Norm	5/ Slug	5/ Slug							
<u>13 Oct 76</u> <u>1430</u>	Norm	4-5/Slug	5/ Slug							
<u>14 Oct 76</u> <u>1450</u>	Norm	1 Dead 4 Slug	Slug							
<u>15 Oct 76</u> <u>1400</u>	Norm	1 Dead 4 Slug	Slug							

EDNA WOOD LABORATORIES
4620 Old Spanish Trail Houston, Texas 77021

Page 2

Bioassay No. 4539-2

Bioassay Work Sheet
Physio-Chemical Observations

Date & Time	Test Tank	Polarograph		pH	Sp. Res. ohms	Temp. °C
		μ amps	O ₂ mg/l			
<u>12 Oct 1976</u> 1440	1 ₁	>10	>13.5	7.1	60	19
	1 ₂	/	/	7.15	59	/
	2 ₁	/	/	7.2	56	/
	2 ₂	/	/	/	/	/
	3 ₁	/	/	/	51	/
	3 ₂	/	/	7.25	52	/
	4 ₁	/	/	7.3	48	/
	4 ₂	/	/	/	/	/
	5 ₁	/	/	/	41	/
	5 ₂	/	/	/	/	/
	C	/	/	7.6	63	/
<u>13 Oct 1976</u> 1440	1 ₁	>10	>13.5	7.5	59	19
	1 ₂	/	/	/	60	/
	2 ₁	/	/	7.4	56	/
	2 ₂	/	/	/	/	/
	3 ₁	/	/	7.49	51	/
	3 ₂	/	/	7.5	/	/
	4 ₁	/	/	/	47	/
	4 ₂	/	/	/	/	/
	5 ₁	/	/	/	42	/
	5 ₂	/	/	/	41	/
	C	/	/	7.45	62	/

0 0 4 8

EDNA WOOD LABORATORIES
4820 Old Spanish Trail Houston, Texas 77021

Page 3

Bioassay No. 4539-2

Bioassay Work Sheet
Physio-Chemical Observations

Date & Time	Test Tank	Polarograph		pH	Sp. Res. ohms	Temp. °C
		μ amps	O ₂ mg/l			
14 Oct 1976 1500	1 ₁	>10	>13.5	7.55	58	20
	1 ₂	✓	✓	7.5	✓	✓
	2 ₁	✓	✓	7.4	55	✓
	2 ₂	✓	✓	✓	✓	✓
	3 ₁	✓	✓	✓	✓	✓
	3 ₂	✓	✓	7.49	52	✓
	4 ₁	✓	✓	7.4	47	✓
	4 ₂	✓	✓	✓	✓	✓
	5 ₁	✓	✓	✓	41	✓
	5 ₂	✓	✓	✓	✓	✓
	C	✓	✓	7.7	62	✓
15 Oct 1976 1430	1 ₁	10	13.5	7.6	58	20
	1 ₂	✓	✓	7.5	59	✓
	2 ₁	✓	✓	7.45	56	✓
	2 ₂	✓	✓	✓	✓	✓
	3 ₁	✓	✓	7.4	52	✓
	3 ₂	✓	✓	7.45	54	✓
	4 ₁	✓	✓	7.5	48	✓
	4 ₂	✓	✓	✓	✓	✓
	5 ₁	✓	✓	✓	42	✓
	5 ₂	✓	✓	7.45	✓	✓
	C	✓	✓	7.6	62	✓

EDNA LOOD LABORATORIES
 4820 Old Spanish Trail Houston, Texas 77021
 Shell Development
 Blossomy No. 4539-2

Sample Marked DPHC Effluent 10/3/76
 Received 10/11/76
 96 hr TLm: Not toxic

Gambusia affinis
 species fish used

5 fish/10 liters

All test fish were approximately
 1 inch long and weighed 0.7 g each.

