



## Technical Service Report

Report No. 2126-79-839-1

To R.E. Laramy

From G.W. Keen

Date November 28, 1979

Subject The GCMS Analysis of Treatment Influent and Effluent Waters from the Lake Charles Vinyl Chloride Plant

Continental Oil Company  
Research and Development Department  
Analytical Research Section  
Ponca City, Oklahoma

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### Object

To determine the levels of the organic components in the plant effluent and treatment influent water samples collected in October, 1979.

### Conclusions

1. The organic components are summarized in Table 1.
2. GC traces and their interpretation are provided for future reference
3. Headspace and direct injection procedures were used to provide data for ethanol, vinyl chloride, 2-chloroethanol, trichloroacetic acid, and dichloroacetic acid. The reported concentrations for these components are approximations. No internal standards were used. A quantitative, direct injection GC method should be developed if the levels of these components are important.

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Senior Research Scientist

jkm

Distribution EAS FK AJL CMS

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### Discussion

The quantitative data in this report have been back-calculated and reported on an original-sample basis. Several assumptions have been made. We have assumed 100% extraction efficiencies, no losses in the extraction and Kuderna-Danish concentration steps, 100% purging efficiencies, 100% trapping efficiencies and equal mass spectrometry response factors.

The concentrations of the extractable organics were calculated through the use of a D<sub>10</sub>-anthracene internal standard. The solutions were injected with a sample size that would insure the detection of this internal standard and priority pollutants at the 5 ppb level. This obviously leads to extrapolation problems when the sample contains contaminants at the several-thousand ppb level. The reported concentrations, especially the high ones, should be considered order-of-magnitude estimates.

Therefore, the quantitative data contained in this report can only be used as a guide to the relative amounts of material in the samples analyzed. The nature of the samples and the intended use of the analytical data obtained do not justify the cost required to obtain strenuous quantitative results.

### Experimental

Base/neutral and acidic extracts were provided by the Separations Analysis Group. These extracts were analyzed with the Finnigan 4023 GCMS-DS. The 30M SP2100 glass capillary column was programmed from 60° to 260°C at 8°/minute.

Internal standards of bromochloromethane, 1-chloro-2-bromopropane and 1,4-dichlorobutane were added to 5 ml of each sample to be analyzed. The organics were purged with helium, trapped in a silica gel-Tenax absorber tube at room temperature and driven onto the packed column injector by heating the Tenax tube rapidly to 180°C. The GC column was a 6' x 1/4" carbowax 1500 (.2%) on Carbowax C preceded by 2' x 1/4" carbowax 1500 (3%) on Chromosorb W. The glass column was held below room temperature during the thermal elution of the sample from the Tenax column. It was heated rapidly to 60°C, then programmed to 170°C at 8°/minute. It was held at 170° until the run was finished. Mass spectra were recorded at 1.1 second intervals.

### References

File of GCMS data: G542A - 551A  
G554A - 555A

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TABLE I

Summary of the Organic Components

| Component                        | Influent to Treatment | Effluent |
|----------------------------------|-----------------------|----------|
| vinyl chloride                   | 60 ppb                |          |
| ethyl chloride                   | 5000-6000 ppb         |          |
| methylene chloride               | 300-600 ppb           |          |
| ethanol                          | * 9000 ppb            |          |
| acetone                          | 0-100 ppb             |          |
| 1,1-dichloroethane               | † 200-1700 ppb        |          |
| 1,1-dichloroethylene             | † 10-300 ppb          |          |
| 1,2-dichloroethylene             | † 70-200 ppb          |          |
| chloroform                       | > 40000 ppb           | 0-10 ppb |
| 1,2-dichloroethane               | > 200000 ppb          | 20 ppb   |
| carbon tetrachloride             | † 50-1200 ppb         |          |
| trichloroacetic acid             | * 3000 ppb            |          |
| trichloroethylene                | 60 ppb                |          |
| 2-chloroethanol                  | * 40000 ppb           |          |
| 1,1,2-trichloroethane            | 3000 ppb              | 10 ppb   |
| tetrachloroethylene              | 10-40 ppb             |          |
| dichloroacetic acid              | * 70000 ppb           |          |
| chlorobenzene                    | 150 ppb               |          |
| toluene                          | 30 ppb                |          |
| 1,1,2,2-tetrachloroethane        | 50 ppb                |          |
| trichlorobutanes                 | 110 ppb               |          |
| tetrachlorobutanes               | 260 ppb               |          |
| tetrachlorobutenes               | 400 ppb               |          |
| pentachlorobutanes               | 100 ppb               |          |
| pentachlorobutenes               | 10 ppb                |          |
| hexachlorobutenes                | 40 ppb                |          |
| dichlorobenzene                  | 40 ppb                |          |
| xylene                           | 30 ppb                |          |
| bis(chloroethyl) ether           | 400 ppb               |          |
| 2-chloroethyldichlorobutyl ether | 28 ppb                | 10 ppb   |
| tetrachlorodioxadecanol          | 15 ppb                | 8 ppb    |
| pentachlorodioxadodecanol        | 9 ppb                 | 12 ppb   |

† The uncertainties in these values were revealed by the headspace and direct injection analyses. The EPA - recommended absorber tubes may be inefficient for 1,1-dichloroethane, dichloroethylenes and carbon tetrachloride.

\* These levels were estimated from a direct injection using no internal standard.

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LCVCP Influent VOA See Figure 1

| Scan#   | Identification            | Concentration |
|---------|---------------------------|---------------|
| 64      | ethyl chloride            | 5000 ppb      |
| 85      | methylene chloride        | 300 ppb       |
| 93      | 1,1-dichloroethylene      | 10 ppb        |
| 100     | acetone                   | 100 ppb       |
| 120     | 1,1-dichloroethane        | 200 ppb       |
| 130     | dichloroethylene          | 70 ppb        |
| 138     | chloroform                | > 40000 ppb   |
| 150-170 | 1,2-dichloroethane        | > 200000 ppb  |
| 177     | carbon tetrachloride      | 50 ppb        |
| 246     | 2-chloroethanol           | 300 ppb       |
| 284     | branched paraffin         | 60 ppb        |
| 303     | 1,1,2-trichloroethane     | 3000 ppb      |
| 477     | tetrachloroethylene       | 30 ppb        |
| 504     | toluene                   | 30 ppb        |
| 508     | 1,1,2,2-tetrachloroethane | 30 ppb        |
| 564     | chlorobenzene             | 70 ppb        |
| 686     | trichlorobutane           | 70 ppb        |
| 741     | tetrachlorobutene         | 170 ppb       |
| 782     | xylenes                   | 30 ppb        |
| 811     | pentachlorobutane         | 160 ppb       |
| 900     | dichlorobenzene           | 40 ppb        |

LCVCP Effluent VOA See Figure 2

| Scan # | Identification        | Concentration |
|--------|-----------------------|---------------|
| 154    | 1,2-dichloroethane    | 20 ppb        |
| 297    | 1,1,2-trichloroethane | 10 ppb        |

VCM Pump Blank Base Neutrals See Figure 3

| Scan# | Identification                 | Concentration |
|-------|--------------------------------|---------------|
| 92    | 4-methyl-4-penten-2-one        | 70 ppb        |
| 110   | 4-methyl-3-penten-2-one        | 1200 ppb      |
| 129   | 4-methyl-4-hydroxy-2-pentanone | 1200 ppb      |
| 182   | tetrachloropropane             | 70 ppb        |
| 257   | 3-methyl-2-cyclohexen-1-one    | 20 ppb        |
| 342   | trimethylhexene                | 20 ppb        |
| 346   | acetone trimer                 | 20 ppb        |
| 549   | silastic tubing impurity       | 150 ppb       |
| 624   | hexachloropentane              | 10 ppb        |
| 743   | diethyl phthalate              | 40 ppb        |
| 1604  | di(2-ethylhexyl) phthalate     | 13 ppb        |

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VCM Pump Blank Acids See Figure 4

| Scan # | Identification                 | Concentration |
|--------|--------------------------------|---------------|
| 90     | 4-methyl-4-penten-2-one        | 30 ppb        |
| 107    | 4-methyl-3-penten-2-one        | 770 ppb       |
| 124    | 4-methyl-4-hydroxy-2-pentanone | 600 ppb       |
| 182    | tetrachloropropane             | 30 ppb        |
| 257    | 3-methyl-2-cyclohexen-1-one    | 30 ppb        |
| 343    | trimethylhexene                | 20 ppb        |
| 744    | diethyl phthalate              | 11 ppb        |

