# ENVIRONMENTAL PROTECTION AGENCY OFFICE OF ENFORCEMENT EPA-330/2-77-013

EVALUATION OF CARBON TETRACHLORIDE DISCHARGES

AT

FMC CORPORATION, SOUTH CHARLESTON EAST PLANT

South Charleston, West Virginia

June 1977

National Enforcement Investigations Center
Denver, Colorado

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#### I. SUMMARY AND CONCLUSIONS

- 1. From 0630 hours, April 13 through 0630 hours April 21, 1977 wastewater discharges from Outfalls 018, 019, 022, the sulfur wash system and the carbon adsorption unit at the FMC South Charleston, W. Va. East Plant were monitored for the discharge of carbon tetrachloride. Outfalls 018, 019 and 022 discharge to the Kanawha River. The sulfur washwater and the water from the carbon adsorption unit are discharged through Outfall 018. Flow was measured and the carbon tetrachloride loads were calculated for each monitoring point. These discharge points were monitored to determine if FMC was in compliance with the March 15, 1977 consent order which limited the carbon tetrachloride discharge from Outfall 018 to 68 kg (150 lb)/day.
- 2. During the eight-day survey, the maximum amount of carbon tetrachloride discharged from Outfall Ol8 was 7.7 kg (17 lb)/day. This maximum discharge occurred during the sampling period O630 April 13 to O630 April 14. From April 14-19, the carbon tetrachloride discharge from Outfall Ol8 was less than 2.7 kg (6 lb)/day. On April 20, the carbon tetrachloride discharge was 6.4 kg (14 lb)/day.
- 3. The maximum amount of carbon tetrachloride discharged from Outfall 018 during this survey was in compliance with the consent order.
- 4. From April 13 through April 19, the carbon tetrachloride discharged from Outfall 019 was less than 0.45 kg (1.0 lb)/day. On April 20, the carbon tetrachloride was 5.4 kg (12 lb)/day. The increase in the carbon tetrachloride discharged from Outfall 019 was due to a spill outside the spill containment system.
- 5. The discharge of carbon tetrachloride from Outfall 019 is not in compliance with the consent order since such discharge is prohibited.

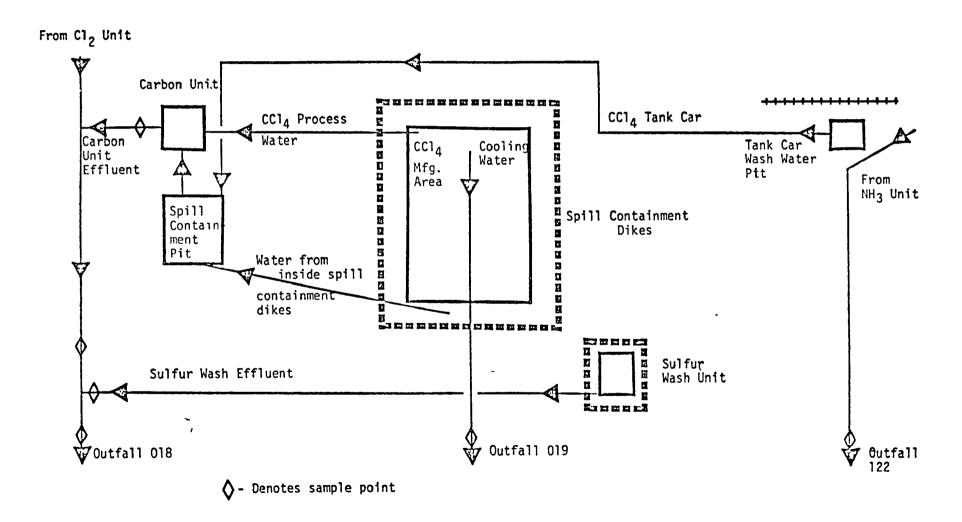
#### II. INTRODUCTION

The FMC East Plant, at South Charleston, West Virginia, manufactures carbon tetrachloride ( $CCl_4$ ) by reacting carbon disulfide with chlorine. Both the carbon disulfide and chlorine are produced in another portion of the East Plant. Waste water from the carbon tetrachloride manufacturing area is discharged from Outfalls 018, 019 and  $022^*$  to the Kanawha River [Figure 1].

In February 1977, excessive amounts of CCl4 were found in the Kanawha and Ohio Rivers and as a result, a temporary restraining order requiring FMC to cease manufacturing CCl4 was issued March 9, 1977 by the United States District Court for the Southern District of West Virginia. Between March 9 and March 15, 1977, the Environmental Protection Agency and FMC negotiated a consent order [Appendix A] that would allow FMC to resume manufacturing CCl4. This consent order was signed by both parties March 15, 1977 and set a discharge limit of 68 kg (150 lb)/day for the discharge of CCl4 from Outfall Ol8. No other CCl4 discharges are allowed from the plant. In addition, the carbon adsorption unit is to be designed to treat wastes, discharged from the decanter unit, to levels of less than 4.5 kg (10 lb)/day of CCl4.

On March 24, 1977, the Environmental Protection Agency, Region III and Headquarters, requested that the National Enforcement Investigations Center (NEIC) monitor CCl<sub>4</sub> discharges from Outfalls 018, 019 and 022 to determine whether FMC was in compliance with the consent order. On March 28, NEIC personnel visited the plant and FMC personnel stated that the plant was not operating because of problems encountered during attempted startups. During the period of March 28-April 15, NEIC personnel made numerous visits to the plant, but these problems

Figure 1 Sketch of Wastewater Discharges From FMC CCl<sub>4</sub> Area



continued until April 9, consequently, the NEIC survey did not begin until April 13, continuing until April 21. Outfalls 018, 019, 022, the discharge from the carbon adsorber and the sulfur wash discharge were monitored during the survey. The carbon adsorber and sulfur waste discharges are discharged through Outfall 018.

#### III. WASTEWATER DISCHARGE POINTS

The wastwater containing carbon tetrachloride that is discharged through Outfall 018 [Figure 1], originates from two areas within the carbon tetrachloride plant. The first stream originates in the CCl<sub>4</sub> purification section of the plant and consists of wash water. Currently, this wash water is treated to remove most of the CCl<sub>4</sub> by routing the wash water through two decanters in series and then through an activated carbon column before discharging it through Outfall 018. The flow rate of this stream ranges from 0.20 and 0.60 m<sup>3</sup> (50 and 150 gal)/ The second  $CCl_A$  containing source originates in the sulfur recovery portion of the plant and consists of wash water from washing the sulfur. This sulfur wash water, containing CCl<sub>4</sub>, is currently discharged to Outfall Ol8 without treatment. By January 1978, a treatment system will be installed to remove the CCl4 from sulfur wash water. The flow rate from the sulfur wash system ranges from 0.90 and 1.70  $\mathrm{m}^3$ (250 and 450 gal)/min. The total discharge rate from Outfall 018 is approximately 122  $m^3$  (32,000 gal)/min. The major portion [119  $m^3$ (31,500 gal)/min] of this waste water originates in the chlorine manufacturing area.

The discharge through Outfall 019 is once-through, non-contact cooling water, used in about 15 to 20 heat exchangers within the CCl<sub>4</sub> plant and this flow rate ranges from 26 and 32 m<sup>3</sup> (6,800 and 8000 gal)/min. There should be no CCl<sub>4</sub> in this, but since most heat exchangers leak, small amounts of CCl<sub>4</sub> are found. This stream is discharged without treatment. FMC has installed a monitoring system to detect CCl<sub>4</sub> at several locations (about 20) where the cooling water discharges to the collection header. Five of these monitoring locations feed samples to an ARCAS total hydrocarbon analyser that monitors the sample for total hydrocarbons. The results of this analysis are transmitted to the carbon tetrachloride control room and printed out on a strip chart.

If the total hydrocarbons for any of the five waste streams exceeds 4 ppm, the operator manually diverts the contaminated waste from Outfall 019 into a spill containment system. All water discharged into the spill containment system is treated by the activated carbon column and discharged through Outfall 018 into the Kanawha River. The hydrocarbon analyzer samples each of the five streams about every 5-6 minutes.

The other approximately 15 monitoring points are conductivity cells. These conductivity cells send a signal to the process control room and, when the conductivity of a stream changes by a certain amount (amount of conductivity change unknown), this stream is manually diverted from Outfall O19 to a spill containment system. Any water entering the spill containment system is subsequently treated by the activated carbon column and discharged via Outfall O18.

The majority of the wastewater discharged from Outfall 022 originates in the ammonia plant and should be free of  $CCl_4$ . Tank cars used for shipping  $CCl_4$  are washed in this area and this wash water containing  $CCl_4$  was formerly discharged via Outfall 022. FMC has modified this system so that all tank car wash water is now discharged through Outfall 018. Even though the tank car wash water is not currently being discharged to Outfall 022, small amounts (100 to 300 ppb) of  $CCl_4$  are present in this discharge.