Blossomy Work Sheet
 Physiological Observations

Sample Concentrations, % by volume

	10%		25%		40%		63%		100%	
	6 ₁	6 ₂	7 ₁	7 ₂	8 ₁	8 ₂	9 ₁	9 ₂	10 ₁	10 ₂
11 Oct 76 1443										
START										
12 Oct 76 1445	Norm	Norm								
13 Oct 76 1430	Norm	Norm								
14 Oct 76 1450	Norm	Norm								
15 Oct 76 1400	Norm	Norm								

EDNA WOOD LABORATORIES
4820 Old Spanish Trail Houston, Texas 77021

Page 2

Bioassay No. 4539-2

Bioassay Work Sheet
Physio-Chemical Observations

Date & Time	Test Tank	Polarograph		pH	Sp. Res. ohms	Temp. °C
		μ amps	O ₂ mg/l			
<u>12 Oct 1976</u> 1445	6 ₁	>10	>13.5	7.1	60	19
	6 ₂	✓	✓	7.35	61	✓
	7 ₁	✓	✓	7.55	56	✓
	7 ₂	✓	✓	✓	✓	✓
	8 ₁	✓	✓	7.5	52	✓
	8 ₂	✓	✓	7.6	✓	✓
	9 ₁	✓	✓	7.55	48	✓
	9 ₂	✓	✓	✓	47	✓
	10 ₁	✓	✓	7.5	42	✓
	10 ₂	✓	✓	7.6	✓	✓
	C	✓	✓	7.7	63	✓
<u>13 Oct 1976</u> 1445	6 ₁	>10	>13.5	7.6	60	19
	6 ₂	✓	✓	7.65	59	✓
	7 ₁	✓	✓	7.8	56	✓
	7 ₂	✓	✓	7.8	✓	✓
	8 ₁	✓	✓	✓	52	✓
	8 ₂	✓	✓	7.85	✓	✓
	9 ₁	✓	✓	7.8	47	✓
	9 ₂	✓	✓	7.75	✓	✓
	10 ₁	✓	✓	7.8	41	✓
	10 ₂	✓	✓	✓	✓	✓
	C	✓	✓	7.45	62	✓

EDNA WOOD LABORATORIES
4820 Old Spanish Trail Houston, Texas 77021

Page 3

Bioassay No. 4539-2

Bioassay Work Sheet
Physio-Chemical Observations

Date & Time	Test Tank	Polarograph		pH	Sp. Res. ohms	Temp. °C
		μ amps	O ₂ mg/l			
14 Oct 1976 1500	6 ₁	>10	>13.5	7.6	57	20
	6 ₂	✓	✓	✓	58	✓
	7 ₁	✓	✓	7.65	55	✓
	7 ₂	✓	✓	7.7	56	✓
	8 ₁	✓	✓	✓	52	✓
	8 ₂	✓	✓	7.75	✓	✓
	9 ₁	✓	✓	7.8	47	✓
	9 ₂	✓	✓	7.7	46	✓
	10 ₁	✓	✓	7.8	41	✓
	10 ₂	✓	✓	✓	✓	✓
	C	✓	✓	7.55	62	✓
15 Oct 1976 1410	6 ₁	>10	>13.5	7.4	60	20
	6 ₂	✓	✓	✓	✓	✓
	7 ₁	✓	✓	7.45	57	✓
	7 ₂	✓	✓	7.5	58	✓
	8 ₁	✓	✓	7.65	52	✓
	8 ₂	✓	✓	7.69	✓	✓
	9 ₁	✓	✓	7.7	48	✓
	9 ₂	✓	✓	✓	✓	✓
	10 ₁	✓	✓	7.75	42	✓
	10 ₂	✓	✓	7.65	✓	✓
	C	✓	✓	7.6	62	✓

APPENDIX IVPROSPECTIVE UNAMBIGUOUS COMPOUNDS TO BE ANALYZED FOR IN CONSENT DECREE INDUSTRIESCompound Name

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

Chlorinated benzenes (other than dichlorobenzenes)

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

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

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

Chloroalkyl ethers (chloromethyl, chloroethyl and mixed ethers)

17. *bis(chloromethyl) ether
18. *bis(2-chloroethyl) ether
19. 2-chloroethyl vinyl ether (mixed ethers)

Chlorinated naphthalene

20. 2-chloronaphthalene

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

21. 2,4,6-trichlorophenol
22. panachlorometa cresols
23. *chloroform (trichloromethane)
24. *2-chlorophenol