VCM Effluent Base/Neutrals See Figure 5

| Scan # | Identification                    | Concentration |
|--------|-----------------------------------|---------------|
| 105    | 4-methyl-3-penten-2-one           | * 300 ppb     |
| 121    | 4-methyl-4-hydroxy-2-pentanone    | * 240 ppb     |
| 180    | tetrachloropropane                | * 30 ppb      |
| 259    | 3-methyl-2-cyclohexen-1-one       | * 10 ppb      |
| 342    | trimethylhexane                   | * 20 ppb      |
| 390    | silastic tubing impurity          | * 60 ppb      |
| 457    | silastic tubing impurity          | * 11 ppb      |
| 525    | 2-chloroethyl dichlorobutyl ether | 10 ppb        |
| 559    | silastic tubing impurity          | *1700 ppb     |
| 584    | tetrachlorodioxadecanol           | 8 ppb         |
| 625    | hexachloropentane (same as blank) | * 10 ppb      |
| 685    | pentachlorodioxadecanol           | 12 ppb        |
| 743    | diethyl phthalate                 | * 9 ppb       |

\* These components were observed in the blank.

VCM Effluent Acids See Figure 6

| Scan # | Identification           | Concentration |
|--------|--------------------------|---------------|
| 105    | 4-methyl-3-penten-2-one  | 120 ppb       |
| 181    | tetrachloropropane       | 11 ppb        |
| 549    | silastic tubing impurity | 90 ppb        |

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VCM Influent to Treatment Base/Neutrals See Figures 7 and 8

| Scan # | Identification                    | Concentration |
|--------|-----------------------------------|---------------|
| 97     | 1,1,2-trichloroethane             | 1200 ppb      |
| 127    | chlorobenzene                     | 40 ppb        |
| 154    | 1,1,2,2-tetrachloroethane         | 50 ppb        |
| 181    | trichlorobutene                   | 20 ppb        |
| 204    | bis(chloroethyl) ether            | 400 ppb       |
| 216    | tetrachlorobutane                 | 30 ppb        |
| 241    | trichlorobutane                   | 90 ppb        |
| 255    | tetrachlorobutane                 | 24 ppb        |
| 261    | tetrachlorobutane                 | 200 ppb       |
| 273    | tetrachlorobutane                 | 11 ppb        |
| 293    | tetrachlorobutene                 | 100 ppb       |
| 307    | tetrachlorobutene                 | 24 ppb        |
| 323    | tetrachlorobutene                 | 60 ppb        |
| 338    | tetrachlorobutene                 | 300 ppb       |
| 350    | tetrachlorobutene                 | 10 ppb        |
| 364    | tetrachlorobutene                 | 16 ppb        |
| 371    | tetrachlorobutene                 | 20 ppb        |
| 392    | silastic tubing impurity          | 120 ppb       |
| 419    | pentachlorobutene                 | 8 ppb         |
| 435    | pentachlorobutane                 | 100 ppb       |
| 458    | silastic tubing impurity          | 15 ppb        |
| 476    | hexachlorobutene                  | 37 ppb        |
| 523    | 2-chloroethyl dichlorobutyl ether | 28 ppb        |
| 560    | silastic tubing impurity          | 1800 ppb      |
| 585    | tetrachlorodioxadecanol           | 15 ppb        |
| 744    | diethyl phthalate                 | 10 ppb        |

Influent to Treatment Acids See Figure 9

| Scan # | Identification           | Concentration |
|--------|--------------------------|---------------|
| 93     | 1,1,2-trichloroethane    | 11 ppb        |
| 183    | tetrachloropropane       | 12 ppb        |
| 315    | bromocyclohexanol        | 12 ppb        |
| 401    | silastic tubing impurity | 50 ppb        |

Influent Direct Injection See Figure 10

| Scan # | Identification                    | Concentration |
|--------|-----------------------------------|---------------|
| 26     | carbon dioxide                    | > 100000 ppb  |
| 53     | ethyl chloride                    | 300 ppb       |
| 95     | ethanol                           | 9000 ppb      |
| 171    | column artifact (caused by water) | 12000 ppb     |
| 193    | chloroform                        | 30000 ppb     |
| 208    | 1,2-dichloroethane                | > 200000 ppb  |
| 293    | trichloroacetic acid              | 3000 ppb      |

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| Scan # | Identification        | Concentration |
|--------|-----------------------|---------------|
| 327    | 2-chloroethanol       | 40000 ppb     |
| 399    | 1,1,2-trichloroethane | 3000 ppb      |
| 440    | dichloroacetic acid   | 70000 ppb     |

\* These concentrations are only estimates. The 1,1,2-trichloroethane concentration was assumed to be 3000 ppb as determined by the purge-and-trap analysis. Others were calculated using their response areas relative to

#### Effluent Direct Injection

Only carbon dioxide and an artifact peak from the large water slug were observed.

#### Effluent Headspace See Figure 11

This analysis was not designed to replace the classical and more accurate GC headspace analysis, but was run at the time of the other analyses in case the Separation Analysis Group had identification problems in their GC headspace analysis.

| Scan # | Identification     | Concentration |
|--------|--------------------|---------------|
| 211    | chloroform         | 10 ppb        |
| 224    | 1,2-dichloroethane | 20 ppb        |
| 395    | syringe impurity   | 50 ppb        |

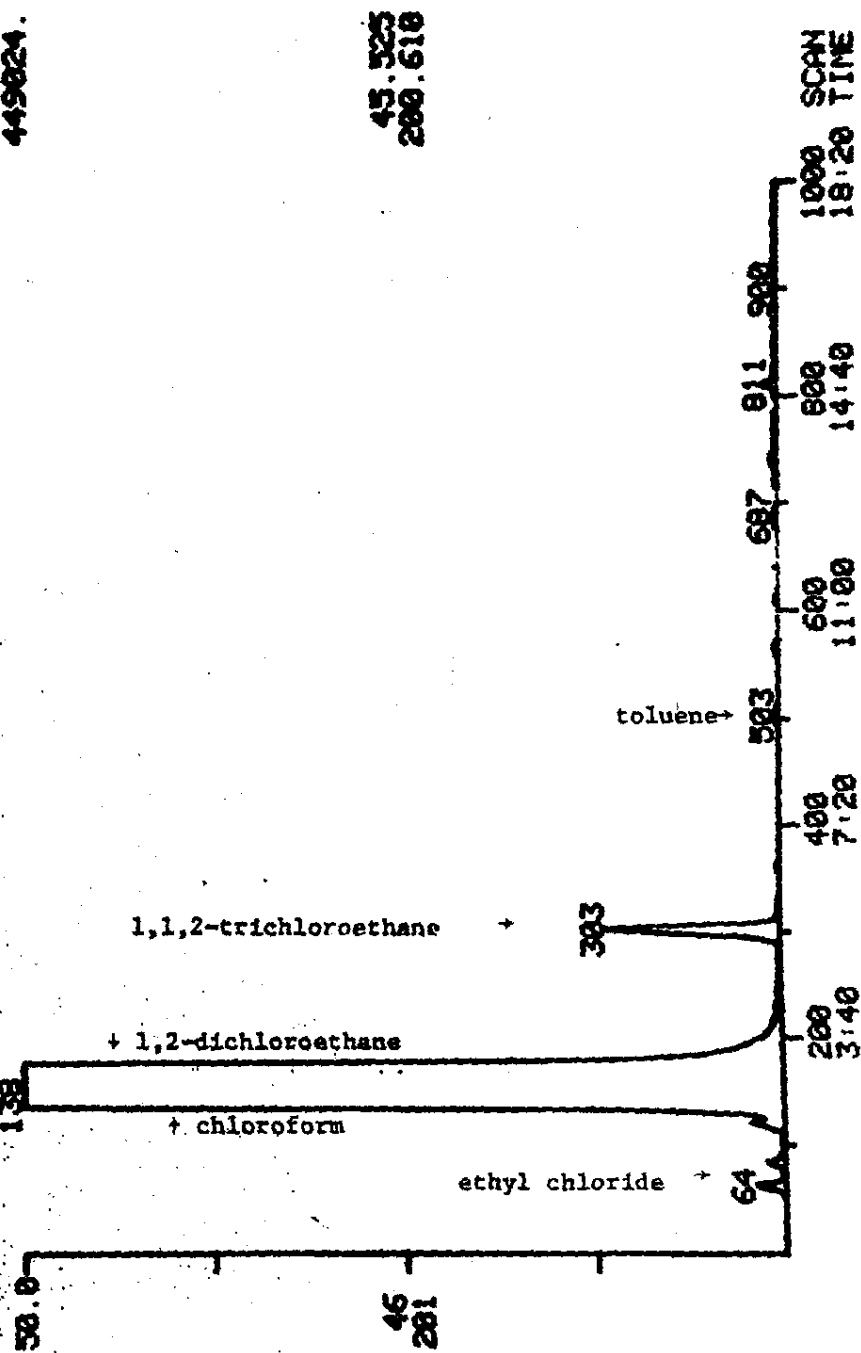
#### Influent to Treatment Headspace See Figure 12