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#### IV. MONITORING PROCEDURES

During the period April 13 through April 20, 1977, samples for CCl<sub>4</sub> analysis were collected and flows were measured at Outfalls 018, 019, 022, the sulfur wash effluent, and the carbon adsorption unit. Chain-of-custody procedures [Appendix B] were followed for the collection of all samples and field data and for laboratory analyses. Samples were collected by FMC personnel at the same time and locations as the NEIC samples were collected.

### FLOW DETERMINATIONS AND MONITORING LOCATIONS

# Outfall 018

The sampling location for Outfall 018 was at the company-installed collar-pipe tap just above the point at which the pipe became submerged in the Kanawha River. Flows were determined by the dilution technique using Lithium Chloride as a tracer [Appendix C]. Company personnel measured the flow using a potassium dichromate solution as the tracer for the dilution technique. Flows were also measured by NEIC (using the Lithium method) at a manhole approximately 15 m (50 ft) upstream of the 018 sample point. This flow measurement was used as a check on the flow from Outfall 018. The concentrated lithium chloride solution was injected into the waste stream approximately 90 m (300 ft) upstream of the 018 sampling site.

# Sulfur Wash Discharge

The sampling location for the sulfur wash area discharge was at a hole in the 20 cm (8 in) pipe about 1.5 m (5 ft) upstream of the entrance to the 018 conduit. Flow determinations were made using

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lithium chloride. The concentrated lithium chloride solution was injected into the waste stream near the sulfur wash area, about 90 m (300 ft) upstream of the sampling site.

# Carbon Adsorption Unit Discharge

The sampling location for the effluent from the carbon adsorption unit was a tap located in the discharge line just downstream from the unit flowmeter. Flows were measured using the company-installed turbo-flowmeter and digital totalizer. Both are direct read-out units. A calibration of these units was not possible; however, the flow determined by the flowmeter was in agreement with the totalizer flow.

# Outfall 019

The sampling location for Outfall 019 was located in a rectangular concrete channel near the bank of the river. Instantaneous flow determinations were made using the Dye-Dilution technique (April 13) and using a Marsh-McBirney velocity meter (April 14 to 20). [Appendix C]. The dye injection station was located at a 90° bend in the channel, approximately 20 m (65 ft) upstream of the 019 sample site. Velocity traverses with the Marsh-McBirney flowmeter were performed at the sample site.

# Outfall 022

Outfall 022 was sampled at the entrance to the company-installed 3-in. Parshall flume prior to where the wastewater is discharged to the river. Flows were measured using the Parshall flume and recorded continuously (April 13-17) by a Manning dipper flowmeter. Instantaneous head measurements at the Parshall flume were also recorded hourly and converted to flow (April 13-20).

# FLOW MEASUREMENT PROCEDURES

Beginning at 0730 on April 13, flow measurements were made every four hours at Outfalls 018 and 019, each flow corresponding to a CCl<sub>4</sub> composite sample (see following section). For example, the 0730 flow corresponds to composite No. 1 (0630-0930), the 1130 flow to No. 2 (1030-1330), etc. At Outfall 022 and the carbon adsorption unit, the hourly instantaneous flows were averaged in six groups of four to correspond to the six CCl<sub>4</sub> composite samples. Flows for the sulfur wash effluent were determined three times daily, i.e. once per shift, at 1130, 1930 and 0330, with each flow corresponding to two of the six CCl<sub>4</sub> composites (1130 to No. 1, and No. 2, etc.).

## SAMPLE COLLECTION PROCEDURE

Beginning at 0630 on April 13, individual grab samples were collected hourly (on the half-hour to coincide with company procedure) from each of the five monitoring locations. All samples were stored in ice and shipped to the NEIC laboratory in Denver, Colorado at the end of each 24-hour period. The CCl<sub>4</sub> samples were composited on an equal volume basis to correspond to the flow measurement frequency for Outfalls 018 and 019 (i.e. every four hours). For example, the hourly samples collected between 0630 and 0930 were combined equally into the No. 1 composite sample, those collected between 1030 and 1330 into No. 2, etc., yielding six composites per location per day. All samples were analyzed in accordance with EPA approved analytical procedures [Appendix D] which are in accordance with the methods of analysis specified in the consent order [Appendix A]. Samples were not collected at the carbon adsorption unit while the unit was down, 1230 April 20 to 0330 April 21.

The loads [kg(lb)/day] of carbon tetrachloride discharged from each sample point were calculated using the average flow rate for each four-hour period and the carbon tetrachloride analysis from the corresponding four-hour composite. These six calculated loads were summed to determine the total load for each 24-hour period.

#### V. MONITORING RESULTS

Monitoring results are summarized by sampling locations [Table 1] and discussed below. Complete physical and chemical data for each sample location are found in Appendix E.

# Outfall 018

The March 15 consent order [Appendix A] specifies that the carbon tetrachloride discharge from Outfall Ol8 be less than 68 kg (150 lb)/day. During the eight day survey, April 13-20, 1977, the maximum amount of carbon tetrachloride discharged from this Outfall was 7.7 kg (17 lb)/day April 13. From April 14-19, the discharge of carbon tetrachloride was less than 2.7 kg (6 lb)/day. On the final day of the survey, April 20, the carbon tetrachloride discharge was 6.4 kg (14 lb)/day.

The maximum carbon tetrachloride discharge rate of 7.7 kg (17 lb)/day is in compliance with the March 15 consent order.

# Outfall 019

The March 15 consent order specified that carbon tetrachloride shall not be discharged from any outfall other than 018. During the eight days of the NEIC survey, carbon tetrachloride was discharged daily from Outfall 019. From April 13 through April 19, the carbon tetrachloride in the wastewater discharge was 0.45 kg (1.0 lb)/day or less [Table 1]; however, on April 20 the carbon tetrachloride discharged from Outfall 019 was 5.4 kg (12 lb)/day. This increase in the carbon tetrachloride discharge was due to a spill that was discharged via Outfall 019. The March 15 consent order does not allow

Table 1

SUMMARY OF MONITORING RESULTS
FMC CORPORATION
April 13-21, 1977

DATE STATION	#/13 m³/day kg/day x 1000 (mgd) (1b/day)	x 1000	4/15 m <sup>3</sup> /day kg/day x 1000 (mgd) (1b/day)	4/16 m <sup>3</sup> /day kq/day x 1000 (mqd) (1b/day)	X 1000	x 1000	# 4/19 m <sup>3</sup> /day kg/day x 1000 (mqd) (1b/day)	x 1000
018	146 7.9 (38.6) (17.0)	160 2.0 (42.3) (4.4)	171 2.5 (45.1) (5.5)	117 1.3 (30.9) (2.8)	152 0.83 (40 1) (1.8)	170 0.90 (45.0) (2.0)	172 2.5 (45.4) (5.5)	170 6 4 (44 8) (14.0)
918 Unstream	(flow only)	174 (45.9)	170 (45 0)	144 (38.0)	151 (40.0)	161 (42.4)	164 (43.4)	159 (42.0)
Carbon** Adsorption Unit	0 27 <0.81* (0.971) <(1.8)		0.31 <0.92* (0.081) <(2.0)	0.30 <0.90* (0.079) <(2.0)	0.28 <0.84* (0.074) <(1.9)	0.27 <0.82* (0 072) <(1.8)	0 27 <0.82* (0 072) <(1.8)	ე.91 ი.89 (ე.724) (2.0)
Sulfur Wash		1.2 2.6 (0.32) (5.8)	1 1 2 5 (0 29) (5 6)	1 2 2 0 (0.31) (4.5)	1.2 1.2 (0.31) (2.5)	1.2 1.1 (0.32) (2.5)	1 3 2.1 (0 34) (4.6)	1.2 2.0 (0.31) (4.4)
019	37.2 <0.30* (9.8) <(0.66)		41.1 <0.15* (10.9) <(0.33)	39 3 <0 28* (10.4) <(0 62)	40 2 <0.21* (10.6) <(0.46)	38.7 0.25 (10.2) (0.55)	3º.2 0.47 (10.4) (1.0)	43.1 <5.6* (11.4) <(12.0)
022	1 0 0.22 (0.26) (0.49)	1 0 0.19 (0 26) (0.42)	2.7 0.28 (0.68) (0.62)	2.4 0 17 (0 62) (0 37)	1.9 0.21 (0.49) (0.46)	1 8 0 44 (0.47) (0 32)		

<sup>\*</sup>Indicates that one or more of 6 daily 4-hour composites was less than detectable limit of 3 ppb; thus, load is shown as "less than." \*\*Loads for carbon adsorption are in grams/day and 10<sup>-3</sup> lb/day

any carbon tetrachloride to be discharged from Outfall Ol9, FMC was not in compliance with the consent order. In addition, the spill on April 20 was not in compliance with the consent order.

# Outfall 022

The carbon tetrachloride discharge from Outfall 022 ranged from 0.10 to 0.28 kg (0.22 to 0.62 lb)/day [Table 1] during the survey. The March 15 consent order does not allow any carbon tetrachloride to be discharged from Outfall 022; FMC was not in compliance with the consent order.

