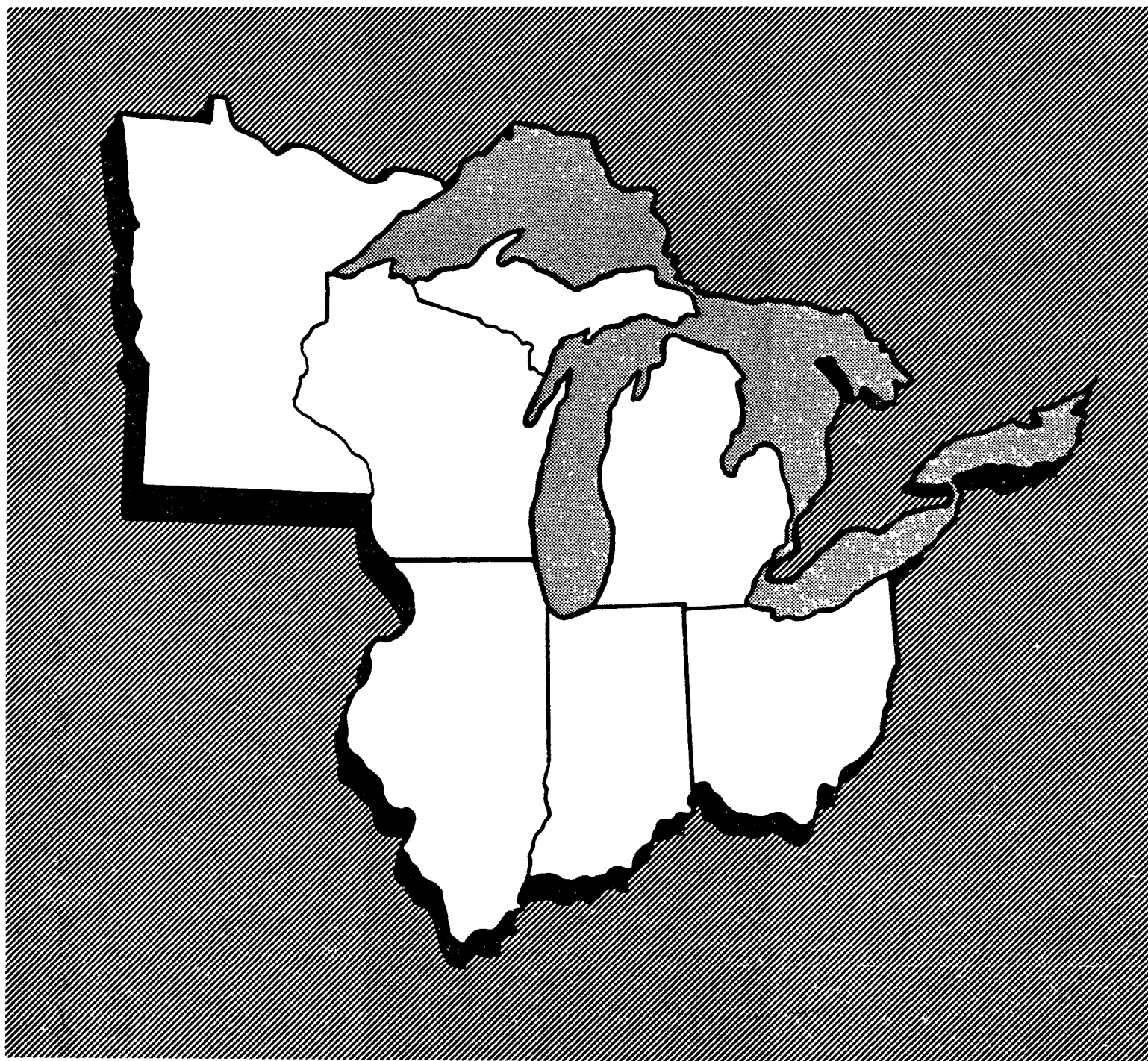




# Test Report for Getty Synthetic Fuels, Inc., Calumet City, Illinois

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TEST REPORT FOR  
GETTY SYNTHETIC FUELS, INC.  
CALUMET CITY, ILLINOIS

Contract No. 68-01-6312  
Work Assignment 54

Submitted to  
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Region V  
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Chicago, Illinois 60604

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## CHAPTER 1

### INTRODUCTION

The U.S. Environmental Protection Agency (EPA), Region V, Air Management Division is interested in characterizing the composition of the feed and discharge streams of the Getty Synthetic Fuels landfill gas process plant in Chicago, Illinois. To this end, Work Assignment 54 of Contract 68-01-6312 was given to Engineering-Science of Fairfax, Virginia, to devise a sampling and analytical program and conduct the field sampling in accordance with the program. Analyses were performed by Radian Corporation of RTP, N.C.

The purpose of this project is to quantify the loadings of the non-criteria pollutants listed in Table 1.1 and to screen for other organic compounds using gas chromatography/mass spectrometry (GC/MS). Three gas streams and one liquid stream were evaluated. The feed gas is primarily composed of methane and carbon dioxide but some non-methane hydrocarbons and metal vapors may be present.

The remainder of this report presents the field testing and analytical results. Field tests were conducted on March 5 and 6, 1984, by personnel from Engineering Science. Representing Getty Synthetic Fuels was Mr. Randy Masukawa; EPA, Region V, was represented by Mr. John Connell. The office of the Illinois State Attorney General had representatives on site during testing. The ES test crew was composed of Messrs. Krask, Gallagher, and Felts, and was headed by Mr. Cottone. Laboratory analyses were under the supervision of Mr. Denny Wagoner of Radian.

Sampling and analytical procedures followed those presented in the Quality Assurance Plan for Getty Synthetic Fuels, Inc., Calumet City, Illinois (April 1984).

TABLE 1.1

EPA NON-CRITERIA AIR POLLUTANTS TO BE QUANTIFIED AT THE  
GETTY SYNTHETIC FUELS FACILITY

---

Acetaldehyde	Manganese <sup>a</sup>
Acrolein	Methyl Chloroform (1,1,1, Trichloroethane)
Acrylonitrile	Methylene Chloride (dichloromethane)
Allyl Chloride	Nickel <sup>a</sup>
Benzyl Chloride	Nitrobenzene
Chromium <sup>a</sup>	Nitrosomorpholine
Cadmium <sup>a</sup>	Perchloroethylene
Carbon Tetrachloride	Phenol
Chlorobenzene	Polychlorinated Biphenyls
Chloroform	Toluene
Chloroprene	Trichloroethylene
o-, m-, p-Cresol	Vinylidene Chloride
p-Dichlorobenzene	o-, m-, p-Xylene
Dimethyl Nitrosamine	Arsenic <sup>a</sup>
Dioxin	Benzene
Epichlorohydrin	Beryllium <sup>a</sup>
Ethylene Dichloride	Mercury <sup>a</sup>
Formaldehyde	Vinyl Chloride
Hexachlorocyclopentadiene	

---

<sup>a</sup> Quantified in the liquid stream only.

## CHAPTER 2

### SUMMARY AND DISCUSSION OF RESULTS

The concentrations of target compounds of the testing of the Getty Synthetic Fuels process at the Chicago/Calumet cities landfill are summarized in Tables 2.1 through 2.7 of this section. Analysis for dioxins, PCB's and nitrogenated compounds (XAD-2 and Thermosorb <sup>TM</sup>/N samples) resulted in none of those compounds being detected. During the analysis of the charcoal samples, allyl chloride (3-chloropropene), methylene chloride and vinylidene chloride (1,1-dichloroethene) could not be separated from the carbon disulfide solvent peak. Aldehyde sampling resulted in no acrolein, propaldehyde or benzaldehyde being detected.

All sample rates, times and volumes are taken from the field data sheets which are contained in Appendix G.

#### Charcoal

Table 2.1 presents the results of the charcoal testing. Analysis of the charcoal tube samples were performed by GC/FID, using carbon disulfide as the desorbition medium. The feed gas stream was characterized as having up to 65 ppm levels of five aromatic compounds including chlorobenzene. Epichlorohydrin was also found in two of the five samples. The CO<sub>2</sub> vent had much lower levels (less than 3 ppm) of benzene, 1,2-dichloroethane, trichloroethylene and perchloroethylene. None of those compounds were found in more than one of the five vent samples. One of the vent samples also had 86 ppm of chloroform. The sale gas had no detectable amounts of any of the compounds analyzed by this method.

The audit gas was collected on charcoal for the determination of benzene and perchloroethylene. Neither of those components were detected. Based on the audit gas sample volume, the minimum detectable benzene and perchloroethylene concentrations would be 0.5 and 0.9 ppm respectively.

#### Tenax<sup>®</sup>

Table 2.2 summarizes the results of the Tenax<sup>®</sup> sampling. GC/MS was the analytical technique employed for Tenax<sup>®</sup>; direct desorbition using the purge and trap procedure was the analytical recovery procedure. Since the Tenax<sup>®</sup> samples were direct injected onto the GC column without dilution the sensitivity was much better than the charcoal method. In the feed gas, chlorobenzene was found but in lower concentrations than detected with the charcoal sample train. Other compounds detected on the Tenax<sup>®</sup> feed gas samples include 1, 1, 1-trichloroethane, vinyl chloride (up to 703 ppb), trichloroethylene, tetrachloroethylene and chloroform. The chloroform was detected at a level which was below the minimum detectable by the charcoal technique.



TABLE 2.1

GETTY SYNTHETIC FUELS  
RESULTS OF ANALYSES OF CHARCOAL SAMPLES  
(GC/FID ANALYSIS, CS<sub>2</sub> DESORPTION)

Sample Location	Sample Volume (liters)	Date	Start Time	(a) ppm	(b) ppm	(c) ppm	(d) ppm	(e) ppm	(f) ppm	(g) ppm	(h) ppm	(i) ppm	(j) ppm	(k) ppm
Feed Gas	0.671(n)	3/6	1033	ND(m)	ND	35.2	ND	23.7	ND	ND	43.5	ND	31.0	34.7
	0.908	3/6	1204	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	1.632(n)	3/6	1452	ND	ND	12.9	ND	9.2	ND	ND	15.5	67.2	11.3	18.2
	2.150	3/6	0858	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	7.273(n)	3/5	1728	ND	ND	6.9	ND	4.7	ND	3.0	ND	45.5	5.8	ND
Sale Gas	2.006	3/6	1203	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	12.148	3/5	1728	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	17.381	3/6	0900	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
CO <sub>2</sub> Vent	1.839	3/6	1216	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	3.649(1)	3/6	1519	ND	ND	ND	ND	ND	ND	ND	2.8	ND	ND	ND
	3.791(1)	3/6	1518	ND	ND	ND	ND	ND	4.3	ND	ND	ND	ND	ND
	5.446(n)	3/6	0901	85.6	ND	ND	1.2	2.2	ND	ND	ND	ND	ND	ND
	10.574	3/5	1731	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Audit Gas	17.332	3/5		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	16.226	3/5		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
				mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg
Feed Gas	Blank	3/5		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Sale Gas	Blank	3/5		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
CO <sub>2</sub> Vent	Blank	3/5		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

(a) Chloroform

(b) Carbon tetrachloride

(c) Chlorobenzene

(d) 1,2-dichloroethane

(e) Benzene

(f) Trichloroethylene

(g) Perchloroethylene

(h) Epichlorohydrin

(i) Toluene

(j) o-xylene

(k) m,p-xylene

(1) Duplicate sample

(m) ND = none detected

(n) Back-half of charcoal tube extracted and analyzed separately with no detectable target compounds

TABLE 2.2

GETTY SYNTHETIC FUELS  
RESULTS OF ANALYSES OF TENAX® SAMPLES  
(GC/MS ANALYSIS, PURGE AND TRAP)

Sample Location	Sample Volume (liters)	Date	Start Time	(a) ppb	(b) ppb	(c) ppb	(d) ppb	(e) ppb	(f) ppb	(g) ppb
Feed Gas	0.296	3/6	1405	21	647	453	624	0 <sup>(k)</sup>	ND	2135
	0.640 <sup>(h)</sup>	3/6	1645	23	703	363	348	1298	ND	ND
	0.627 <sup>(h)</sup>	3/6	1645	35	103	3650	4014	0 <sup>(k)</sup>	ND	16380
	0.966	3/5	1950	25	ND <sup>(i)</sup>	291	256	1940	ND	1278
	2.918	3/6	1059	69	30	861	ND	55	ND	ND
Sale Gas	0.524 <sup>(1)</sup>	3/6	1405	ND	283	186	ND	2742	ND	ND
	9.606	3/6	1059	2	88	ND	ND	15	ND	ND
	18.421	3/5	1953	2	67	0 <sup>(k)</sup>	ND	113	ND	ND
CO <sub>2</sub> Vent	0.922	3/6	1406	26	1143	108	ND	662	ND	100
	6.266	3/5	1956	6	1278	19	11	53	ND	37
	8.116	3/6	1100	4	1229	16	9	160	ND	53
Audit Gas	12.930	3/5		NA <sup>(j)</sup>	NA	NA	NA	NA	NA	NA
	13.996	3/5		1.0	22	0 <sup>(k)</sup>	8	1210	7	ND
				µg	µg	µg	µg	µg	µg	µg
Feed Gas		3/5		.078	ND	0.534	ND	9.806	ND	ND
		3/6		ND	ND	ND	ND	1.043	ND	ND
Sale Gas		3/6		ND	ND	0.551	0.445	0.641	0.339	ND
CO <sub>2</sub> Vent		3/5		ND	ND	ND	ND	1.574	ND	ND
		3/6		ND	ND	ND	ND	0.576	ND	ND

(a) 1,1,1-trichloroethane

(b) Vinyl chloride

(c) Trichloroethylene

(d) Tetrachloroethylene

(e) Chloroform

(f) Carbon tetrachloride

(g) Chlorobenzene

(h) Duplicate sample

(i) ND = none detected

(j) NA = not analyzed

(k) Value equal to or less than zero after blank correction made

(1) This sample had a broken charcoal/Tenax® tube. Reported results are from analysis of Tubes 1, 2, and 3 of the four tube sampling train.

Carbon tetrachloride was not detected at any of the sites by either method. The Tenax® samples from the CO<sub>2</sub> vent gas contained higher levels of vinyl chloride than the feed gas site. Other compounds were found in similar or lower concentrations than in the feed gas stream.

The sale gas Tenax® samples had ranges of 67 to 283 ppb of vinyl chloride, 15 to 2742 ppb of chloroform and 2 ppb of 1,1, 1-trichloroethane. One sample had 186 ppb of trichloroethylene. Otherwise, there were no detectable compounds at that site.

It should be noted that chloroform was detected in all of the Tenax® modules including the blanks. The samples were corrected using the blank values specific to each site but it seems that contamination did occur. Since chloroform extractions were performed on the DNPH in the field, laboratory, this could provide a possible explanation for the high blank values.

The results of the audit gas sampled using the Tenax® train are also included in Table 2.2. Those samples were collected at the test location as was observed by the EPA project officer.

#### DNPH

Table 2.3 summarizes the results of the aldehyde sampling. The procedure for the analysis of aldehydes was a high performance liquid chromatography (HPLC) technique. Formaldehyde and acetaldehyde were both detected fairly consistently in the feed gas stream. The CO<sub>2</sub> vent and sale gas each had one sample with fairly high formaldehyde levels. Those tests were conducted concurrently with the feed gas run yielding the highest formaldehyde level. Little or no acetaldehyde was found in samples from those two sites.

#### Condensate

Table 2.4 summarizes the metal analyses of the hydrocarbon condensate stream. ICAP and AA were the analytical techniques employed for metals analysis. Beryllium, arsenic and mercury were below the minimum detectable levels of the method. A GC/MS screening and quantification of selected compounds in the hydrocarbon condensate is presented in Table 2.5.

#### Summary of Results

Table 2.6 presents the observed range of loadings of each component in each of the three gas streams tested. The values listed are in pounds per hour and were determined based on the ES measured concentrations and flow rates provided by Getty.

Table 2.7 presents GC/MS screening for selected XAD-2 and charcoal tube samples. By comparing the results of the charcoal tube GC/FID analyses to the charcoal tube GC/MS analyses, it can be observed that there exists a favorable correlation between the two analytical methods.

TABLE 2.3

GETTY SYNTHETIC FUELS  
RESULTS OF ANALYSES OF ALDEHYDES SAMPLES<sup>a</sup>  
(HPLC)

Sample Location	Sample Volume (l)	Date	Start Time	(b) ppm	(c) ppm
Feed Gas	2.137	3/6	1334	14.1	ND
	13.258 <sup>(d)</sup>	3/6	1530	7.5	0.8
	18.767 <sup>(d)</sup>	3/6	1530	8.6	1.0
	19.222	3/6	1129	8.4	0.4
	58.114	3/5	1618	3.1	0.3
Sale Gas	2.140	3/6	1331	17.1	ND
	20.242	3/6	1129	ND <sup>(e)</sup>	ND
	55.265	3/5	1418	ND	ND
CO <sub>2</sub> Vent	1.923	3/6	1334	27.6	ND
	18.846	3/6	1129	2.5	1.0
	51.797	3/5	1620	ND	ND
Feed Gas	Blank	-	-	ND	ND
Sale Gas	Blank	-	-	ND	ND
CO <sub>2</sub> Vent	Blank	-	-	ND	ND

(a) Acrolein, propaldehyde, and benzaldehyde analyzed for and not detected

(b) Formaldehyde

(c) Acetaldehyde

(d) Duplicate sample

(e) ND = less than three times standard deviation of blank (i.e., 5 µg total)

TABLE 2.4

METAL RESULTS OF HYDROCARBON CONDENSATE SAMPLES  
(ICAP AND AA)

Radian No.		Parts Per Million (ppm)						
ID No.	Date	Beryllium	Cd	Cr	Mn	Ni	As	Hg
6084								
3:45 pm	3/5	<.0005	.089	.80	.21	.25	<.003	<.002
6086								
1710 hrs	3/5	<.0005	.044	.46	.21	.056	<.003	<.002
6087								
1100 hrs	3/6	<.0005	.020	.27	.039	.035	<.003	<.002
6085								
1230 hrs	3/6	<.0005	.044	.46	.21	.056	<.003	<.002
6088								
1630 hrs	3/6	<.005	.016	.19	.05	.027	<.003	<.002

TABLE 2.5

## GC/MS SCREENING OF HYDROCARBON CONDENSATE SAMPLES

Radion No. ID No.	Date	(a)	(b)	(c)	(d)	(e)	(f)	(g)	(h)	(i)	(j)	(k)	(l)	(m)	(n)
parts per million (ppm)															
6084 3:45 pm	3/5	43,200	<10	100	<10	3030	<10	650	81,050	<25	19,150	1650	70,050	32,700	650
6086 1710 hrs	3/5	<25	<10	<5	<10	<15	<10	<5	36,750	<25	2,400	<10	70,300	28,050	<5
6087 1100 hrs	3/6	35,800	<10	105	<10	3165	<10	885	86,800	<25	21,600	2100	75,000	34,450	435
6085 1230 hrs	3/6	40,800	<10	160	<10	4035	<10	925	86,900	<25	21,200	2200	78,550	35,700	605
6088 1630 hrs	3/6	27,650	<10	70	<10	2340	<10	480	72,800	<25	18,450	1200	68,350	32,250	545

(a) 1,1-Dichloroethene  
 (b) Chloroform  
 (c) 1,2-Dichloroethane  
 (d) 1,1,1-Trichloroethane  
 (e) Benzene  
 (f) CCl<sub>4</sub>  
 (g) Trichloroethene

(h) Toluene  
 (i) Epichlorohydrin  
 (j) Chlorobenzene  
 (k) Tetrachloroethene  
 (l) m,p-Xylene  
 (m) o-Xylene  
 (p) Dichlorobenzene

TABLE 2.6

## ESTIMATED LOADINGS AT GETTY SAMPLING LOCATIONS

	POUNDS PER HOUR		
	FEED GAS	SALE GAS	CO <sub>2</sub> VENT
<u>Charcoal Samples</u>			
Chloroform	0	0	0-1.4
Carbon Tetrachloride	0	0	0
Chlorobenzene	0-1.8	0	0
1, 2-Dichloroethane	0	0	0-0.02
Benzene	0-0.7	0	0-0.02
Trichloroethylene	0	0	0-0.08
Perchloroethylene	0-0.2	0	0
Epichlorohydrin	0-1.8	0	0-0.04
Toluene	0-1.5	0	0-0.04
O-Xylene	0-1.5	0	0
m, p-xylene	0-1.6	0	0
<u>Tenax® Samples</u>			
1,1,1-Trichloroethane	0.0012-0.0041	0.0001	0-0.0004
Vinyl chloride	0-0.0178	0.0013	0-0.0117
Trichloroethylene	0.0173-0.0504	0	0-0.0019
Tetrachloroethylene	0-0.0455	0	0-0.0003
Chloroform	0-0.1037	0.0004	0-0.0106
Carbon Tetrachloride	0	0	0
Chlorobenzene	0-0.1056	0	0-0.0016
<u>DNPH Samples</u>			
Formaldehyde	0.04-0.19	0-0.12	0-0.11
Acetaldehyde	0.01-0.03	0	0-0.01

TABLE 2.7

## GC/MS SCREENING OF SELECTED CHARCOAL AND XAD-2 SAMPLES

Sample Location	Sample Vol.	Sample Date	Start Time	(a) ppm	(b) ppm	(c) ppm	(d) ppm	(e) ppm	(f) ppm	(g) ppm	(h) ppm	(i) ppm	(j) ppm	(k) ppm	(l) ppm	(m) ppm	(n) ppm
CHARCOAL TUBES																	
CF531 Feed Gas	7.273	3/5	1728	<1.7	<0.5	<0.3	<0.5	3.1	<0.4	0.4	60.6	<1.8	6.0	<0.4	37.6	12.8	<0.2
CC661 CO <sub>2</sub> Vent	3.791	3/6	1518	<3.3	<0.1	<0.6	<1.0	<2.4	<0.8	<0.5	0.0	<3.4	<1.7	<0.8	<0.6	<0.6	<0.4
CC641 CO <sub>2</sub> Vent	5.446	3/6	0901	<2.3	<0.8	<0.4	<0.7	<5.7	<0.6	<0.3	0.0	<2.4	<1.2	<0.5	<0.4	<0.4	<0.3
XAD-2 CARTRIDGES																	
XF64 Feed Gas	5.360	3/6	1003	<1.2	<0.4	<0.2	<0.3	<0.9	<0.3	<0.2	64.5	<1.2	18.0	<0.3	90.3	41.7	1.3
XP64 Sale Gas	42.270	3/6	1003	<0.2	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.03	<0.2	<0.1	<0.1	<0.1	<0.1	<0.1
XC64 CO <sub>2</sub> Vent	37.721	3/6	1006	<0.2	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.13	<0.2	<0.1	<0.1	0.07	0.05	<0.1

(a) 1,1-Dichloroethene  
 (b) Chloroform  
 (c) 1,2-Dichloroethane  
 (d) 1,1,1-Trichloroethane  
 (e) Benzene  
 (f) CCl<sub>4</sub>  
 (g) Trichloroethene

(h) Toluene  
 (i) Epichlorohydrin  
 (j) Chlorobenzene  
 (k) Tetrachloroethene  
 (l) m,p-Xylene  
 (m) o-Xylene  
 (p) Dichlorobenzene



## Discussion of Results

In reviewing the tables in this section, several points bear consideration. First is the observation of the relative "cleanliness" of the sale gas in relation to the feed gas. For the sale gas charcoal tube samples (GC/FID analysis), no compounds of interest were detected. For the sale gas Tenax® samples (GC/MS analysis), compounds detected had concentrations in the parts per billion range (excluding chloroform in the sample collected 1405 on March 6). As explained elsewhere, there exists the possibility that there was chloroform contamination of the Tenax® samples. A single aldehyde sale gas sample indicated the presence of formaldehyde (1331 on March 6).

It appears that the condensate system is an effective hydrocarbon removal process. In reviewing Table 2.6, it will be observed that rather substantial concentrations of certain compounds exist in the hydrocarbon condensate. 1,1-dichloroethene was present in the concentration range of 2.7% to 4.3%, exclusive of one sample. Toluene concentration was as high as 8.7%, while xylene (m,p,o) was approximately 10% of the total hydrocarbon condensate volume. Chlorobenzene was present in the range of 0.2% to 2.1% of the condensate. Exclusive of 1,1-dichloroethene, all of the aforementioned compounds in the hydrocarbon condensate were present in the feed gas at high concentrations. As explained at the beginning of this chapter, for the gas samples, 1,1-dichloroethene (vinylidene chloride) could not be separated from the carbon disulfide peak, and hence, could not be quantified. It is pertinent to note, based on that GC/MS screen of the hydrocarbon condensate, that a substantial percentage of this condensate is chlorinated hydrocarbons (approximately 4.5%).

## CHAPTER 3

### FACILITY DESCRIPTION

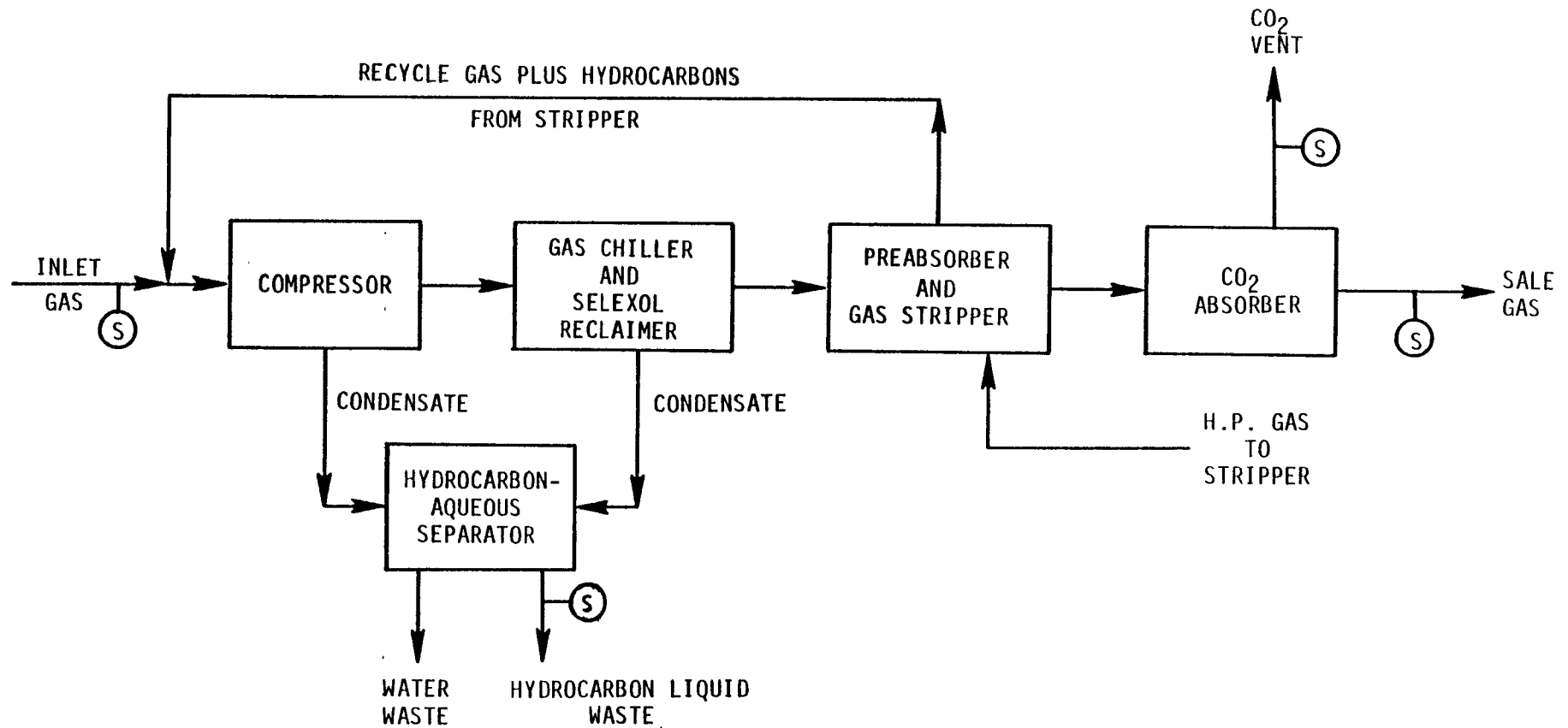
The Getty Synthetic Fuels facility collects and compresses gases generated by decomposition of materials in the Calumet City, C.I.D. sanitary landfill. A block diagram of the process is shown in Figure 3.1. The gas is pulled from the land fill at a vacuum ranging from 2 to 6 inches of mercury, gage. Compression of the gases results in condensation of water and organic compounds. The liquid and gas streams leaving the compressors are both treated further. The liquid stream is stored in tanks and the aqueous and organic layers are allowed to separate. The organic phase is then decanted off the aqueous layer. The organic liquid is sold as a low grade fuel. The aqueous layer is now being discharged into the city sewer system.