| Scan # | Identification        | Concentration |
|--------|-----------------------|---------------|
| 54     | vinyl chloride        | 60 ppb        |
| 64     | ethyl chloride        | 6000 ppb      |
| 107    | methylene chloride    | 600 ppb       |
| 128    | dichloroethylene      | 300 ppb       |
| 171    | 1,1-dichloroethane    | 1700 ppb      |
| 195    | dichloroethylene      | 200 ppb       |
| 215    | chloroform            | >40000 ppb    |
| 232    | 1,2-dichloroethane    | >70000 ppb    |
| 262    | carbon tetrachloride  | 1200 ppb      |
| 309    | trichloroacetic acid  | 10 ppb        |
| 357    | trichloroethylene     | 60 ppb        |
| 413    | 1,1,2-trichloroethane | 3000 ppb      |
| 599    | tetrachloroethylene   | 40 ppb        |
| 695    | chlorobenzene         | 150 ppb       |

Fig 1  
Influent Volatile Organics (Purge and Trap)

MASS CHROMATOGRAM  
10/10/79 11:24:00  
DATA: C343A #1  
CALI: C911F #1  
SAMPLE: UCN 598 INFLUENT UOA  
RANGE: G 1.1500 LABEL: N 0.4.0 QUAN: A 0.1.0 BASE: U 20. 3  
449024.

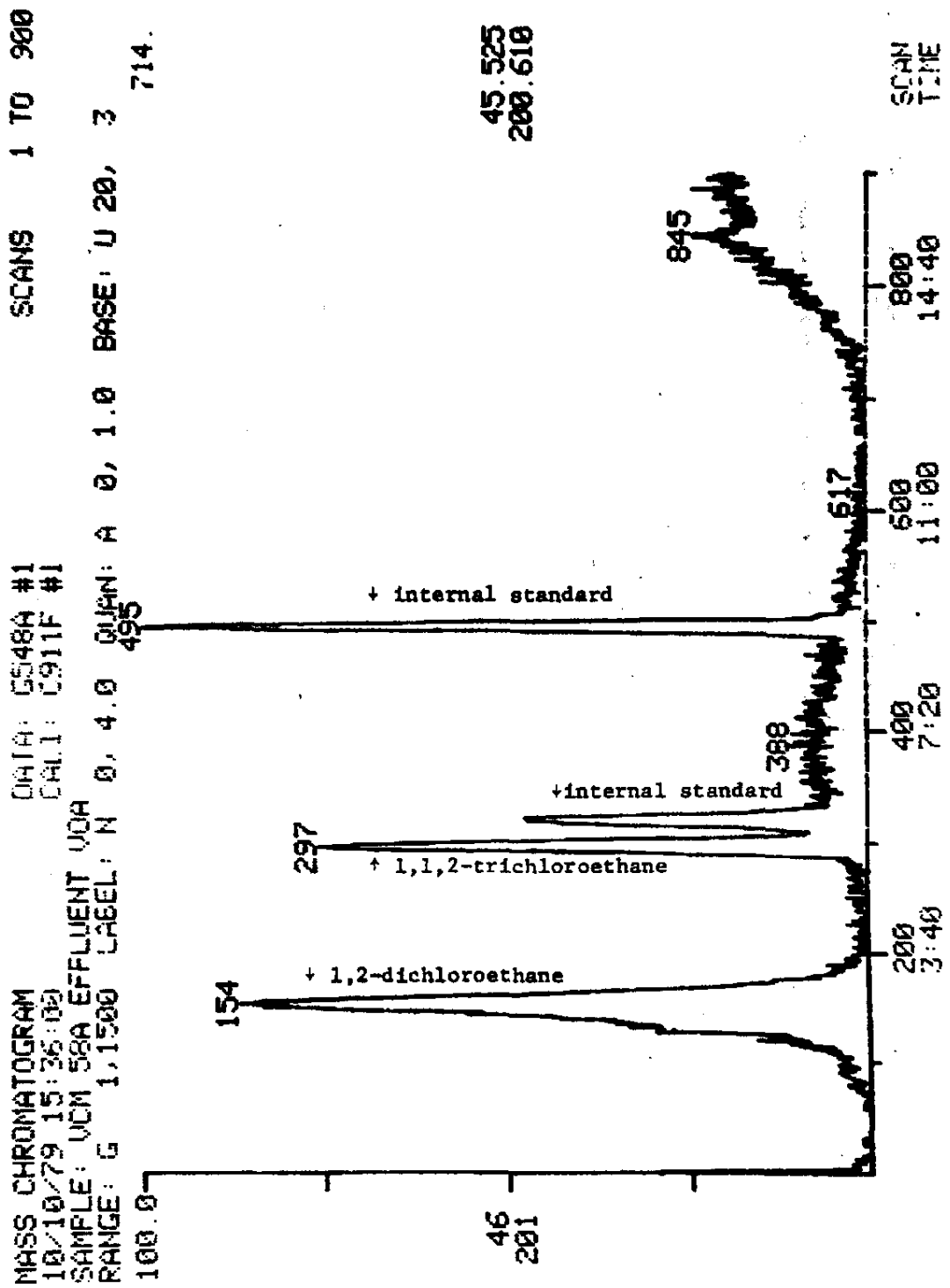
SCANS 1 TO 1000



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Figure 1  
Effluent Volatile Organics (Purge and Trap)