## OTHER MONITORING POINTS

The discharge from the sulfur wash system was monitored during this survey as a partial check on the 018 discharge. The sulfur wash wastewater contains the majority of the carbon tetrachloride that is discharged through Outfall 018. The maximum amount of carbon tetrachloride discharged via the sulfur wash system was 2.6 kg (5.8 lb)/day [Table 1]. The amount of carbon tetrachloride in the sulfur wash stream was approximately equal to the carbon tetrachloride in Outfall 018 except on April 13 and April 20. The difference in the loads from the sulfur wash stream and Outfall 018 for April 20 is explained by the carbon adsorption unit not being in operation on this date, but the large difference detected on April 13 cannot be explained.

The average carbon tetrachloride discharge from the carbon adsorber was very low, less than 0.0009 kg (0.002 lb)/day, [Table 1] during the survey. The maximum carbon tetrachloride concentration in this wastewater stream was 19 ppb with a flow rate of 0.09 m<sup>3</sup> (25 gal)/min. This is a maximum load of 0.003 kg (0.006 lb)/day.

The March 15 consent order requires that the carbon column effluent contain less than 4.5 kg (10 lb)/day carbon tetrachloride; therefore, the discharge from the carbon adsorber was in compliance with the consent order.

# SPILL CONTAINMENT SYSTEM

The consent order required that a spill containment system be installed around the carbon tetrachloride process area before the process was put into operation and this system was completed prior to any attempted start-ups. The spill containment system was in compliance with the consent order.

APPENDIX A
March 15 Consent Order

V

# UNITED STATES DISTRICT COUPT FOR THE SOUTHERN DISTRICT OF WEST VINGINIA

#### RUNTINGTON

UNITED STATES OF AMERICA,

Plaintiff,

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CIVIL ACTION NO.

PIK COPPORATION,

Defendant.

#### COMPUNT OPDER

The complaint having been filed on Harch 9, 1977, and with plaintiff and defendant by their respective attorneys having consented without trial or adjudication of any issue of fact or law herein to the entry of this Order, and without this Order constituting any evidence or admission by any party hereto with respect to any issue of fact or law herein:

NOW, THEREFORE, before the taking of any testimony, upon the pleadings, and without adjudication of any issue of fact or law herein, and upon the consent of the parties hereto, it is CROLRED, ADJUDGED and DECREED as follows:

I

This court has jurisdiction of the subject ratter and of the parties consenting hereto.

( ...

The provisions of this Order shall apply to and be birding upon the defendant, its officials, directors, agents, servants, enployers, successors and assigns and all persons, firms and corporations acting under, through or for it and upon those persons, firms and corporations in active concert or participation with it.

III

The defendant (hercafter "FHC") may commence and continue the operation of its smiff chlorine reaction system at its South Charleston, West Virginia plant (the "Plant") when and so long as it has:

- A. Erected a containment system sufficient to contain approximately 21,000 gallons, around the smitf chlorine reaction system and plugged all sewers, conduits, pipes or other conveyances from within the contained area in a manner designed to prevent any spills, leaks or other types of discharges from the smiff chlorine system from reaching the Kanawha River or other waters of the United States;
- B. Provided twenty-four (24) hour visual surveillance to detect and eliminate the presence of any spills, leaks or any other type of discharge of carbon tetrachloride into the contained area; and

C. Made adequate provision, including but not limited to twenty-four (24) hour visual observation, for the storage, transport, and disposal of the first stage carbon tetrachloride product mix produced thereby in a manner designed to prevent any spill, leak or other discharge into the Eanawha River or other waters of the United States and so stores, transports, and disposes of such product mix. Transfer of such product mix to temporary storage shall only occur during daylight hours and shall be in a "batch" mode and under continuous visual surveillance.

IV

PMC may commence and continue the manufacture of 'carbon tetrachloride at the Plant when and so long as it has:

- A. Installed treatment facilities on the discharge line from the decenter unit designed to treat wastes discharged therefrom to levels less than 10 pounds of carbon tetrachloride on a single day, provided however, that:
- 1. The treatment facilities shall consist of a single stage activated carbon column system with the apparts-nances required for treatment of all process waste waters discharged from the decenter unit;
- 2. The treatment facilities shall be operated at all times to minimize the discharge of carbon tetrachloride.

discharge from the treatment facilities by taking flow proportioned composited samples at least once every four hours, shall average the amounts of carbon tetrachloride in all composited samples taken during each day, shall split samples with EPA on request, shall analyze the samples using the analytical procedure attached hereto as Exhibit A, shall submit the results of such analysis to EPA weekly, and shall allow EPA or its contractor to monitor carbon tetrachloride in the discharge from the treatment facilities.

- 4. Whenever, after the completion of the second charge of carbon to the treatment facilities, the treatment facilities have been operated for a period of ten consecutive days such the total discharge during any one day of that period does not exceed the average of the daily discharges of all of the ten days during that period by more than 100%, an effluent limitation is thereby established equal to twice the average of the daily discharges on all of the ten days during that period.

  FMC shall thereafter not discharge wastes from the decanter unit containing carbon tetrachloride in excess, on any day, of the effluent limitation so established, and
- 5. FMC may, at any time, replace the treatment facilities with alternative treatment facilities which it has demonstrated to MPA will result in a discharge of carbon

tetrachloride equal to or less than the amounts established in paragraph IV-A-4 herein for the discharge of carbon tetrachloride from the decenter unit treated waste water stream and the discharge limitations established in paragraph IV-A-4 herein shall be modified accordingly to a lesser number.

B. Erected a containment system around the entire carbon tetrachloride production facility at the Plant and plugged all sewers, conduits, pipes or other conveyances from within the contained area, except into outfall Ols, in a manner designed to prevent any spills, leaks, or other types of discharges from the carbon tetrachloride production facility from reaching the Kanawha River or other waters of the United States.

V

- A. FMC shall operate its treatment facilities at all times to minimize the discharge of carbon tetrachloride and in no event shall it discharge carbon tetrachloride from cutfall 013 at the Plant to the Kanawha River in excess of 150 pounds on any single day (the "total discharge allowed"); provided, however, that:
- 1. On or before January 1, 1978, TMC shall install a dual stage carbon treatment system on the discharge line from the sulfur wash unit designed to treat wastes discharged therefrom to levels less than 15 pounds per day.

1. A. A.

- 2. FMC shall monitor carbon tetrachloride in the discharge from the treatment facilities by taking flow proportioned composited samples at least once every four hours, shall average the amounts of carbon tetrachloride in all composited samples taken during each day, shall split samples with EPA on request, shall smalyze the samples using the analytical procedure attached hereto as Exhibit A, shall submit the results of such analysis to EPA weekly, and shall allow EPA or its contractor to monitor carbon tetrachloride in the discharge from the treatment facilities.
- installation of the treatment facilities on the discharge line from the sulfur wash unit, the treatment facilities have been operated for a period of ten consecutive days such that the total discharge during any one day of that period does not exceed the average of the daily discharges of all of the ten days during that period by more than 100%, an effluent limitation is thereby established equal to twice the average of the daily discharges on all of the ten days during that period. FMC shall thereafter not discharge waste waters from the sulfur wash unit containing carbon tetrachloride in excess, on any day, of the effluent limitation so established.
- 4. Whenever an effluent limitation is established under paragraph V h-3 herein, an effluent limitation shall be '7' y established on outfall 018 at the Plant equal to the sum of

the effluent limitation on the discharge from the decenter unit established under paragraph IV A-4 herein and the effluent limitation on the discharge from the sulfur wash unit established under paragraph V A-3 herein. FMC shall thereafter not discharge carbon tetrachloride from the Flant in excess, on any day, of that effluent limitation.

- 5. In the event that the flow of the Kanawka River at the Charleston gauge is less than 2890 cubic feet per second ("cfs") prior to the time that the treatment facilities required by paragraph V herein are installed and operating, FMC shall cease discharging carbon tetrachloride from the Plant until such time as the flow of the Kanwaha River exceeds 2890 cfs at the Charleston gauge.
- B. FMC shall discharge all process waste waters containing carbon tetrachloride from the Plant through outfall 016. There shall be no spill, leak, or any other type of discharge of carbon tetrachloride from the Plant other than as specified in this paragraph V. In the event that any discharge other than as set forth herein shall occur, FMC shall not be relieved from liability under this Order, unless and until FMC demonstrates by clear and convincing proof to the satisfaction of this Court that circumstances beyond its control, including employee negligence, which could not be prevented or foreseen, caused such discharge.

C. Notwithstanding any other provision of this Order, in the event that carbon tetrachloride is spilled, leaked or otherwise discharged to the Kanawha kiver from the Plant other than as specified in this paragraph V, or appears likely to be discharged, FMC shall immediately take all steps necessary to minimize or, if possible, eliminate the quantity so spilled, leaked or otherwise discharged. In the event of such a spill, leak or other discharge, FMC shall immediately notify LPA and shall relate to it the circumstances leading to the event and the remadial measures taken. Compliance with the provisions of this Order shall not relieve FMC from legal liability for any spill, leak or other discharge.

