



Technical Service Report

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

Report No. 2118-79-848-0

To R.E. Laramy

From G.W. Keen

Date November 8, 1979

Subject The GCMS Analysis of Treatment Influent and Effluent Water
from Aberdeen

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Object

To identify and compare the levels of the impurities in the water samples taken before and after the waste treatment facility.

Conclusions

1. The major components are summarized in Table 1.
2. GC traces and their interpretations are provided.
3. Phthalate half-esters disproportionated or thermally decomposed during the analysis to produce phthalic anhydride, alcohols and diesters.
4. All of the impurities were significantly lower in plant effluent than they were in the influent to treatment.

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Distribution EAS FK AJL

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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 D₁₀-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

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 Date: G471A-G479A
G485A-G492A

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TABLE I

Summary of the Major Impurities

	Influent to Treatment	Effluent
vinyl chloride	2000 ppb	2 ppb
ethylbenzene	70 ppb	6 ppb
xylene	400 ppb	45 ppb
1-hexanol	2200 ppb	
2-ethylhexanal	130 ppb	
α -methylstyrene	300 ppb	5 ppb
2-ethylhexanol	* 80000 ppb	† 30 ppb
1-octanol	* 14000 ppb	
2-phenyl-2-propanol	80 ppb	
isodecyl alcohols	* 30000 ppb	
1-decanol	* 10000 ppb	
tridecane	200 ppb	
phthalic anhydride	* 50000 ppb	18 ppb
di(2-ethylhexyl) ether	100 ppb	
2-hydroxymethylbenzoic acid lactone	1500 ppb	
tetradecane	900 ppb	
hexyl octyl ether	400 ppb	
BHT	80 ppb	
n-octyl 2-ethylhexyl ether	30 ppb	
dioctyl ether	2000 ppb	1 ppb
hexyl decyl ether	200 ppb	
2-ethylhexyl benzoate	60 ppb	1 ppb
2-ethylhexyl decyl ether	20 ppb	
octyl decyl ether	2300 ppb	3 ppb
didecyl ether	800 ppb	1 ppb
phthalate half esters	* 85000 ppb	90 ppb
dihexyl phthalate	160 ppb	
n-hexyl n-octyl phthalate	240 ppb	4 ppb
isooctyl phthalate	160 ppb	
di(2-ethylhexyl) phthalate	* 66000 ppb	† 90 ppb
n-octyl 2-ethylhexyl phthalate	2100 ppb	† 4 ppb
di(n-octyl) phthalate	* 28000 ppb	† 40 ppb
di(isodecyl) phthalate	* 76000 ppb	† 130 ppb
2-ethylhexyl n-decyl phthalate	1900 ppb	† 2 ppb
n-octyl n-decyl phthalate	* 37000 ppb	† 70 ppb
di(n-decyl) phthalate	* 16000 ppb	† 20 ppb
decyl dodecyl phthalate	300 ppb	
triethyl trimellitate	300 ppb	

* These concentrations were so high that the quantitative data are suspect.
It was necessary to make a 1:90 split injection.

† Portions of these components may have resulted from phthalate half ester decomposition and disproportionation during work-up and injection. They had lower concentrations in the base/neutral fraction than in the acid fraction.

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Interpretations of the Attached GC Traces

Aberdeen Influent Volatile Organics. See Figure 1

Scan #	Identification	Concentration
92	vinyl chloride	2000 ppb
634	ethylbenzene	70 ppb
754	xylene	200 ppb
773	xylene	190 ppb
878	α -methylstyrene	300 ppb
896	2-ethylhexanol	140 ppb

Aberdeen Effluent Volatile Organics. See Figure 2

96	vinyl chloride	2 ppb
628	ethylbenzene	6 ppb
748	xylene	23 ppb
767	xylene	22 ppb
871	α -methylstyrene	5 ppb
892	* 2-ethylhexanol	80 ppb

* The Ponca City water was high in 2-ethylhexanol on the day the glass ware was cleaned for this run. This value may be high. The purge and trap blank was high in 2-ethylhexanol.

Aberdeen Pump Blank (base/neutral) See Figure 3

Scan #	Identification	Concentration
108	4-methyl-3-penten-2-one	19 ppb
182	tetrachloropropane	17 ppb
258	3-methyl-2-cyclohexene-1-one	3 ppb
343	trimethylhexene	9 ppb
458	silastic tubing impurity	1 ppb
553	silastic tubing impurity	140 ppb
625	hexachloropentane	60 ppb
725	diethyl phthalate	10 ppb
1049	dibutyl phthalate	1 ppb
1551	dicyclohexyl phthalate	2 ppb
1610	di(2-ethylhexyl) phthalate	12 ppb
1977	silastic tubing impurity	11 ppb

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Aberdeen Pump Blank (acids) See Figure 4

Scan #.	Identification	Concentration
104	4-methyl-3-penten-2-one	11 ppb
180	tetrachloropropane	5 ppb
257	3-methyl-2-cyclohexen-1-one	3 ppb
341	trimethylhexene	3 ppb
743	diethyl phthalate	5 ppb
1503	di(2-ethylhexyl) adipate	1 ppb
1590	dicyclohexyl phthalate	1 ppb
1656	di(2-ethylhexyl) phthalate	4 ppb
2060	silastic tubing impurity	2 ppb

Aberdeen Influent to Treatment (base/neutrals) See Figures 5, 6 and 7

This sample was so loaded with organic materials that it was necessary to make a split (low sensitivity) injection. The following data were from that split injection. The split ratio was about 1:90.