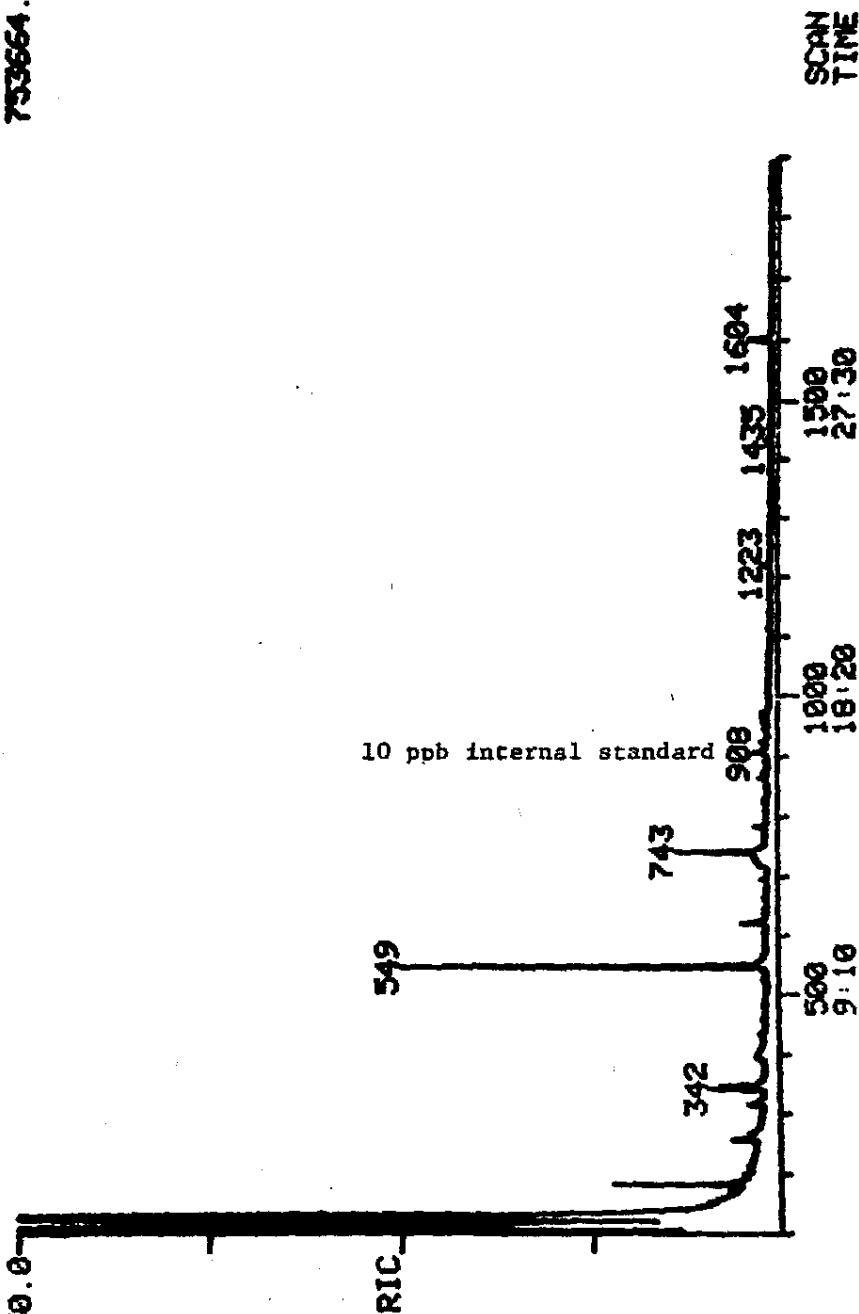
VEV-266076

VVC 000012822

Fig. 3

Pump Blank Base/Neutrals

RIC 10/15/79 10:02:00 DATA: G557A #1 SCANS 100 TO 1900  
10/15/79 10:02:00 CALI: C1015A #1  
SAMPLE: VCM PUMP BLANK (B/N) 10-79  
RANGE: C 1.3500 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3 753664.



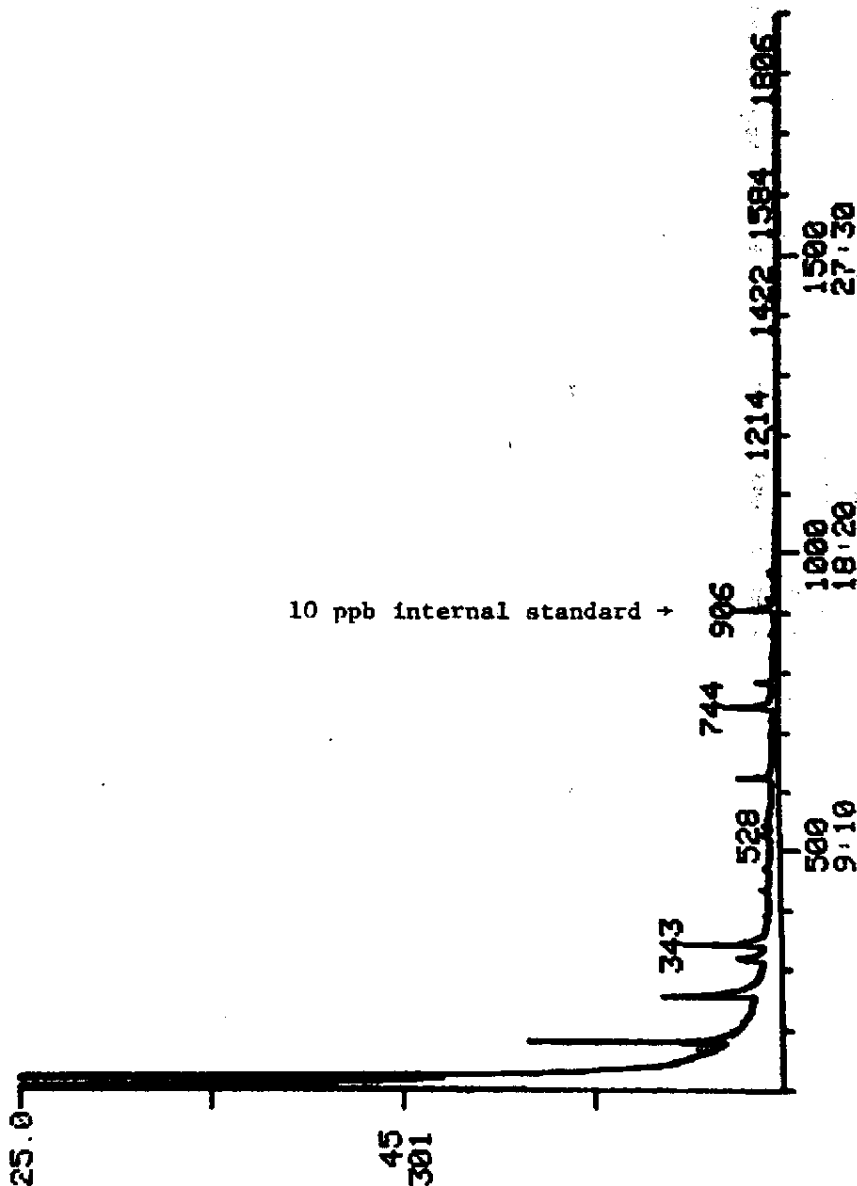
VVC 000012823

VEV-266077

Fig 4

Pump Blank Acids

MASS CHROMATOGRAM DATA: G538A #1  
10/15/79 11:16:00 CALI: C1015A #1  
SAMPLE: UCM PUMP BLANK (ACIDS) 10-79  
RANGE: G 1.3500 LABEL: N 0.4.0 QUAN: A 0.1.0 BASE: U 20. 3  
25.0 200704.