The compressed gas stream is passed through a preabsorber and gas stripper and then through a CO<sub>2</sub> absorber. The product or "sale gas" stream is at approximately 350 psi. The CO<sub>2</sub> vent is approximately at ambient pressure. The company plans to add a CO<sub>2</sub> production plant in the future.

Getty provided analyses of the feed or inlet gas and the product or "sale gas". These analyses are summarized in Tables 3.1 and 3.2 respectively. Getty states that the feed gas flow rate averages  $125 \times 10^3$  cubic meters per day (M<sup>3</sup>/d) (4.4 million cubic feet per day). Sale gas and CO<sub>2</sub> vent gas streams average approximately  $59.5 \times 10^3$  M<sup>3</sup>/d (2.1 MCFD) and  $28.3 \times 10^3$  M<sup>3</sup>/d (1.0 MCFD), respectively. Actual flow rates during this test program are presented in Table 3.3.

The feed gas stream has a loading of approximately 127 kg (280 pounds) per hour of non-methane hydrocarbons while the CO<sub>2</sub> vent reportedly has 3.6 kg (8 pounds) per hour. The gas volumes are 5,200 and 1,200 cubic meters per hour respectively for the feed and CO<sub>2</sub> vent gas lines indicating the feed concentration may be approximately 24 g per M<sup>3</sup> compared to the CO<sub>2</sub> vent concentration which may be approximately 3 g per M<sup>3</sup>. The non-methane hydrocarbon concentration of the sale gas is probably between these two concentrations.

# BLOCK FLOW DIAGRAM OF GETTY SYNTHETIC FUELS PLANT



## LEGEND:

S = Sample locations

TABLE 3.1  
ANALYSIS OF GETTY SYNTHETIC FUELS  
FEED GAS<sup>a</sup>

Composition	Actual Composition		Synthesized Composition	
	Mols/Hr	Lbs/Hr	Mols/Hr	Lbs/Hr
CO <sub>2</sub>	206.4668	---	193.110	---
H <sub>2</sub>	2.1849	---	---	---
N <sub>2</sub>	3.2774	---	1.020	---
CH <sub>4</sub>	334.2792	---	316.830	---
C <sub>2</sub> H <sub>6</sub>	0.1092	3.284	---	---
C <sub>3</sub> H <sub>8</sub>	0.0071	0.313	---	---
C <sub>4</sub> H <sub>10</sub>	0.0011	0.064	---	---
C <sub>6</sub> H <sub>14</sub>	0.0632	5.447	0.0400	3.447
C <sub>7</sub> H <sub>16</sub>	0.0939	9.409	0.0700	7.014
C <sub>8</sub> H <sub>18</sub>	0.1895	21.647	0.2300	26.273
C <sub>9</sub> H <sub>20</sub>	0.1845	23.664	0.0200	2.565
C <sub>10</sub> H <sub>22</sub>	0.1593	22.665	0.0500	7.114
C <sub>11</sub> H <sub>24</sub>	0.0066	1.032	---	---
C <sub>6</sub> H <sub>6</sub>	0.0807	6.303	0.0510	3.984
C <sub>7</sub> H <sub>8</sub>	0.4064	37.446	0.4100	37.777
C <sub>8</sub> H <sub>10</sub>	0.4032	42.808	0.1000	10.617
CH <sub>2</sub> Cl <sub>2</sub>	0.1324	11.245	0.0800	6.795
C <sub>2</sub> HCl <sub>3</sub>	0.0829	10.892	0.0200	2.628
C <sub>2</sub> Cl <sub>4</sub>	0.2246	37.245	---	---
C <sub>6</sub> H <sub>13</sub> OH	---	---	1.1000	112.323
Oxygenated Hydrocarbons <sup>b</sup>	0.5293	45.104	---	---
Alkyl	0.275	3.315	---	---
Benzenes <sup>b</sup>				
Nitrogen Compounds <sup>b</sup>	0.0176	2.112	---	---
		284.535		220.537

<sup>a</sup> Provided by Getty Synthetic Fuels.

<sup>b</sup> Composition of these compounds was not determined or quantified and assumptions were made as to composition, distribution of components and properties for purposes of calculating emission data.

TABLE 3.2

ANALYSIS OF GETTY SYNTHETIC FUELS PRODUCT OR "SALE GAS"<sup>a</sup>

Component	Mole Percent
Helium	0
Hydrogen	0.174
Oxygen	0.143
CO <sub>2</sub>	2.052
Nitrogen	2.429
Methane	95.199
Ethane	0
Propane	0.003
Isobutane	0
n-Butane	0
Isopentane	0
n-Pentane	0
Hexane	0

<sup>a</sup> Provided by Getty Synthetic Fuels.

TABLE 3.3  
PROCESS STREAM GAS  
FLOW RATES

	March 5, 1984	March 6, 1984
Feed Gas (Inlet) (MSCFD)	4138.104	4062.283
Sale Gas (Outlet) (MSCFD)	2206.104	2206.104
CO <sub>2</sub> Vent Discharge (MSCFD)	1353.927	1227.034

## CHAPTER 4

### SAMPLING LOCATIONS

There are three gas sampling locations and one liquid stream sampling location. The sites were identified during this program as follows:

- o Feed Gas - the raw gas tapped from the landfill site.
- o Sale Gas - compressed gas product from the facility.
- o CO<sub>2</sub> Vent - the vented CO<sub>2</sub> gas stream stripped from the compressed gas stream.
- o Hydrocarbon Discharge - the hydrocarbon condensate stream resulting from compressing and chilling of the gas stream.

Since the process streams could not be opened to the atmosphere during testing, stainless steel manifolds with shutoff valves were used for connecting the gas sampling systems to the sampling locations. This eliminated the need for glass lined sample probes as specified in the methods discussed below.

#### Feed Gas

The feed gas stream was under negative pressure ranging from -5 to -15 centimeters of mercury. The feed gas line was approximately 40 centimeters (16 inches) in diameter. A 1/4 inch female pipe thread connection on an existing 1 inch valve was used for the sample collection port.

A gas meter was on the inlet gas stream. Strip charts were obtained from Getty as a record of the gas flow and are included in Appendix F.

#### Sale Gas

The sale gas sampling point was under 25 kg/cm<sup>2</sup> (approximately 350 psi) positive pressure. The sale gas sample point was in a 40 centimeter (16 inch) diameter pipe. A valve with 1/4 inch female pipe thread was used for sample collection. Sale gas volumes were carefully monitored by the Getty Synthetic Fuels company.

#### CO<sub>2</sub> Vent

The CO<sub>2</sub> vent was at slightly positive pressure. The size of the vent was approximately 6 cm (2.5 inches) inside diameter and was lo-

cated approximately 12 feet above grade. Gas flow rates from the CO<sub>2</sub> vent were provided by the plant engineers.

#### Hydrocarbon Condensate Discharge

The hydrocarbon liquid stream is collected in a holding tank then discharged into a tank truck. Samples were collected from the discharge line to the truck. The hydrocarbon liquid stream flow was provided to the test team by Getty.



## CHAPTER 5

### SAMPLING AND ANALYTICAL PROCEDURES

Due to the qualitative and quantitative data requirements of this project, several different sampling and analytical methods were required. Those methods were discussed in the QA document for this project and were included there by reference or as an appendix.

#### SAMPLING METHODOLOGIES

##### Liquid Samples

The hydrocarbon condensate discharge sample was collected from a tap in the discharge line. The sample was collected directly into a borosilicate glass bottle with a Teflon® lined screw cap. The bottles were cleaned with acetone prior to the test program.

A single liquid sample was collected to represent each tank load before discharge. A total of five liquid samples were collected. The total sample volume was approximately 500 ml for each sample.

##### Gas Sampling

Table 5.1 lists the target compounds of interest and the adsorption media that was used for collection of each component.

The DNPH sampling method is summarized in Appendix A. The sample rate used was varied from 1 to 1.5 liters per minute. Sample time was varied from 10 to 40 minutes. These variations were used to allow collection of sample volumes ranging from 10 to 60 liters which provided some insurance against all samples from a given sampling location having too much or too little aldehyde. The sample train operation and leak check procedures which were used were those specified in EPA Reference Method 6. Reagent preparation, glassware cleaning, and sample recovery were as specified in Appendix A. No probe or heated filter was used for this test program. This is a deviation from the method provided in Appendix A which is designed for sampling combustion off-gases. The sample gas at the Getty site was relatively free of particulate material and was at ambient temperature (approximately 32F°).

The sampling and analytical methods used for nitrogenated compound determinations is summarized in Appendix B. The method presented there is designed for industrial hygiene investigations. The sample apparatus

TABLE 5.1

## TARGET COMPOUNDS IN GAS STREAMS AND PLANNED SAMPLING METHODS

Compound	Adsorbtion Media
Acetaldehyde	DNPH
Acrolein	DNPH
Acrylonitrile	Thermosorb <sup>™</sup> /N
Allyl Chloride	Charcoal
Benzyl Chloride	Tenax <sup>®</sup>
Carbon Tetrachloride	Tenax <sup>®</sup>
Chlorobenzene	Tenax <sup>®</sup>
Chloroform	Tenax <sup>®</sup>
Chloroprene	Tenax <sup>®</sup>
o-, m-, p-Cresol	XAD-2
p-Dichlorobenzene	XAD-2
Dimethyl Nitrosamine	Thermosorb <sup>™</sup> /N
Dioxin	XAD-2
Epichlorohydrin	Tenax <sup>®</sup>
Ethylene Dichloride	Charcoal
Formaldehyde	DNPH
Hexachlorocyclopentadiene	XAD-2
Methyl Chloroform	Tenax <sup>®</sup>
Methylene Chloride	Charcoal
Nitrobenzene	Thermosorb <sup>™</sup> /N
Nitrosomorpholine	Thermosorb <sup>™</sup> /N
Perchloroethylene	Tenax <sup>®</sup>
Phenol	XAD-2
Polychlorinated Biphenyls	XAD-2
Toluene	Charcoal
Trichloroethylene	Tenax <sup>®</sup>
Vinylidene Chloride	Charcoal
o-, m-, p-Xylene	Charcoal
Benzene	Charcoal
Vinyl Chloride	Charcoal

was modified slightly using teflon fittings to allow attachment to the sample manifold installed at each of the three gas sample sites. Heat tape was used to heat the manifold to 120°F to prevent condensation upstream of the tube. An EPA Reference Method 6 control module was used to control and measure sample volumes.

Three Tenax® and one Tenax®/charcoal tubes were used in series for that system. Each Tenax®-GC cartridge contains approximately 1.6 grams of sorbent. The reported 100% saturation level of the material for several compounds are listed below:

<u>Compound</u>	<u>Micrograms/g</u>
n-Hexane	93
n-Octane	900
n-Decane	18,200
Benzene	198
Toluene	3,030
p-Xylene	1,680
Ethylbenzene	3,700
n-Propylbenzene	76,400

<sup>a</sup> "Characterization of Sorbent Resins for Use in Environmental Sampling", U.S. EPA, IERL, EPA-600/7-78-054, March 1978.

If the two extreme values for n-hexane and n-propylbenzene are considered not representative, an average saturation value of approximately 4,600 micrograms per gram results. For a single tube containing 1.6 grams of Tenax®, a maximum of 7,400 micrograms of hydrocarbons can be collected.

As discussed in Section No. 1, the expected loadings at the feed and CO<sub>2</sub> vent locations were 24 and 3 grams of non-methane hydrocarbon per cubic meter, respectively. These loadings indicated that the maximum sample volumes for the feed and CO<sub>2</sub> vent streams through a single Tenax®-GC module would be 0.3 and 2.5 liters, respectively. In order to allow an increase of the feed gas sample volume and to provide a margin of safety against overloading the Tenax®-GC sorbent, the test procedures used included three 1.6 gram Tenax®-GC modules in series followed by a Tenax®/charcoal module (1 gram of each). This provided a total of 5.8 grams of Tenax® which has a maximum adsorption capacity of approximately 27 milligrams of hydrocarbon based on the assumptions made above. The resulting maximum sample volumes would then be 1 and 9 liters at the feed and CO<sub>2</sub> vents, respectively. The actual sample volumes used ranged from approximately 0.3 to 3.0 liters at the feed gas site and 1 to 8 liters at the CO<sub>2</sub> Vent. This sample volume reduction and the 1 gram of charcoal in the backup tube were assumed to provide a safety margin. According to the manufacturers specifications (Appendix C), charcoal has a saturation point at approximately 100 milligrams per gram of sorbent.

Since there is no specific information on the non-methane hydrocarbon loading of the sale gas stream, the sample volumes at that site were varied from 0.5 to 18 liters.

Appendix C summarizes a NIOSH sampling method (P&CAM 127) which was used as a guide for collecting the charcoal tube samples. The metering system used met EPA Reference Method 6 specifications. The NIOSH method describes lower detection levels and minimum and maximum sample volumes. These limits are based on OSHA standards which did not necessarily represent the concentrations to be encountered. Breakthrough levels for five compounds are presented below. These were based on the OSHA standard and the maximum sample volume at levels five times the standard and the compound molecular weight.

	<u>OSHA Standard (ppm)</u>	<u>Maximum Sample Volume (liters)</u>	<u>Mole- cular Weight</u>	<u>Maximum Collectable (mg)</u>
Acetone	1000	7.7	58	94
Benzene	10	55	78	9
Carbon Tetrachloride	10	60	80	10
Ethylene Dichloride	50	12	97	12

The above maximum collectable amounts are on a 150 mg charcoal tube. The current sampling effort used 600 milligram charcoal tubes which have a capacity of 60 milligrams of hydrocarbons as reported by the manufacturer.

XAD-2 resin was used for collection of dioxins and PCB's. The XAD-2 module size is designed to allow collection of a large sample volume. No filter was used in the train since particulate loading was not of interest. The XAD-2 module was attached directly to the heated sample manifold.

The XAD-2 module contains approximately 30 grams of resin. Saturation levels for several compounds on XAD-2 are provided below:

<u>Compound</u>	<u>Micrograms/g of XAD-2<sup>a</sup></u>
n-Hexane	269
n-Octane	10,700
n-Decane	121,000
Benzene	170
Toluene	990
p-Xylene	4,000
Ethylbenzene	249
n-Propylbenzene	23,000

<sup>a</sup> "Characterization of Sorbent Resins for Use in Environmental Sampling", U.S. EPA, IERL, EPA-600/7-78-054, March 1978.

An average saturation value determined after elimination of the extreme values for n-decane and benzene is approximately 6,500 micrograms per gram of XAD-2 resulting in a total capacity per 30 gram module of approximately 195 milligrams. Based on the estimated loadings of the feed gas and CO<sub>2</sub> vent, maximum sample volumes of approximately 8 and 65 liters, respectively, can be collected through a single module before overloading occurs at those two sites. Sample rates of 0.5 to 1 liter per minute were used for this procedure. Sample volumes ranged from 1 to 6 liters at the feed gas site and from 4 to 40 liters at the CO<sub>2</sub> vent. The sample volume at the sale gas site was varied between 1 and 40 liters. Since the gas volumes to be measured were much lower than those recommended by Method 5, the metering device was replaced with a Method 6 type sample console.

Charcoal and Thermosorb<sup>™</sup>/N tubes were prepared by the respective manufacturers and sealed. These units remained sealed until immediately prior to sampling.

Tenax<sup>®</sup>, XAD-2, and DNPH adsorbents were prepared by the subcontractor laboratory and shipped by air to ES just prior to shipment of the test equipment to the test site.

#### ANALYTICAL PROCEDURES

Most of the analytical procedures used are summarized in Test Methods for Evaluating Solid Waste Physical /Chemical Methods, U.S. EPA Office of Solid Waste and Emergency Response, SW-846, July 1982.

The methods employed and the compounds of interest are listed in Table 5.2.

The Tenax<sup>®</sup> and XAD-2 samples were desorbed by SW-846 Methods 5030 and 3540, respectively. Charcoal media samples were desorbed and analyzed according to NIOSH P&CAM 127. Thermosorb<sup>™</sup>/N samples were analyzed according to the procedures specified in Appendix B, as previously mentioned.

One set of Tenax<sup>®</sup> modules from each of the feed gas and CO<sub>2</sub> vent test sites was analyzed individually so that loadings for each module could be determined. The remaining sets of modules were analyzed with the first three tubes as one sample fraction and the Tenax<sup>®</sup>/charcoal tube as a second fraction.

Tenax<sup>®</sup> and XAD-2 modules were cleaned according to procedures in SW-846. DNPH solution was prepared by the subcontract laboratory. Blanks of each of the sample media prepared by Radian was analyzed and determined to be free of contamination prior to shipment to ES.

TABLE 5.2

## ANALYTICAL PROCEDURES AND COMPOUNDS OF INTEREST

Method Description	Method Number	Method Source	Sample Media	Compounds of Interest
Purge and Trap	5030	SW-846	Tenax®	N.A. - Desorbition Procedure
Soxhlet Extraction	3540	SW-846	XAD-2	N.A. - Desorbition Procedure
HPLC Analysis	8310	SW-846	DNPH and Liquid	Acetaldehyde, Acrolein, Formaldehyde
GC/MS	8270	SW-846	XAD-2 and Liquid	o-, m-, p-Cresol, Phenol
GC/ECD	8080	SW-846	XAD-2 and Liquid	p-Dichlorobenzene, Dioxins, Hexachlorocyclopentadiene, Polychlorinated Biphenyls
GC/MS	8240	SW-846	Tenax® and Liquid	Benzyl Chloride, Carbon Tetrachloride, Chlorobenzene, Chloroform, Chloroprene, Epichlorohydrin, Methyl Chloroform, Perchloroethylene Trichloroethylene
GC/FID	P&CAM 127	NIOSH	Charcoal and Liquid	Allyl Chloride, Ethylene, Dichloride, Methylene Chloride, Toluene, Vinylidene Chloride, o-, m-, p-Xylene, Benzene, Vinyl Chloride
GC/TEA	Not Assigned	Thermosorb Sampler Instructions (Appendix B)	Thermosorb™/N	Acrylonitrile, Dimethyl Nitrosamine, Nitrobenzene, Nitrosomorphdine
AA	7060	SW-846	Liquid	Arsenic
AA	7090	SW-846	Liquid	Beryllium
AA	7130	SW-846	Liquid	Cadmium
AA	7190	SW-846	Liquid	Chromium
AA	7470	SW-846	Liquid	Mercury
AA	7520	SW-846	Liquid	Nickel
AA	303A	Standard Methods for the Examination of Water and Wastewater	Liquid	Manganese

## CALIBRATION PROCEDURES

### Field Sampling Equipment

Gas sampling metering systems met standard EPA Reference Method 5 (XAD-2 samples) and EPA Reference Method 6 (Tenax®, DNPH, charcoal, and Thermosorb™/N) requirements. These units were calibrated according to Reference Methods 5 and 6 (40 CFR 60 Appendix A) procedures and APTD-0576. Sampling equipment was calibrated within two weeks of the field test program. Calibration was performed both before and after the test program.

### Laboratory Instruments

All instruments used for analysis of samples collected during this program were calibrated with standard solutions of the compounds of interest. General calibration procedures are described in the specific methods listed in Table 5.2. Stock standard solutions for atomic adsorption analyses were prepared as described in the SW846 procedures under the reagent section. Appropriate dilutions were made for AA analyses which bracket the sample concentrations.

Upon receipt by the laboratory, the Tenax® cartridges were spiked with an internal standard and thermally desorbed at 180-200°C with organic-free nitrogen in a thermal desorption unit. The desorbed sample gas was bubbled through 5 ml of organic free water and trapped on an analytical sorbent trap.

After the 10 minute sample accumulation, the analytical sorbent trap was heated to 180°C and the carrier gas flow reversed so that the effluent from the analytical trap was directed into the GC/MS. The volatile compounds were separated by temperature programmed gas chromatography and detected by low resolution mass spectrometry. The concentrations were calculated using the internal standard technique. Details of the purge and trap GC/MS analysis are described in EPA SW846 Method 5030. Included are calibration procedures.

Calibration standards were prepared at three concentration levels for several compounds of interest to bracket the expected sample concentrations. The calibration standards were prepared by spiking a blank trap with a methanolic solution of the calibration standard (including an internal standard) using the flash evaporation technique. The trap was analyzed according to the purge and trap chromatographic procedures described in SW846 5030. Calibration curves are presented in Appendix D of this report.

After analysis of the calibration standards, the area response of the characteristic ions of each analyte were tabulated against the concentration of each and the internal standard. A response factor (RF) was calculated for each calibration compound by:

$$RF = A_S C_{IS} / A_{IS} C_S$$

where:  $A_S$  = area of the characteristic ion for the analyte to be measured  
 $A_{IS}$  = area of the characteristic ion for the internal standard  
 (characteristic ion for PFB is  $M/Z = 186$ )  
 $C_{IS}$  = amount (ng) of the internal standard  
 $C_S$  = amount (ng) of the volatile in calibration standard

After the sample cartridges are analyzed, the amount of a specific analyte in the cartridge is calculated by:

$$\text{Amount of Compound} = A_S C_{IS} / A_{IS} RF$$

where:  $A_S$  = area of the characteristic ion for the analyte to be measured  
 $A_{IS}$  = area for the characteristic ion of the internal standard  
 $C_{IS}$  = amount (ng) of internal standard

Results were tabulated in ng/cartridge as presented in Appendix D.

XAD and charcoal tubes are desorbed using solvent extraction. Samples were analyzed on gas chromatographs equipped with FID and ECD. These samples were also spiked with an internal standard as discussed above for the Tenax® tubes.

Nitrogenated compounds were analyzed using a Thermal Energy Analyzer detector. Calibration principles were similar to that used for GC/MS except that no internal standard was injected onto the sorbent. The laboratory report of results is presented in Appendix E.

Calibration of the HPLC was done in a similar manner to the Tenax® samples. Both internal and external standards were used.

Specific compounds to be used for the primary standards for all instruments were determined after an initial screening of the samples was made. Target components were identified during the screening process. Drift was compensated for by the relative response of internal standards.

#### DATA REDUCTION, VALIDATION, AND REPORTING

Process gas volumes obtained from the plant personnel were at standard conditions and dry.

Sample gas volumes were corrected as follows:

$$V_m(\text{STD}) = V_m Y \frac{T_{\text{STD}}}{T_m} \frac{P_m}{P_{\text{STD}}}$$

where:  $V_m(\text{STD})$  = volume of the meter corrected to standard conditions  
 $V_m$  = volume of the meter at meter conditions  
 $Y$  = dry gas meter calibration factor



$T_m$  = the temperature of the gas meter  
 $P_m$  = pressure of the gas meter

The laboratory data were submitted as component concentrations per sample, final calculations to determine gas concentration were as follows:

$$C_x = \frac{C_s}{V_m(\text{STD})} \times \frac{35.3 \text{ cubic feet}}{\text{cubic meter}}$$

where:  $C_x$  = component concentration in micrograms per cubic meter  
 $C_s$  = component concentration in micrograms per sample  
 $V_m(\text{STD})$  = sample volume in standard cubic feet

Pollutant concentrations were then converted to parts per million by volume as follows:

$$\text{ppm}_v = C_x \frac{0.024}{\text{MW}}$$

where: MW = component molecular weight

#### DATA VALIDATION

During sample collection, sample recovery and analysis, notes were maintained on the respective data sheets regarding any events which were observed that may affect the results in adverse or unusual ways. These observations included poor post-test leak checks, sample spillage, process upsets, excursions from routine sampling, or analytical procedures and others.

The laboratory report includes the results of analyses of spiked samples, surrogate sample recovery, external standard curves, and internal standard response.

All records of instrument calibrations, sample collection, recovery and analysis, and computerized and manual calculations will be maintained for a minimum of two years following completion of the final report and formal acceptance of the same by EPA.

## CHAPTER 6

### INTERNAL QUALITY CONTROL CHECKS

The methods that are used are retested during the measurement process by analysis of reagent quality (blanks), spiked samples, duplicates (splits), and synthetic or reference standards. These samples are prepared and submitted for analysis by someone other than the person performing the analysis.

TENAX®, CHARCOAL, XAD, DNPH, AND NITROGENATED HC TUBES

#### Blanks

Analysis of blanks for each type of media were used to demonstrate the absence of field or lab contamination, or the level at which it occurred, and were analyzed with the samples. Four types of blanks were analyzed:

- o Baseline blank - a sorbent tube that was analyzed immediately after conditioning. No results of these blanks are presented here, if contamination was found, samples were reconditioned and the baseline blank checked again. The cycle was repeated until satisfactory results were obtained.
- o Lab blank - a sorbent tube remained in the laboratory and was analyzed with the samples.
- o Field blank - a sorbent tube was opened at the field site, capped, and returned to the laboratory. DNPH solution, used for aldehyde sampling, was transferred to clean impingers and then recovered into sample bottles.
- o Trip blank - a sorbent tube that went from the laboratory to the field and back unopened and was analyzed with the samples.

The charcoal and Thermosorb™/N sample media were prepared by commercial suppliers of those media. The baseline and laboratory blanks were not included for those sorbents.

#### Splits

Two sample trains were operated in parallel to collect duplicate samples on all sample media. The results were used to represent reproducibility of the overall sampling and analytical technique.

### Spiking

Prior to analysis, each sorbent tube was spiked with an internal standard as an instrument calibration check.

### PERFORMANCE AND SYSTEMS AUDITS

The performance audits are an evaluation of the entire sample collection, recovery, and analytical system. Two sets of the internal QC checks described in Section No. 9 provided some measure of system performance. These include the duplicate samples. Duplicate samples provided a measure of reproducibility in data acquisition.

The EPA Project Office obtained an audit cylinder from the Quality Assurance Division of the Emission Monitoring Systems Laboratory in Research Triangle Park, North Carolina. The audit cylinder contains five compounds included in Table 5.1. The compounds are benzene, carbon tetrachloride, chloroform, perchloroethylene and vinyl chloride and were analyzed from the samples collected on Tenax® and charcoal. ES collected two samples of the audit gas on each of these two types of media. The audit samples were collected at the test site during the field sampling program. The true concentration of the compounds have not been revealed to ES.

APPENDIX A  
ALDEHYDE DNPH METHOD

## SAMPLING PARAMETERS AND METHODOLOGY FOR ALDEHYDE COLLECTION

### INTRODUCTION:

Three different absorbing solutions will be used in a midjet impinger train to collect aldehydes from incinerator sources.

The sampling methodology (i.e., flow rates, sample volumes, probe, and filter temperatures) will remain the same, while recovery procedures vary and are specified in the attached procedures for each method.

### I. FACTORS COMMON TO ALL METHODS

A. Sampling train design is identical to EPA Method 6

B. Supplies and Equipment

1. Sampling - midjet impingers, u-tubes, ice bath, vacuum pump capable of 2-3 l/min flowrate, heated filtration device, flowmeter, gas meter, chilled reagents.
2. Sample recovery - sample bottles (60 or 100 ml vol), chilled reagents, d.H<sub>2</sub>O

C. Sampling Parameters

1. Two-holed, 4" port caps to permit simultaneous runs from the same sampling port
2. Probe and filter temperatures to be maintained at 350°F±5°
3. Filter material - quartz
4. Flow rate - 1 to 1.5 l/min
5. Sample volume - collect ~ 1-2 ft<sup>3</sup>
6. Purging - all samples to be purged for approximately 1/2 the total sampling time

7. Samples for each run to include: probe, filter, filter number, first impinger, second impinger, third impinger, reagent blank (note: for each run)
8. Reagents to be kept on ice
9. Following recovery, samples to be kept on ice until analysis
10. Monitor: flow rate, total sample volume, probe temperature, filter temperature, stack temperature

#### Special Notes

1. Labelling system must be waterproof to endure storage in ice chests with H<sub>2</sub>O for long periods of time.
2. Sufficient levels of ice in ice bath must be maintained during sampling.
3. Probe and filter temperatures must be maintained at the prescribed level.