VI

At commencement of the manufacture of carbon tetrachloride at the Plant, FMC shall commence continuous monitoring of carbon tetrachloride (1) in the waste discharge line from the treatment system following the decanter unit; (2) in the waste water discharge line from the sulfur wash system (after treatment, once treatment is installed); and (3) in the water discharged from outfall 018 to the Kanawha River and shall report the results of same to EPA once a week until April 15, 1977, and once a month thereafter. Continuous means sampling at least once every hour. Reports shall be in terms of pounds of carbon tetrachloride discharged per day.

PMC shall, within 14 days of the effective date of this Order, rotain the services of an outside engineering firm utilizing registered professional engineers who are competent to perform a detailed survey of the carbon tetrachloride unit at the Plant. This firm will evaluate the physical integrity of all tamks, vessels, pumps, piping and related equipment containing carbon tetrachloride in the carbon tetrachloride unit. The contractor will further review the concept, design and installation of permanent facilities for the prevention, containment and control of accidental carbon tetrachloride discharges. Upon completion of this study the contractor will submit a written report recommending corrections of any conditions which could result in a discharge of carbon tetrachloride to the river. FMC will take action on such recommendations to correct the deficiency. Findings of the contractor and FMC's progress on necessary remedial action will be transmitted to UPA.

#### VIII

FMC will provide representatives of the Environmental Protection Agency access at all times to the Plant in accordance with the following procedure:

A. The representatives shall call the rain Plant telephone number. They shall identify themselves and during the hours of 8:00 a.m. to 5:00 p.m.

shall ask for the resident plant manager or his designee. During the hours of 5:00 p.m. to 8:00 a.m. they shall only identify themselves to the operator. After identifying themselves they shall announce the area of the Plant to which they desire access. Access to the carbon tetrachloride area shall be accorded within thirty minutes from the time the area to be inspected is designated. Access to other areas of the Plant shall be accorded within sixty minutes from the time the area is designated.

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B. FMC Corporation reserves the right to deny access
to such representatives in the event that circumstances
would endanger the health or lives of such representatives. In such event, FMC Corporation shall advise
the representatives in detail of such circumstances.
Immediately after the removal or passage of such
circumstances, access shall be granted to such
representatives.

IX

This Order is not and shall not be interpreted to be a permit under section 402 of the Federal Water Pollution Control Act (33 U.S.C. Sac. 1342), nor shall it in any way relieve the defendant of any obligation imposed by the Federal Water Pollution Control Act or in any permit issued

thereunder, nor shall it in any way relieve the defendant of its obligation to comply with any other local, state or federal law in any way related to the subject of this Order.

X

Jurisdiction is retained by the Court for the purpose of enabling either party to this Order to apply to the Court at any time for such further Orders or directions as may be appropriate for the construction or carrying out of this Order or for the modification of the terms of any of the provisions hereof or for the enforcement or dissolution hereof.

We hereby consent to the entry of this COMSENT ORDER without further notice:

UNITED STATES OF AFTIRICA

DATE: 3/15/)

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Assistant United Tatas Attorney

7MC Corporation

DATE: 3/15/77

Judgment entered in accordance with the foregoing Consent

Order, this \_\_\_ day of March, 1977.

CHARLES W. HADEN, II

United States District Judgo

# Industrial Chemical Division Technical Department Control Laboratory South Charleston

SC11-21 (3-77) -1-

SC Technical Princeton Central Research-

Analytical Method SC11-21(3-77)
Waste Water
Method for CCL4 (Gas Chromatograph)

#### Based On:

This procedure is based on work done at ICRD, Princeton, New Jersey and at South Charleston, West Virginia.

Carbon tetrachloride in water is measured by direct injection of the aqueous sample into a chromatograph equipped with an electron capture detector. Familiarity with the instrument and detector is assumed.

#### Calibration:

Prepare four solutions containing

pg/g CCL4 in methanol. Caution: All glassware must be cleaned with soap and water, rinsed with pure water, and baked in an over at 400°C for 2 hours. Store in a clean desiccator until ready to use. Prepare a series of about ten hypo bottles (50 ml) equipped with teflon lined rubber seals and aluminum crimp caps or with miniert valves. Fill the bottle completely full (no air bubbles), seal and weigh.

Inject microliter quantities from the strong standards and mix well. Glass beads or a teflon stir bar facilitate the mixing. These aqueous standards should be prepared to cover a range of 1 ppb to 1 ppm.

Inject 5 µl from each standard into the chromatograph and calculate a response factor from the average. This factor is programmed into the calculator-integrator in accordance with the manufacturers instructions.

## Procedure:

Inject 5 µl of samples and read the ppb CCL4 from the integrator print out.

LA Suttle

Attachment

"EXHIBIT A"

1:

Attachment No. 1

#### Instrumental Conditions

Hewlett-Packard (Model 5738) Gas-Liquid Chromatograph with ECD Detector - Nickel63 15 m Ci.

Column, 8 feet glass 6 mm ID packed with Tenax 60/80 mesh.

Carrier Gas - 5% methane in argon 60 PSI 60 mls/min flow.

Injector - on column 250°C.

Column - 165°C.

Detector - 300°C vented to hood with exhaust fan.

Integrator-recorder Hewlett Packard #3380A programmed per
instruction manual.

HA Martin

APPENDIX B
Chain of Custody Procedures

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# ENVIRONMENTAL PROTECTION AGENCY NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

# CHAIN OF CUSTODY PROCEDURES June 1, 1975

#### **GENERAL**

The evidence gathering portion of a survey should be characterized by the minimum number of samples required to give a fair representation of the effluent or water body from which taken. To the extent possible, the quantity of samples and sample locations will be determined prior to the survey.

Chain of Custody procedures must be followed to maintain the documentation necessary to trace sample possession from the time taken until the evidence is introduced into court. A sample is in your "custody" if:

- 1. It is in your actual physical possession, or
- 2. It is in your view, after being in your physical possession, or
- It was in your physical possession and then you locked it up in a manner so that no one could tamper with it.

All survey participants will receive a copy of the survey study plan and will be knowledgeable of its contents prior to the survey. A pre-survey briefing will be held to re-appraise all participants of the survey objectives, sample locations and Chain of Custody procedures. After all Chain of Custody samples are collected, a de-briefing will be held in the field to determine adherence to Chain of Custody procedures and whether additional evidence type samples are required.

#### SAMPLE COLLECTION

- 1. To the maximum extent achievable, as few people as possible should handle the sample.
- Stream and effluent samples shall be obtained, using standard field sampling techniques.
- 3. Sample tags (Exhibit I) shall be securely attached to the sample container at the time the complete sample is collected and shall contain, at a minimum, the following information: station number, station location, data taken, time taken, type of sample, sequence number (first sample of the day sequence No. 1, second sample sequence No. 2, etc.), analyses required and samplers. The tags must be legibly filled out in ballpoint (waterproof ink).
- 4. Blank samples shall also be taken with preservatives which will be analyzed by the laboratory to exclude the possibility of container or preservative contamination.
- 5. A pre-printed, bound Field Data Record logbook shall be maintained to record field measurements and other pertinent information necessary to refresh the sampler's memory in the event he later takes the stand to testify regarding his actions during the evidence gathering activity. A separate set of field notebooks shall be maintained for each survey and stored in a safe place where they could be protected and accounted for at all times. Standard formats (Exhibits II and III) have been established to minimize field entries and include the date, time, survey, type of samples taken, volume of each sample, type of analysis, sample numbers, preservatives, sample location and field measurements such as temperature, conductivity,

DO, pH, flow and any other pertinent information or observations. The entries shall be signed by the field sampler. The preparation and conservation of the field logbooks during the survey will be the responsibility of the survey coordinator. Once the survey is complete, field logs will be retained by the survey coordinator, or his designated representative, as a part of the permanent record.

- 6. The field sampler is responsible for the care and custody of the samples collected until properly dispatched to the receiving laboratory or turned over to an assigned custodian. He must assure that each container is in his physical possession or in his view at all times, or locked in such a place and manner that no one can tamper with it.
- 7. Colored slides or photographs should be taken which would visually show the outfall sample location and any water pollution to substantiate any conclusions of the investigation. Written documentation on the back of the photo should include the signature of the photographer, time, date and site location. Photographs of this nature, which may be used as evidence, shall be handled recognizing Chain of Custody procedures to prevent alteration.

#### TRANSFER OF CUSTODY AND SHIPMENT

- 1. Samples will be accompanied by a Chain of Custody Record which includes the name of the survey, samplers' signatures, station number, station location, date, time, type of sample, sequence number, number of containers and analyses required (Fig. IV). When turning over the possession of samples, the transferor and transferee will sign, date and time the sheet. This record sheet allows transfer of custody of a group of samples in the field, to the mobile laboratory or when samples are dispatched to the NEIC Denver laboratory. When transferring a portion of the samples identified on the sheet to the field mobile laboratory, the individual samples must be noted in the column with the signature of the person relinquishing the samples. The field laboratory person receiving the samples will acknowledge receipt by signing in the appropriate column.
- 2. The field custodian or field sampler, if a custodian has not been assigned, will have the responsibility of properly packaging and dispatching samples to the proper laboratory for analysis. The "Dispatch" portion of the "Chain of Custody Record shall be properly filled out, dated, and signed.
- 3. Samples will be properly packed in shipment containers such as ice chests, to avoid breakage. The shipping containers will be padlocked for shipment to the receiving laboratory.
- 4. All packages will be accompanied by the Chain of Custody Record showing identification of the contents. The original will accompany the shipment, and a copy will be retained by the survey coordinator.
- 5. If sent by mail, register the package with return receipt requested. If sent by common carrier, a Government Bill of Lading should be obtained. Receipts from post offices, and bills of lading will be retained as part of the permanent Chain of Custody documentation.
- 6. If samples are delivered to the laboratory when appropriate personnel are not there to receive them, the samples must be locked in a designated area within the laboratory in a manner so that no one can tamper with them. The same person must then return to the laboratory and unlock the samples and deliver custody to the appropriate custodian.