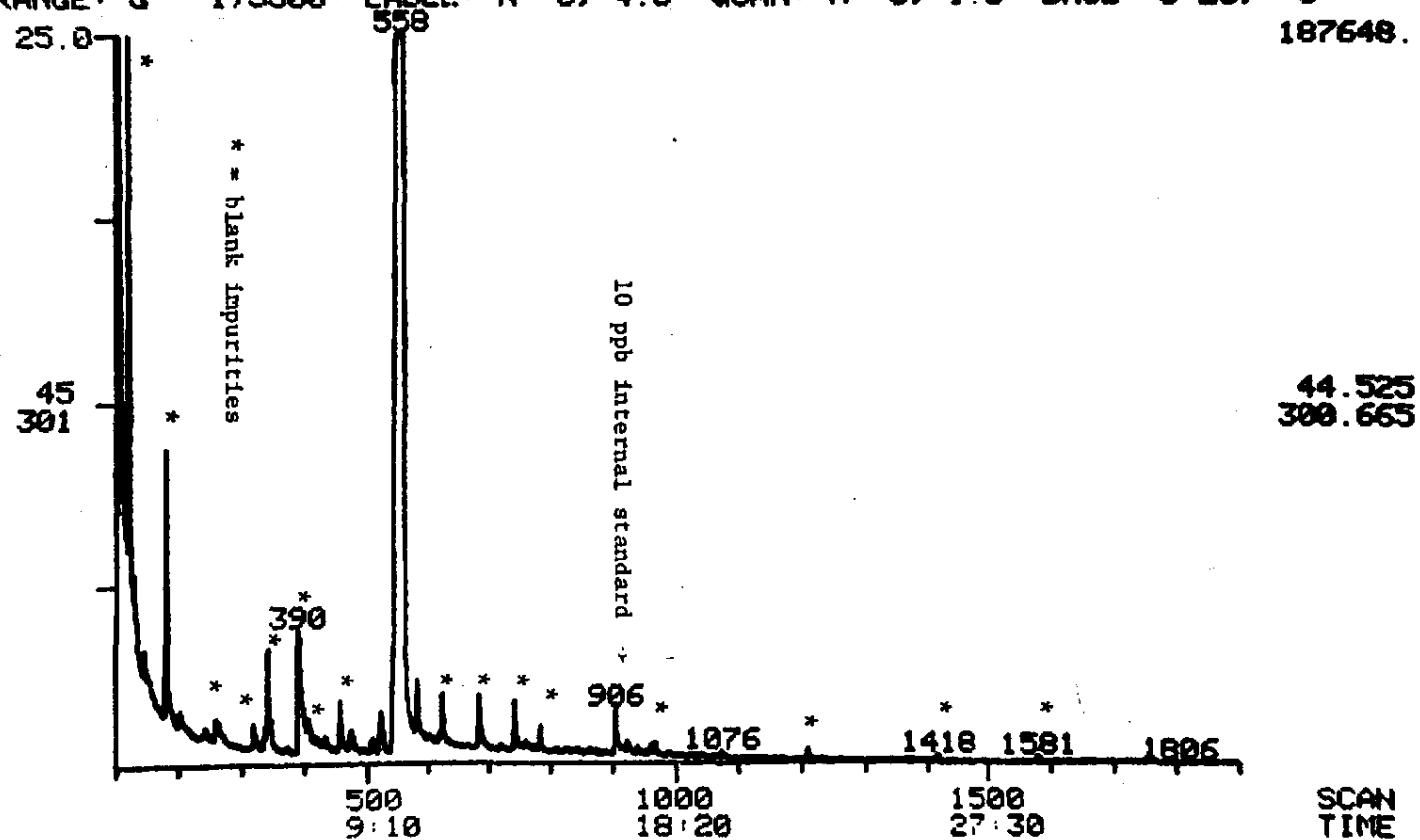


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VVC 000012824

### **Effluent Extract Base/Neutrals**

**187648.**



CHRO:

VVC 000012825

VEV-266079

Effluent Extract Acids

MASS CHROMATOGRAM  
 10/15/79 13:50:00  
 DATA: G560A #1  
 CALI: C1015A #1  
 SAMPLE: UCM EFFLUENT (ACIDS) 10-79  
 RANGE: G 1.3500 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3  
 25.0 549  
 SCANS 100 TO 1900  
 69120.

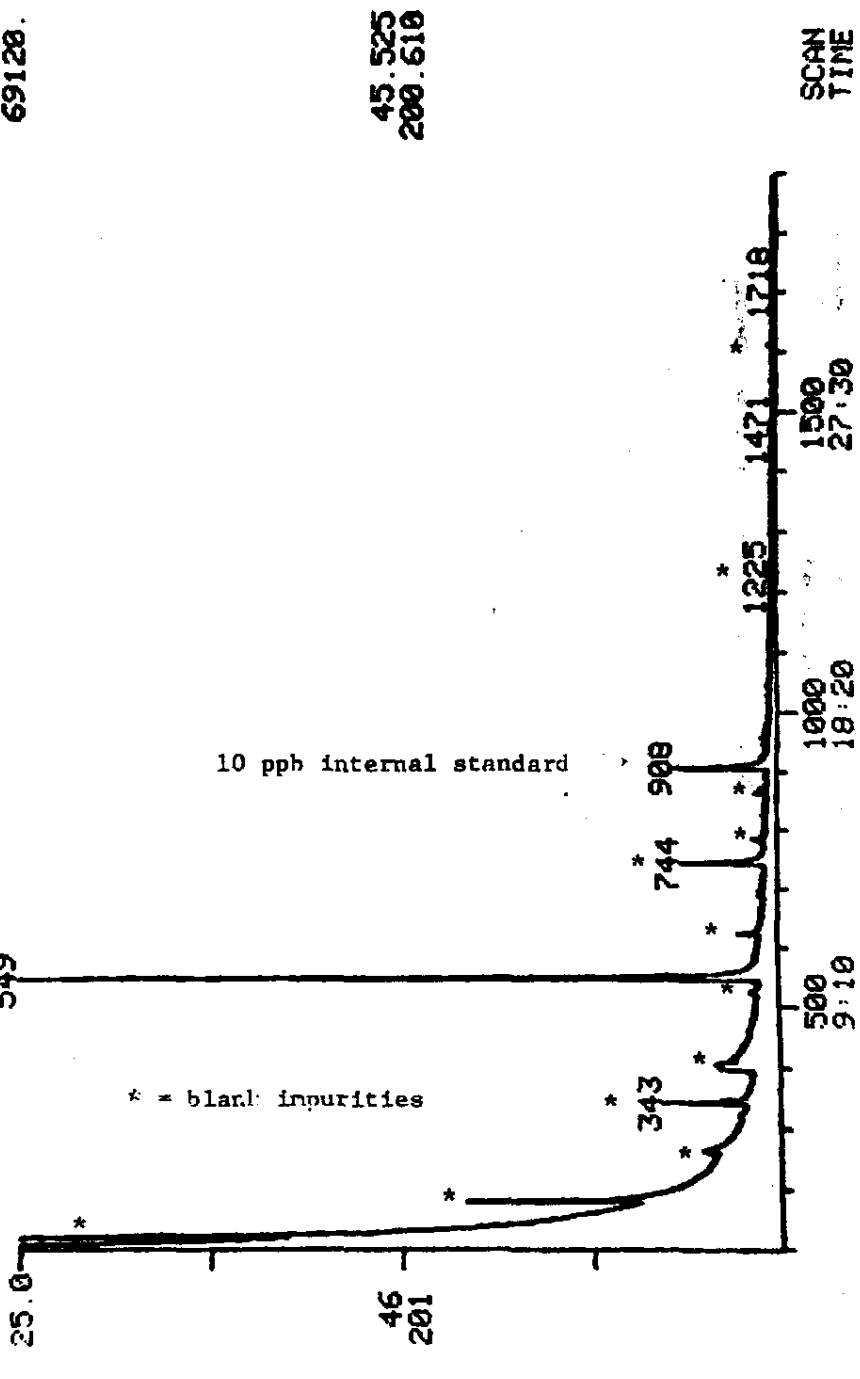
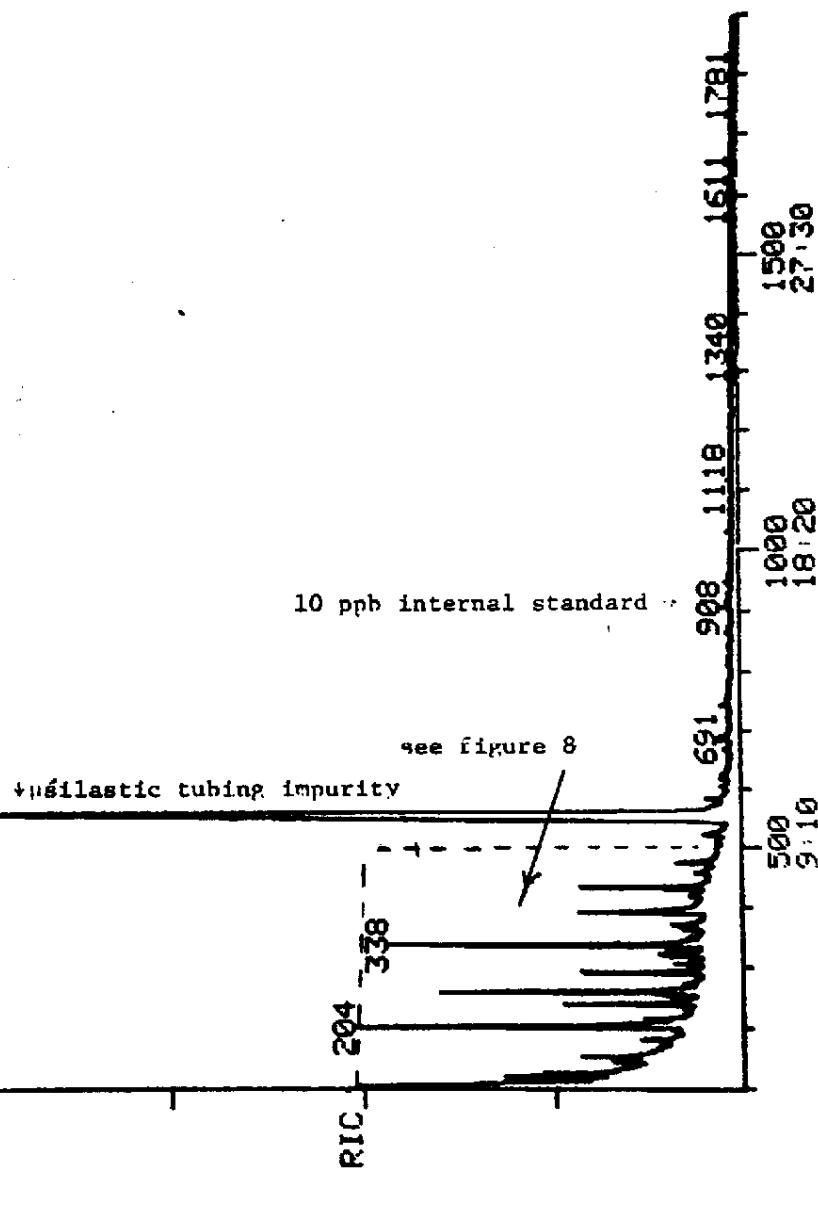


Figure 7  
Influent Extract Base/Neutrals

RIC  
 10/15/79 15:04:00  
 SAMPLE: UCM INFLUENT TO TREATMENT (B/N) 10-79  
 RANGE: G 1.3500 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3  
 100.0  
 DATA: G361A #1  
 CALI: C1015A #1  
 SCANS 100 TO 1900  
 1146870.