## II. METHODS

### A. 2,4-Dinitrophenylhydrazine (DNPH)

Principle: Aldehydes in the sample gas react to the DNPH in 2NHCl to form soluble and insoluble 2,4-DNP-hydrazone derivatives.

#### 1. Preparation of DNPH (Eastman Kodak No. 1866)

To a 1 l volumetric flask containing 500 ml distilled H<sub>2</sub>O, add 163 ml of concentrated HCl and 2.5 gm DNPH crystals. It will be necessary to utilize a magnetic stirrer and TFE stir bar for approximately 1 hr to effect partial solution. After this time, fill to the mark with d.H<sub>2</sub>O, mix well, and filter the DNPH through Whatman No. 1 filter paper.

#### 2. Clean-up of DNPH

The filtered DNPH solution must be solvent-extracted in order to remove interfering peaks from the HPLC traces. This is carried out by using a 2 l separatory funnel, into which the entire 1 l of

DNPH solution has been added, plus 100 ml of chloroform (Burdick and Jackson - HPLC quality). The mixture is shaken for 5 minutes; the layers allowed to separate, and the  $\text{CHCl}_3$  drained off. Five additional, 100 ml  $\text{CHCl}_3$  extractions are then performed, with the final (6th) extraction standing overnight to affect total separation of layers. For each batch of DNPH prepared, two 50 ml portions of the  $\text{CHCl}_3$  and two 50 ml portions of the DNPH will be collected in sample bottles, and labeled "blank," with appropriate date, run #, etc. At the conclusion of the DNPH sampling, additional blanks of the  $\text{CHCl}_3$  and DNPH will be collected.

### 3. Stability and storage limits of DNPH

After the DNPH solution is initially prepared, filtered, and extracted with  $\text{CHCl}_3$  (in the same day), the solution must be kept near  $0^\circ\text{C}$  until used for the sample run. Discard the solution if crystals begin to form. Stability of this solution usually does not exceed 8 days.

### B. Sodium Bisulfite (Fisher No. S-654)

Principle: Carbonyls are collected in the bisulfite absorbing medium and form stable bisulfite addition compounds.

A 1% solution is prepared by dissolving 5 gm  $\text{NaHSO}_3$  in 500 ml d. $\text{H}_2\text{O}$

### C. Basic Peroxide

Principle: Derivatives of the aldehydes are to be analyzed by ion chromatography.

A 0.05N NaOH solution is prepared using 2 gm NaOH to 1 l of 3%  $\text{H}_2\text{O}_2$

## SAMPLING FLOW CHART

- a. 10 ml of reagent are placed in each of the first 3 impingers, with the fourth impinger left dry to protect the pump (follow sampling parameters on Page 1).
- b. After collection of the desired sample volume, the system is to be purged, by pulling ambient air through an activated charcoal filter connected to the front end of the impinger train. (The probe and filter have been disconnected at this point.) The charcoal filter can be simply constructed by using a common plastic drying tube fitted with glass wool plugs in each end filled with activated charcoal, and with an appropriately-sized ground glass ball joint fitted to the front end of the impinger train. Total Purge time should be equivalent to ~ 1/2 the total sampling time.
- c. The probe, filter, and filter holder are to be recovered separately, using the specific reagent for that run. Following the collection of these samples, it will be necessary to "dry" the probe and filter holder with a solvent such as 100% isopropanol to prepare for the following run.

DNPH	BISULFITE	BASIC H <sub>2</sub> O <sub>2</sub>
<p>d. Midget impingers are to be recovered <u>separately</u> using a small amount of d.H<sub>2</sub>O</p> <p>e. Each impinger is then rinsed with approximately 5 ml of CHCl<sub>3</sub>, collecting this CHCl<sub>3</sub> wash in the appropriate sample bottle.</p>	<p>d. Midget impingers are to be recovered together (combined) in one sample bottle, rinsing each impinger with a small amount of d.H<sub>2</sub>O, and adding the rinsings to the sample bottle.</p>	<p>d. Midget impingers are to be recovered together (combined) in one sample bottle, rinsing each impinger with a small amount of d.H<sub>2</sub>O and adding the rinsings to the sample bottle.</p>
<p>e. Impingers must be cleaned after each recovery using a small amount of chloroform, followed by a rinse using DNPH.</p>	N/A	N/A



APPENDIX B

THERMOSORB™/N SAMPLING AND ANALYTICAL PROCEDURES

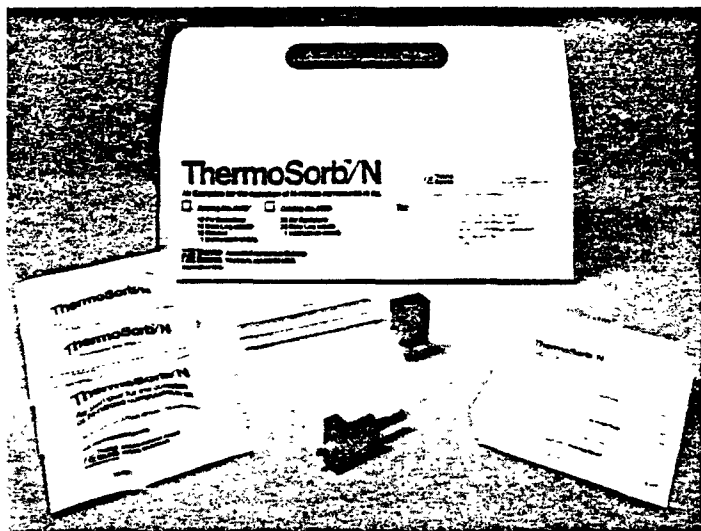
# ThermoSorb/N Air Sampler

## Instructions for Monitoring

### Introduction

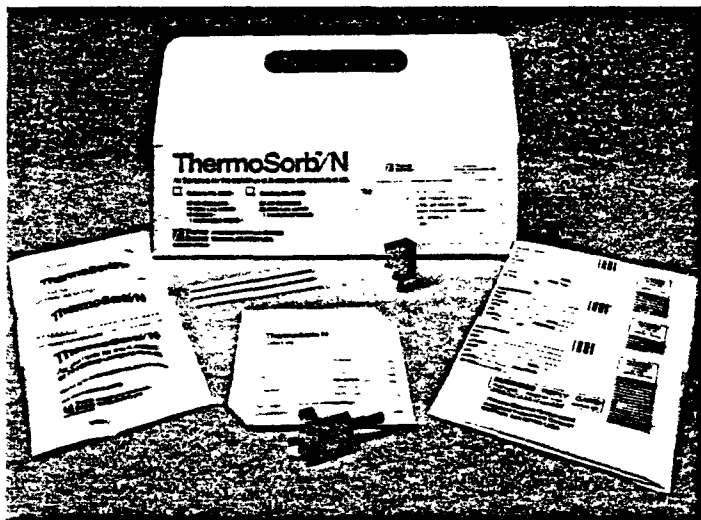
Air is drawn through a proprietary sorbent with a suitable air sampling pump. The N-nitroso compounds are absorbed with high efficiency. After sampling is complete, the sorbent is eluted with solvent to remove the N-nitroso compounds. The solvent is then analyzed by combined gas-liquid chromatography with TEA Analyzer. Detection limits of better than 0.05  $\mu\text{g}/\text{m}^3$  are possible when sampling for one hour at 2.0 L/min.

The ThermoSorb/N air sampler contains:



**Catalogue Number 6533**  
**Contents**

- 20 ThermoSorb/N air samplers in foil pouches
- 20 Foil pouch clips
- 20 Data log work sheets
- 1 Instruction sheet



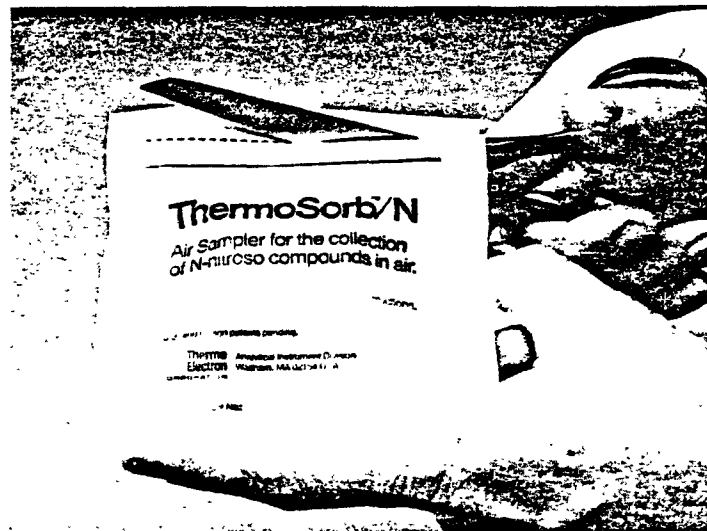
**Catalogue Number 6525**  
**Contents**

- 10 ThermoSorb/N air samplers in foil pouches
- 10 Foil pouch clips
- 10 Data log work sheets
- 10 Mailing envelopes for
- 10 Analyses at the Analytical Services
- Laboratory of Thermo Electron Corporation
- 1 Instruction sheet

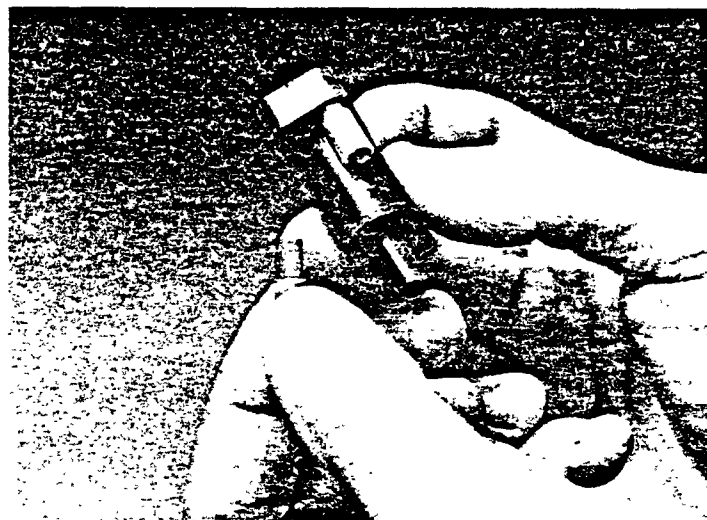
### Other Equipment Needed for Monitoring:

1. Air sampling pump (high flow or low flow)
2. Battery charger
3. Pump calibration — soap bubble tower
4. Tubing, 1/4 in., flexible
5. Stopwatch
6. Scissors

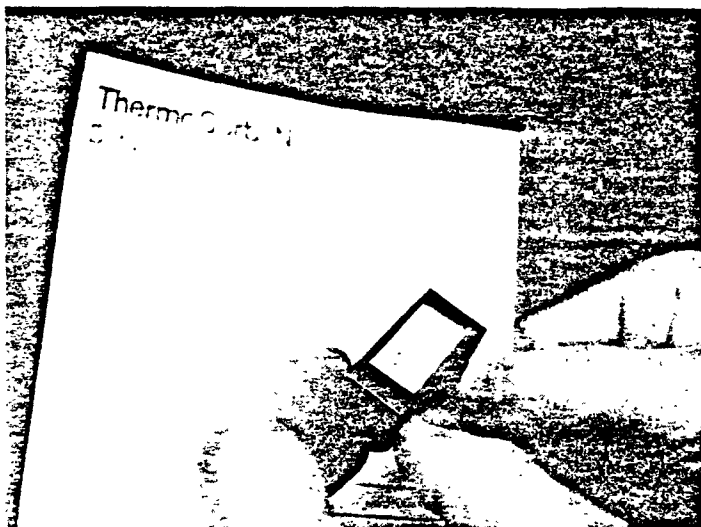
### Preparation Before Sampling:



1. Remove the ThermoSorb/N air sampler from the foil pouch. Use scissors to cut open the foil pouch. Save the foil pouch for re-use.



2. Remove the red end caps from the inlet and outlet ports. The red caps can be stored on the ThermoSorb/N air sampler in the brackets under the "AIR IN" sign.



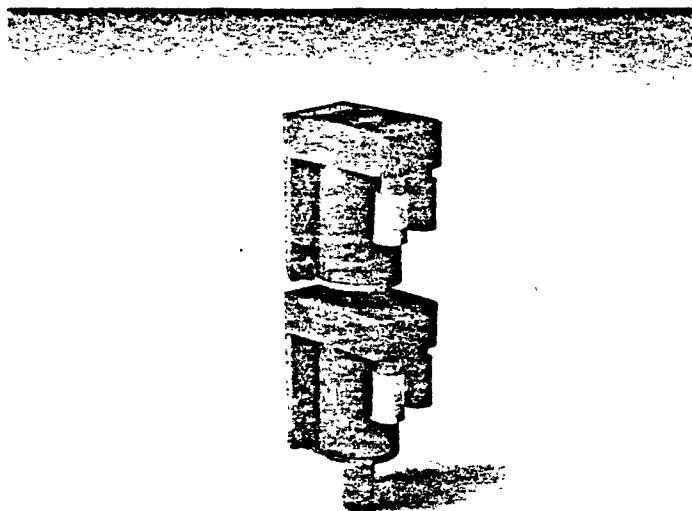
3. Label the ThermoSorb/N air sampler with the peel-off "Air Sampler" label provided on the "Data Log" worksheet. The molded clip of the ThermoSorb/N air sampler provides a flat surface to affix the label.
4. Attach the ThermoSorb/N air sampler to the sampling pump using an appropriate length of 1/4 in. flexible tubing.
5. Calibrate the pump with the ThermoSorb/N air sampler attached. Use a stopwatch and bubble tower to determine the air flow. A 2.0 L/min flow rate is suggested for general monitoring. Flow rates for the ThermoSorb/N air sampler can vary between 0.2 L/min to 4.0 L/min without affecting collection efficiency.
6. Record the air flow and all other appropriate data on the "Data Log" worksheet. The "Data Log" worksheet can be readily applied to a laboratory notebook page.

#### Sampling:

1. Attach the ThermoSorb/N air sampler



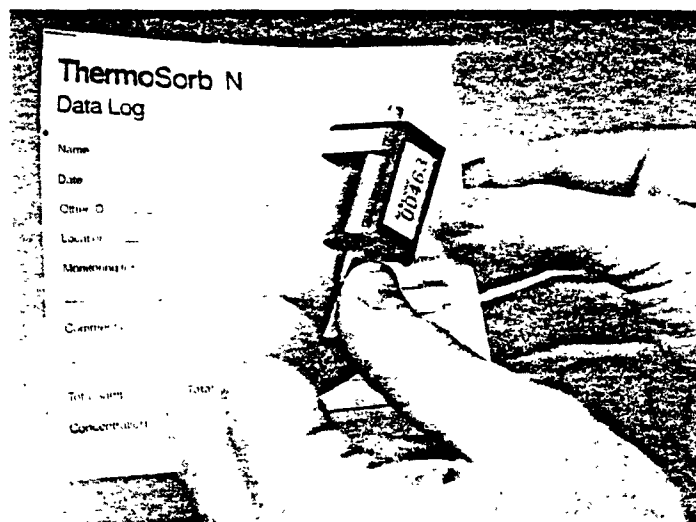
- a) In the breathing zone of the worker to be monitored. The molded clip attaches easily to pockets or collars.
- b) Near the process to be monitored. The molded clip provides a flat surface so that the ThermoSorb/N air sampler can be easily oriented toward the area of interest.
- c) On the pump, in the area to be monitored.
2. Sample for an appropriate period of time. 100L of air total volume is the recommended sample size.
  - a) For time weighted averages (TWA's) use 0.2 L/min for 8 hrs.
  - b) For process sampling use 2.0 L/min for 50 min or 4.0 L/min for 25 min.



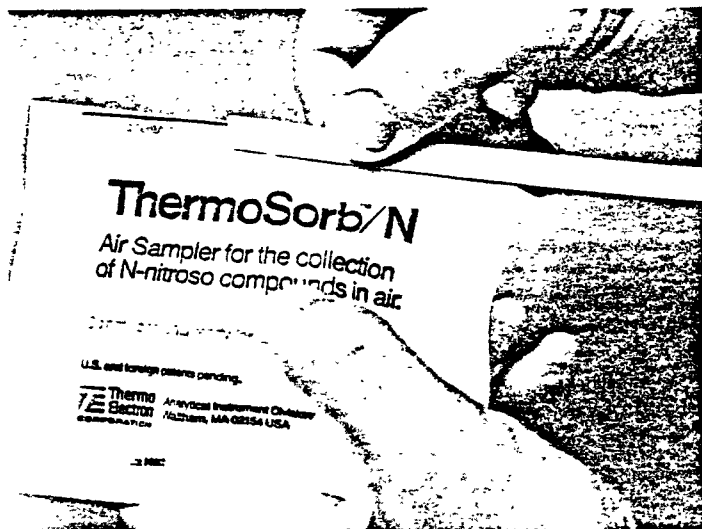
3. If high concentrations of nitrosamines are expected (i.e., over 1500  $\mu\text{g}$ ) use another ThermoSorb/N air sampler as a "back-up" section.
4. Note changes in monitoring conditions (the "Air Sampler" label on the "Data Log" worksheet can be used for notes in the field), for example:
  - a) Obstructions in the ThermoSorb/N air sampler.
  - b) Changes in flow rate.
  - c) Changes in ambient temperature, barometric pressure, or relative humidity.
5. Remove the ThermoSorb/N air sampler from the monitoring site.

#### After Sampling:

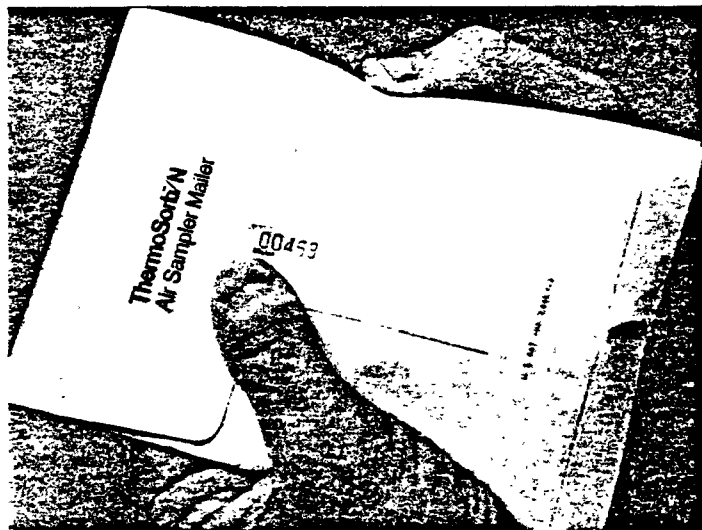
1. Calibrate the pump with the ThermoSorb/N air sampler attached.
2. Detach the ThermoSorb/N air sampler from the pump.
3. Replace the red end caps on the inlet and outlet ports of the ThermoSorb/N air sampler.
4. Record the appropriate data, be sure to include the flow rate after sampling.



5. Affix the "Analysis Vial" label from the "Data Log" worksheet to the ThermoSorb/N air sampler. This can be done on the side of the "AIR IN" sign. The laboratory will then be able to label the analysis vial with a label numbered the same as the ThermoSorb/N air sampler.



6. Replace the ThermoSorb/N air sampler in the foil pouch. Fold the pouch and seal it with the clip provided.
7. Place the sealed foil pouch into a mailer and submit for analysis.



8. Affix the "Mailer Label" from the "Data Log" worksheet as a seal on the back flap of the mailer.

#### Analysis by Thermo Electron:

1. The Analytical Services Laboratory at Thermo Electron Corporation will report results of analysis within 3 working days after the sample is received.
2. The Laboratory reports results in nanograms.
3. Calculate the concentration of the substance monitored by dividing the results in nanograms by the average volume of air sampled in liters. Results will be in  $\mu\text{g}/\text{m}^3$ .

For information on analysis, request publication IS-33, "ThermoSorb/N Air Sampler Analysis Instructions".

For further information:

Call: (617) 890-8700

Write: Thermo Electron Corporation  
Analytical Instruments  
Waltham, MA 02154 U.S.A.

Telex: 92-3473

Ask for Order Entry, Analytical Instruments, regarding purchase of additional ThermoSorb/N air samplers.

Ask for ThermoSorb/N Customer Service for questions regarding air monitoring.

Ask for Analytical Services Laboratory for questions regarding analysis of the ThermoSorb/N air samplers.

# ThermoSorb/N Air Sampler Analysis Instruction

## Introduction

Air is drawn through a proprietary sorbent with a suitable air sampling pump. The N-nitroso compounds are absorbed with high efficiency. After sampling is complete, the sorbent is eluted with solvent to remove the N-nitroso compounds. The solvent is then analyzed by combined gas-liquid chromatography with TEA Analyzer. Detection limits of better than 0.05  $\mu\text{g}/\text{m}^3$  are possible when sampling for one hour at 2.0 L/min.

## Equipment Needed:

1. Glass syringe, 5.0 ml with male luer adapter.
2. Industrial blunt needle, 20 gauge with female luer adapter.
3. Auto sampler vial, 1.8 ml with Teflon coated crimp tops.
4. Crimping tool.
5. Assorted laboratory glassware.
6. Single column, temperature programmable gas chromatograph and accessories (auto sampler and integrator are optional).
7. TEA Analyzer (Thermo Electron Corporation).
8. Helium, chromatographic grade.

## Reagents Needed:

1. A mixture of 25% methanol, analytical grade in 75% dichloromethane, analytical grade.  
**NOTE:** Dichloromethane is irritating to the eyes, can cause dermatitis upon prolonged contact, is a mild narcotic, a weak mutagen and a suspect carcinogen.  
Methanol affects the nervous system and chronic exposure may cause headaches, dizziness, dermatitis, the feeling of intoxication, and blurred vision.  
These are dangerous chemicals, large doses can be fatal.
2. Nitrosamine Standards, 1 ng/ $\mu\text{l}$  solutions (available from Thermo Electron Corp., Analytical Service Laboratory).

## Preparation of Sample:

1. Remove the ThermoSorb/N air sampler from the mailer and foil pouch. (see Fig. 1)
2. Label analysis vial with the label on the ThermoSorb/N air sampler. (see Fig. 2)
3. Remove the red end caps, store them in the bracket under the "AIR IN" sign. (see Fig. 3)
4. Attach a syringe needle to the male luer fitting of the ThermoSorb/N air sampler.
5. Attach a syringe barrel to the female luer fitting of the ThermoSorb/N air sampler.
6. Elute by "Backflushing" the ThermoSorb/N air sampler with the mixture of dichloromethane and methanol. Collect 1.5-1.8 ml of the eluent in a labelled auto sampler vial. The exact volume should be known. The optimum elution rate is 0.5 ml/min. A manual procedure is:
  - a) Add 1 ml of the dichloromethane/methanol to the syringe barrel attached to the ThermoSorb/N air sampler.
  - b) Allow the solvent to flow into the ThermoSorb/N air sampler.
  - c) If flow is not immediate, gently push the solvent through with the plunger of the syringe.
  - d) After 30 seconds, repeat steps a through c until 1.5-1.8 ml of sample is collected. (see Fig. 4)
7. Crimp the cap on the vial.

## Establish the following gas chromatographic conditions:

Inlet Temperature: 150°C  
Column Temperature: 140°C to 200°C at 4°C per min.  
Column: 10' long x 1/8" O.D. stainless steel  
Packing: 10% Carbowax 20M + 2% KOH on Chromosorb W-AW, 80/100 mesh  
Carrier: Helium @ 30 cc/min.

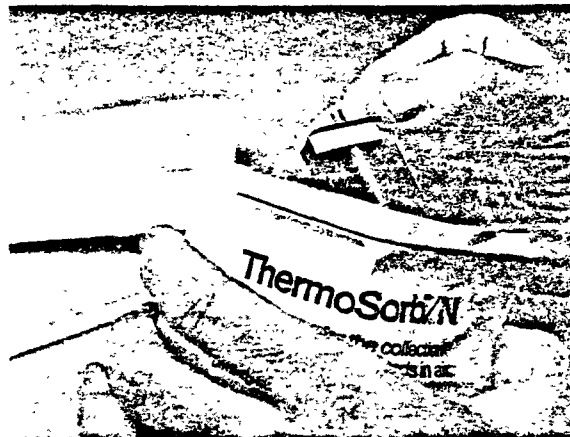


Figure 1



Figure 2



Figure 3

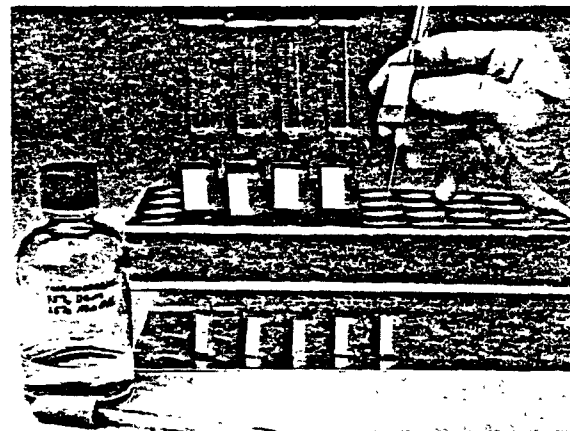


Figure 4

**Establish the following TEA Analyzer conditions:**

Pyrolyzer: 475°C  
Oxygen: 5 cc/min.  
Pressure: 1.0 TORR  
Attenuation: x4

**Analytical Procedure:**

1. Inject 4 µl aliquots of the N-nitroso standard of interest until acceptable reproducibility is obtained.
2. Inject 4 µl aliquots of the solvent eluted from the ThermoSorb/N air sampler, and calculate results as described below. Duplicate injections of samples are recommended.
3. To assure consistent results, the N-nitroso standard of interest should be injected periodically.

**Calculations:**

1. Calibration: A response factor, KF, is calculated as follows:

$$KF = \frac{PA}{N}$$

Where: P is the peak area resulting from injection of the standard.

A is the attenuation of the instrument.

N is the amount, in nanograms, of standard injected.

2. The volume of air sampled is:

$$V = \frac{Ft}{1000}$$

Where: V is the volume of air sampled in m<sup>3</sup>.

F is the flow rate in L/min.

t is the sampling time in min.

3. The concentration of nitrosamine in the air is:

$$C = \frac{paL}{1000 (KF)IV}$$

Where: p is the peak area resulting from injection of the sample.

a is the attenuation setting of the instrument.

L is the final volume of eluent in microliters (usually 1800 µl).

I is the volume injected, in microliters.

C is in µg/m<sup>3</sup>.

For information on monitoring, request publication IS-25, "ThermoSorb/N Air Sampler Instructions for Monitoring".

For further information:

Call: (617) 890-8700

Write: Thermo Electron Corporation  
Analytical Instruments  
Waltham, MA 02154 U.S.A.

Telex: 92-3473

Ask for Order Entry, Analytical Instruments regarding purchase of additional ThermoSorb/N air samplers.

Ask for ThermoSorb/N Customer Service for questions regarding air monitoring.

Ask for Analytical Services Laboratory for questions regarding analysis of the ThermoSorb/N air samplers.

The TEA Analyzer is covered by several foreign and one or more of the following U.S. patents: 3,973,910; 3,996,002; 3,996,003; 3,996,004; 3,996,008; 3,996,009; 4,066,411.

ThermoSorb air sampler has U.S. and foreign patents pending.

ThermoSorb and TEA are trademarks of Thermo Electron Corporation.

 **Analytical Instruments**

IS-33  
February 1980  
Printed in U.S.A.

## APPENDIX C

### CHARCOAL TUBE SAMPLING METHOD



# Charcoal Sample Collection Tubes

---

**for organic hydrocarbons**

**600 mg. size**

**part number 463072**

**store at temperature below 100°F.**

## **Description:**

These tubes are useful for sampling the many industrial solvents known to be absorbed and retained on charcoal for later analysis. They are not suitable for collecting strong oxidants, substances with high vapor pressures, or very polar compounds such as methanol and acetone. The activated charcoal which is used as the sample absorber in the tube has a very high collection efficiency for solvent vapors.

The sample is obtained by drawing a known volume of air through the tube. Each tube has two separate sections of active charcoal. The larger section is the test or sample portion and when properly used all of the solvent vapors will be absorbed in this section. The smaller section is used as a reference or blank.