#### LABORATORY CUSTODY PROCEDURES

- 1. The laboratory shall designate a "sample custodian." An alternate will be designated in his absence. In addition, the laboratory shall set aside a "sample storage security area." This should be a clean, dry, isolated room which can be securely locked from the outside.
- 2. All samples should be handled by the minimum possible number of persons.
- 3. All incoming samples shall be received only by the custodian, who will indicate receipt by signing the Chain of Custody Sheet accompanying the samples and retaining the sheet as permanent records. Couriers picking up samples at the airport, post office, etc. shall sign jointly with the laboratory custodian.
- 4. Immediately upon receipt, the custodian will place the sample in the sample room, which will be locked at all times except when samples are removed or replaced by the custodian. To the maximum extent possible, only the custodian should be permitted in the sample room.
- 5. The custodian shall ensure that heat-sensitive or light-sensitive samples, or other sample materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained.
- 6. Only the custodian will distribute samples to personnel who are to perform tests.
- 7. The analyst will record in his laboratory notebook or analytical worksheet, identifying information describing the sample, the procedures performed and the results of the testing. The notes shall be dated and indicate who performed the tests. The notes shall be retained as a permanent record in the laboratory and should note any abnormalties which occurred during the testing procedure. In the event that the person who performed the tests is not available as a witness at time of trial, the government may be able to introduce the notes in evidence under the Federal Business Records Act.
- 8. Standard methods of laboratory analyses shall be used as described in the "Guidelines Establishing Test Procedures for Analysis of Pollutants," 38 F.R. 28758, October 16, 1973. If laboratory personnel deviate from standard procedures, they should be prepared to justify their decision during cross-examination.
- 9. Laboratory personnel are responsible for the care and custody of the sample once it is handed over to them and should be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the tests were run.
- 10. Once the sample testing is completed, the unused portion of the sample together with all identifying tags and laboratory records, should be-returned to the custodian. The returned tagged sample will be retained in the sample room until it is required for trial. Strip charts and other documentation of work will also be turned over to the custodian.
- 11. Samples, tags and laboratory records of tests may be destroyed only upon the order of the laboratory director, who will first confer with the Chief, Enforcement Specialist Office, to make certain that the information is no longer required or the samples have deteriorated.

ا دور	Station No.	Date	Time	Sequence No.
* GPO WARM	Station Location	on .		Grab
	BOD Solids COD Nutrients	MetalsOil and GreaseD.OBactOther	Roma	arks/Preservative: ,
Sa	mplers.			

ENVIRONMENTAL PROTECTION AGENCY

OFFICE OF ENFORCEMENT

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

BUILDING 53, BOX 25227, DENVER FEDERAL CENTER

DENVER, COLORADO 80225

Back

PHENOL

REMARKS

EXHIBIT II

E IT

	Samplors:
FIELD DATA RECORD	<del></del>
	<del></del>

STATION	NUMBER	DATE	TIME	TEMPERATURE °C	CONDUCTIVITY	pH S.U.	D.O. mg/l	Gage H1. or Flow F1. or CFS
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### **EXHIBIT IV**

## ENVIRONMENTAL PROTECTION AGENCY Office Of Enforcement

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

### CHAIN OF CUSTODY RECORD

SURVEY				SAN	IPLER:	S: (Sign	alure)		
STATION NUMBER	STATION LOCATION	DATE	TIME	<u> </u>	MPLE TY Ter Grob	PE Aír	SEQ NO	NO OF	ANALYSIS REQUIRED
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Method of S	Shipment:			l					

Distribution: Orig. - Accompany Shipment

1 Copy - Survey Coordinator Field Files

# APPENDIX C Flow Measurement Techniques

### FLOW MEASUREMENT USING LITHIUM CHLORIDE

Flow determinations at two monitoring locations (Outfall O18 and the sulfur wash effluent) were made using the lithium chloride tracer method. In this technique a solution of lithium chloride of known concentration is injected at a constant rate an adequate distance upstream of the sampling point to insure complete mixing. Samples are then collected at the monitoring location to determine the lithium concentration in the waste stream. The flow is then calculated from the known injection rate, the concentration of the injected lithium and the lithium concentration after mixing in the wastewater.

Samples were analyzed for lithium at the NEIC laboratory in Denver on a Perkin-Elmer Model 403 Atomic Absorption spectrophotometer. Calibration of the instrument was performed daily using lithium standards of known concentration. Frequent checks were made during the day to insure accuracy.

A preliminary study of the two monitoring locations whose flows were measured by lithium chloride tracer was conducted prior to the start of the survey. In this study, samples from these two sites were analyzed for background levels of lithium to determine the concentration necessary for the injected lithium and the appropriate injection rate. During the initial set-up period in the field, and after one week of sampling, these studies were repeated with similar results. The preliminary field study also tested each outfall to determine the injection time required for development of the concentration plateau. A factor of safety was then included in the collection time at each station to insure that sampling occurred after the concentration plateau had been reached. Background samples were taken with each flow sample prior to injection of the lithium and the concentration of lithium in each flow sample was corrected for the background concentration. Also, a sample of the injected lithium solution was taken with each

flow sample and analyzed to obtain the concentration. Other quality control procedures included: 1) measurement of pump injection rates at least 4 times per day, 2) a check to determine the length of time required for the lithium concentration to return to background concentrations after the injection pumps were shut off.

### DYE DILUTION TECHNIQUE FOR FLOW MEASUREMENT

Flow determinations were made at Outfall 019 (April 13-14) using the dye dilution technique with fluorometric detection. In this procedure, a fluorescent dye is introduced into the flow stream, and the concentration of the dye is monitored downstream at the sampling point. The dye injection station is located far enough upstream to insure complete mixing at the sampling site. The dye is premixed gravimetrically to a known concentration and is injected at a known and constant rate. The flow at the sampling point is determined from the injection rate, the known concentration of the dye, and the measured concentration of the dye in the flow stream.

The dye used in this technique is Rhodamine WT, which exhibits high sensitivity, a low sorptive tendency, and stability under varying pH conditions. The fluorescence assay equipment consists of the G.K. Turner Model III fluorometer, with a far UV lamp, a high sensitivity kit, a standard door and a matched set of cuvettes. The fluorometer is calibrated once per day by measuring the fluorescence of gravimetrically prepared standards of known concentrations.

A preliminary study of the Outfall was conducted during the initial set-up period of the survey. Samples were taken and analyzed for background fluorescence. A background sample was also taken with each flow sample, and the fluorescence of the flow sample was corrected for the fluorescence of the background sample. Quenching and sorption studies were also conducted, and results indicated no significant loss

of dye through chemical change or physical adsorption. When low pH values quenched the fluorescence of a sample, the sample was neutralized to pH 7 (with a powdered buffer to insure no concentration change) to recover the fluorescence, and then reanalyzed.

Other quality control measures included: 1) measurement of dye injection rate at each station at least 4 times per day; 2) a mixing check at the sampling site to insure a complete mix; 3) use of polyethylene gloves when handling concentrated dye to reduce the possibility of contamination; 4) frequent fluorometer reference to zero using a dummy cuvette; and 5) triple-rinsing of cuvettes with distilled water before each use.

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### FLOW MEASUREMENT USING THE MARSH-McBIRNEY ELECTRO-MAGNETIC FLOW METER

The flow meter is provided with a Faraday type electro magnetic flow sensor which has two perpendicular axes so that both X and Y directional vectors can be measured. The operator can obtain from the measurement both the direction and magnitude of the water velocity. All sensors are cylindrically shaped and have no moving parts which are susceptible to clogging or fouling.

The sensor consists of a cylinder containing an electro-magnet internally and two pairs of external electrodes in contact with the water. Flow around the cylinder probe intersects magnetic flux lines causing voltages to be generated which are linearly proportional to the water velocity. The voltages are detected by the electrodes, processed at the signal conditioner and presented as analog voltages linearly proportional to the X and Y components of the velocity vector.

The accuracy of an electro-magnetic sensor includes linearity, zero drift, and absolute calibration. Linearity allows direct reading without a correction chart and accurate recording of steady flow in the presence of fluctuations. All Marsh McBirney instruments are accurate to at least  $\pm$  2% of reading or  $\pm$  0.07 feet per second, whichever is larger.