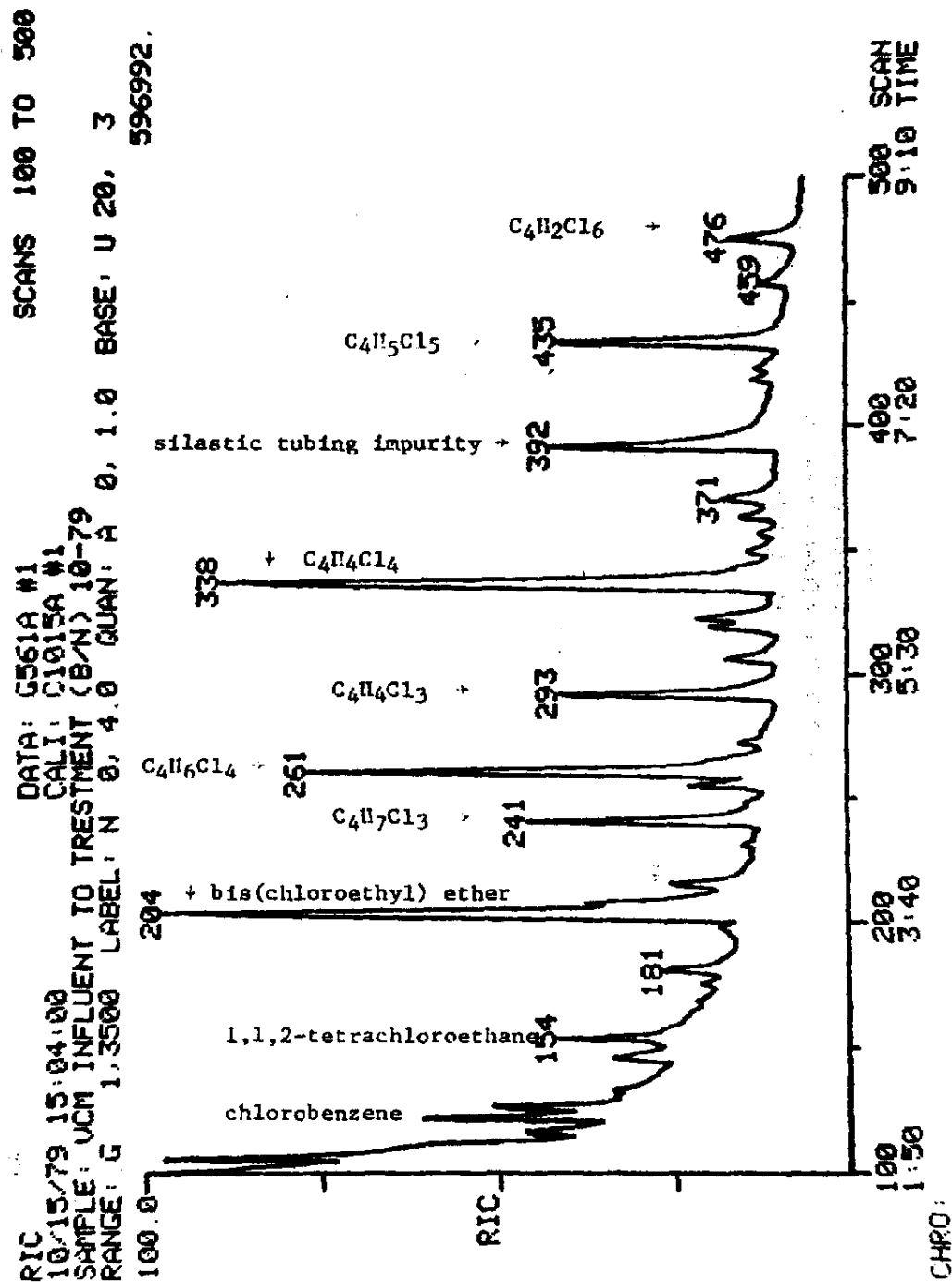


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VEV-266081

Figure 8

Influent Extract Base/Neutrals



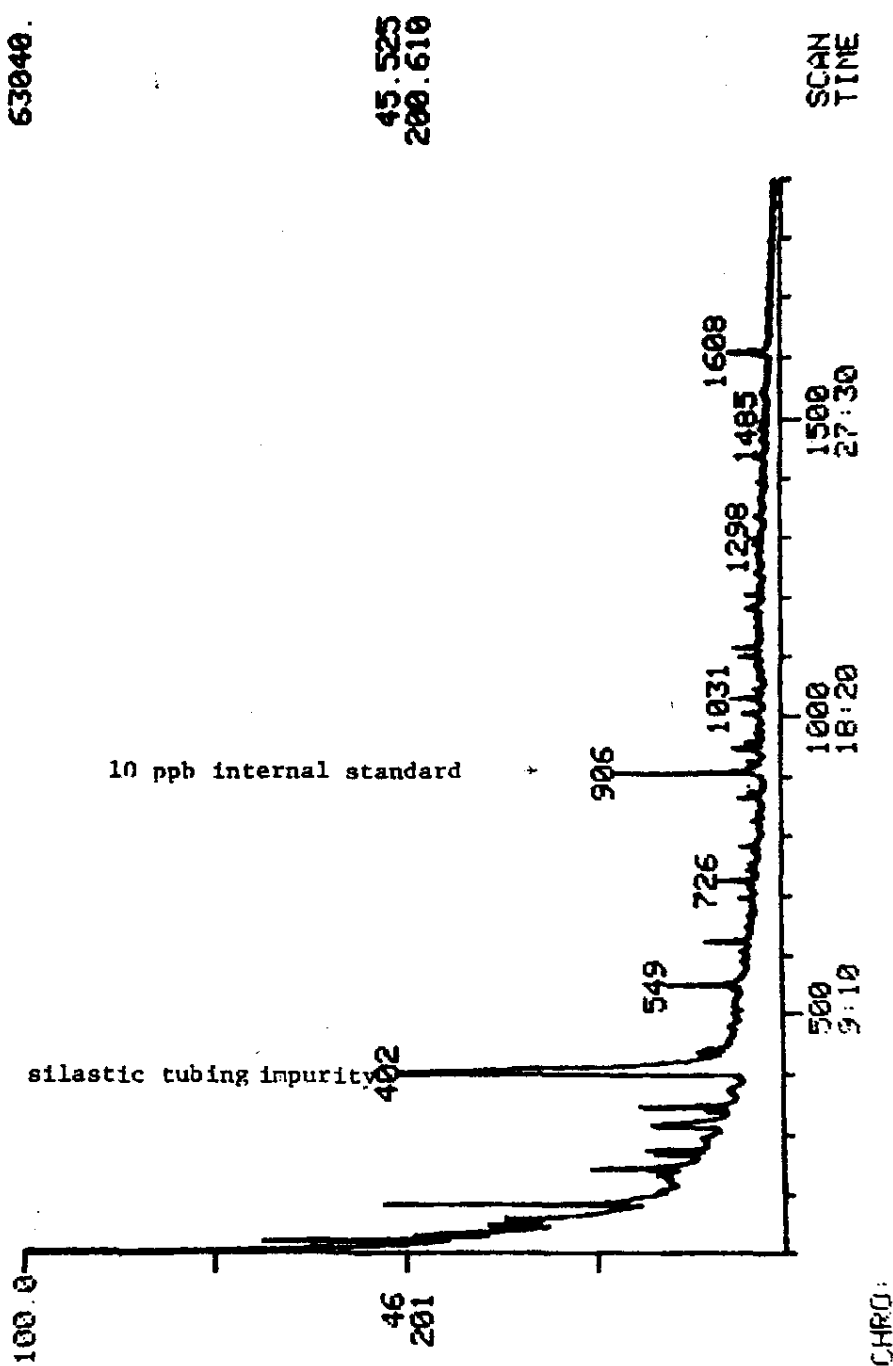
VEV-266082

VVC 000012828

Page 3

Influent Extract Acids

MASS CHROMATOGRAM  
10/16/79 3:23:00  
DATA: G562A #400  
CALI: C1015A #1  
SAMPLE (CON INFLUENT TO TREATMENT (ACIDS) 10-79  
RANGE: C 1.3500 LABEL: N 0, 4.0 GUAN: A 0, 1.0 BASE: U 20, 3  
SCANS 100 TO 1900  
63040.