After using a tube to obtain a solvent sample the two charcoal sections are analyzed separately. If any significant amount of sample is found in the reference it should be assumed that the absorption limit of the charcoal tube has been exceeded. In this case another sample should be collected with a fresh tube using a smaller sample volume and possibly a lower sample flow rate until an acceptable sample is obtained. (The saturation limit of the sample portion is approximately 60 milligrams of total sample which is roughly equivalent to a 40 liter sample of 500 parts per million concentration of total solvents). In some cases when sampling mixtures of solvents it may be impossible to obtain a clear blank. This may be due to the displacement of the more polar compound into the second section as the less polar one is absorbed in the first.

## **LIMITATIONS**

**Temperature.** Do not use for sampling air at temperatures above 125°F.

**Relative Humidity.** Collection efficiency is not affected by relative humidity below 95%.

(over)



#### TO PREPARE AND MAKE TEST:

1. Remove one charcoal sampling tube from the box. Break off both glass tips from the tube; CAUTION, wear safety glasses for protection against any glass particles which may be scattered by this procedure.
2. Install the sampling tube in the MSA Charcoal Tube Holder part number 463093.  
NOTE: The arrow on the tube indicates the correct direction of sample flow and should point toward the pump.
3. Connect the sampling line of the tube holder to a suitable pump or metered vacuum source and adjust the flow rate to obtain the desired sample period. For example: if a 20 liter sample is to be taken over a 100 minute period the flow rate should be 200 milliliters per minute.

NOTE: During the sample period the tube should be in a vertical position.

NOTE: The flow rate through the tube should not be over one liter per minute.

4. When the desired sample volume has passed through the tube shut off the sample flow. Remove the charcoal tube from the holder and install a plastic cap onto each end of the tube to protect the sample from contamination or loss until it is analyzed.

#### REFERENCES AND ANALYTIC PROCEDURES:

1. Kupel, Richard E., et al: Qualitative Detection Limits for Specific Compounds Utilizing Gas Chromatographic Fractions, Activated Charcoal and a Mass Spectrometer. American Industrial Hygiene Association Journal 32:383 (1971).
2. Kupel, Richard E., et al: A Convenient Optimized Method for the Analysis of Selected Solvent Vapors in the Industrial Atmosphere. American Industrial Hygiene Association Journal 31:225 (1970).
3. Halpin, Walter R. and Reid, Frank H.: Determination of Halogenated Hydrocarbons in Air by Charcoal Tube and Gas Chromatography. American Industrial Hygiene Association Journal 29:390 (1968).
4. Hermann, Edward R. and Fraust, Charles L.: Charcoal Sampling Tubes for Organic Vapor Analysis by Gas Chromatography. American Industrial Hygiene Association Journal 27:68 (1966).
5. Otterson, E. J. and Guy, C. U.: A Method of Atmospheric Solvent Vapor Sampling on Activated Charcoal in Connection with Gas Chromatography. Transactions of the Twenty-Sixth Annual Meeting of the American Conference of Governmental Industrial Hygienists, Philadelphia, Pa. page 37, American Conference of Governmental Hygienists, Cincinnati, Ohio (1964).

Manufactured by



A6711 (L) REV. 8

**MINE SAFETY APPLIANCES COMPANY**  
PITTSBURGH, PENNSYLVANIA, U.S.A., 15208

463091

# ORGANIC SOLVENTS IN AIR

## Physical and Chemical Analysis Branch

### Analytical Method

Analyte:	Organic Solvents (See Table 1)	Method No.:	P&CAM 127
Matrix:	Air	Range:	For the specific compound, refer to Table 1
Procedure:	Adsorption on charcoal desorption with carbon disulfide, GC		
Date Issued:	9/15/72	Precision:	10.5% RSD
Date Revised:	2/15/77	Classification:	See Table 1

#### 1. Principle of the Method

- 1.1 A known volume of air is drawn through a charcoal tube to trap the organic vapors present.
- 1.2 The charcoal in the tube is transferred to a small, graduated test tube and desorbed with carbon disulfide.
- 1.3 An aliquot of the desorbed sample is injected into a gas chromatograph.
- 1.4 The area of the resulting peak is determined and compared with areas obtained from the injection of standards.

#### 2. Range and Sensitivity

The lower limit in mg/sample for the specific compound at  $16 \times 1$  attenuation on a gas chromatograph fitted with a 10:1 splitter is shown in Table 1. This value can be lowered by reducing the attenuation or by eliminating the 10:1 splitter.

#### 3. Interferences

- 3.1 When the amount of water in the air is so great that condensation actually occurs in the tube, organic vapors will not be trapped. Preliminary experiments indicate that high humidity severely decreases the breakthrough volume.
- 3.2 When two or more solvents are known or suspected to be present in the air, such information (including their suspected identities), should be transmitted with the sample, since with differences in polarity, one may displace another from the charcoal.
- 3.3 It must be emphasized that any compound which has the same retention time as the specific compound under study at the operating conditions described in this method is an interference. Hence, retention time data on a single column, or even on a number of columns, cannot be considered as proof of chemical identity. For this reason it is important that a sample of the bulk solvent(s) be submitted at the same time so that identity(ies) can be established by other means.

TABLE 1  
Parameters Associated With P&CAB Analytical Method No. 127

Organic Solvent	Method Classification	Detection limit (mg/sample)	Sample Volume (liters)		GC Column Temp. (°C)	Molecular Weight	
			Minimum <sup>(a)</sup>	Maximum <sup>(b)</sup>			
Acetone	D		0.5	7.7	60	58.1	1000
Benzene	A	0.01	0.5	55	90	78.1	10
Carbon tetrachloride	A	0.20	10	60	60	154.0	10
Chloroform	A	0.10	0.5	13	80	119	
Dichloromethane	D	0.05	0.5	3.8	85	84.9	
p-Dioxane	A	0.05	1	18	100	88.1	
Ethylene dichloride	D	0.05	1	12	90	99.0	50
Methyl ethyl ketone	B	0.01	0.5	13	80	72.1	
Styrene	D	0.10	1.5	34	150	104	
Tetrachloroethylene	B	0.06	1	25	130	166	
1,1,2-trichloroethane	B	0.05	10	97	150	133	
1,1,1-trichloroethane (methyl chloroform)	B	0.05	0.5	13	150	133	
Trichloroethylene	A	0.05	1	17	90	131	
Toluene	B	0.01	0.5	22	120	92.1	200
Xylene	A	0.02	0.5	31	100	106	

(a) Minimum volume, in liters, required to measure 0.1 times the OSHA standard

(b) These are breakthrough volumes calculated with data derived from a potential plot (11.2) for activated coconut charcoal. Concentrations of vapor in air at 5 times the OSHA standard (11.3) or 500 ppm, whichever is lower, 25°C, and 760 torr were assumed. These values will be as much as 50% lower for atmospheres of high humidity. The effects of multiple contaminants have not been investigated, but it is suspected that less volatile compounds may displace more volatile compounds (See 3.1 and 3.2)

- 3.4 If the possibility of interference exists, separation conditions (column packing, temperatures, etc.) must be changed to circumvent the problem.

#### 4. Precision and Accuracy

- 4.1 The mean relative standard deviation of the analytical method is 8% (11.4).
- 4.2 The mean relative standard deviation of the analytical method plus field sampling using an approved personal sampling pump is 10% (11.4). Part of the error associated with the method is related to uncertainties in the sample volume collected. If a more powerful vacuum pump with associated gas-volume integrating equipment is used, sampling precision can be improved.
- 4.3 The accuracy of the overall sampling and analytical method is 10% (NIOSH-unpublished data) when the personal sampling pump is calibrated with a charcoal tube in the line.

#### 5. Advantages and Disadvantages of the Method

- 5.1 The sampling device is small, portable, and involves no liquids. Interferences are minimal, and most of those which do occur can be eliminated by altering chromatographic conditions. The tubes are analyzed by means of a quick, instrumental method. The method can also be used for the simultaneous analysis of two or more solvents suspected to be present in the same sample by simply changing gas chromatographic conditions from isothermal to a temperature-programmed mode of operation.
- 5.2 One disadvantage of the method is that the amount of sample which can be taken is limited by the number of milligrams that the tube will hold before overloading. When the sample value obtained for the backup section of the charcoal tube exceeds 25% of that found on the front section, the possibility of sample loss exists. During sample storage, the more volatile compounds will migrate throughout the tube until equilibrium is reached (33% of the sample on the backup section).
- 5.3 Furthermore, the precision of the method is limited by the reproducibility of the pressure drop across the tubes. This drop will affect the flow rate and cause the volume to be imprecise, because the pump is usually calibrated for one tube only.

#### 6. Apparatus

- 6.1 An approved and calibrated personal sampling pump for personal samples. For an area sample, any vacuum pump whose flow can be determined accurately at 1 liter per minute or less.
- 6.2 Charcoal tubes: glass tube with both ends flame sealed, 7 cm long with a 6-mm O.D. and a 4-mm I.D., containing 2 sections of 20/40 mesh activated charcoal separated by a 2-mm portion of urethane foam. The activated charcoal is prepared from coconut shells and is fired at 600°C prior to packing. The absorbing section contains 100 mg of charcoal, the backup section 50 mg. A 3-mm portion of urethane foam is placed between the outlet end of the tube and the backup section. A plug of silylated glass wool is placed in front of the absorbing section. The pressure drop across the tube must be less than one inch of mercury at a flow rate of 1 lpm.
- 6.3 Gas chromatograph equipped with a flame ionization detector.
- 6.4 Column (20 ft × 1/8 in) with 10% FFAP stationary phase on 80/100 mesh, acid-washed DMCS Chromosorb W solid support. Other columns capable of performing the required separations may be used.

- 6.5 A mechanical or electronic integrator or a recorder and some method for determining peak area.
- 6.6 Microcentrifuge tubes, 2.5 ml, graduated.
- 6.7 Hamilton syringes: 10  $\mu$ l, and convenient sizes for making standards.
- 6.8 Pipets: 0.5-ml delivery pipets or 1.0-ml type graduated in 0.1-ml increments.
- 6.9 Volumetric flasks: 10 ml or convenient sizes for making standard solutions.

## 7. Reagents

- 7.1 Spectroquality carbon disulfide (Matheson Coleman and Bell).
- 7.2 Sample of the specific compound under study, preferably chromatquality grade.
- 7.3 Bureau of Mines Grade A helium.
- 7.4 Prepurified hydrogen.
- 7.5 Filtered compressed air.

## 8. Procedure

- 8.1 **Cleaning of Equipment:** All glassware used for the laboratory analysis should be detergent washed and thoroughly rinsed with tap water and distilled water.
- 8.2 **Calibration of Personal Pumps.** Each personal pump must be calibrated with a representative charcoal tube in the line. This will minimize errors associated with uncertainties in the sample volume collected.
- 8.3 **Collection and Shipping of Samples**
  - 8.3.1 Immediately before sampling, the ends of the tube should be broken to provide an opening at least one-half the internal diameter of the tube (2 mm).
  - 8.3.2 The small section of charcoal is used as a back-up and should be positioned nearest the sampling pump.
  - 8.3.3 The charcoal tube should be vertical during sampling to reduce channeling through the charcoal.
  - 8.3.4 Air being sampled should not be passed through any hose or tubing before entering the charcoal tube.
  - 8.3.5 The flow, time, and/or volume must be measured as accurately as possible. The sample should be taken at a flow rate of 1 lpm or less to attain the total sample volume required. The minimum and maximum sample volumes that should be collected for each solvent are shown in Table 1. The minimum volume quoted must be collected if the desired sensitivity is to be achieved.
  - 8.3.6 The temperature and pressure of the atmosphere being sampled should be measured and recorded.
  - 8.3.7 The charcoal tubes should be capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.
  - 8.3.8 One tube should be handled in the same manner as the sample tube (break, seal, and transport), except that no air is sampled through this tube. This tube should be labeled as a blank.
  - 8.3.9 Capped tubes should be packed tightly before they are shipped to minimize tube breakage during shipping.

- 8.3.10 Samples of the suspected solvent(s) should be submitted to the laboratory for qualitative characterization. These liquid bulk samples should not be transported in the same container as the samples or blank tube. If possible, a bulk air sample (at least 50 l air drawn through tube) should be shipped for qualitative identification purposes.

#### 8.4 Analysis of Samples

- 8.4.1 Preparation of Samples. In preparation for analysis, each charcoal tube is scored with a file in front of the first section of charcoal and broken open. The glass wool is removed and discarded. The charcoal in the first (larger) section is transferred to a small stoppered test tube. The separating section of foam is removed and discarded; the second section is transferred to another test tube. These two sections are analyzed separately.
- 8.4.2 Desorption of Samples. Prior to analysis, one-half ml of carbon disulfide is pipetted into each test tube. (All work with carbon disulfide should be performed in a hood because of its high toxicity.) Tests indicate that desorption is complete in 30 minutes if the sample is stirred occasionally during this period.
- 8.4.3 GC Conditions. The typical operating conditions for the gas chromatograph are:
1. 85 cc/min. (70 psig) helium carrier gas flow.
  2. 65 cc/min. (24 psig) hydrogen gas flow to detector.
  3. 500 cc/min. (50 psig) air flow to detector.
  4. 200°C injector temperature.
  5. 200°C manifold temperature (detector).
  6. Isothermal oven or column temperature — refer to Table 1 for specific compounds.
- 8.4.4 Injection. The first step in the analysis is the injection of the sample into the gas chromatograph. To eliminate difficulties arising from blowback or distillation within the syringe needle, one should employ the solvent flush injection technique. The 10  $\mu$ l syringe is first flushed with solvent several times to wet the barrel and plunger. Three microliters of solvent are drawn into the syringe to increase the accuracy and reproducibility of the injected sample volume. The needle is removed from the solvent, and the plunger is pulled back about 0.2  $\mu$ l to separate the solvent flush from the sample with a pocket of air to be used as a marker. The needle is then immersed in the sample, and a 5- $\mu$ l aliquot is withdrawn, taking into consideration the volume of the needle, since the sample in the needle will be completely injected. After the needle is removed from the sample and prior to injection, the plunger is pulled back a short distance to minimize evaporation of the sample from the tip of the needle. Duplicate injections of each sample and standard should be made. No more than a 3% difference in area is to be expected.
- 8.4.5 Measurement of area. The area of the sample peak is measured by an electronic integrator or some other suitable form of area measurement, and preliminary results are read from a standard curve prepared as discussed below.

#### 8.5 Determination of Desorption Efficiency

- 8.5.1 Importance of determination. The desorption efficiency of a particular compound can vary from one laboratory to another and also from one batch of charcoal to another. Thus, it is necessary to determine at least once the percentage of the specific compound that is removed in the desorption process for a given compound, provided the same batch of charcoal is used. NIOSH has found that the desorption efficiencies for the compounds in Table 1 are between 81% and 100% and vary with each batch of charcoal.

8.5.2 Procedure for determining desorption efficiency. Activated charcoal equivalent to the amount in the first section of the sampling tube (100 mg) is measured into a 5-cm, 4-mm I.D. glass tube, flame-sealed at one end (similar to commercially available culture tubes). This charcoal must be from the same batch as that used in obtaining the samples and can be obtained from unused charcoal tubes. The open end is capped with Parafilm. A known amount of the compound is injected directly into the activated charcoal with a microliter syringe, and the tube is capped with more Parafilm. The amount injected is usually equivalent to that present in a 10-liter sample at a concentration equal to the federal standard.

At least five tubes are prepared in this manner and allowed to stand for at least overnight to assure complete absorption of the specific compound onto the charcoal. These five tubes are referred to as the samples. A parallel blank tube should be treated in the same manner except that no sample is added to it. The sample and blank tubes are desorbed and analyzed in exactly the same manner as the sampling tube described in Section 8.4.

Two or three standards are prepared by injecting the same volume of compound into 0.5 ml of CS<sub>2</sub> with the same syringe used in the preparation of the sample. These are analyzed with the samples.

The desorption efficiency equals the difference between the average peak area of the samples and the peak area of the blank divided by the average peak area of the standards, or

$$\text{desorption efficiency} = \frac{\text{Area sample} - \text{Area blank}}{\text{Area standard}}$$

## 9. Calibration and Standards

It is convenient to express concentration of standards in terms of mg/0.5 ml CS<sub>2</sub> because samples are desorbed in this amount of CS<sub>2</sub>. To minimize error due to the volatility of carbon disulfide, one can inject 20 times the weight into 10 ml of CS<sub>2</sub>. For example, to prepare a 0.3 mg/0.5 ml standard, one would inject 6.0 mg into exactly 10 ml of CS<sub>2</sub> in a glass-stoppered flask. The density of the specific compound is used to convert 6.0 mg into microliters for easy measurement with a microliter syringe. A series of standards, varying in concentration over the range of interest, is prepared and analyzed under the same GC conditions and during the same time period as the unknown samples. Curves are established by plotting concentration in mg/0.5 ml versus peak area.

NOTE: Since no internal standard is used in the method, standard solutions must be analyzed at the same time that the sample analysis is done. This will minimize the effect of known day-to-day variations and variations during the same day of the FID response.

## 10. Calculations

10.1 The weight, in mg, corresponding to each peak area is read from the standard curve for the particular compound. No volume corrections are needed, because the standard curve is based on mg/0.5 ml CS<sub>2</sub> and the volume of sample injected is identical to the volume of the standards injected.

10.2 Corrections for the blank must be made for each sample.

$$\text{Correct mg} = \text{mg}_s - \text{mg}_b$$

where:

$mg_a$  = mg found in front section of sample tube

$mg_b$  = mg found in front section of blank tube

A similar procedure is followed for the backup sections.

10.3 The corrected amounts present in the front and backup sections of the same sample tube are added to determine the total measured amount in the sample.

10.4 This total weight is divided by the determined desorption efficiency to obtain the corrected mg per sample.

10.5 The concentration of the analyte in the air sampled can be expressed in mg per  $m^3$ .

$$mg/m^3 = \frac{\text{Corrected mg (Section 10.4)} \times 1000 \text{ (liters}/m^3)}{\text{Air volume sampled (liters)}}$$

10.6 Another method of expressing concentration is ppm (corrected to standard conditions of 25°C and 760 mm Hg).

$$ppm = mg/m^3 \times \frac{24.45}{MW} \times \frac{760}{P} \times \frac{(T + 273)}{298}$$

where:

P = pressure (mm Hg) of air sampled

T = temperature (°C) of air sampled

24.45 = molar volume (liter/mole) at 25°C and 760 mm Hg

MW = molecular weight

760 = standard pressure (mm Hg)

298 = standard temperature (°K)

## 11. References

- 11.1 White, L. D., D. G. Taylor, P. A. Mauer, and R. E. Kupel, "A Convenient Optimized Method for the Analysis of Selected Solvent Vapors in the Industrial Atmosphere", Am Ind Hyg Assoc J 31:225, 1970.
- 11.2 Young, D. M. and A. D. Crowell, Physical Adsorption of Gases, pp. 137-146, Butterworths, London, 1962.
- 11.3 Federal Register, 37:202:22139-22142, October 18, 1972.
- 11.4 NIOSH Contract HSM-99-72-98, Scott Research Laboratories, Inc., "Collaborative Testing of Activated Charcoal Sampling Tubes for Seven Organic Solvents", pp. 4-22, 4-27, 1973.



APPENDIX D

RADIAN LABORATORY REPORT

ANALYTICAL RESULTS OF SAMPLES FROM  
GETTY SYNTHETIC FUELS, INC.  
CALUMET CITY, ILLINOIS

IN SUPPORT OF WORK FOR  
ENGINEERING-SCIENCE

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## 1.0 SUMMARY

Samples were received from Engineering-Science for analysis according to protocols outlined in the "Quality Assurance Plan for Getty Synthetic Fuels, Inc., Calumet City, Illinois.

The samples received were: NIOSH Charcoal tubes (600 mg size); VOST Samples (Tenax/Charcoal tubes); XAD-2 Resin samples; Aldehyde samples (DNPH reagent); and Liquid Condensate samples.

## 2.0 RESULTS AND DISCUSSION

### 2.1 CHARCOAL TUBE ANALYSIS

The front and back sections of each charcoal tube were transferred to separate vials and desorbed with two (2) milliliters of OmniSolve brand "distilled in glass" grade carbon disulfide. The vials were then allowed to stand for at least 30 minutes with slight agitation. A one microliter aliquot was analyzed by gas chromatography with flame ionization detection. All front sections were analyzed and inspected, then the corresponding back sections were analyzed from the samples containing the highest components to determine if breakthrough occurred. There were no detectable compounds found in any of the back sections analyzed. The procedure reference followed was the NIOSH P&CAM 127 method for organic solvents. Analysis was performed on a Varian 3700 gas chromatograph and Vista 402 data system. The column used was a fused silica capillary (SPB-1) from Supelco Inc. A split flow of 20 mL/min. was used and an oven temperature of 40°C for 5 min., then programmed to 240°C at 10°C per minute. The analysis of Allyl Chloride (3-chloropropene), methylene chloride, and Vinylidene chloride (1,1-dichloro-ethylene) could not be done because they would not resolve from the carbon disulfide solvent peak. Vinyl chloride also was not analyzed for because it could not be detected at the 1 ppm level. Chloroprene (2-chloro-1,3-butadiene) was not analyzed for because we were unable to locate a supplier of the primary standard. The following compounds were analyzed for by this method: Benzyl chloride, Carbon Tetrachloride, Chlorobenzene, Chloroform, o-,m-,p-Cresols, p-Dichlorobenzene, Epichlorohydrin, Ethylene Dichloride (1,2-Dichloroethane), Hexachlorocyclopentadiene, Methyl Chloroform (1,1,

1-Trichloroethane), Perchloroethylene, Toluene, Trichloroethylene, o-xylene, Benzene. Table 1 shows the calibration curve data and graphs for each compound. The highest charcoal tube sample was also analyzed by GC/MS fused silica capillary to confirm the GC/FID results. Carbon tetrachloride and epichlorohydrin were shown not to be present. However, the peak at carbon tetrachloride's retention times, was identified as 2-ethyl-4-methyl-1-pentanol.

## 2.2 XAD-2 RESIN SAMPLES

Each XAD-2 resin sample was transferred to a Soxhlet extractor and extracted in Burdick & Jackson brand hexane for a period of 24 hours. During the transfer process each sample was spiked with surrogate standards consisting of 1,4-Bromofluorobenzene; phenol-d<sub>6</sub>; and phenanthrene-d<sub>10</sub>. The hexane extracts were then reduced to 1 mL using a Kurderna-Danish Evaporative Concentrator followed by nitrogen blow-down. For quality assurance purposes each XAD-2 resin sample was then extracted a second time with Fisher Scientific brand GC/MS grade methylene chloride for another 24 hours, then reduced to 1 mL as before. All sample extracts were analyzed by gas chromatography using fused silica capillary columns with flame ionization detection. The hexane extracts were also screened by electron capture for polychlorinated biphenyls and dioxins. The recoveries for the 14 XAD-2 samples are summarized as follows:

<u>Standard Deviation</u>		
BFB	Avg. 52%	10
Phenol-d <sub>6</sub>	Avg. 33%	7
Phenanthrene-d <sub>10</sub>	Avg. 66%	13

The EPA reference "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater" EPA-600/4-82-057, July 1982; states that acceptable recoveries and standard deviations for phenol and phenathrene are 36%, standard deviation 21; and 76%, standard deviation 22, respectively.

Table 1. Calibration Curves for Engineering Science VOST Samples

	<u>Corr. Cor</u>	<u>Intercept</u>	<u>Slope</u>
Vinyl Chloride	.9995	-32171	203.98
Chloroform	.9625	-69589	148.21
Trichloroethane	.9956	-3432	81.74
Carbon Tetrachloride	.9738	-14207	53.81
Trichloroethene	.9690	-41509	92.80
Tetrachloroethene	0.9588	-26536	65.16
Chlorobenzene	.9794	-32076	148.78

The instrument conditions used for the XAD-2 samples are as follows:

Varian 3700 Gas Chromatograph

Vista 402 data system

Flame Ionization Detector

SPB-1 fused silica capillary

Split flow 20 ml/min.

Carrier helium 14 psig

Make-up nitrogen 30 ml/min.

Oven 40°C for 5 min., programmed to 240°C, hold 11 min.

Air 300 ml/min.; H<sub>2</sub> 30 ml/min.

Inj. temp. 260°C; det. 280°C

Electron Capture Detector

SE-54 fused silica capillary

Split flow 20 ml/min.

Carrier & make-up N<sub>2</sub> 14 psig, 30 ml/min.

Oven 40°C for 5 min., programmed to 220°C, hold for 22 min.

Inj. temp. 260°C; det. 300°C.

## 2.3 ALDEHYDES ANALYSIS

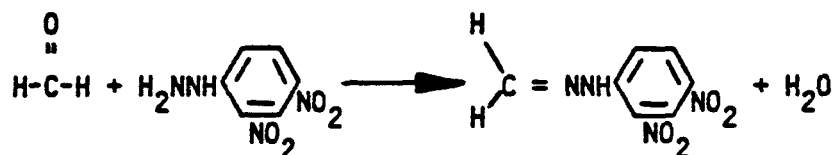
The procedure used for the analysis of aldehydes was a high performance liquid chromatography (HPLC) technique from the reference "Determination of Aliphatic and Aromatic Aldehydes in Polluted Airs as their 2,4-Dinitrophenylhydrazones by High Performance Liquid Chromatography", by Kuwata, Uebori, and Yamasaki, published in the Journal of Chromatographic Science, May 1979.

### 2.3.1 Principle of Method

Gaseous aldehydes are drawn through an impinger system and allowed to react with a solution of 2,4-dinitrophenylhydrazine to form the 2,4 dinitrophenylhydrazone derivatives. These derivatives are extracted from the sampling solution with chloroform and concentrated by evaporation.



The residue is then dissolved in acetonitrile and analyzed by high performance liquid chromatography (HPLC). The balanced reaction equation for formaldehyde is given below:



where 1 mole of aldehyde reacts with 1 mole of hydrazine to form 1 mole of hydrazone derivative and 1 mole of water.

### 2.3.2 Preparation of Standards

The hydrazone derivatives were prepared and purified by the procedure given in Systematic Identification of Organic Compounds, by Shriner, Fuson, and Curtin, John Wiley, 1964.

Two grams of 2,4 dinitrophenylhydrazine were placed in a 500 ml Erlenmeyer flask to which was added 10 ml of concentrated sulfuric acid and up to 15 ml of deionized water to effect dissolution. With vigorous swirling, 50 ml of 95% ethanol was added in approximately 10 ml aliquots. (The solution may appear to be cloudy at this point). A previously prepared solution of approximately 2.5 g of aldehyde in 100 ml of ethanol (aldehyde must be in excess over the hydrazine) is slowly added to this warm, vigorously swirled solution. The newly formed derivative will be present as a colored precipitate which is recovered by filtration utilizing a 60 ml coarse fritted glass filter funnel. All derivatives except that of acrolein and benzaldehyde were recrystallized from 95% ethanol. The addition of acetone to the ethanol was necessary to effect the dissolution of these two less soluble derivatives. Compounds were recrystallized a minimum of three times until pure as determined by HPLC and were considered 99% pure when the sum of the peak areas of all impurities was less than 1% of the total. The newly prepared derivatives were stored in a vacuum desiccator in the dark.

Standards of each compound were prepared in acetonitrile and stored at sub-ambient temperature. Stock solutions were individually prepared by accurately weighing (nearest 0.1 mg) 50 mg of each compound into a

100 ml volumetric flask and filling to the mark with acetonitrile. Working standards were prepared by transferring 20, 50, 100, 200, 300 microliters of each stock solution to five separate 10 ml volumetric flasks. The concentrations of the working standards covered the linear range of 10, 25, 50, 100 and 150 nanograms per 10 microliter injection.

#### 2.3.3 Preparation of Sampling Solution

The sampling solution is prepared by placing 1 grams of 2,4 dinitrophenylhydrazine in a 1 liter volumetric flask containing approximately 500 ml of deionized water and 166 ml of concentrated hydrochloric acid. (166ml of HCl diluted to 1 liter prepares a 2 N solution.) The solution is vigorously agitated until the hydrazine is dissolved and then diluted to 1 liter with deionized water. This solution is transferred to a separatory funnel and extracted six times with 100 ml aliquots of chloroform. If the sampling solution is not used within four days, additional extractions may be necessary to remove decomposition impurities as determined by a HPLC analysis of the "blank" sampling solution.