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APPENDIX D
Analytical Quality Control

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### ANALYTICAL QUALITY CONTROL SUMMARY FOR FMC CCl4 SURVEY ANALYSES

Quality control consisted of measurement of three parameters to monitor the accuracy, repeatability and limit of detectability. Also, the method was evaluated and found to be linear over a working range of 0 to 200 ppb CCl<sub>4</sub> and checked occasionally during the analyses of samples.

### Accuracy

No accuracy data from measurement of independently prepared standards are available. However, stock standard solutions were prepared daily by addition of 6.3  $\mu$ l of pure CCl4 from a 10  $\mu$ l syringe to 1 liter of reagent water to yield a solution of 10 mg CCl4/l. The amount of CCl4 delivered from the syringe was measured as 9.88 mg with a relative standard deviation (RSD) of 1.31 percent for 10 determinations. These data show a negative bias of 1.2% on the accuracy of the standard and thus a 1.2% positive bias of the results.

### Repeatability

Positive calibration of the analyses was performed using a 200 ppb CCl4 in reagent water standard. The standard was prepared daily and run at least three times during the sample analysis runs that day. The daily results of these analyses are shown below:

Sampling Date	<u>n</u>	mean	Percent RSD
April 13-14	3	197	11
April 14-15	4	201	6.7
April 15-16	5	202	3.7
April 16-17	5	203	5.2
April 17-18	5	200	4.3
April 18-19	7	196	7.9
April 19-20	9	194	8.4
April 20-21	4	201	3.0

Duplicate analyses of samples were performed occasionally to supplement the above data. Results for three duplicate analyses are comparable with the % RSD data shown above.

<u>Initial</u>	Second	Percent Deviation
186 ppb	199 ppb	6.5
27 ppb	30 ppb	10
25 ppb	23 ppb	8.7

### Limit of Detectability

Detection limits were determined as two times the standard deviation of blank at the retention time of CCl<sub>4</sub>. Initial limits were set from analysis of six reagent water blanks. The standard deviation was 1.2 ppb and the detection limit calculated to be 3 ppb (rounded up from 2.4 ppb for convenience of reporting data). Reagent water blanks were measured daily during analyses and were 3 ppb or less.

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### ANALYTICAL METHOD AND EVALUATION FOR MEASUREMENT OF CC1

The analyses for carbon tetrachloride (CCl<sub>4</sub>) were performed using the direct aqueous injection technique described by FMC in the consent decree dated 15 March 1977. This technique was evaluated to determine detection limits, linear range, and precision at NEIC prior to use for this survey. Occurrances of high concentrations of CCl<sub>4</sub> were confirmed by combined gas chromatography-mass spectrometry.

The direct aqueous injection technique for CCl $_4$  described by FMC in the consent decree of 15 March 1977 was evaluated for use by NEIC. The only significant deviations from the FMC method was that 2  $\mu$ l in instead of 5  $\mu$ l injections were made and that standards were prepared daily in aqueous solution.

### Instrument Conditions

GC: Hewlett Packard (HP) Model 5738 with 15 mCi Nickel

63 detector

Column: 8 feet by 1/8 inch stainless steel packed with 60/80

mesh Tenax GC

Carrier Gas: 10% methane in argon, 60 psig and 60 ml/min flow rate

Injector: 250°C Column: 165°C Detector: 300°C

Peak Measurement: Either peak height or area integration with HP3352

data system

Linearity: Standard solutions of CCl<sub>4</sub> in water over a range of 0-600 ppb were analyzed. Figure 1 shows that above

0-600 ppb were analyzed. Figure 1 shows that above 200 ppb the response is no longer linear. This is probably due to the large amounts of CCl<sub>4</sub> causing the detector to recover slower, thus increasing the area. To minimize errors, samples above 200-250 ppb CCl<sub>4</sub> were diluted with distilled water and reanalyzed. Figure 2 shows that linearity is maintained at the low end of the analysis range. Peak height measure-

ments yield comparable results.

Detection Limit: Six distilled water samples were analyzed. At the

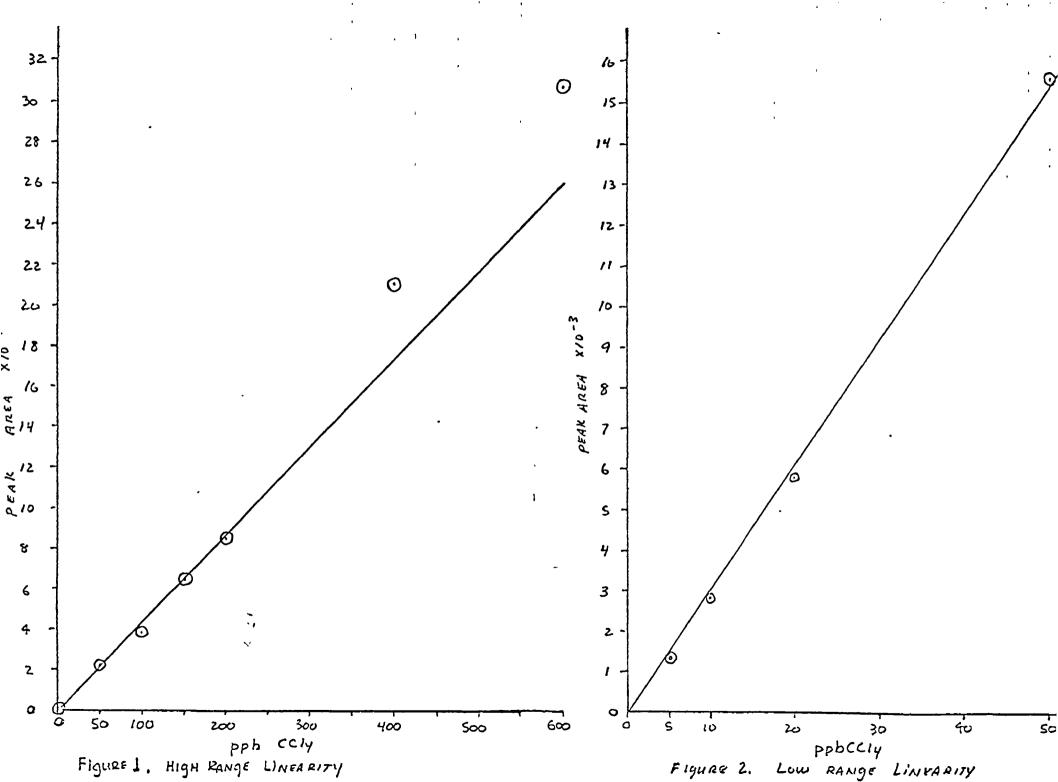
retention time equivalent to CCl4, the baselines were measured to determine the detection limit. The standard deviation  $(\sigma)$  was 1.2 ppb. The detection limit

 $(2\sigma)$  was averaged up to 3 ppb.

Precision: The average percent standard deviation for the 200 ppb

 $CC1_A$  standard was 5.3 for 7 days of analyses and 5 to 6

measurements of the standard per day.



APPENDIX E
Monitoring Results

17 3

Table A MONITORING RESULTS OUTFALL 018 FMC CORPORATION April 13-21, 1977

Date	Composite	Time	Flo	DW	CCl4	CCl4	cc14	018 Upstr	am Flow
	Number		m <sup>3</sup> /day x 1000	mqd	ppb	kg/day	1b/day	m <sup>3</sup> /day x 1000	mqd
4/13	1	0730	150 151 <u>1</u> /	39 6	15	2.4	5.3		
	2 3	1130 1530	151 <del>-</del> 7 152	40.0 40.1	13	2 0 1 8	4.3 4 0		
	3 A	1930	120	34.2	300 <u>3</u> /	39	85 6		
	τ 5	2330	129 142 <u>1</u> /	37 4	9 1	1 3	2.8		
	6	0330	154,	40 6	5.2	0.8	1.8		
4/14	ĭ	0730	1601/	42.2	5.4	0.9	1.9	168	44.4
•	2	1130	165	43.7	7.1	1 2	26	191	50.4
	3	1530	200	52.9	8.3	1.7	3 7	231	60.9
	4	1930	104	27.5	5.1	- 053	1 2	153	40 4
	5	2330	$145^{-1}$	38.3	5.9	0.86	19	144	38.1
	6	0330	186 183 <u>1</u> /	49.1	36	6.7	15	157	41.4
4/15	1	0730	1831/	48.4	17	3.1	6 9	177	46 7
	2	1133	181 187 <u>1</u> /	47.7	8.6	16	3 4	162	42 5
	3 4	1500 1920	192	49.3 50.9	8.0 7.9	1 5 1.5	3.3 3.4	155 186	41.2 49.2
	<del>4</del> 5	2330	132	36 2	17.9	2 3	5.1	100	45.2
	5 6	0330	137 <sub>1</sub> /	38.2	34	4.9	11		
4/16	<sup>6</sup> 2/	9739	152	40 2	18	2.7	6.0	142	37.4
,	2	1130	146	38 5	8.0	1.2	2 6	138	36 5
	3	1530	74 80 <u>1</u> /	19 6	58	0 43	0 95	138	36 5 36 4
	4	1930	8017	21 2	6.0	0.48	11	148	39.0
	5	2330	86	22 7	4.9	0.42	0 93	156	41.3
	62/	0330	163	43.0	14	2 3	5 0	142	37 5
4/17	1-	0730	116	30 6	<sup>-</sup> 6 6	0.76	1 7	100	42
	2	1130	151 147	39 9	5.9	0.89	2.0	165	43 7 37 5
	5 /*	1530 1930	764	41 5 43 4	- 6.5 4.2	1.0 0.69	2 3 1 5	142 1-6	ა/ : 38 მ
	<u> </u>	2330	170	43 4	5 4	0.59	2 0	151	39 8
	6 .	_ 0330	153	40.5	4.8	0.74	1.6	153	49.4

If Lithium sample lost or contaminated, flow is average of preceding and following flows.