Scan #	Identification	Concentration
135	1-hexanol	1900 ppb
179	isooctenes	10 ppb
186	2-ethylhexanal	130 ppb
208	α -methylstyrene	200 ppb
222	isooctanol	130 ppb
256	2-ethylhexanol	32000 ppb
289	1-octanol	13000 ppb
295	2-phenyl-2-propanol	80 ppb
350-450	isodecyl alcohols	16000 ppb
479	1-decanol	10000 ppb
517	tridecane	200 ppb
525	di(2-ethylhexyl) ether	100 ppb
528	2-hydroxymethylbenzoic acid lactone	150 ppb
535	5-, 6- and 7-methyltridecane	60 ppb
554	n-heptylcyclohexane	80 ppb
564	2-methyltridecane	80 ppb
584	3-methyltridecane	90 ppb
591	branched C ₁₅ paraffin	50 ppb
610	tetradecane	860 ppb
628	1-dodecanol	35 ppb
650	n-octylcyclohexane	26 ppb
665	hexyl octyl ether	440 ppb
747	diethyl phthalate	17 ppb
771	octyl 2-ethylhexyl ether	30 ppb
834	dioctyl ether	2000 ppb
836	hexyl decyl ether	200 ppb
859	2-ethylhexyl benzoate	60 ppb
885	octyl isodecyl ether	20 ppb
936	2-ethylhexyl decyl ether	240 ppb
1001	octyl decyl ether	2200 ppb
1092	decyl isodecyl ether	20 ppb
1174	didecyl ether	800 ppb
1216	hexyl phthalate	100 ppb

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1525	hexyl octyl phthalate	240 ppb
1588	isooctyl phthalate	150 ppb
1660	di(2-ethylhexyl phthalate	* 62000 ppb
1749	octyl 2-ethylhexyl phthalate	2000 ppb
1875	di(n-octyl) phthalate	* 27000 ppb
1900-2400	diisodecyl phthalate	* 72000 ppb
2006	2-ethylhexyl decyl phthalate	1800 ppb
2160	n-octyl n-decyl phthalate	* 35000 ppb
2489	di(n-decyl) phthalate	* 15000 ppb
2982	decyl dodecyl phthalate	300 ppb
3500	trioctyl trimellite	300 ppb

* overloaded component

Aberdeen Influent to Treatment (acids) (split injection) See figures 8, 9 and 10

Scan#	Identification	Concentration
138	1-hexanol	300 ppb
256	2-ethylhexanol	* 46000 ppb
290	1-octanol	1200 ppb
350-450	isodecyl alcohols	14000 ppb
504	phthalic anhydride	* 50000 ppb
525	2-hydroxymethylbenzoic acid lactone	1300 ppb
833	dioctyl ether	100 ppb
600-1100	phthalic anhydride and alcohols (from half ester decomposition	* 85000 ppb
999	octyl decyl ether	110 ppb
1172	didecyl ether	30 ppb
1626	dihexyl phthalate	160 ppb
1641	di(2-ethylhexyl) phthalate	4000 ppb
1747	n-octyl 2-ethylhexyl phthalate	100 ppb
1682	di-(n-octyl) phthalate	1300 ppb
2004	2-ethylhexyl n-decyl phthalate	100 ppb
2137	n-octyl n-decyl phthalate	1800 ppb
2473	di(n-decyl) phthalate	500 ppb
2000-2400	di(isodecyl) phthalate	3900 ppb

Aberdeen effluent (base/neutrals) See Figures 11, 12 and 13

Scan #	Identification	Concentration
257	3-methyl-2-cyclohexen-1-one	21 ppb
319	3,5,5-trimethyl-2-cyclohexen-1-one	7 ppb
392	silastic tubing impurity	16 ppb
551	silastic tubing impurity	40 ppb
1633	di(2-ethylhexyl) phthalate	10 ppb
1856	di(n-octyl) phthalate	2 ppb
2132	n-octyl n-decyl phthalate	6 ppb
2469	di(n-decyl) phthalate	2 ppb
1900-2350	di(isodecyl) phthalate	28 ppb