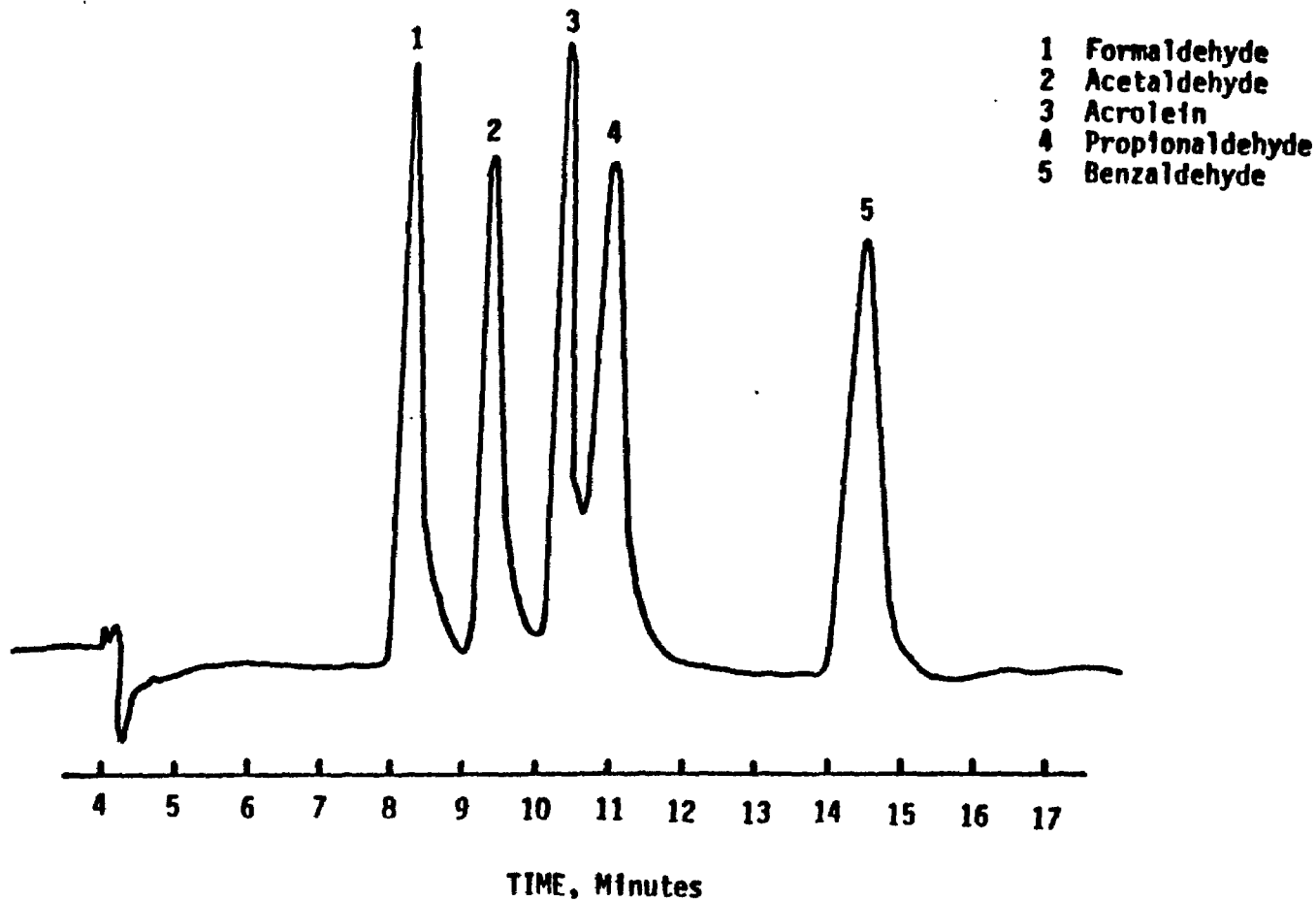
#### 2.3.4 Chromatographic Parameters

The samples were analyzed by high performance liquid chromatography utilizing a 5 micron, monomeric C<sub>18</sub> reverse phase column maintained at 30°C. The mobile phase was an isocratic mixture of 30% water and 70% acetonitrile flowing at a rate of 0.5 ml/min. The compounds were detected with a variable UV-Vis detector set at a wave length of 254 nanometers approximately midway in the sensitivity range. Quantitation was based on peak areas as determined by an electronic integrator with the chromatograms displayed on a strip chart recorder. Figure 1 shows a typical chromatogram of the five aldehyde derivatives.

#### 2.3.5 Sample Preparation

The samples were received packaged in ice. After an initial examination, the samples were placed in a refrigerator and maintained at sub-ambient temperatures. Prior to extraction, the volume of each sample was determined and transferred to a glass separatory funnel. The chloroform used for

Figure 1. Chromatogram of Aldehyde Derivative Standards.



sample extraction was used to rinse the sample container and then added to the flask. Depending on the sample volume varying amounts of chloroform were used but always in the ratio of 1 ml of chloroform per 6 ml of sample. Each sample was extracted three times with equal volumes of chloroform, evaporated to dryness, and redissolved in a known volume of acetonitrile (typically 50 ml). Further dilutions were made when necessary.

#### 2.3.6 Apparatus

A Varian Instrument Model 5061 high performance liquid chromatograph (HPLC) was utilized for the separation and detection of the individual compounds. It was coupled to a variable wavelength UV-Visible detector and maintained at a wavelength of 254 nm. Integration and retention times were determined electronically with a Varian Vista 402 data system. Samples were injected automatically via a Varian Series 8000 autosampler.

#### 2.3.7 Reagents

Acetonitrile, Fisher HPLC

Water, Fisher HPLC

Chloroform, Fisher HPLC

Acetone, Fisher HPLC

Ethanol, Fisher 95%

Sulfuric Acid, Fisher Reagent grade

2,4 Dinitrophenylhydrazine, Eastman

Formaldehyde, Fisher, Certified, ACS 37% W/W

Acetaldehyde, Fisher, Certified

Acrolein, Eastman

Propionaldehyde, Eastman

Benzaldehyde, Fisher, Certified

#### 2.3.8 Glassware Clean-Up

All glassware was originally washed with water and detergent, rinsed with deionized water and followed with a chloroform rinse. After each use, the glassware was rinsed with hot tap water followed with deionized water and chloroform rinses.

#### 2.4 GC/MS ANALYSIS OF XAD-2 SAMPLES

The XAD-2 extracts (1 mL) were analyzed by GC/MS using an on-column injection technique to maximize the minimum detection limits (MDL). The instrument used was a Finnigan 4000 with INCOS data system. The analytical column was a SPB-5 bonded fused silica capillary column, 30 meter by 0.25 mm. The oven was at 10°C for 5 minutes, then programmed to 275°C at 8° per min. The injector and separator oven temperatures were kept at 280°C. The data system scan rate was set at 0.95 seconds, and scanned between 40 and 450 AMU. The electron energy was set on 70 volts. Anthracene-d<sub>10</sub> was used as an internal standard for all injections.

#### 2.5 VOLATILE ORGANIC SAMPLING TRAIN (VOST) SAMPLES

All Tenax/Charcoal sorbent cartridges were analyzed according to the recent EPA document EPA-600/8-84-007, March 1984, "Protocol for the Collection and Analysis of Volatile POHCs Using VOST". This protocol is the GC/MS procedure used and was followed with no exceptions. Each sorbent cartridge analyzed was desorbed separately because of the high amounts of organics found. Calculations for each run were done by summing up each individual cartridge results for that run. As required by the protocol each cartridge was spiked with the internal standards 1,4-bromofluorobenzene and benzene-d<sub>6</sub>. The column used for these analyses was a glass SP-1000 1% on Carbopack B 60/80, 6 ft. by 2 mm. The carrier gas was helium set at a flow of 25 mL/min. The oven temperature was initially at 30°C and programmed to 190°C at 15°/min. The scan range was 33 to 260 AMU at 2 sec/scan. The injector and separator temperatures were 190 and 200°C. Desorption of the "inside-inside" cartridges was done by connecting them directly to the inlet of a Tekmar LSC-1 purge and trap apparatus. Each cartridge was heated to 180°C and purged with helium at 30 mL/min for 10 minutes to reconcentrate the volatiles in the Tekmar's analytical trap. The trap was then heated to 180°C and switched in line with the GC/MS. The calibration curves and graphs are presented in Section 3.0.

## 2.6 LIQUID SAMPLES

The hydrocarbon condensate samples (100 mL) were digested with concentrated nitric acid followed by oxidation with hydrogen peroxide. The final volumes were 100 mL and analysis by ICAP and AA of the following metals was performed: Arsenic, Beryllium, Cadmium, Chromium, Mercury, Nickel, and Manganese.

## 2.7 DIOXIN SCREENS

XAD-2 samples and liquid condensate samples were screened for Dioxins using GC/MS selected ion monitoring and GC/Electron Capture. Selected samples representing the highest levels found from each sampling location were reduced to 100 microliters by nitrogen blow-down. These extracts were then screened by GC/Electron Capture Detection using fused silica capillary chromatography. An interfering peak was found by GC/ECD in all samples including the blank. The sample extracts were then screened by single ion monitoring GC/MS. The Dioxins screened for were: Dibenzo-p-dioxin, 1-chlorodibenzo-p-dioxin, 2-chlorodibenzo-p-dioxin, 2,7-dichlorodibenzo-p-dioxin, 1,2,4-trichlorodibenzo-p-dioxin, and octachlorodibenzo-p-dioxin. The GC/MS screening procedure was achieved by injecting the sample directly on-column while scanning. The Finnigan 4000 GC/MS/INCOS data system was programmed from 140°C to 275°C at 20°C per minute. The separator temperature was maintained at 280°C. Two (2) microliters of sample was introduced on-column on a SPB-5 fused silica capillary column, 30 meter x 0.25 mm. The selected ions monitored were 257, 320, and 322 for 0.21 seconds each with an electron energy of 70 eV. The screening level was 1 picogram per microliter. This correlates to 100 picograms per 100 microliters, which represents the total sample. No Dioxins were found at this level.

The liquid condensate samples were extracted by using Florosil as a clean-up medium, where 1 ml of the sample was eluted. A 1% methylene chloride in hexane solution was then eluted to remove interferences, followed by 20% methylene chloride in hexane. This fraction was screened for Dioxins as described.

### 3.0: APPENDICES

Charcoal Tube Results

VOST GC/MS Results

Aldehyde Results

Metal Results of Liquid Condensate

XAD-2 Extract Percent Recoveries

GC/FID Calibration Data and Curves

GC/MS VOST Calibration Data and Curves

CHARCOAL TUBE RESULTS: TOTAL MICROGRAMS

Radian I.D.	Field I.D.	Chloroform	1,2-Dichloro-ethane	1,1,1 Tri-chloroethane	Benzene	CCl <sub>4</sub>	Trichloro-ethylene	Epichloro-hydrin	Toluene	Perchloro-ethylene	Chloro-benzene	o-Xylene	Benzyl Chloride	m,p-Xylene
6007 Front	C-T-F-1-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6008 Front	C-C-5-1-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6009 Front	C-C-5-3-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6010 Front	C-C-6-4-1*	2349.3	27.5	ND	35.4	ND	ND	ND	ND	ND	ND	ND	ND	ND
6011 Front	C-C-6-5-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6012 Front	C-C-6-6-1	ND	ND	ND	ND	ND <sup>†</sup>	88.9	ND	ND	ND	ND	ND	ND	ND
6013 Front	C-C-6-6-11	ND	ND	ND	ND	ND	ND	40.6	ND	ND	ND	ND	ND	ND
6014 Front	C-F-5-1-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6015 Front	C-F-5-3-1*	ND	ND	ND	100.3	ND <sup>†</sup>	ND	ND	1286.0	150.5	240.4	188.4	ND	ND
6016 Front	C-F-6-4-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6017 Front	C-F-6-5-1*	ND	ND	ND	46.3	ND <sup>†</sup>	ND	113.9	ND	ND	112.2	93.1	ND	104.3
6018 Front	C-F-6-6-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6019 Front	C-F-6-7-1*	ND	ND	ND	43.7	ND	ND	98.9	431.0	ND	100.3	82.3	ND	133.1
6020 Front	C-P-5-1-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6021 Front	C-P-5-3-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6022 Front	C-P-6-4-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6023 Front	C-P-6-5-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6024 Front	C-S-5-1-1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
6025 Front	C-S-5-1-2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Detection Limits**		135.6	27.5	10.7	29.3	647.2	74.0	27.9	33.7	113.2	53.8	38.9	115.0	77.4

\* Sample backs were run and none was detected at the given detection limits.

\*\* ND = None Detected at the following detection limits based on charcoal tube desorbed in 2 ml CS<sub>2</sub>.

† GC/MS identified peak as 2-ethyl-4-methyl-1-pentanol.



## VOST GC/MS: TOTAL MICROGRAMS

Field I.D.	Vinyl Chloride	Chloroform	Epichlorohydrin	1,1,1-Tri-chloroethane	CCl <sub>4</sub>	Trichloro-ethene	Tetrachloro-ethene	Chloro-benzene	Benzyl Chloride	Chloroprene
T-C-5-1	ND	1.574	ND	ND	ND	ND	ND	ND	ND	ND
T-C-6-2	ND	0.576	ND	ND	ND	ND	ND	ND	ND	ND
T-C-6-4	26.087*	7.590*	ND	0.171	ND	0.699	0.519	2.025	ND	ND
T-C-6-5	2.741	4.151	ND	0.133	ND	0.550	ND	0.440	ND	ND
T-F-5-1	ND	9.806	ND	.078	ND	0.534	ND	ND	ND	ND
T-F-5-3†	ND	14.727	ND	0.173	ND	1.823	1.730	5.784	ND	ND
T-F-6-2	ND	1.043	ND	ND	ND	ND	ND	ND	ND	ND
T-F-6-4	0.230	6.229	ND	1.166	ND	14.184	ND	ND	ND	ND
T-F-6-5	0.498	3.716	ND	.074	ND	1.010	1.274	2.959	ND	ND
T-P-6-2	ND	0.641	ND	ND	0.339	0.551	0.445	ND	ND	ND
T-P-6-4	2.222	1.342	ND	0.100	ND	ND	ND	ND	ND	ND
T-C-5-3	20.813	3.258	ND	0.207	ND	0.657	0.502	1.093	ND	ND
T-P-5-3	3.245	11.044	ND	.167	ND	.207	ND	ND	ND	ND
T-F-6-6 -11-14	.168	3.985	ND	.121	ND	12.78	17.26	48.1	ND	ND
T-F-6-6-1-4	1.170	9.549	ND	.137	ND	1.536	2.275	ND	ND	ND
Audit T-S-5-2	0.792	10.505	ND	0.084	1.009	0.532	1.269	ND	ND	ND
Detection Limit	.020 µg	.015 µg	2.0 µg <sup>B</sup>	.010 µg	.012 µg	.010 µg	.012 µg	.20 µg	.2.5 µg <sup>C</sup>	A,B

A Note - The chloroprene standard was not available.

B Note - Epichlorohydrin and Chloroprene are water soluble.

C Note - Benzyl Chloride would not elute from Tenax. The boiling point is 179°C.

D Note - Results are the sum of four VOST tubes.

†One of the stainless plugs was shipped loose on tube #1.

\*Revised.

SELECTED ALDEHYDES, TOTAL MIROGRAMS COLLECTED

Field ID	Formal	Acetal	Acrolein	Propal	Benzal
DC5312	ND	ND	ND	ND	ND
DC533	ND	ND	ND	ND	ND
DC641	59.3	34.1	ND	ND	ND
DC651	67.2	ND	ND	ND	ND
DF5312	186	30.6	ND	ND	ND
DF533	39.7	ND	ND	ND	ND
DF641	206	46.8	ND	ND	ND
DF651	38.2	ND	ND	ND	ND
DF661	126	20.5	ND	ND	ND
DF6611	205	13.4	ND	ND	ND
DP5312	ND	ND	ND	ND	ND
DP533	ND	ND	ND	ND	ND
DP641	ND	ND	ND	ND	ND
DP651	46.4	ND	ND	ND	ND
C. Coeff.	0.99972	0.99997	0.99998	0.9998	0.9999
Y. Inter.	-203	-20.2	-130	5.8	-424
Slope	0.887	0.802	1.135	0.7484	0.8292

ND = three times standard deviation of blank (i.e., 5 µg total).

# METAL RESULTS OF LIQUID COMPOSITE SAMPLES

Radian No. ID No.	Beryllium	Cd	Parts Per Million (ppm)				As	Hg
			Cr	Mn	Ni			
6084 3:45 pm	<.0005	.089	.80	.21	.25	<.003	<.002	
6085 1230 hrs	<.0005	.049	.37	.041	.12	<.003	<.002	
6086 1710 hrs	<.0005	.044	.46	.21	.056	<.003	<.002	
6087 1100 hrs	<.0005	.020	.27	.039	.035	<.003	<.002	
6088 1630 hrs	<.0005	.016	.19	.05	.027	<.003	<.002	

XAD-2  
EXTRACT PERCENT RECOVERIES

<u>BFB</u>	<u>1435 ng Phenol-d<sub>6</sub></u>	<u>1000 ng Phenanthrene-d<sub>10</sub></u>
42	29	62
50	37	56
55	38	68
51	34	64
43	27	51
56	20	76
72	18	80
70	32	98
45	36	68
38	30	56
50	40	58
44	38	54
53	40	63
59	37	74
Avg.: 52%	33%	66%
S.D.: 10	7	13

---

Acceptable EPA Values:      36%  $\pm$  21 S.D.                      76%  $\pm$  22 S.D.

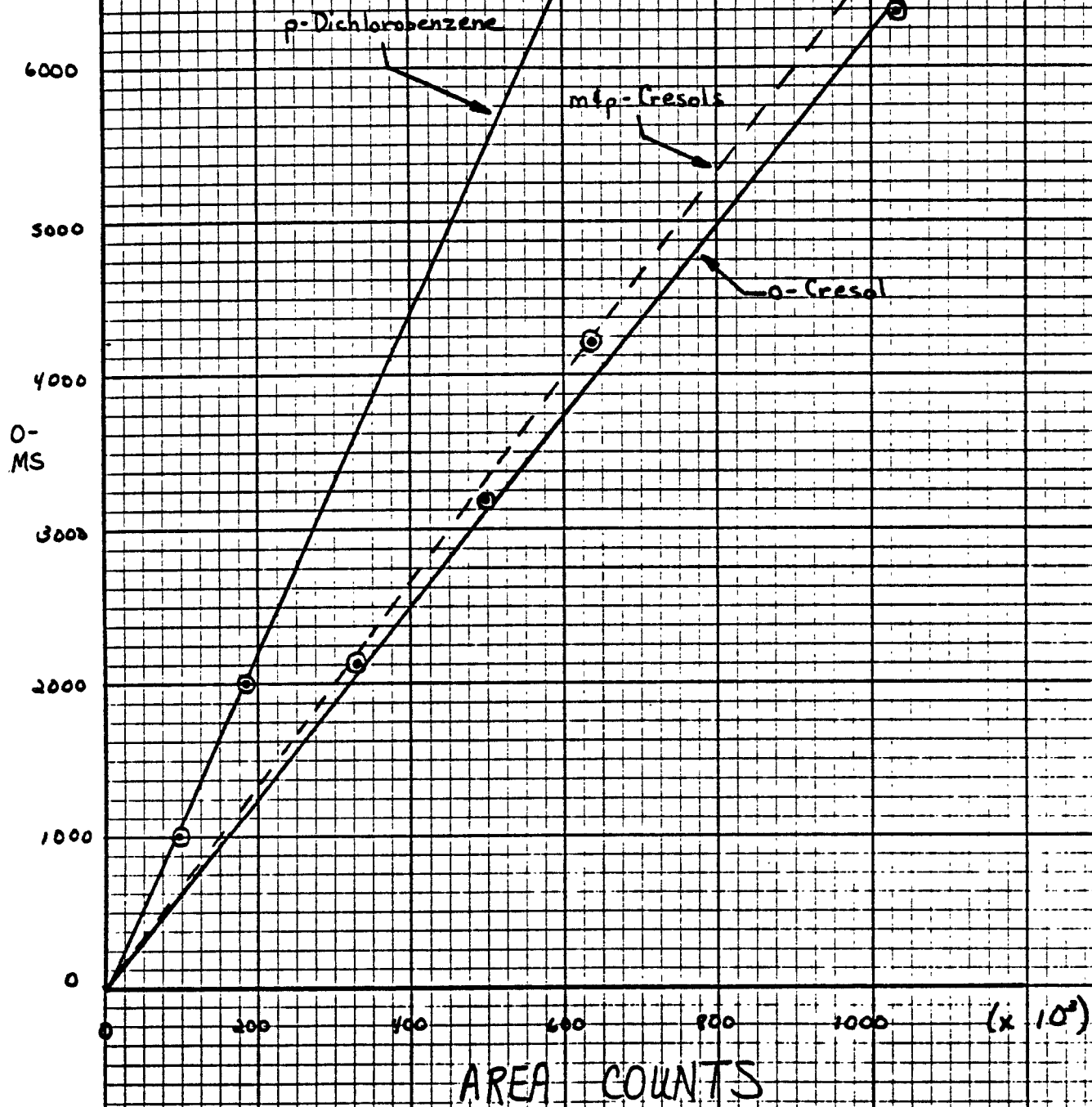
S.D. = Standard Deviation

GC/FID CALIBRATION DATA  
LINEAR REGRESSIONS

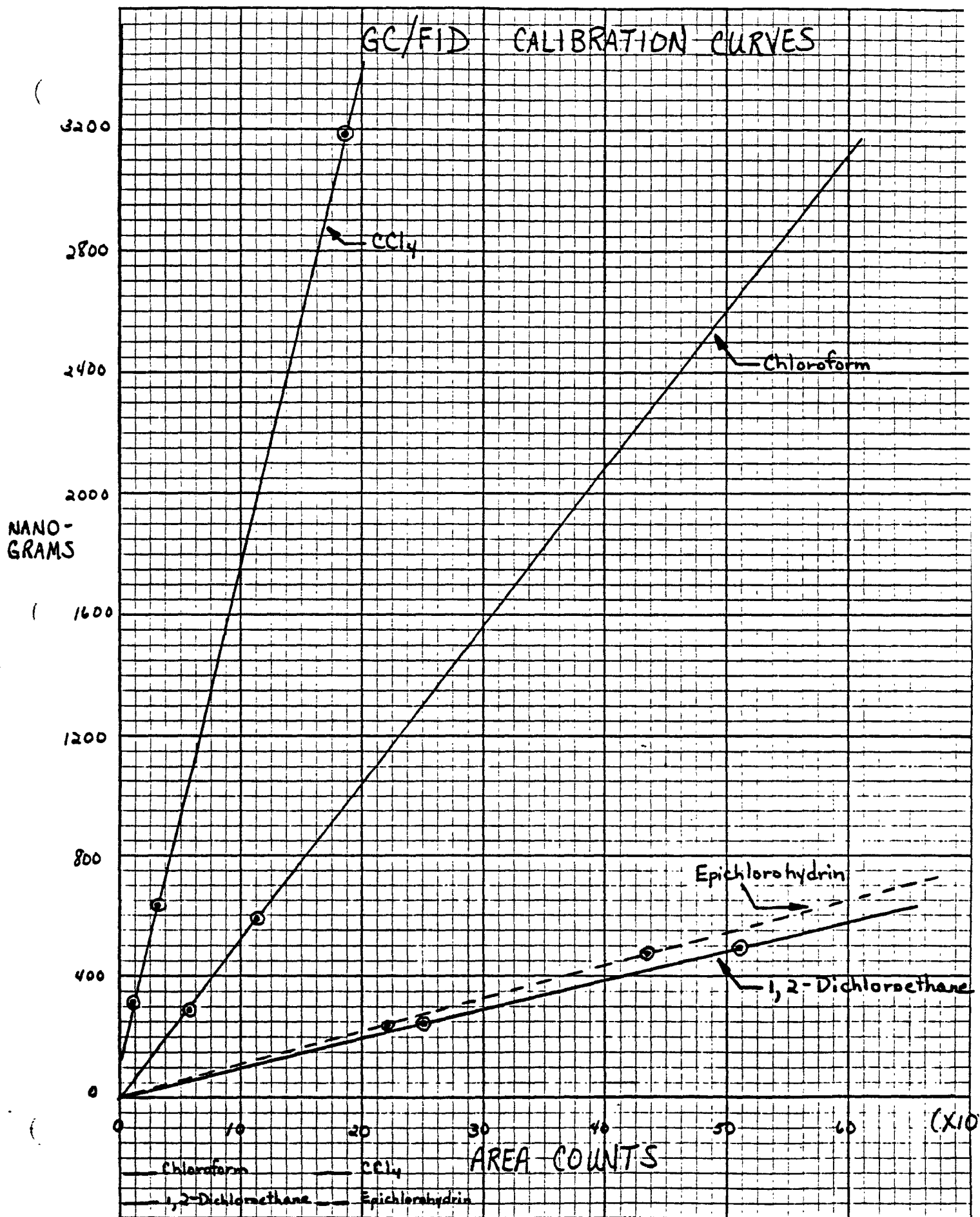
	<u>Chloroform (using 0,0)</u>	<u>Trichloroethylene</u>	<u>m-p-Xylene</u> <sup>a</sup>
correlation r:	0.9999	0.9999	
intercept i:	43	-2558	
slope s:	19.2	82.1	
	<u>1,2-Dichloroethane (using 0,0)</u>	<u>Epichlorohydrin (using 0,0)</u>	<u>o-Xylene</u>
	0.9999	0.9999	0.9999
	-115	72	-6308
	102.6	92.2	284.6
	<u>1,1,1 Trichloroethane</u>	<u>Toluene</u>	<u>Benzyl Chloride</u>
	0.9997	0.9999	0.9999
	2672	-6501	-9166
	40.8	336.1	129.5
	<u>Benzene</u>	<u>Perchloroethylene</u>	
	0.9999	0.9999	
	-5360	-2941	
	334.5	58.4	
	<u>CCl<sub>4</sub></u>	<u>Chlorobenzene</u>	
	0.9999	0.9999	
	-999	-5083	
	6.2	175.5	

<sup>a</sup>Note: No linear regression data available due to inability to integrate peak. (Response factor was used to calculate sample results.)