Dichlorobenzenes

- 25. *1,2-dichlorobenzene
- 26. *1,3-dichlorobenzene
- 27. *1,4-dichlorobenzene

Dichlorobenzidine

- 28. 3,3-dichlorobenzidine

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

- 29. *1,1-dichloroethylene
- 30. *1,2-trans-dichloroethylene
- 31. *2,4-dichlorophenol

Dichloropropane and dichloropropene

- 32. 1,2-dichloropropane
- 33. 1,3-dichloropropene (1,3-dichloropropene)
- 34. *2,4-dimethylphenol

Dinitrotoluene

- 35. 2,4-dinitrotoluene
- 36. 2,6-dinitrotoluene

- 37. *1,2-diphenylhydrazine
- 38. *ethylbenzene
- 39. *fluoranthene

Haloothers (other than those listed elsewhere)

- 40. *4-chlorophenyl phenyl ether
- 41. *4-bromophenyl phenyl ether
- 42. *bis-2-chloroisopropyl ether
- 43. *bis-2-chloroethoxy methane

Halomethanes (other than those listed elsewhere)

- 44. *methylene chloride (dichloromethane)
- 45. *methyl chloride (chloromethane)
- 46. *methyl bromide (bromomethane)
- 47. *bromoform (tribromomethane)
- 48. *dichlorobromomethane
- 49. *trichlorofluoromethane
- 50. *dichlorodifluoromethane
- 51. chlorodibromomethane
- 52. *hexachlorobutadiene
- 53. *hexachlorocyclopentadiene

- 54. *isophorone
- 55. *naphthalene
- 56. *nitrobenzene

Nitrophenols (including 2,4-dinitrophenol and dinitrocresol)

- 57. 2-nitrophenol
- 58. 4-nitrophenol
- 59. *2,4-dinitrophenol
- 60. 4,6-dinitro-o-cresol

Nitrosamines

- 61. N-nitrosodimethylamine
- 62. N-nitrosodiphenylamine
- 63. N-nitrosodi-n-propylamine

- 64. *pentachlorophenol
- 65. *phenol

Phthalate esters

- 66. bis(2-ethylhexyl) phthalate
- 67. butyl benzyl phthalate
- 68. di-n-butyl phthalate
- 69. diethyl phthalate
- 70. dimethyl phthalate

Polynuclear aromatic hydrocarbons

- 71. 1,2-benzanthracene
- 72. benzo (a)phrene (3,4-benzopyrene)
- 73. 3,4-benzofluoranthene
- 74. 1,12-benzofluoranthene
- 75. chrysene
- 76. acenaphthylene
- 77. anthracene
- 78. 1,12-benzoperylene
- 79. fluorene
- 80. phenanthrene
- 81. 1,2:5,6-dibenzanthracene
- 82. ideno (1,2,3-C, D) pyrene
- 83. pyrene
- 84. *2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD)
- 85. *tetrachloroethylene
- 86. *toluene
- 87. *trichloroethylene
- 88. *vinyl chloride (chloroethylene)

*Compounds specifically named in Appendix A of Consent Decree.

PROSPECTIVE PESTICIDES AND METABOLITES TO BE ANALYZED FOR IN CONSENT DECREE INDUSTRIES

Pesticides and Metabolites

- *aldrin
- *dieldrin
- *chlordane (technical mixture and metabolites)

DDT and metabolites

- *4,4'-DDT
- 4,4'-DDE (p,p'-DOX)
- 4,4'-DDD (p,p'-TDE)

Endosulfan and metabolites

- *a-endosulfan
- *B-endosulfan
- endosulfan sulfate

Endrin and metabolites

- *endrin
- endrin aldehyde
- endrin ketone

Heptachlor and metabolites

- *heptachlor
- heptachlor epoxide

Hexachlorocyclohexane (all isomers)

- *a-BHC
- *B-BHC
- *Y-BHC (lindane)
- *S-BHC

Polychlorinated biphenyls (PCB's)

- PCB-1242 (Arochlor 1242)
- PCB-1254 (Arochlor 1254)

- *toxaphene

- *asbestos (fibrous)
- *beryllium (total)
- *cadmium (total)
- *chromium (total)
- *copper (total)
- *cyanide (total)
- *lead (total)

*mercury (total)
*nickel (total)
*selenium (total)
*silver (total)
*thallium (total)
*zinc (total)

*Specific compounds and chemical classes as listed in the Consent Decree.