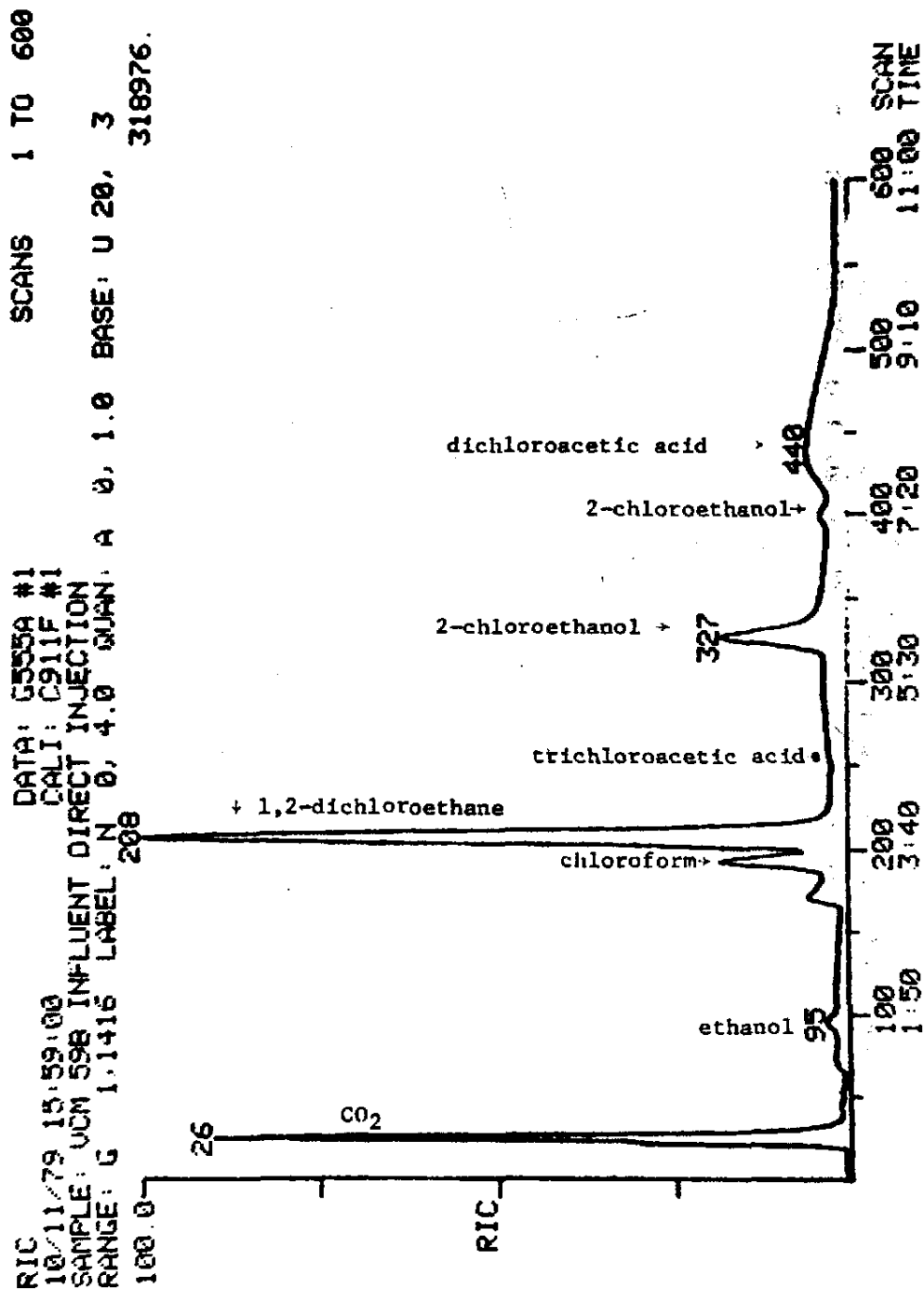


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VEV-266083

Figure 10

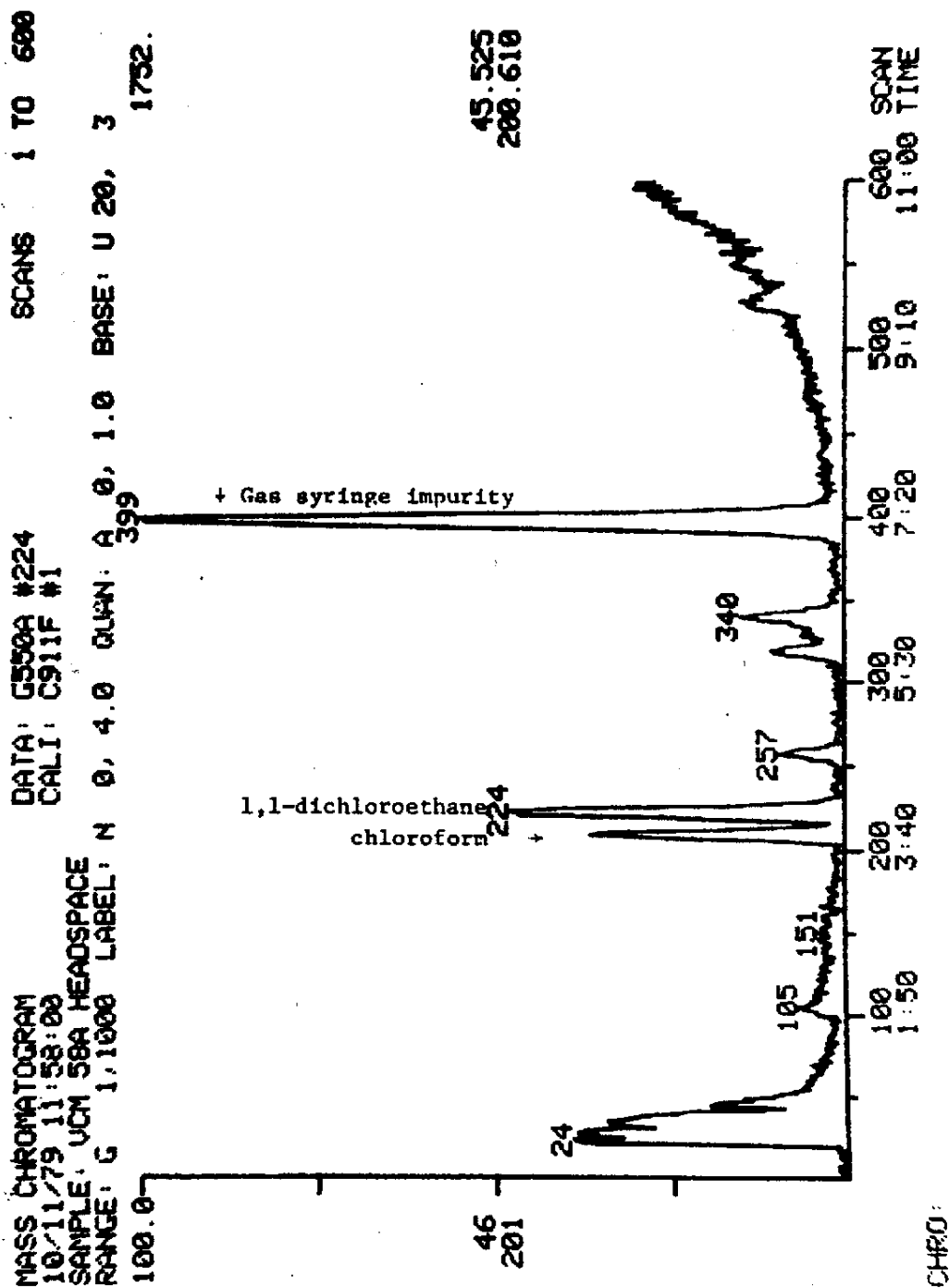
Influent Direct Injection



VEU-266084

VVC 000012830

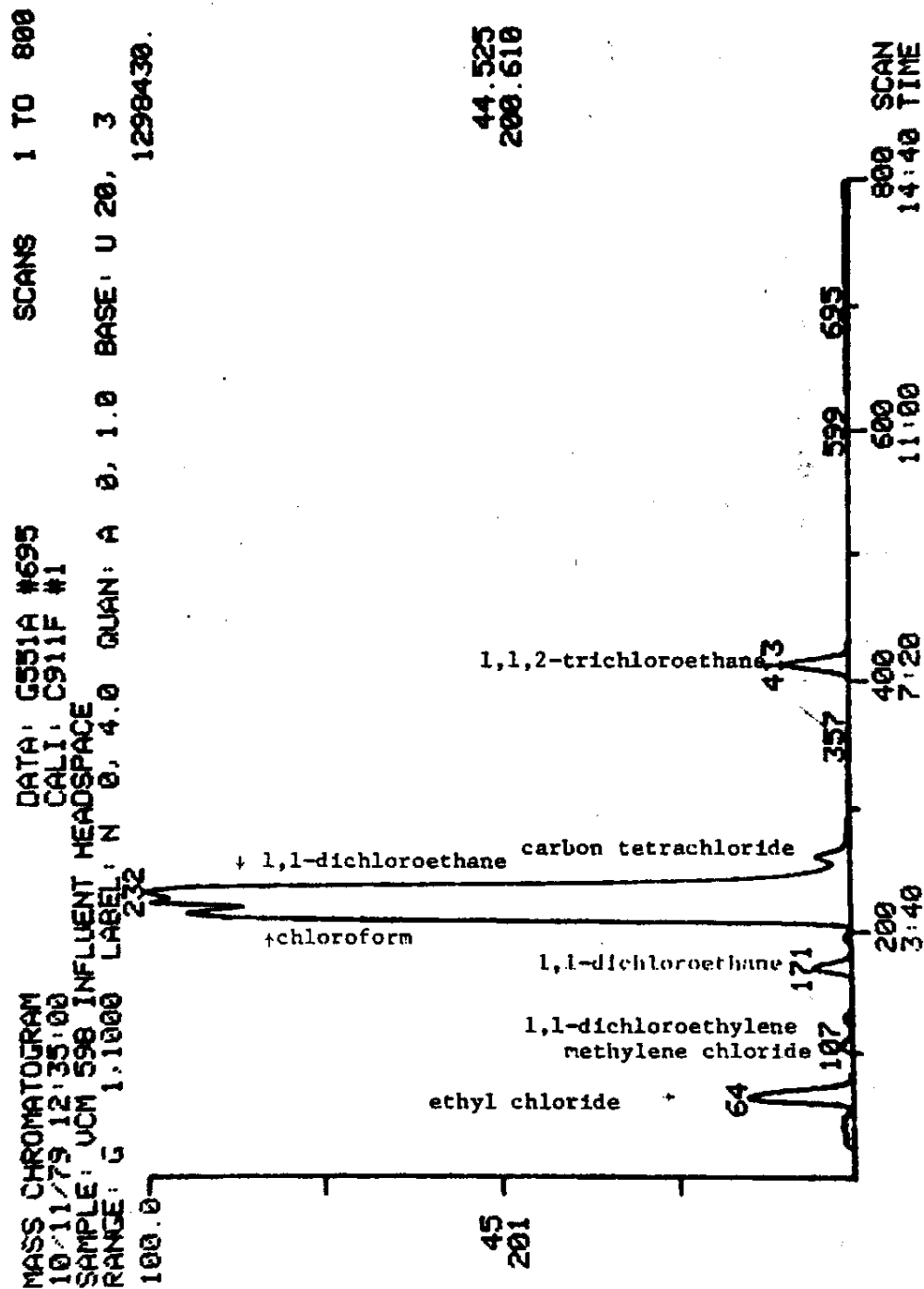
Effluent Headspace Injection



VVC 000012831

VEV-266085

Figure 3  
Influent Headspace Injection



ANALYSIS REPORT  
SPECTROSCOPY GROUP

Name R. E. Laramy Analysis No. 47092, 47093, 48498-48501  
 Sample No. See Below Notebook No. ICAP-1 pg 31, 32  
 Spectrum, Film or Plate No. \_\_\_\_\_  
 Type of Analysis Requested ESICAP

| Sample No.  |        |        |        |         |         |        |
|-------------|--------|--------|--------|---------|---------|--------|
| Element     | NIT-11 | NIT-12 | VCM-51 | VCM-52A | VCM-52B | VCM-53 |
| Al          | 0.35   | <0.15  | <0.15  | <0.15   | <0.15   | 0.33   |
| Ba          | 0.094  | <0.005 | 0.22   | <0.005  | <0.005  | 0.31   |
| Ca          | 22     | 0.21   | >330   | 0.74    | 0.90    | >330   |
| Cd          | <0.015 | <0.015 | <0.015 | <0.015  | <0.015  | <0.015 |
| Cr          | 1.5    | <0.03  | <0.03  | <0.03   | <0.03   | <0.03  |
| Cu          | <0.015 | <0.015 | <0.015 | <0.015  | <0.015  | 0.026  |
| Fe          | 1.2    | <0.03  | 0.54   | <0.03   | <0.03   | 11     |
| Mg          | 8.8    | <0.10  | 55     | <0.10   | <0.10   | 46     |
| Mn          | 0.13   | <0.01  | 0.090  | <0.01   | <0.01   | 0.50   |
| Mo          | <0.05  | <0.05  | <0.05  | <0.05   | <0.05   | <0.05  |
| Ni          | <0.075 | <0.075 | <0.075 | <0.075  | <0.075  | <0.075 |
| Pb          | <0.25  | <0.25  | <0.25  | <0.25   | <0.25   | <0.25  |
| Sn          | <0.10  | <0.10  | 0.12   | <0.10   | <0.10   | <0.10  |