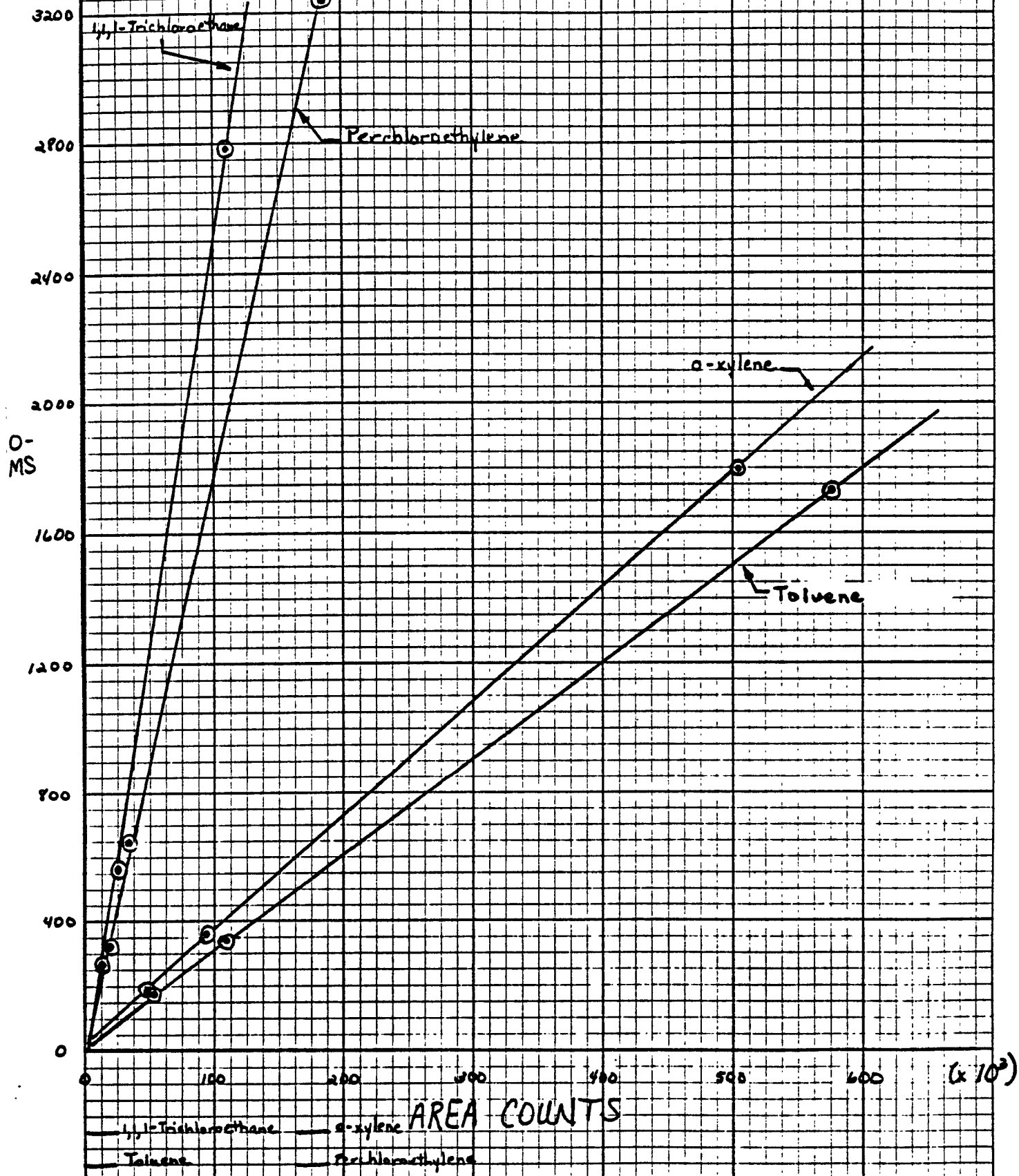
# GC/FID CALIBRATION CURVES



— p-Dichlorobenzene  
 - - - o-Cresol  
 - - - m,p-Cresols

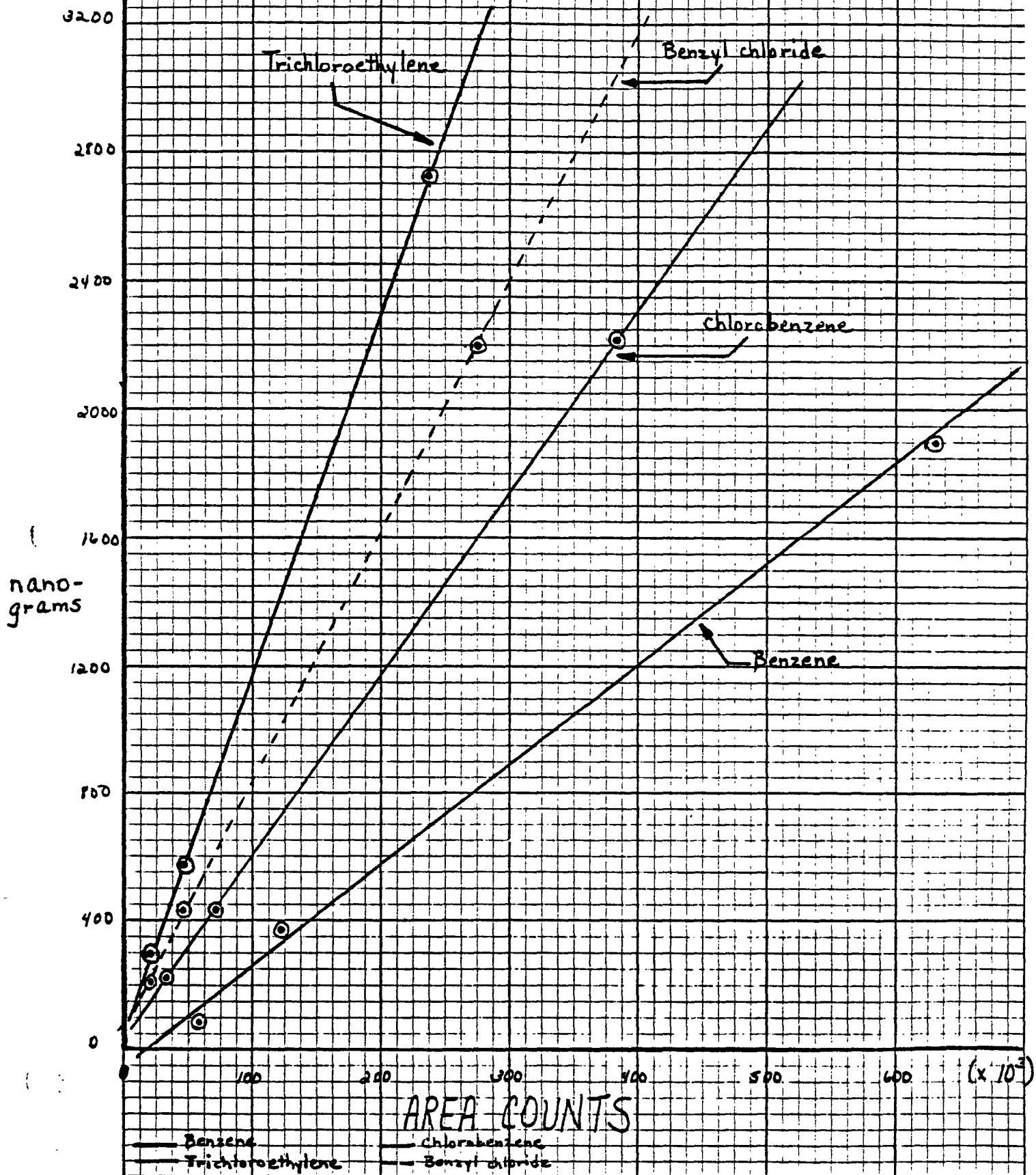


# GC/FID CALIBRATION CURVES





# GC/FID CALIBRATION CURVES



GC/FID CALIBRATION CURVE  
HEXACHLOROCYCLOPENTADIENE

46 1320

0  
IMS

800

700

600

500

400

300

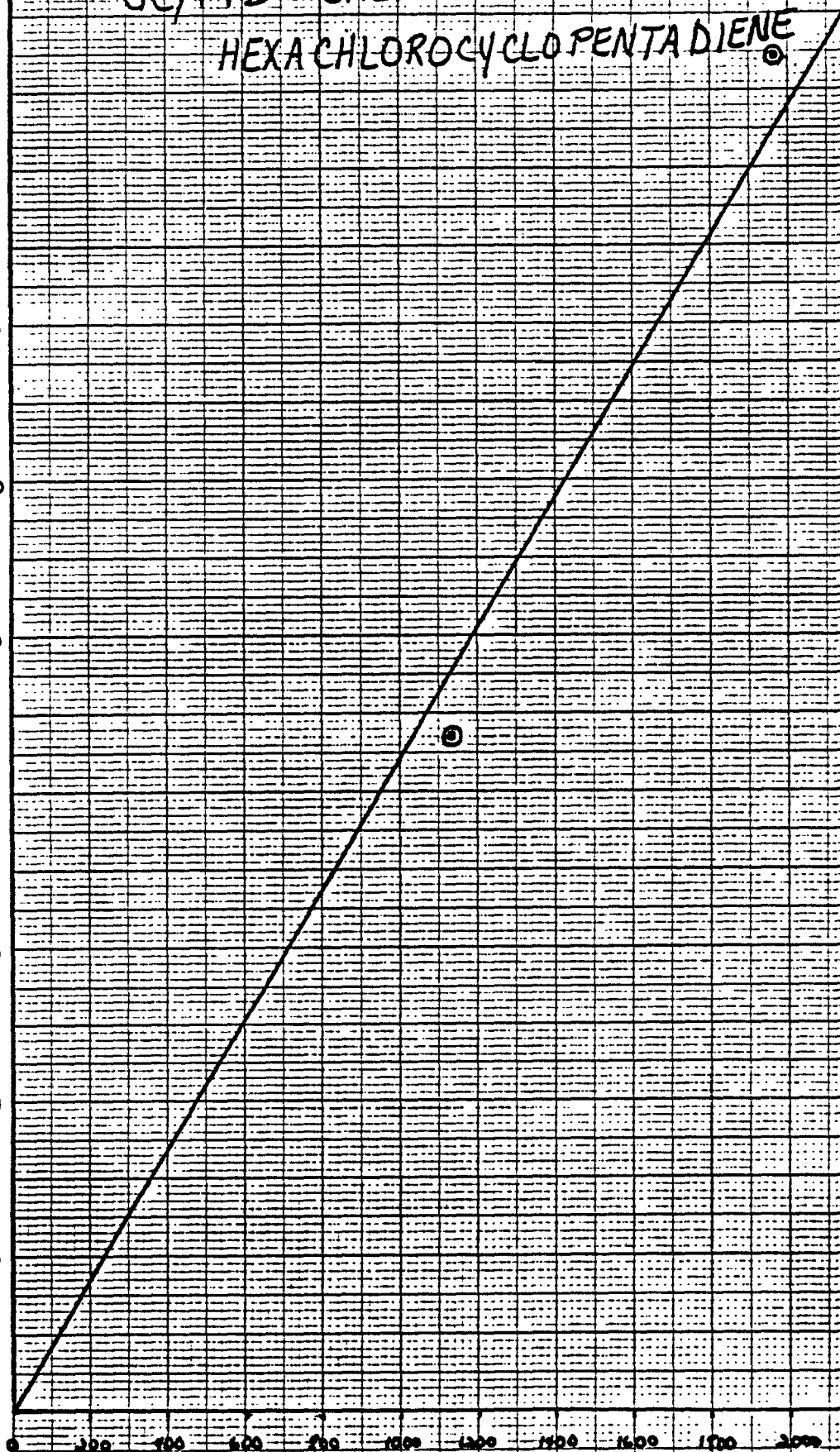
200

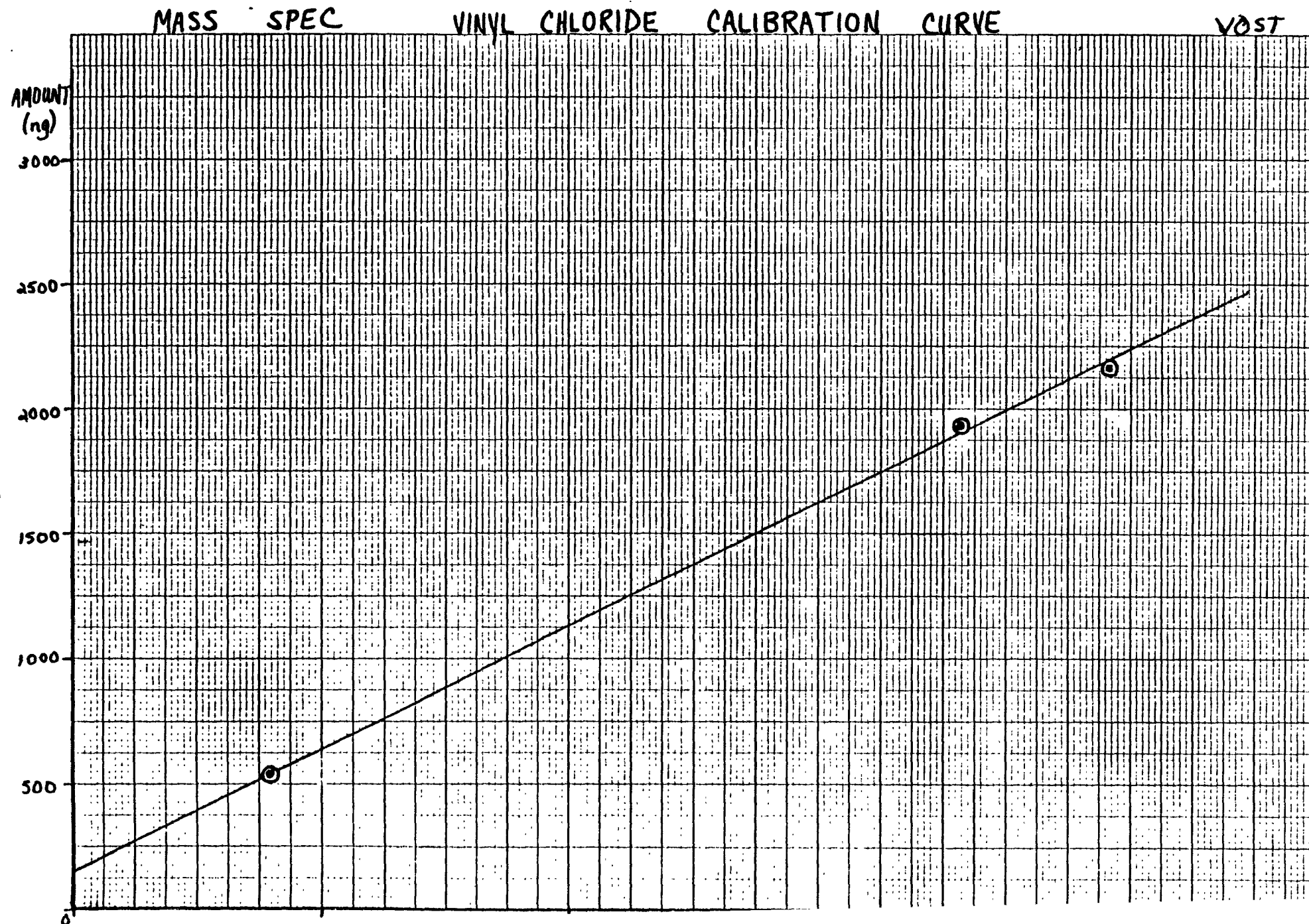
100

0

0 200 400 600 800 1000 1200 1400 1600 1800 2000

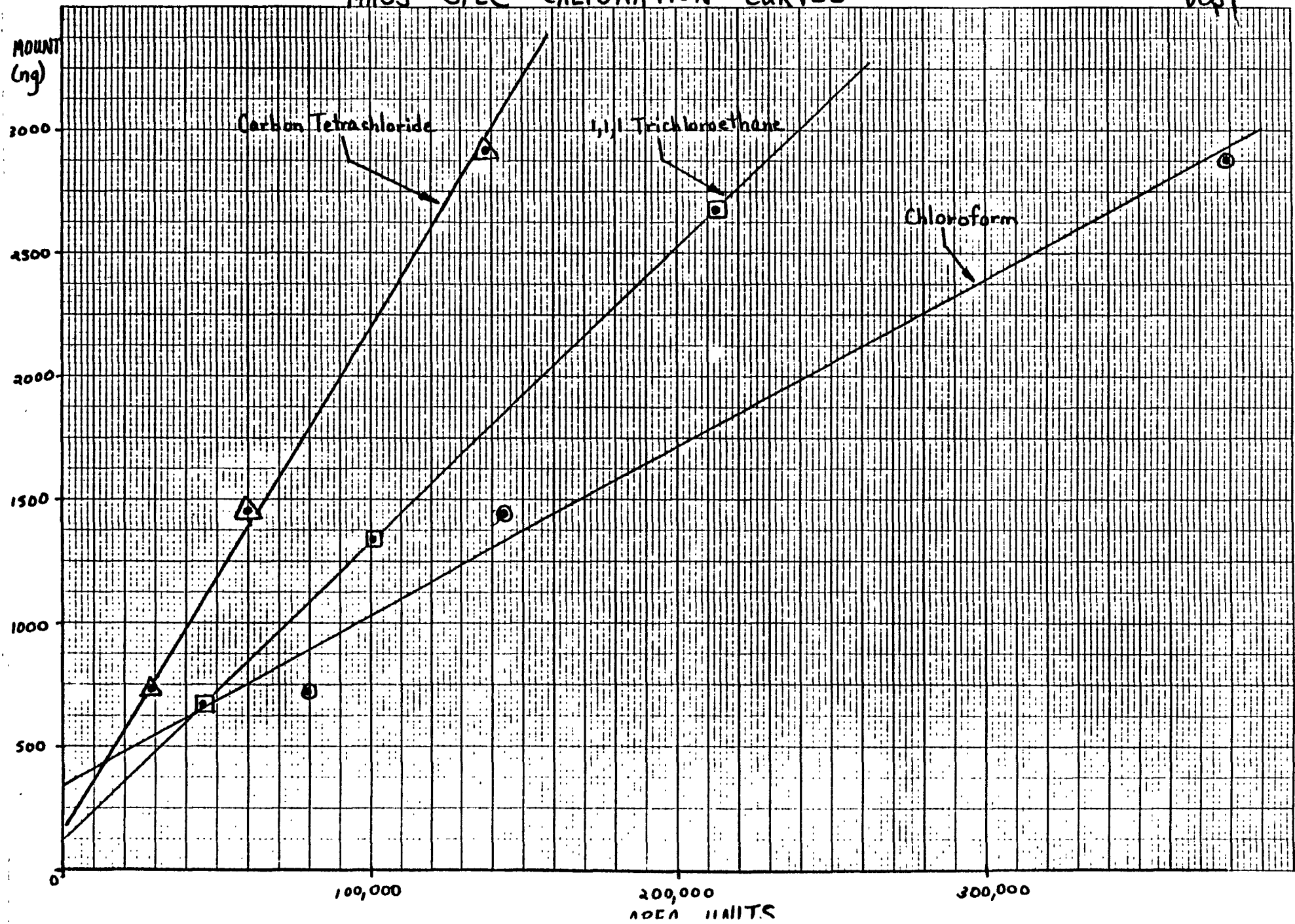
AREA COUNTS





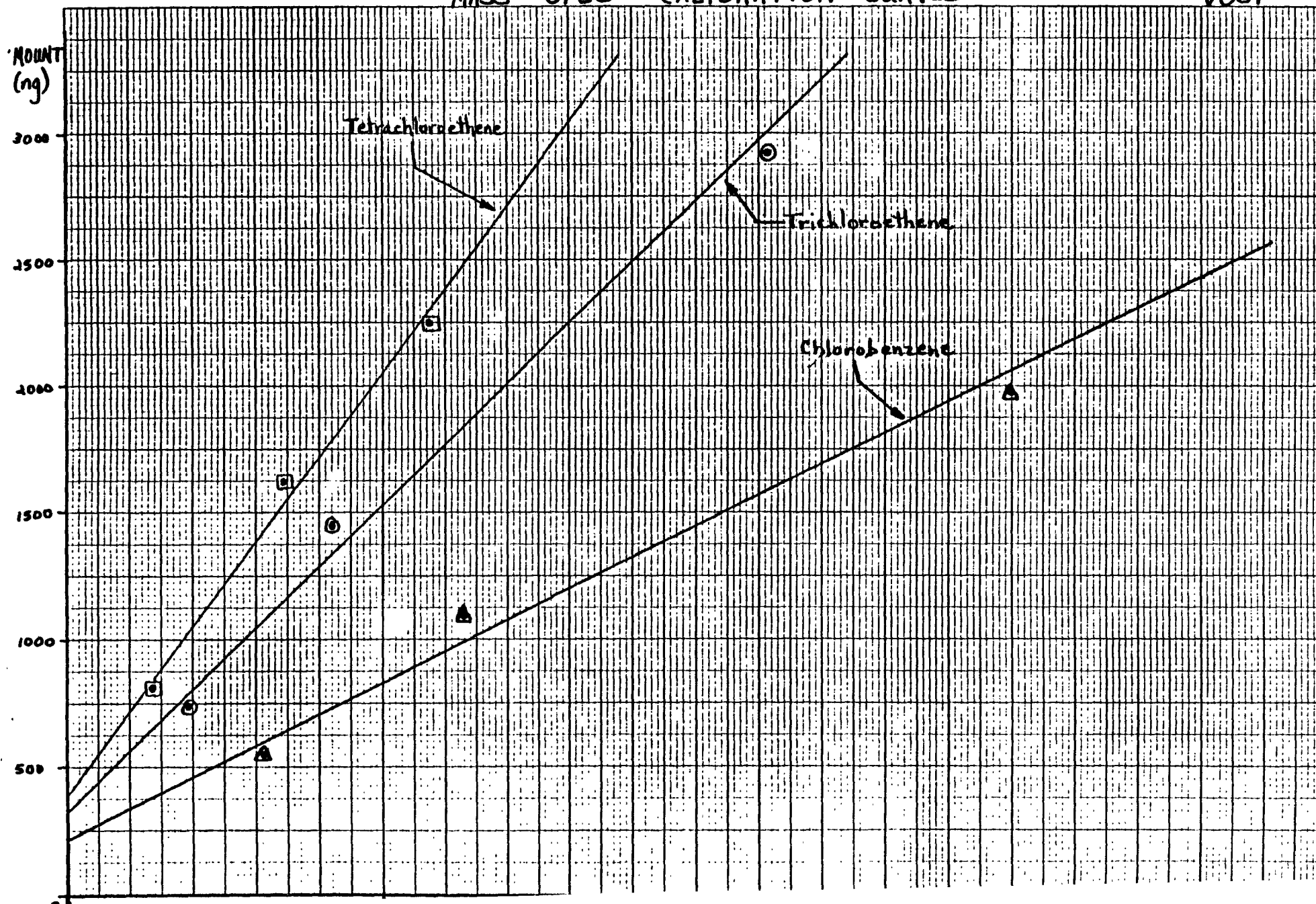
## MASS SPEC CALIBRATION CURVES

VOST



# MASS SPEC CALIBRATION CURVES

VOST



**APPENDIX E**

**THERMO ELECTRON LABORATORY REPORT**



## Analytical Services Laboratory

---

125 Second Avenue  
Waltham, Massachusetts 02154  
(617) 890-8700

Telex: 92-3473  
Cable: TEECORP

**FORMAL REPORT OF ANALYSIS**  
**FOR**  
**N-NITROSO COMPOUNDS**

**Prepared for:**

Engineering-Science, Inc.  
10521 Rosehaven Street  
Fairfax, Virginia 22030  
ATTN: Mr. Larry Cottone

**Date:**

29 March 1984

**Report No.:**

5450-5184

**Notebook Page:**

A1001-42

**Approved by:** \_\_\_\_\_

A handwritten signature, likely of a laboratory official, written over a horizontal line.

SUMMARY OF RESULTS

## Thermosorbs

Customer Sample Number	NDMA <sup>1</sup> ng	NDEA <sup>1</sup> ng	NDPA <sup>1</sup> ng	NDBA <sup>1</sup> ng	NPIP <sup>1</sup> ng	NPYR <sup>1</sup> ng	NMOR <sup>1</sup> ng
A10064	--- 2	--- 2	--- 2	--- 2	--- 2	--- 2	--- 2
A10065	---	---	---	---	---	---	---
A10066	---	---	---	---	---	---	---
A10067	---	---	---	---	---	---	---
A10068	---	---	---	---	---	---	---
A10069	---	---	---	---	---	---	---
A10070	---	---	---	---	---	---	---
A10071	---	---	---	---	---	---	---
A10072	---	---	---	---	---	---	---
A10073	---	---	---	---	---	---	---
A10074	---	---	---	---	---	---	---
A10075	---	---	---	---	---	---	---
A10076	---	---	---	---	---	---	---
A10077	---	---	---	---	---	---	---
A10078	---	---	---	---	---	---	---
A10079	---	---	---	---	---	---	---

1. N-nitroso compounds in nanograms per ThermoSorb cartridge.

2. Not detected

Limit of detection (LOD): 5 ng per cartridge for NDMA

8 ng per cartridge for NDEA, NDPA, NPIP, NPYR, and NMOR

10 ng per cartridge for NDBA

Date Sample Received: 3/15/84

Date of Analysis: 3/26/84

Method of Analysis: GC-TEA

Analyst: Linda Cantor



Analytical Services Laboratory



ABBREVIATIONS:

NDMA - N-nitrosodimethylamine  
NDEA - N-nitrosodiethylamine  
NDPA - N-nitrosodipropylamine  
NDBA - N-nitrosodibutylamine  
NPIP - N-nitrosopiperidine  
NPYR - N-nitrosopyrrolidine  
NMOR - N-nitrosomorpholine  
NMVA - N-nitrosomethylvinylamine  
NMEA - N-nitrosomethylethylamine  
NEPA - N-nitrosoethylpropylamine  
NPBA - N-nitrosopropylbutylamine  
NMPA - N-nitrosomethylpropylamine  
NMBA - N-nitrosomethylbutylamine  
NEBA - N-nitrosoethylbutylamine  
NMBZA - N-nitrosomethylbenzylamine  
NPHBZA - N-nitrosophenylbenzylamine  
NDAA - N-nitrosodiamylamine  
NDCHA - N-nitrosodicyclohexylamine  
NDPHA - N-nitrosodiphenylamine  
NMDDA - N-nitrosomethyldodecylamine  
NMDDA - N-nitrosomethyltetradecylamine  
NMPHA - N-nitrosomethylphenylamine  
NDELA - N-nitrosodiethanolamine  
NDPLA - N-nitrosodipropanolamine  
NDIPLA - N-nitrosodiisopropanolamine  
NNN - N-nitrosornicotine  
NMU - N-nitrosomethylurea  
NEU - N-nitrosoethylurea  
NPU - N-nitrosopropylurea  
NMUT - N-nitrosomethylurethane  
NPRO - N-nitrosoproline  
NHPRO - N-nitrosohydroxyproline  
NSAR - N-nitrososarcosine

APPENDIX F

GETTY SYNTHETIC FUELS  
PROCESS DATA



Getty Synthetic Fuels, Inc. | 1467 Ring Road, Calumet City, IL 60409 • Telephone (312) 868-3700/3701

---

March 14, 1984

Mr. Larry Cattone  
Engineering Science  
No. 2 Flint Hill  
10521 Rosehaven Street  
Fairfax, VA 22030-2899

Dear Mr. Cattone:

Here are the zerox copies of plant records which covered the March 6, 1984, sampling of our facility. We utilized the readings from these charts to calculate the plant inlet and sales volumes. The discharge vent readings are from an indicator and do not have a 24 hour chart recorder.

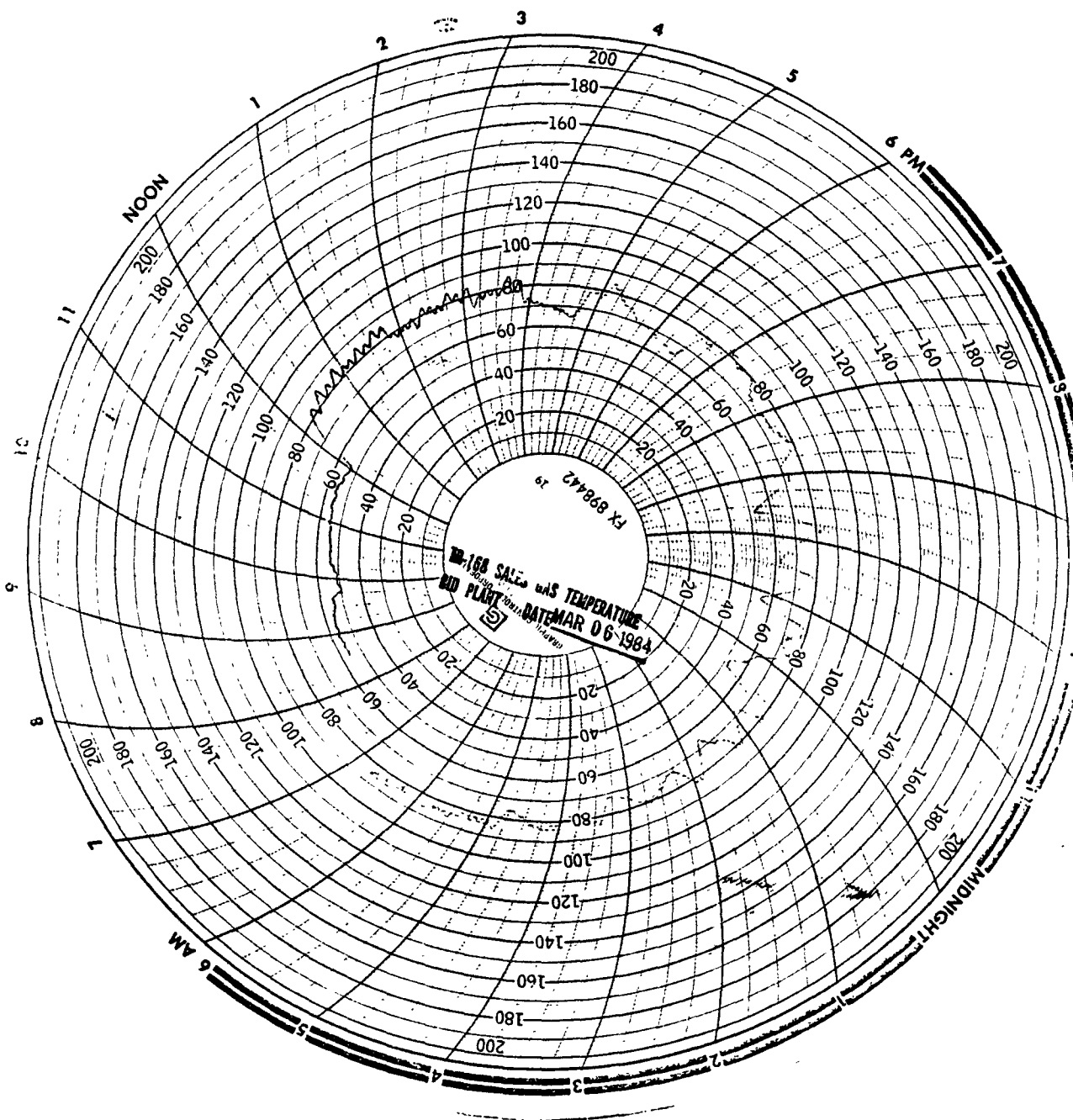
If you need any additional information, please feel free to call me or our California office (213-595-4964).