 $<sup>\</sup>frac{2}{1}$  bottle broken; composite consists of 3 samples.  $\frac{3}{1}$  Confirmed by mass spectrometry.

Table A (continued)

MONITORING RESULTS OUTFALL 018 FMC CORPORATION April 13-21, 1977

Date	Composite	Time	F.	low	CC14	CC14	CC14	0]8 Hostre	eam Flow
	Number		m <sup>3</sup> /day x 1000	mgd	opb	kg/day	1b/day	m <sup>3</sup> /dav x 1000	mgd
4/18	1	0730	170	44 9	5.5	0.93	2 1	164	43.4
	2	1130	159	41.9	5.5	0.87	19	131	34.5
	3	1530	169	44.6	5 4	0.91	2.0	161	42.5
	4	1939	157	41.5	5 6	0 83	1.9	178	<i>t</i> 6 9
	5	2330	191,,	50 5	45	0.86	19	173	45.7
	6	0330	176 <del>"</del> /	46.4	5.5	0 97	2.1	158	41 7
4/19	1	0730	160	42.3	4.4	0 70	1 6	157	41 5
	2	1130	174	46 J	8.8	15	3 4	166	43.9
	3	1530	169	44 7	12	2 0	4 5	170	45 0
	4	1930	161	42.4	21	3.2	7.1	154	40 6
	5	2330	171	45.2	17	2.9	6 4	155	41.0
	6	0330	197	519	21	4.7	10	184	43.5
4/20	Ì	0730	182	48 2	30	5.5	12	166	13.8
	2	1130	169	44 5	130	22	48	157	41 4
	3	1530	177	46.7	17	3 0	6 6	170	45 0
	4	1930	161	42 5	16	2.5	5 7	157	41.5
	5	2330	154	40.7	11	1.7	3 7	156	41.2
	6	0330	174	45.7	22	3.8	é 4	147	38.5

 $<sup>\</sup>frac{1}{2}$  Lithium sample lost or contaminated; flow is average of preceding and following flows.  $\frac{2}{2}$  1 bottle proken; composite consists of 3 samples.

Table B MORITORING RESULTS OUIFALL 019 FIR CORPORATION April 13-21, 1977

Date	Composite	Time	F1c	):1	cc14	CCl4	CCla
	Number		m <sup>3</sup> /day x 1000	mad	opb <sup>4</sup>	kg/day	CC1 <sub>4</sub> 1b/day
4/13	1 2 3 4 5	0730 1139 1530 1939 2339	38 2 34.8 36 32/ 37.8 34.1	10.1 9.2 9.62/ 10.0 9.0	16 <3 <3 5 9 9.8	0.61 <0.10 <0.11 0.22 0.33	1.3 0.23 0.24 0.49
4/14	6 1 2 3 4 5	0330 0730 1130 1530 1930 2330	42 4 27 5 36 7 41.3 45.0 45.8	11.2 7 8 9 7 10.9 11 9 12.1	9.8 5.1 <3 3 7 5 3	0.42 0.15 <0.11 0.15 0.24 0.64	0.92 0.33 0.24 0.34 0.53 1.4
4/15	6 1 2 3 4 5	0330 0730 1130 1530 1930 2330	47.3 38.2 31.8 37.5 46.2 46.2	12.5 10 1 8.4 9.9 12 2 12.2	5 0 8.0 <3 <3 <3 <3	0.24 0.11 <0.095 <0.11 <0.14 <0.14	0.52 0.25 0.21 0.25 0.31 0.31
4/16	1 <sup>1</sup> / 2 3 4 5	0330 ;730 1130 1530 1930 2330	47.3 35.2 35.6 36.7 45.8 43.5	12.5 9 3 9.4 9.7 12.1	6.5 7.0 18 <3 5.3 5.0	0.31 0 25 0.64 <0.11 0.24 0.22	0 68 0 54 1.4 0 24 0.54 0.48
4/17	6 1 2 3 <u>1</u> / 5	0330 0730 1130 1530 1930 2330	39.0 34.1 37.8 49.3 <u>2</u> / 42.8 43.5	10.3 9 0 10.0 10.6 11.3 11.5	4.9 5.1 3.2 3 7.6 7.3	0.19 0.17 0.12 <0 12 0.33 0.32 0.22	0.42 0.38 0 28 0 27 0 72 0.70 0 49
4/18	6 1 2 3 4 5	0330 0730 1130 1530 1930 2330	42.8 35 6 34.8 34.1 40.9 44.3	.9.4 9.2 9 0 10.8 11.7	5.2 4.2 7.1 12 5.5 6.0	0 15 0.25 0.41 0.22 0 27	0.33 0.55 0.90 0.50 0.59
4/19	6 1 2 3 4	0330 0730 1130 1530 1930 2330	42 4 36.0 34.4 37.3 <sup>2</sup> / 40.1 41 6	11.2 9.1 9 1 9.8 10.6	5.2 3.7 6.1 20 35 3.1	0.22 0.13 9.21 1.75 1.4 0.13	0.29 0.46 1.6 3.1 0.28
4/20	6 1 2 3 4 5 6	0330 0730 1130 1530 1930 2330 0330	45.8 37 1 38.2 37.8 53.3 50.7 41 6	12.1 9.8 10 1 10.0 14.1 13.4 11 0	4 2 11 5.9 <3 400 160 87	0.19 0.41 0.23 <0.11 21 8.1 3.6	0.42 0.90 0.50 0.25 47 18 8.0

Thottle broken; composite consists of 3 samples.
Flow measurement not taken due to toxic atmosphere, flow is average of preceding and following flows

Table C
MONITORING RESULTS
OUTFALL 022
FMC CORPORATION
April 13-21, 1977

Date	Composite Number	Time	m³/day x 1000	nod mod	CC1 <sub>4</sub>	CC1 <sub>4</sub> Eg/day	CC1 <sub>4</sub> 1b/day
4/13	1 ? 3 4 5	0730 1130 1539 1930 2339	0 89 0 93 1.0 1.0	0 236 0 246 0 272 0 272 0.281	229 240 200 230 240	0 20 0 22 0 21 0 24 0 26	0 43 0 49 0 45 0 52 0 56
4/14 4/14	6 1 2 3 4 5	0330 0730 1130 1530 1930 2330	1.1 0 98 0.91 0 93 0.98 1 0	0.283 0 259 0 240 0 247 0.260 0 269	190 220 190 240 210 170	0.20 0 22 0 17 0 22 9 21 0 17	0 45 0 48 0 38 0 49 0 46 0 38
4/15	6 1 2 <u>1</u> / 3 4 5 6	0330 0730 1130 1530 1930 2330 0330	1.0 0 89 2 5 2 8 3 0 3 2 3 0	0 268 0.236 0 673 0 749 0 802 0 835 0 781	160 160 250 60 94 79 68	0 16 0.14 0 64 0 17 0 29 0 25 0 20	0 36 0 32 1 4 0.38 0.63 0.55 0.44
4/16	1 2 3 4 5 6	0730 1130 1530 1930 2330 0330	2 9 2.9 2 4 1.9 2 0 1.8	0.759 0.765 0.646 0.508 0.530 0.484	64 54 72 91 90 84	0.18 0 16 0 18 0 17 0 18 0.15	0.41 0.34 0.39 0.39 0.40 0.34
4/17	1 2 3 4 5 <u>1</u> / 6	0730 1130 1530 1930 2330 0330	1.8 2.0 1 8 1 9 1.9 1 8	0.479 0.526 0.484 0.498 0.498 0.498	74 220 110 93 72 80	0.13 0.44 0.20 0.18 0.14 0.15	0.30 0.97 0.44 0 39 0 30 0.32
4/18	1 2 3 4 5 6	0730 1130 1530 1930 2330 0330	1.8 1.8 1.8 1.8 1.8	0.472 0.472 0.472 0.472 0.472 0.472	83 78 96 80 72 79	0 15 0.14 0.17 0 14 0.13 0.14	0.33 0 31 0.38 0.32 0.28 0.31
4/19	1 2 3 4 5 6	0730 1130 1530 1930 2330 0330	1.8 1.9 0 37 0.40 0 37 0 37	0.481 0.489 0.099 0.105 0.099	95 87 330 340 230 220	0 17 0.16 0.12 0.14 0 086 0.082	0.38 0.36 0.27 0.30 0.19 0.18
4/20	1 2 3 4 5 6	0730 1130 1530 1930 2330 0330	0.32 0.96 0.36 0.36 0.34 0.32	0.084 0.254 0.094 0.094 0.089 0.085	320 130 300 270 250 270	0.10 0.13 0.11 0.096 0.084 0.087	0.22 0.28 ,0.24 0.21 0.19

Mottle broken, composite consists of 3 samples.