| Tl          | <0.50  | <0.50  | <0.50  | <0.50   | <0.50   | <0.50  |
| V           | <0.025 | <0.025 | <0.025 | <0.025  | <0.025  | <0.025 |
| Zn          | 0.52   | <0.025 | 0.079  | <0.025  | <0.025  | 0.68   |
| Na          | 37     | 0.52   | >1000  | 0.77    | 0.60    | >1000  |
| -----       |        |        |        |         |         |        |
| AA ANALYSIS |        |        |        |         |         |        |
| Ag          | 2.02   | 2.02   | <0.02  | <0.02   | <0.02   | 0.02   |
| Bz          | 2.01   | 2.01   | <0.01  | <0.01   | <0.01   | <0.01  |
| Sb          | <0.2   | <0.2   | 0.4    | <0.2    | <0.2    | <0.2   |
|             |        |        |        |         |         |        |
|             |        |        |        |         |         |        |
|             |        |        |        |         |         |        |

\*No line was observed for this element. The < value given is the lowest concentration standard for this element in our calibration. All < or > values are outside our calibration range. Other analytical data are needed contact the Emission Laboratory.

Signed Rhea Hambrell  
 Date 10/18/79

VVC 000012833

UEV-266087

ANALYSIS REPORT  
SPECTROSCOPY GROUP

Name R E Laramy Analysis No. 48498-48501  
Sample No. See below Notebook No. HMS-1 pg 85  
Spectrum, Film or Plate No. \_\_\_\_\_

Type of Analysis Requested AAAS AAHG AASE

[illegible]

\*No line was observed for this element. The < value given is the lowest concentration seen for this element in our calibration. All < or > values are outside our calibration range. For other analytical data are needed contact the Emission Laboratory.

Signed R. C. Train  
Date 10-17-79