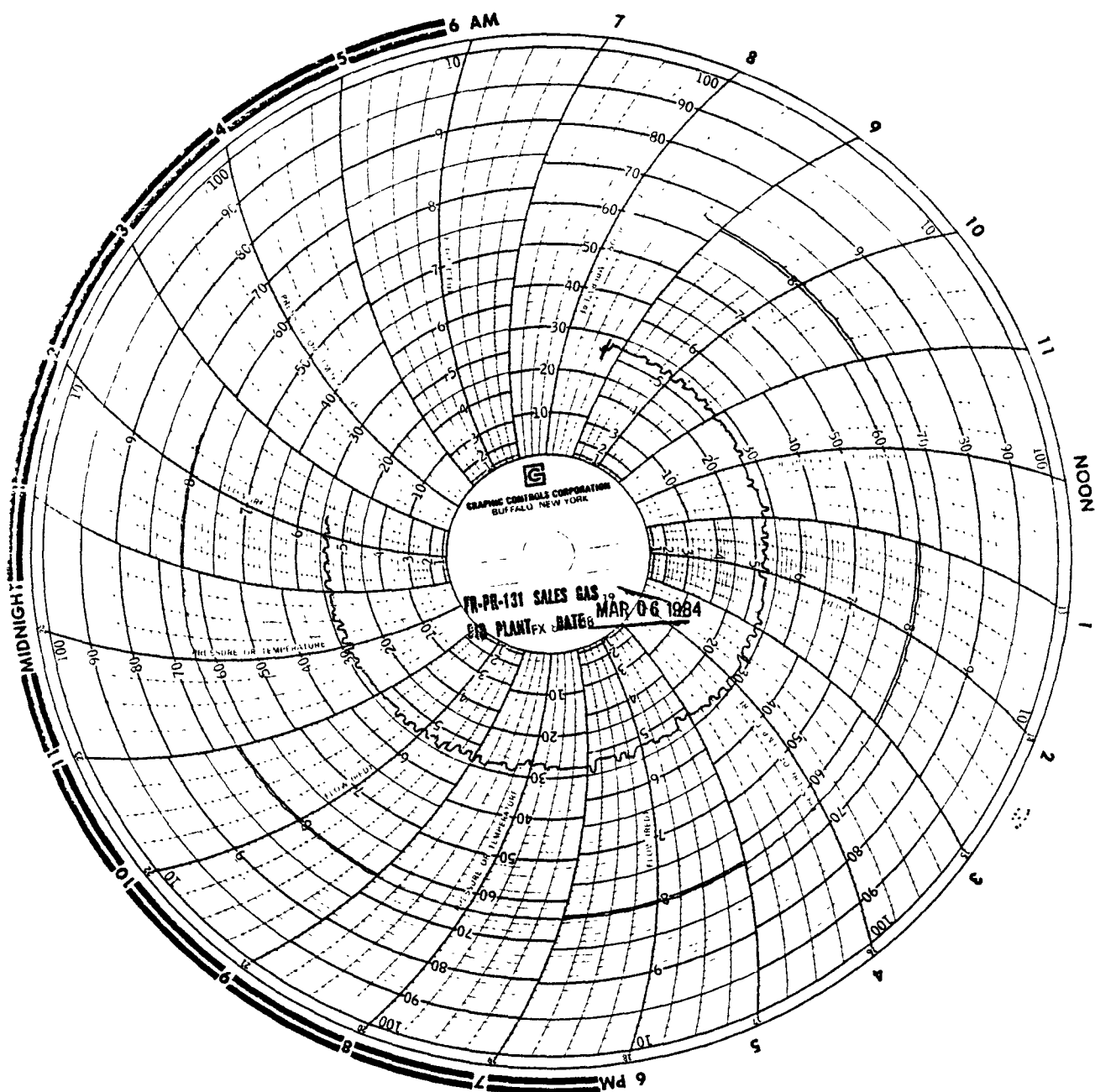
Sincerely,

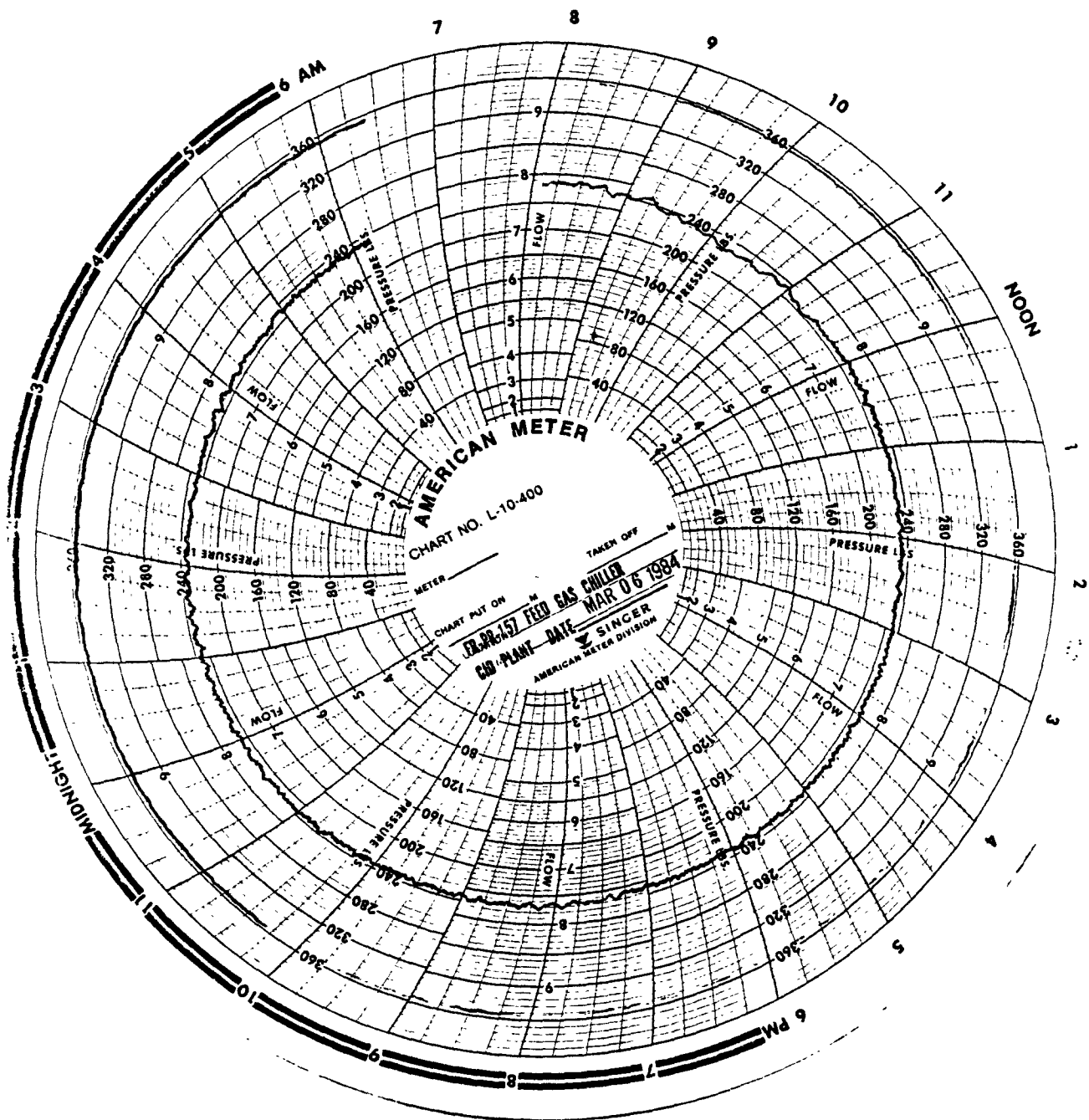
A handwritten signature in cursive script that reads "James A. Greenwell, Jr.".

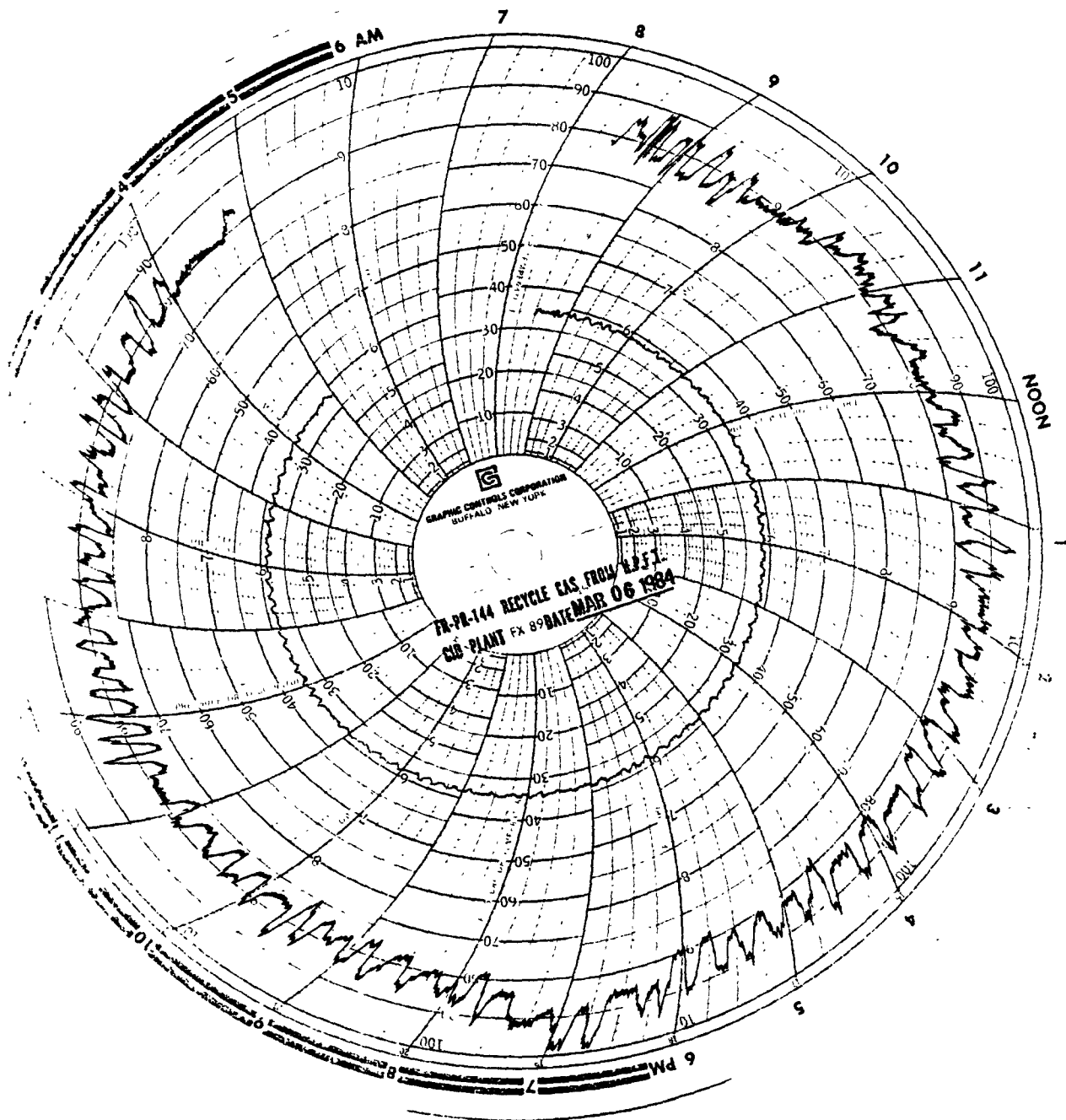
James A. Greenwell, Jr.  
Engineering Technician  
CID Plant  
Calumet City, Illinois 60409

JAG:jp









RANDY MASUKAWA  
EX 420

3-6-84

DATA FOR U.S. E.P.A. TESTING  
ON 3-5-84 TO 3-6-84

3-5-84

AVE INLET (MSCFD)	4138.104
AVE SALES (MSCFD)	2206.104
AVE VENT DISCHARGE (MSCFD)	1353.927

3-6-84

AVE INLET (MSCFD)	4062.283
AVE SALES (MSCFD)	2206.104
AVE VENT DISCHARGE (MSCFD)	1227.034

$\approx 10-15$  gpm total less than 1 gpm  
 $\frac{1}{2} - 1$  gpm - Check w/ Dick Jenkins on gas

Figures are an average for the total test period, and are volume figures for the test points where sampling occurred.



## APPENDIX G

### FIELD DATA SHEETS WITH GAS VOLUME CORRECTION TO STANDARD CONDITIONS

- o DNPH
- o XAD
- o Tenax®
- o Charcoal
- o Thermosorb™/N

**DNPH**

Run Number	Date	Start Time	End Time	Actual Gas Volume (Liters)	Meter Temp. (F°)	Meter V	Sample Gas Volume (Dry Stand- ard Liters)
D-F-6-5	3-6	1334	1339	2.095	37	0.96	2.137
D-F-6-6	3-6	1530	1546	13.000	37	0.96	13.258
D-F-6-6	3-6	1530	1546	18.062	38	0.98	18.767
D-F-6-4	3-6	1129	1153	19.075	43	0.98	18.767
D-F-5-3	3-5	1618	1658	57.784	44	0.96	58.114
D-C-6-5	3-6	1334	1336	1.886	37	0.96	1.923
D-C-6-4	3-6	1131	1144	18.813	46	0.96	18.846
D-C-5-3	3-5	1620	1700	49.843	43	0.99	51.797
D-P-6-5	3-6	1131	1133	1.970	26	1.00	2.140
D-P-6-4	3-6	1129	1142	18.900	33	1.00	20.242
D-P-5-3	3-5	1418	1458	51.602	33	1.00	55.265

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Gatty  
 DATE 3/6/84  
 RUN NUMBER D-F-6-5-1  
 OPERATOR Michael Crahan  
 AMBIENT TEMPERATURE (°F) \_\_\_\_\_  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) No Leak @ 19.5" Hg

OTHER DATA/NOTES \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1334	0	<del>0000.000</del> 825.195		37							1.0	6	
		1339	2	2.182											

\*This column for moisture determinations.

COMMENTS

104.7%  
 (0.4)

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Getty  
 DATE 3/6/84  
 RUN NUMBER D-F-6-6-1  
 OPERATOR Michael Gallagher  
 AMBIENT TEMPERATURE (°F) \_\_\_\_\_  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) No Leak @ 16" Hg  
96

OTHER DATA/NOTES \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

	Sorbent Tube No.	Clock Time (24 hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1530	0	0000.000		36								6	
		1534	4	3.800		37								6	
		1538	8	6.600		37								6	
		1542	12	-		39								6	
		1546	16	13.542		37								6	
Final leak check = ok @ 16.5" Hg															
				13.000											
				13.542											
TOTAL		.	.	.	.	.	.	.	.	.	.	.	.	.	.
AVERAGE		.	.	.	.	.	.	.	.	.	.	.	.	.	.

\*This column for moisture determinations.

COMMENTS

msd (ok)

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Getty O. 1  
 DATE 3/16/84  
 RUN NUMBER D-F-6-41  
 OPERATOR Michael Gallagher  
 AMBIENT TEMPERATURE (°F) 32  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) No Leak @ 15" Hg  
γ = 0.96

OTHER DATA/NOTES \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g <sup>a</sup>
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1129	0	5046.425		41								6	
		1132	3	-		42								6	
		1135	6	-		42								6	
		1138	9	5052.48		43								6	
		1141	12	5054.5		43								6	
		1144	15	5056.60		43								6	
		1147	18	5057.2		44								6	
		1150	21	5062.3		45								6	
		1153	24	5065.500											
				5065.500											
				Final	Leak check = ok @ 18"Hg										
				19.215											
				18.312											
TOTAL		.	.	.	.	.	.	.	.	.	.	.	.	.	.
AVERAGE		.	.	.	.	.	.	.	.	.	.	.	.	.	.

\*This column for moisture determinations.

COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Cetty  
DATE 3/6/84  
RUN NUMBER D-F66-11  
OPERATOR Michael Gallagher  
AMBIENT TEMPERATURE (°F) 29  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) No Leak @ 16" Hg  
98

**OTHER DATA/NOTES**

**SHEET** \_\_\_\_\_ **OF** \_\_\_\_\_

[illegible]

\*This column for moisture determinations.

## CONTENTS

fol mid





# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-6-84  
RUN NUMBER D-C-6-5-1  
OPERATOR KRASK  
AMBIENT TEMPERATURE (°F) 24  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 9"Hg, 30sec.

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

$V_{OST} = 0.96$   
 FLOW RATE  $\sim 1 \text{ lpm}$

[illegible]

\*This column for moisture determinations.

## CONTENTS

94.3% Co

# VOST FIELD DATA SHEET

LEAK CHECK (RATE) NH @ 6.5" Hg, 30 sec.

\_\_\_\_\_

\_\_\_\_\_

[illegible]

## COMMENTS

m.d  
OK

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT CETTY SYNTHETIC FUELS  
 DATE 3-5-84  
 RUN NUMBER D-C-5-3-1  
 OPERATOR KRASK  
 AMBIENT TEMPERATURE (°F) 32  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) ND @ 15" Hg for 30 seconds

OTHER DATA/NOTES \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
METER BOX - ES-11,  $\beta = 0.99$   
FLOW RATE  $\sim 1.5$  lpm  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

	Sorbent Tube No.	Clock Time (24 hr, Clock)	Sampling Time, Min.	704.663 I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*	
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)			
							Salt Water Out	Sample Gas Out								
		1620	0	704.663		43								0		
			5	704.88		43								0		
			10	705.10		43								0		
			15	705.324		43								0		
			20	705.54		43								0		
			25	705.77		43								0		
			30			43								0		
			35	706.21		45								0		
		1700	40	706.441		45								0		
			Purge rate ~1.5 lpm, begin 1704, end 1725													
TOTAL		•			•	•	•	•	•	•	•	•	•	•		
AVERAGE		•	•	•							•	•	•	•		

\*This column for moisture determinations.

COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Betty  
DATE 3/6/61  
RUN NUMBER D.P. 6 5-1  
OPERATOR Loggins  
AMBIENT TEMPERATURE (°F) 25  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) OK @ 1.60" H<sub>2</sub>O

OTHER DATA/NOTES \_\_\_\_\_

**SHEET      OF**

[illegible]

\*This column for moisture determinations.

## COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT 5. atty  
DATE 3/6/84  
RUN NUMBER D. P. 6.4.1  
OPERATOR LOTTM  
AMBIENT TEMPERATURE (°F) 36  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES D.P.W. 4.1  
TEST #2 DNP/N NT  
solar gas rate

SHEET 1 OF 1

 $\gamma = 100$ [illegible]

\*This column for moisture determinations.

## COMMENTS

Mid

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT SCOTT  
DATE 5-1-84  
RUN NUMBER P-P-E-3-1  
OPERATOR COTEAU  
AMBIENT TEMPERATURE (°F) 32°  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES ALL GAS - DNP  
TEST, run if

SHEET 1 OF 1

RATE	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
1.0		1418	0	67.188		33.									
1.0		1423	5			33									
1.0		1428	10	72.50		33									
		1433	15	74.30		33									
		1438	20	80.00		33									
		1443	25	92.00		33									
		1448	30	96.50		33.									
		1453	35	102.30		33									
		1458	40	111.790											

\*This column for moisture determinations.

## CONTENTS

Run Number	Date	Start Time	End Time	Actual Gas Volume (Liters)	Meter Temp. (F°)	Meter V	Sample Gas Volume (Dry Stand- ard Liters)
X-F-6-5	3-6	1430	1432	0.887	37	0.96	0.905
X-F-5-3	3-5	1914	1916	1.559	38	0.96	1.587
X-F-6-4	3-6	1003	1017	5.277	39	0.96	5.360
X-C-5-3	3-5	1916	1924	3.766	46	0.99	3.890
X-C-6-5	3-6	1426	1448	22.203	37	0.96	22.644
X-C-6-4	3-6	1006	1046	37.432	43	0.96	37.721
X-P-6-5	3-6	1428	1430	0.992	25	1.00	1.080
X-P-5-3	3-5	1914	1918	1.980	32	1.00	2.125
X-P-6-6	3-6	1612	1623	14.230	22	1.00	15.588
X-P-5-3	3-6	1609	1621	12.623	22	1.00	13.828
X-P-6-4	3-6	1003	1033	39.308	31	1.00	42.270

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Betty  
DATE 3/6/82  
RUN NUMBER X-F-6-5-1  
OPERATOR Michael Gallagher  
AMBIENT TEMPERATURE (°F) 30  
BAROMETRIC PRESSURE 29.49  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) No Leak @ 13.5" Hg  
X = .96

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_\_ OF \_\_\_\_\_

[illegible]

\*This column for moisture determinations.

## COMMENTS

85.2% to



# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Getty Oil  
DATE 3/5/84  
RUN NUMBER X-R-5-31  
OPERATOR Michael Gallagher  
AMBIENT TEMPERATURE (°F) \_\_\_\_\_  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) OK @ 11.5" Hg

OTHER DATA/NOTES \_\_\_\_\_

**SHEET**      **OF**      **\_\_\_\_\_**

[illegible]

\*This column for moisture determinations.

## COMMENTS

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Betty Oil  
 DATE 3/6/84  
 RUN NUMBER ~~1000~~ X-F-6-4-1  
 OPERATOR Michael Gallagher  
 AMBIENT TEMPERATURE (°F) \_\_\_\_\_  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) No Leak @ 11.5" Hg  
X 076

OTHER DATA/NOTES \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

	Sorbent Tube No.	Clock Time (24 hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1003	0	5033.417		37							1.0	5.5	
		1005	2	5034.035		38							1.0	5.5	
		1007	4	5034.8		38							1.0	5.5	
		1009	6	5035.583		38							1.0	5.5	
		1011	8	5036.83		39							1.0	5.5	
		1013	10	5037.29		40							1.0	5.5	
		1015	12	5038.0		40							1.0	5.5	
		1017	14	5038.914		41								5.5	
				No Leak @		11" Hg.									
							</								

\*This column for moisture determinations.

COMMENTS

88% FH

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-5-84  
RUN NUMBER X-C-5-3-1  
OPERATOR KRASK  
AMBIENT TEMPERATURE (°F) 31  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 7 1/4"

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_\_ OF \_\_\_\_\_

METER BOX ES-11 T=0.99  
FLOW RATE  $\sim 0.5$  gph

[illegible]

\*This column for moisture determinations.

## COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-6-84  
RUN NUMBER X-C-6-5-1  
OPERATOR ILR ASK  
AMBIENT TEMPERATURE (°F) 74  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 8" Hg, 3054

OTHER DATA/NOTES \_\_\_\_\_

**SHEET      OF**

WOST<sup>o</sup>C  $\gamma = 0.96$   
FLOW RATE  $\sim 1$  lpm

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final LITERS Gas Meter Reading -Ft <sup>3</sup>	TEMPERATURES (°C)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1426	0	0.000		2								0	
		1431	5	4.46		2								0	
		1436	10	9.44		3								0	
		1441	15	14.54		4								0	
		1446	20	19.56		4								0	
			22.5	22.203		4								0	
				21.515											
TOTAL		•			•	•	•	•	•	•	•	•	•	•	
AVERAGE		•	•	•							•	•	•	•	

\*This column for moisture determinations.

## COMMENTS

# ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-6-84  
RUN NUMBER X-C-104-1  
OPERATOR KIRASIK  
AMBIENT TEMPERATURE (°F) 26  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 7.5" Hg, 30 sec.

OTHER DATA/NOTES \_\_\_\_\_

SHEET OF

METER BOX - VAST °C,  $T = 0.96$   
FLOW ~ 1 LPM

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final  LITERS Gas Meter Reading <del>Ft</del>	TEMPERATURES ( $^{\circ}$ F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (l/min)		
							Salt Water Out	Sample Gas Out							
		1006	0	0.000		5								0	
		1011	5	4.35		7								0	
		1016	10	9.16		5								0	
		1021	15	14.		6								0	
		1026	20	18.57		6								0	
		1031	25			7								0	
		1036	30	27.94		7								0	
		1041	35	32.96		7								0	
		1046	39 $\frac{2}{4}$ c	37.950		8								0	
TOTAL		.		36.432	.	.	.	.	.	.	.	.	.	.	
AVERAGE		.	.	.							.	.	.	.	

\*This column for moisture determinations.

## COMMENTS

91.1% H

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT 6atty  
DATE 3/6/84  
RUN NUMBER X-8-6-5-1  
OPERATOR Cottone  
AMBIENT TEMPERATURE (°F) 25  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) OK @ 14" H<sub>2</sub>O

OTHER DATA/NOTES:

**SHEET OF**

X-P. 6-5-1  
Third Test XAP-2  
Sakayan'

[illegible]

\*This column for moisture determinations.

## CONTENTS

29 1/2 . 04

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT 6thly  
DATE 3/6/24  
RUN NUMBER X-P-6-6-1  
OPERATOR COTTON  
AMBIENT TEMPERATURE (°F) 22  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_\_ OF \_\_\_\_\_

Duplicate MS-2  
Sale gas

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g <sup>a</sup>
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
1.0		2412	0	647.678		22									
		2417	5	655.00		22									
		2421	9.0	659.00											
		2423	11.0	661.908		22									
														</	

\*This column for moisture determinations.

### COMMENTS

ok mid

# VOST FIELD DATA SHEET

LEAK CHECK (RATE)

---

**SHEET      OF**

	Sorbent Tube No.	Clock Time (24 hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
10		4:09	0	300.577		22									
		4:14	5	305.56											
1.0		4:14	10	311.40		22									
		4:21	12	313.22		22									

AA 12 OK



# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Getty  
DATE 2/5/84  
RUN NUMBER X-P-5-3-1  
OPERATOR Lottave  
AMBIENT TEMPERATURE (°F) 32  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES X-P-5-3-1  
Side gas - XAD-2 TEST 1

SHEET \_\_\_\_\_ OF \_\_\_\_\_

[illegible]

\*This column for moisture determinations.

## COMMENTS

# VOST FIELD DATA SHEET

PLANT Catty  
DATE 3/6/84  
RUN NUMBER X-P-6.4.1  
OPERATOR COTTON  
AMBIENT TEMPERATURE (°F) 26  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES X-P-64-1  
VAD-2 TEST 2 Asy Scale  
3rd

SHEET 1 OF 1

$$Y \approx 1.00$$

Sample Label	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Fe <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water Out	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
L.O		1023	0	195.542											
L.O		1808	5	201.70		31									
L.O		1013	10	208.50		30									
L.O		1018	15	215.55		31									
L.O		1023	20	222.60		31									
L.O		1028	25	224.60		31									
L.O		1033	30	234.850		21									
TOTAL		.		39.308	.	.	.	.	.	.	.	.	.	.	
AVERAGE		.	.	.							.	.	.	.	

\*This column for moisture determinations.