Table D

MONITORING RESPETS
SULTUP WASH FEELUENT
FMC CORPORATION
April 13-21, 1977

Date 4/13	Composite Number	71me 	m <sup>3</sup> /day x 1000	mgd	CC14 ppb	CC1 <sub>4</sub> kg/day	CC1 <sub>4</sub> 1b/day
4/13	2	0730					
	2		1.21/		1600	3 79	
		1130	1.2	0.32	1300	1.7	3 8
	4	1530 1930	ι 2 <u>1</u> /	0.32	1300 490	1,1	2 4
	5	2330		17.32	420	1,,,	2, 4
	Ğ	0330	۱ ۶ <u>۱</u> /	0.32	600	0.61	1.3
4/14	1	0/30	17		1300		
	2	1130	1.21/	0.32	1400	1 6	3.6
	3	1520	1.21/	0.32	1400 1300	1 6	3 6
	4 5	1930 2330	1.2	0.32	1400	1 0	3 0
	6	0330	1 2 <u>1</u> /	0.32	6400	4.7	10
4/15	ĭ	0730	, ,	7.02	3300		
•	2	1130	0.61	0.16	1700	1.5	3 4
	3	1530			1900		
	4 5	1930	1.4	0.37	1500	2.4	5.2
	5	2330	1 2	0.25	2300 5100	3.7	8.2
4/16	6 1 <u>3</u> /	0330 0730	1.3	0.35	3200	3.7	0.2
7, 10		1130	1.3	0.34	1200	2.9	6.3
	3	1530	1.3	0.51	1100	2.3	0.0
	2 3 4 5 6	1930	1.1	0.29	1200	1.3	2.8
	5	2330			620	_	
		0330	12	0.31	2500	1.9	4.1
4/17	1	0730		0 20	1500	1 6	2.4
	2	1130 1530	1.2	0.32	1100 990	1.6	3.4
	3 4 <u>3</u> /	1930	1.1	0.29	650	0.90	2.0
	5	2330	•••	0.25	740	0.50	
	5 6	0330	1.2	0.31	840	0.95	2.1
4/18	1	0730		•	960		
	2	1130	1.2 <u>2</u> /	0.31	1300	1.4	3.0
	2 3 4	1530	1.0	0 01	1100	1.2	2.6
	4 r	1930	1.2	0.31	850 620	1.2	2.6
	5 6	2330 0330	1.32/	0.34	620	0.80	1.8
4/19	1	0730		0.54	430	0.07	
., .,	ż	1130	1.3 <sup>2/</sup>	0.34	530	0 62	1.4
	3	1530			1600		
	4	1930	1.4	0.38	3300	3.4	7.6
	5	2330			3100	0.0	4.0
4/20	6 1	0330	1.2	0.31	510 760	2.2	4.8
4/20		0730 1130	1.2	0.32	2900	.2.	4.8
	2 3 4	1530	1.6	0. JL	1300	, •	
	4	1930	1.2	0.31	2300	0 92	2.0
	5 6	2330			750		
	6	0337	1.2	0.30	3900	2.8	6.2

<sup>1/</sup> Flows not measured with LiCl for April 13-14, flow used is average of April 15-21 flows.

 $<sup>\</sup>frac{2}{3}$  Lithium sample lost or contaminated, flow is average of preceding and following.  $\frac{3}{1}$  bottle broken, composite consists of 3 samples.

Table E

MONITORIGG RESULTS
CARBO L ADSORPTION UNIT
FMC CORPORATION
April 13-21, 1977

Date	Composite Number	Time	m <sup>3</sup> /day x 1000	mqd	CC1 <sub>4</sub> pob	CC1 <sub>1</sub> g/day	CC14 1b/day y10-3
4/13	1 2 3	0730 1130 1530	0.27 0 26 0.27	0 0/2 0 068 0 072	<3 <3 <3	<0.82 <0.78 <0.82	<1.8 <1.7 <1.8
	4 5	1930 2330	0.27 0.27	0 072	<3 <3,	<0.82 <0.82	<1 8 <1 8
	6	03 )	0.27	0 070	<3	< 0 80	<18
4/14	1	0750	0.27	0.072	<3	<0.82	<1.8
	2	1130 1530	0.33 0.27	0 088 0 072	<3 <3	<1.0 <0.82	<2.2 <1.8
	3 4	1930	0.27	0 072	<3	< 0.80	<1.8
	5	2330	0.25	0 065	<3	< 0.74	<1.6
	6	0330	0.25	0 065	< 3	< 0.74	<16
4/15	1	0730	0.27	0 070	<3	<0 80	<18
	2	1130	0.36	0 095 0 090	<3 <3	<1.1 <1.0	<2 4 <2 2
	3 4	1530 1930	0 34 <b>0.</b> 30	0.078	< 3	<0.89	<2.0
	5	2330	0.29	0.076	<3	<0.86	<1.9
	5 6 1 <u>1</u> /	0337	0.30	0.079	<3	<0.90	<2 0
4/16	17/	0739	0.27	0.072	<3	<0.82	<1.8
	2	1130	0.35	0 094	<3	<1.1	<2 4
	2 3 4	1530 1930	0.32 0.27	0 085 0 072	<3 <3	<0.96 <0.82	<2 1 <1.8
	5	2330	0.27	0 072	<3	<0.82	<1.8
	6	0330	0.29	0.078	< 3	<0.88	<1.9
4/17	1	0739	0.27	0.072	<3	<0.82	<1.8
	2	1130	0.31	0.083	<3	<0.94	<2.1
	3	1530	0.27	0.072	<3 <3	<0.82 <0.78	<1.8 <1.7
	4 5	1930 2330	0.26 0.27	0 068 0 072	<3	<0.82	<1.8
	6	0330	0.28	0.074	<3	< 0 84	<1.9
4/18	ĭ	0730	0.27	0.072	<3	< 9.82	<1.8
	2	1130	0.27	0 072	<3	<0.82	<1.8
	3	1530	0.27	0 072 0 072	<3 <3	<0 82 <0.82	<1.8 <1.8
	4 5	1930 2330	0.27 0.27	0.072	<3	< 0.82	<1.8
	6	0337	0.27	0 072	<3	< 0 82	<18
4/19	1	0730	0.27	0 072	<3	<0.82	<1.8
	2	1130	0 27	0.072	<3	<0.82	<1.8
	3	1530	0.27	0.072	<3 <3	<0.82 <0.82	<1.8 <1.8
	4 5	1930 2330	0.27 0.27	0.072 0.072	<3	<0.82	<1.8
	5 6	0330	0.27	0.072	<3	<0.82	<1.8
4/20	1	0730	0.27	0.072	3 4	0.93	2.1
	2 3	1130	0.14	0.036	13	1.8	4 0
	3	1530	0	0	-	0	, ; 0,
	4 5	1930 2330	0 0	0 0	-	0 0	0
	5 6	0330	0.014	0.036	19	0 2.6	5.7

<sup>1/ 1</sup> bottle broken; composite consists of 3 samples.

Table F

SUMMARY OF FIELD MEASUREMENTS 1/
FMC CORPORATION
April 13-21, 1977

	4/13	3	4/14		4/15		4/16	i	4/17	,	4/18		4/19		4/20	
Station	рН	Temp	рН	Тетр	рН	Temp	рН	Temp	рН	Temp	рН	Temp	рН	Temp	рН	Temp
019	3.1 10.6	22 25	10.3	22 25	10.4 11.7	22 24	10.5 12.0	23 25.5	10.4 11.6	24 26	10.0 10.8	25 26	8.7 10 0	25 26	6.1 11.4	25 26.5
Carbon Assorption Unit	12.5 13.0	35 43	12 2 12.7	32 39	12.5 13.0	25 40	12.3 13.0	33 43	12.5 12 9	27 38	12.6 12.8	28 41	12 0 12.9	21 33	12.0 12.5	29 33
Sulfur Wash	1.4	15 23.5	2.4 7.5	15 20	0 5 2.9	- 18 26	1.2 3 7	17 25	2 8 3.7	18 21	1.4 3.1	22 23	1.1	22 23	2 6 4 0	19 23
019	6.4 9.7	14 18	7.6 9 1	14 17	7 6 9.4	17 20	7.3 9 9	16 20	7.4 8.4	17 18	6 5 9 3	19 22	4.0 9 1	1 <i>7</i> 19	7.6 9.9	19 20
022	10.2 11.3	14 16	9.7 11.0	14 16	8 8 12,4	14 25	9.6 10.3	14.5 16.0	9.8 10.2	16 11	10.2 10.7	17 17.	8.8 10 5	18 19	9.7 10.4	18 19

<sup>1/</sup> pH and temperature are presented in ranges of S.U. and °C