## COMMENTS

98.32 Hi

Run Number	Date	Start Time	End Time	Actual Gas Volume (Liters)	Meter Temp. (F°)	Meter V	Sample Gas Volume (Dry Stand- ard Liters)
T-S-5-1	3-5	1238	1258	12.048	32	1.00	12.930
T-S-5-2	3-5	1333	1353	12.110	32	1.00	12.996
T-F-6-5	3-6	1405	1407	0.290	37	0.96	0.296
T-F-6-6	3-6	1645	1648	0.623	44	0.98	0.640
T-F-6-6	3-6	1645	1648	0.625	45	0.96	0.627
T-F-5-3	3-5	1950	1955	0.945	36	0.96	0.966
T-F-6-4	3-6	1059	1105	2.884	41	0.96	2.918
T-C-6-5	3-6	1406	1410	0.875	36	0.99	0.922
T-C-5-3	3-5	1856	1904	6.054	45	0.99	6.266
T-C-6-4	3-6	1100	1115	8.086	45	0.96	8.116
T-P-6-5	3-6	1405	1407	0.482	26	1.00	0.524
T-P-6-4	3-6	1059	1112	8.951	32	1.00	9.606
T-P-5-3	3-6	1953	2006	17.095	30	1.00	18.421

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Quarry  
 DATE 3/5/84  
 RUN NUMBER T-5-5-1-1 through 4  
 OPERATOR W. T. W.  
 AMBIENT TEMPERATURE (°F) 32°F  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) 0.008 LPM at sample rate

OTHER DATA/NOTES E14 = T-5-5-1-1  
E15 = T-5-5-1-2  
E22 = T-5-5-1-3  
E3 = T-5-5-1-4  
HAIR 123

SHEET 1 OF 1

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g <sup>a</sup>
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
	506	1238	0	00.520		32			32						
		1243	5	03.60		32			32						
		1248	10	06.57		32			32						
		1253	15	09.50		32			32						
		1258	20	12.568											

<sup>a</sup>This column for moisture determinations.

COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Getty  
DATE 3/5/84  
RUN NUMBER T-5-5-2-1 through 4  
OPERATOR Corrigan  
AMBIENT TEMPERATURE (°F) 32  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) 0.018 @ pump rate

OTHER DATA/NOTES		
TS 5-2-1		E 28
TS 5-2-2		E 22
TS 5-2-3		E 21
TS 5-2-4		E 11

**SHEET OF**

Time	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final	TEMPERATURES ( <sup>o</sup> F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final
				Gas Meter Reading Ft <sup>3</sup>	1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		Weight Silica Gel, g*
							Salt Water Out	Sample Gas Out							
0.55	SEA NCE	1333	0	17.560			32								
0.60		1348	5	18.40											
0.58		1343	10	21.60			32								
0.57		1341	15	24.60											
0.55		1353	20	27.670			32								
TOTAL		.	20	. . .	.	.	.	.	.	.	.	.	.	.	
AVERAGE		.	.	+ + + + +							.	.	.	.	

<sup>a</sup>This column for moisture determinations.

## COMMENTS

# ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

PLANT Geth

DATE 3/6/84

RUN NUMBER T-F-6-J-1,2,3,4

OPERATOR Michael Gallagher

AMBIENT TEMPERATURE (°E)

### BAROMETRIC PRESSURE

RELATIVE HUMIDITY

LEARN CHECK (DATE) 1-1-16 @ 105" H.

 $\delta = .96$ 

\_\_\_\_\_

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\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

.....

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1-339

2 - 3/7

3-097

4-088

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final <i>Dry</i> Gas Meter Reading <i>ft<sup>3</sup></i>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g <sup>a</sup>
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1405	0	0000.000		37								5.5	
		1406.8	1.8	0002.920											
				0000.290											
				<i>Final</i>	<i>Leak check =</i>	<i>sh @ 14" Hg</i>									

## COMMENTS

92.8% to  
(5K)

# VOST FIELD DATA SHEET

PLANT Getty  
DATE 3/6  
RUN NUMBER T-E-6-6-11, 12, 13, 14  
OPERATOR \_\_\_\_\_  
AMBIENT TEMPERATURE (°F) \_\_\_\_\_  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) No Leak @ 14" Hg

OTHER DATA/NOTES:

**SHEET OF**

1 041  
2 046  
3 124  
4 072

[illegible]

\*This column for moisture determinations.

### COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

**SHEET      OF**

1 191  
2 099  
3 110  
4 019

[illegible]

## CONTENTS

Mid OK



# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Gottly J.I  
 DATE 3/5/84  
 RUN NUMBER T-F-5-3  
 OPERATOR Michael Gallagher  
 AMBIENT TEMPERATURE (°F) 30°  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) OK (2) 10.5" Hg  
δ = 0.96

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

1 E5  
 2 E35  
 3 E40  
 4 E31

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	Salt Water Out	Sample Gas Out	Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
		1950	0	5023.780		36							.2	5.5	
		1952	2	5024.3		36							.2	5.5	
		1954	4	5024.6		36							.2	5.5	
		1955	5	5024.764											
Final Leak Check = OK (2) 17" Hg															
TOTAL		.	.	.	.	.	.	.	.	.	.	.	.	.	.
AVERAGE		.	.	.	.	.	.	.	.	.	.	.	.	.	.

\*This column for moisture determinations.

COMMENTS

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Getty Oil  
 DATE 3/6/84  
 RUN NUMBER T.F-6-4  
 OPERATOR Michael Gallagher  
 AMBIENT TEMPERATURE (°F) 32.0  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) No Leak @ 21.5"Hg

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

1 10 21  
 2 10 21  
 3 10 21  
 4 10 21

8-96

	Sorbent Tube No.	Clock Time (24 hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		10 59	0	5042.768		41			44				6		
		11 01	2	5043.7		41			44				6		
		11 03	4	5044.8		41			44				6		
		11 05	6	5045.772											
Final leak check = ok @ 13.5" Hg															

\*This column for moisture determinations.

COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT \_\_\_\_\_  
DATE 3-6-84  
RUN NUMBER T-C-6-5-(1-4)  
OPERATOR KIRASK  
AMBIENT TEMPERATURE (°F) 24  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 5' Hg, 30 sec.

OTHER DATA/NOTES

TUBES 273, 336, 143, E22

VOID %  $\gamma = 0.99$

Flow rate  $\approx 0.2$  gpm

**SHEET OF**

[illegible]

\*This column for moisture determinations.

## CONTENTS

7.5% Lo

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-5-84  
RUN NUMBER T-C-5-3-(1-4)  
OPERATOR ICRASK  
AMBIENT TEMPERATURE (°F) 30  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 10.5" Hg

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_\_ OF \_\_\_\_\_

METER BOX LS-11  $T=0.99$   
FLOWRATE  $\sim 1$  gpm

[illegible]

\*This column for moisture determinations.

## COMMENTS

OK and

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-6-84  
RUN NUMBER T-C-6-4-(1-4)  
OPERATOR KRASK  
AMBIENT TEMPERATURE (°F) \_\_\_\_\_  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 6.5" Hg, 30sec

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_\_ OF \_\_\_\_\_

TUBES E-7A E8, E26, E-19

METER BOX - VOLT  $^{\circ}\text{C}$   $T = 0.96$   
Flow  $\sim 0.5 \text{ lpm}$

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final <i>LITERS</i> Gas Meter Reading <i>Ft<sup>3</sup></i>	TEMPERATURES ( $^{\circ}$ C)						PROBE COOLING FLOWS			Pump Vacuum (In Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	Salt Water Out	Sample Gas Out	Ambient (VOST)	Leak Check Vacuum (in Hg)	Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
		1100	0	0000		7								0	
		1105	5	2.21		8								0	
		1110	10	4.66		7								0	
		1115	15	7.19		8								0	
			17 <sup>23</sup> / <sub>60</sub>	8.423		7								0	
TOTAL	.	.	.	8.086	.	.	.	.	.	.	.	.	.	.	
AVERAGE	.	.	.	.							.	.	.	.	

\*This column for moisture determinations.

## COMENTE

89.9% 1di

# ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

PLANT 601  
DATE 3/6/84  
RUN NUMBER 1-1-6 5-1 T-1 TP-6-5 4  
OPERATOR Catone  
AMBIENT TEMPERATURE (°F) 26  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_\_ OF \_\_\_\_\_

T-P-6-5-1	E 157
T-P-6-5-2	E 079
T-P-6-5-3	E 080
T-P-6-5-4	E 13

[illegible]

\*This column for moisture determinations.

## COMMENTS

96.4% LD  
OK

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Betty  
DATE 3/6/84  
RUN NUMBER T-8-6-4-1 through 4  
OPERATOR COSTONE  
AMBIENT TEMPERATURE (°F) 28  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) OK @ 1.56" H<sub>2</sub>O  
Y=1.00

OTHER DATA/NOTES

T.P. 6.4.1 = E 43

T.P. 6.4.2 = E 25

T.P. 6.4.3 = E 34

T.P. 6.4.4

SHEET 1 OF 1

<i>Samp Label</i>	Sorbent Tube No.	Clock Time (24 hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
<i>LPM</i>															
0.5		1059	0	237.85U		31									
0.5		1104	5	237.30		32									
0.5		1109	10	241.60		32									
0.5		1112	13	273.80F		32									
TOTAL		.	.	2.951	.	.	.	.	.	.	.	.	.	.	
AVERAGE		.	.	.							.	.	.	.	

\*This column for moisture determinations.

AA: 2

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Betty  
DATE 3/5/84  
RUN NUMBER T.P. 5-32  
OPERATOR Cottrell  
AMBIENT TEMPERATURE (°F) 31  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

$$Y = 1.00$$

OTHER DATA/NOTES TENIX Sol<sub>2</sub> TEST 1

T-P-5-3-1	= E18
T-P-5-3-2	= E37
T-P-5-3-3	= E16
T-P-5-3-4	= E42

**SHEET      OF**

<i>Sample Rate L/min</i>	Sorbent Tube No.	Clock Time (24 hr Clock)	Sampling Time, Min.	I = Initial F = Final	TEMPERATURES ( $^{\circ}\text{F}$ )					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final
				Gas Meter Reading Ft <sup>3</sup>	1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (l/min)		Weight Silica Gel, g <sup>a</sup>
							Salt Water Out	Sample Gas Out							
1.0		1953	0	160.360			30								
1.0		1958	5	167.20			30								
		2003	10	174.40											
		2006	13	177.455											
TOTAL		.			.	.	.	.	.	.	.	.	.	.	
AVERAGE		.	.	.						.	.	.	.		

\*This column for moisture determinations.

## COMMENTS

~~100~~ 95% H<sub>2</sub>



CHARCOAL

Run Number	Date	Start Time	End Time	Actual Gas Volume (Liters)	Meter Temp. (F°)	Meter V	Sample Gas Volume (Dry Stand- ard Liters)
C-S-5-1	3-5	1405	1420	16.150	32	1.00	17.332
C-S-5-2	3-5	1428	1443	15.12	32	1.00	16.226
C-F-6-5	3-6	1033	1034	0.665	42	0.96	0.671
C-F-6-6	3-6	1204	1209	0.906	46	0.96	0.908
C-F-6-7	3-6	1452	1457	1.603	38	0.96	1.632
C-F-6-4	3-6	0858	0911	2.070	28	0.96	2.150
C-F-5-3	3-5	1728	1758	7.146	38	0.96	7.273
C-C-6-5	3-6	1216	1225	1.825	43	0.96	1.839
C-C-6-6	3-6	1518	1535	3.732	39	0.96	3.791
C-C-6-6	3-6	1519	1536	3.532	46	0.99	3.649
C-C-6-4	3-6	0901	0931	5.340	37	0.96	5.446
C-C-5-3	3-6	1731	1801	10.195	44	0.99	10.574
C-P-6-5	3-6	1203	1208	1.850	27	1.00	2.006
C-P-5-3	3-5	1728	1758	11.32	32	1.00	12.148
C-P-6-4	3-6	0900	0940	16.13	30	1.00	17.381

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Betty  
DATE 3-5-84  
RUN NUMBER C-5-5-1-1  
OPERATOR COTTONE  
AMBIENT TEMPERATURE (°F) 32  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) 0.015 Lpm @ 1 Lpm. T

OTHER DATA/NOTES LAIRIT 1245

SHEET 1 OF 1

[illegible]

\*This column for moisture determinations.

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Gitty  
 DATE 3/5/84  
 RUN NUMBER C-5-5-2-1  
 OPERATOR COTTON  
 AMBIENT TEMPERATURE (°F) \_\_\_\_\_  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) 0.013 L/min @ 1072

OTHER DATA/NOTES C-5-5-2-1  
ANALYST GAS  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET 1 OF 1

LATE (LPM)	Sorbent Tube No.	Clock Time (24 hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (l/min)		
1	235-2-1	1428	0	42422		32									
		1433	5	4810											
		1438	10	5520		12									
		1443	15	59.038											
TOTAL		•	15	1002	•	•	•	•	•	•	•	•	•	•	
AVERAGE		•	•	55.0							•	•	•	•	

\*This column for moisture determinations.

COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Gatty Oil  
DATE 3/6/84  
RUN NUMBER C-F-6-5-1  
OPERATOR Michael Gallagher  
AMBIENT TEMPERATURE (°F) 31°  
BAROMETRIC PRESSURE 29.48  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) No Leak @ 12" Hg  
 $\delta = .96$

OTHER DATA/NOTES \_\_\_\_\_

**SHEET**      **OF**

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final	
				Gas Meter Reading ft³	1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H₂O (cc/min)	Air (L/min)		Weight Silica Gel, g <sup>a</sup>	
							Salt Water Out	Sample Gas Out								
		1033	0	5039.470			42									
			1.5	5040.163												
				Final leak check = ok @ 19" Hg												
							</									

\*This column for moisture determinations.

## CONTENTS

# VOST FIELD DATA SHEET

LEAK CHECK (RATE) No Leak @ 15" Hg

.....

SHEET \_\_\_\_\_ OF \_\_\_\_\_

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)				Leak Check Vacuum (in Hg)	Flow Rate PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g <sup>a</sup>	
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE			Ambient (VOST)	Salt Water (gpm)	H <sub>2</sub> O (cc/min)			Air (L/min)
							Salt Water Out	Sample Gas Out							
	1728	0	0	5012.564		35						2	5.5		
	1738	10	10	5015.8		38						2	5.5		
	1748	20	20	5017.9		42						2	5.5		
	1758	30	30	5020.008											
				Final leak check = 100% leak @ 10" Hg											
							</								

\*This column for moisture determinations.

## COMMENTS

## ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

## PLANT

DATE \_\_\_\_\_

**RUN NUMBER**

**OPERATOR**

AMBIENT TEMPERATURE (°F) 29

### **BAROMETRIC PRESSURE**

## RELATIVE HUMIDITY

**LEAK CHECK (RATE)**

OTHER DATA/NOTES

**SHEET      OF**

$$\gamma = 0.96$$
**TOTAL**

## AVERAGE

\*This column for moisture determinations.

1/2 1/2

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Getty  
DATE 3/6/64  
RUN NUMBER C.F-6-7-1  
OPERATOR Michael Gallagher  
AMBIENT TEMPERATURE (°F) 31  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) No leak @ 13.5" Hg

OTHER DATA/NOTES:

SHEET \_\_\_\_\_ OF \_\_\_\_\_

[illegible]

\*This column for moisture determinations.

## COMMENTS

OK Mid



# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT Getty O.I.  
 DATE 3/6/84  
 RUN NUMBER C-F-6-4-1  
 OPERATOR Michael Gallagher  
 AMBIENT TEMPERATURE (°F) 27°  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) No Leak @ 12.5" Hg  
δ = 0.96

OTHER DATA/NOTES \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		0858	0	5025.108		28							.2	5.5	
		0900	2	5025.4		27							.2	5.5	
		0902	4	5025.82		28							.2	5.5	
		0904	6	5026.21		28							.2	5.5	
		0906	8	5026.55		28							.2	5.5	
		0908	10	5026.8		29							.2	5.5	
		0910	12	5027.17		29							.2	5.5	
		0910.5	12.5	5027.264		30							.2	5.5	
				Final leak check - No Leak @ 11" Hg											
TOTAL		•		2.070	•	•	•	•	•	•	•	•	•	•	
AVERAGE		•	•	•							•	•	•	•	

\*This column for moisture determinations.

80% H<sub>2</sub>O

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-6-84  
RUN NUMBER C-C-10-5-1  
OPERATOR KRASIS  
AMBIENT TEMPERATURE (°F) 24  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 7" Hg 30 sec

OTHER DATA/NOTES \_\_\_\_\_

**SHEET OF**

$T_{OST} = 0.96$   
 Flowrate  $\sim 0.2$  gpm

[illegible]

\*This column for moisture determinations.

## COMMENTS

91.2% to  
(OK)

**SHEET OF**

$V_{pST} = 0.96$   
Flow rate  $\sim 0.2$  L/min

[illegible]

\*This column for moisture determinations.

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
 DATE 3-6-84  
 RUN NUMBER C-C-6-6-11  
 OPERATOR KIRASIK  
 AMBIENT TEMPERATURE (°F) 24  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) ND @ 10" Hg, 30 SLG.

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

METER BOX ES-11  $\gamma=0.99$   
Flow rate ND, 2 Lp n-

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1519	0	728.801		47								0	
		1524	5	728.859		47								0	
		1529	10	728.890		46								0	
		1534	15	728.902		45								0	
		1536 1/2	17.15	728.927											
				0.125											
				0.115											
TOTAL		•		3.532	•	•	•	•	•	•	•	•	•	•	
AVERAGE		•	•	•							•	•	•	•	

\*This column for moisture determinations.

COMMENTS

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-6-84  
RUN NUMBER C-C-6-4-1  
OPERATOR KRASK  
AMBIENT TEMPERATURE (°F) 22  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 14" Hg for 30 sec.

OTHER DATA/NOTES \_\_\_\_\_

SHEET \_\_\_\_\_ OF \_\_\_\_\_

METAL BOX - WOST °C  
FLOW RATE - ~ 0.2 L/min  
 $V = 0.96$

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final  LITERS Gas Meter Reading	TEMPERATURES (°C)				Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*	
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE			Ambient (VOST)	Salt Water (gpm)	H <sub>2</sub> O (cc/min)			Air (L/min)
							Salt Water Out	Sample Gas Out							
		0901	0	0.000		0							0		
		0904	5	0.82		0							0		
		0911	10	1.65		1							0		
		0916	15	2.6		2							0		
		0921	20	3.55		3							0		
			25	4.58		4							0		
		0931	30.5	5.562		4							0		
						</									

\*This column for moisture determinations.

29% OK

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-5-84  
RUN NUMBER C-C-5-3-1  
OPERATOR KR Ask  
AMBIENT TEMPERATURE (°F) 32  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 15" Hg for 30 sec.

OTHER DATA/NOTES \_\_\_\_\_

**SHEET** \_\_\_\_\_ **OF** \_\_\_\_\_

METER BOX - ES-11  $T = 0.99$   
FLOW RATE -  $\sim 0.2$  gpm

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final)  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final)  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (l/min)		
							Salt Water Out	Sample Gas Out							
		1734	<del>0</del>	706.461		45								0	
			5	706.52		44								0	
			10	706.58		44								0	
			15	706.64		44								0	
			20	706.70		44								0	
			25	706.76		44								0	
			30	706.820		45								0	
											</				

\*This column for moisture determinations.

## CONTENTS

1011 to  
1012 to 1013

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Battery  
DATE 3/6/64  
RUN NUMBER C-P-6-5-1  
OPERATOR Cotton  
AMBIENT TEMPERATURE (°F) 26  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES

**SHEET** 1 **OF** 1

Charcoal  
Test 3  
Soda gas

$$Y = 1.00$$
[illegible]

\*This column for moisture determinations.

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# VOST FIELD DATA SHEET

**LEAK CHECK (RATE)**

Water  $\gamma = 1.00$

[illegible]

## COMMENTS

(9)



# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT CORTY  
 DATE 3/6/84  
 RUN NUMBER C.P. 6 4-1  
 OPERATOR COSTA  
 AMBIENT TEMPERATURE (°F) 27  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) OK @ 16" H<sub>2</sub>O  
Y=1.00

OTHER DATA/NOTES C.P. 6-4-1  
Sale gas 2<sup>ND</sup> TEST.  
Charcoal

SHEET 1 OF 1

Sample into LPM	Sorbent Tube No.	Clock Time (24 hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (l/min)		
0.2		0930	0	177.452		28									
0.2		0935	5	178.90		30									
0.2		0940	10	180.40		30									
0.2		0945	15	183.10		31									
0.2		0950	20	185.16		31									
0.2		0955	25	187.20		30									
0.2		1000	30	187.20		30									
0.2		1005	35	191.20		30									
0.2		1010	40	193.590											
TOTAL				16.132											
AVERAGE															

\*This column for moisture determinations.

on 7% H<sub>2</sub>O

THERMOSORB™/N

Run Number	Date	Start Time	End Time	Actual Gas Volume (Liters)	Meter Temp. (F°)	Meter V	Sample Gas Volume (Dry Stand- ard Liters)
N-F-6-4	3-6	0949	0951	1.926	41	0.98	1.989
N-F-6-5	3-6	1310	1320	9.012	37	0.96	9.191
N-F-5-3	3-5	1829	1853	24.355	44	0.98	25.004
N-C-6-4	3-6	0951	0954	1.920	35	0.96	1.966
N-C-6-5	3-6	1312	1324	10.938	37	0.96	11.155
N-C-5-3	3-5	1831	1856	20.825	45	0.99	21.556
N-P-6-4	3-6	0948	0950	1.953	30	1.00	2.104
N-P-6-5	3-6	1309	1317	9.958	25	1.00	10.840
N-P-6-6	3-6	1457	1515	22.583	25	1.00	24.585
N-P-6-3	3-5	1829	1854	35.268	32	1.00	37.849

**A10073**

OTHER DATA/NOTES

**SHEET      OF**

\*This column for moisture determinations.

## COMMENTS

94% LO  
(OK)

# ENGINEERING-SCIENCE VOST FIELD DATA SHEET

# A10076  
Water

PLANT Refinery Oil  
 DATE 3/6/84  
 RUN NUMBER N-F-6-5-1  
 OPERATOR Michael Gallagher  
 AMBIENT TEMPERATURE (°F) 29  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 RELATIVE HUMIDITY \_\_\_\_\_  
 LEAK CHECK (RATE) No Leak @ 135" Hg  
80.96

OTHER DATA/NOTES \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SHEET \_\_\_\_ OF \_\_\_\_

	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (In Hg)	PROBE COOLING FLOWS			Pump Vacuum (In Hg)	I = Initial F = Final Weight Silica Gel, g <sup>a</sup>
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
		1310	0	5170.262		36							1.0	6.5	
		1312	2	5171.9		37							1.0	6.5	
		1314	4	5174.0		38							1.0	6.5	
		1316	6	—		36							1.0	6.5	
		1318	8	5177.8		36							1.0	6.5	
		1320	10	5179.650											
				Final Leak Check at @ 12" Hg											
				9.012											
TOTAL		•			•	•	•	•	•	•	•	•	•	•	
AVERAGE		•	•	•							•	•	•	•	

<sup>a</sup>This column for moisture determinations.

# ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

PLANT Croton oil

DATE 3/5/84

RUN NUMBER N-1-5-3-1

OPERATOR *Michael Gallagher*

AMBIENT TEMPERATURE (°F) 31°F J

**BAROMETRIC PRESSURE** \_\_\_\_\_

### RELATIVE HUMIDITY

LEAK CHECK (RATE)  $1\frac{1}{2}$  Lbs. @ 15" Hg

OTHER DATA/NOTES \_\_\_\_\_

SHEET OF

[illegible]

\*This column for moisture determinations.

## CONTENTS

N-F-5-1-1 = A10064

**A10064**

**A10072**

OTHER DATA/NOTES

METER BOX - VOLT  $\epsilon$   $T=0.96$   
FLOW RATE - 1 gpm

**SHEET      OF**

[illegible]

\*This column for moisture determinations.

## CONTENTS

# ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-6-84  
RUN NUMBER N-C-6-5-1  
OPERATOR KR ASIK  
AMBIENT TEMPERATURE (°F) 25  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) ND @ 14" Hg, 30 sec

OTHER DATA/NOTES \_\_\_\_\_

SHEET 1 OF 1

WOST °C  $\gamma = 196$   
FLOW RATE  $\sim 1.0 \text{ gpm}$

### ii. Air Sampler

Air Sampler  
A10078

11 ~~AT 100-75~~  
Mallory

[illegible]

\*This column for moisture determinations.

## COMMENTS

Mid  
Ck



# ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

PLANT GETTY SYNTHETIC FUELS  
DATE 3-5-84  
RUN NUMBER N-C-5-3-1  
OPERATOR KRASK  
AMBIENT TEMPERATURE (°F) 31  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) MD @ 11"Hg for 30 sec.

OTHER DATA/NOTES \_\_\_\_\_

**SHEET      OF**

METER BOX ES-11  $\tau = 0.99$   
FLOW RATE  $\sim 1 \text{ gpm}$

[illegible]

\*This column for moisture determinations.

## CONTENTS

# ENGINEERING-SCIENCE

# VOST FIELD DATA SHEET

## PLANT

DATE \_\_\_\_\_

**RUN NUMBER**

**OPERATOR**

AMBIENT TEMPERATURE (°F)

### BAROMETRIC PRESSURE

## RELATIVE HUMIDITY

**LEAK CHECK (RATE)** $\gamma = 1.00$ 

OTHER DATA/NOTES

Thermistor, Sale gas

Test #2

**A10071**

SHEET \_\_\_\_\_ OF \_\_\_\_\_

[illegible]

## COMMENTS

97.7%  $\text{L}_{\text{OH}}$

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT 6. City  
DATE 3/6/84  
RUN NUMBER N.P. 6-5-1  
OPERATOR Cotton  
AMBIENT TEMPERATURE (°F) 26  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES \_\_\_\_\_

SHEET OF

Thermosalt Test 3  
Salt gas

**A10074**

[illegible]

\*This column for moisture determinations.

ok mid

# VOST FIELD DATA SHEET

**LEAK CHECK (RATE)**

**A10079**

SHEET OF

				I = Initial F = Final	TEMPERATURES ( <sup>o</sup> F)						PROBE COOLING FLOWS				I = Initial F = Final
	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	Gas Meter Reading Ft <sup>3</sup>	1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)	Leak Check Vacuum (in Hg)	Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (l/min)	Pump Vacuum (in Hg)	Weight Silica Gel, g <sup>a</sup>
							Salt Water Out	Sample Gas Out							
L <sub>9</sub> M															
10		2:57	0	277.442		25									
		3:02	5	283.60		25									
1.0		3:06	10	288.60		25									
1.0		3:12	15	295.20		25									
		13:17	20	300.575		25									
				22.585											
TOTAL		.			.	.	.	.	.	.	.	.	.	.	
AVERAGE		.	.	.							.	.	.	.	

## COMMENTS

903/5 H  
(oh)

# ENGINEERING-SCIENCE

## VOST FIELD DATA SHEET

PLANT Gethy  
DATE 3/6/84  
RUN NUMBER N-1-6-11  
OPERATOR Cotton  
AMBIENT TEMPERATURE (°F) 24  
BAROMETRIC PRESSURE \_\_\_\_\_  
RELATIVE HUMIDITY \_\_\_\_\_  
LEAK CHECK (RATE) \_\_\_\_\_

OTHER DATA/NOTES \_\_\_\_\_

SHEET OF

P-P-6-6-11  
Thermozet duplicate  
A10077  
Müller

$$Y = 1.00$$
[illegible]

\*This column for moisture determinations.

# VOST FIELD DATA SHEET

**LEAK CHECK (RATE)**

A10070

08

Sample Lab (L/M)	Sorbent Tube No.	Clock Time (24 Hr. Clock)	Sampling Time, Min.	I = Initial F = Final  Gas Meter Reading Ft <sup>3</sup>	TEMPERATURES (°F)					Leak Check Vacuum (in Hg)	PROBE COOLING FLOWS			Pump Vacuum (in Hg)	I = Initial F = Final  Weight Silica Gel, g*
					1st Condenser Outlet	Dry Gas Meter Outlet	PROBE		Ambient (VOST)		Salt Water (gpm)	H <sub>2</sub> O (cc/min)	Air (L/min)		
							Salt Water Out	Sample Gas Out							
1.0		1824	0	123.1/2		32									
1.0		1834	5	130.10		32									
1.0		1839	10	137.70		32									
1.0		1844	15	143.00		32									
1.0		1849	20	157.70		32									
1.0		1854	25	158.300											

## COMMENTS

**A10065**  
Matter