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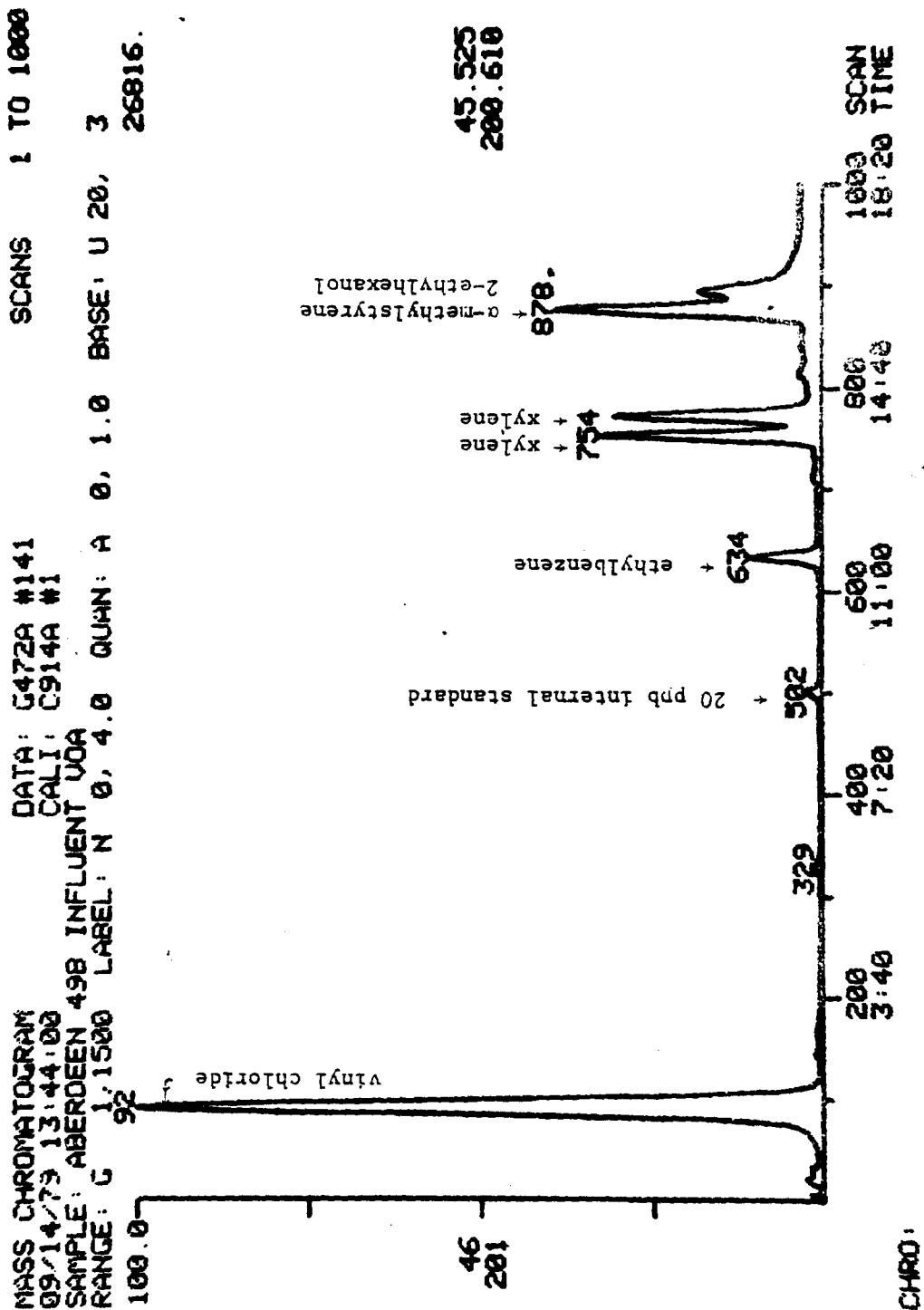
Aberdeen Effluent (acids) See Figures 14, 15 and 16

Scan #	Identification	Concentration
252	2-ethylhexanol	34 ppb
393	silastic tubing impurity	45 ppb
486	phthalic anhydride	18 ppb
551	silastic tubing impurity	16 ppb
701	pentadecane	2 ppb
600-1100	half ester decomposition products	90 ppb
834	dioctyl ether	1 ppb
859	2-ethylhexyl benzoate	1 ppb
999	octyl decyl ether	3 ppb
1171	didecyl ether	1 ppb
1482	di(2-ethylhexyl) adipate	3 ppb
1614	hexyl octyl phthalate	4 ppb
1632	di(2-ethylhexyl) phthalate	90 ppb
1731	n-octyl 2-ethylhexyl phthalate	4 ppb
1845	di(n-octyl) phthalate	36 ppb
1982	2-ethylhexyl n-decyl phthalate	2 ppb
2114	n-octyl n-decyl phthalate	60 ppb
2444	di(n-decyl) pythalate	21 ppb
1900-2400	di(isodecyl) phthalate	100 ppb

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Figure 1

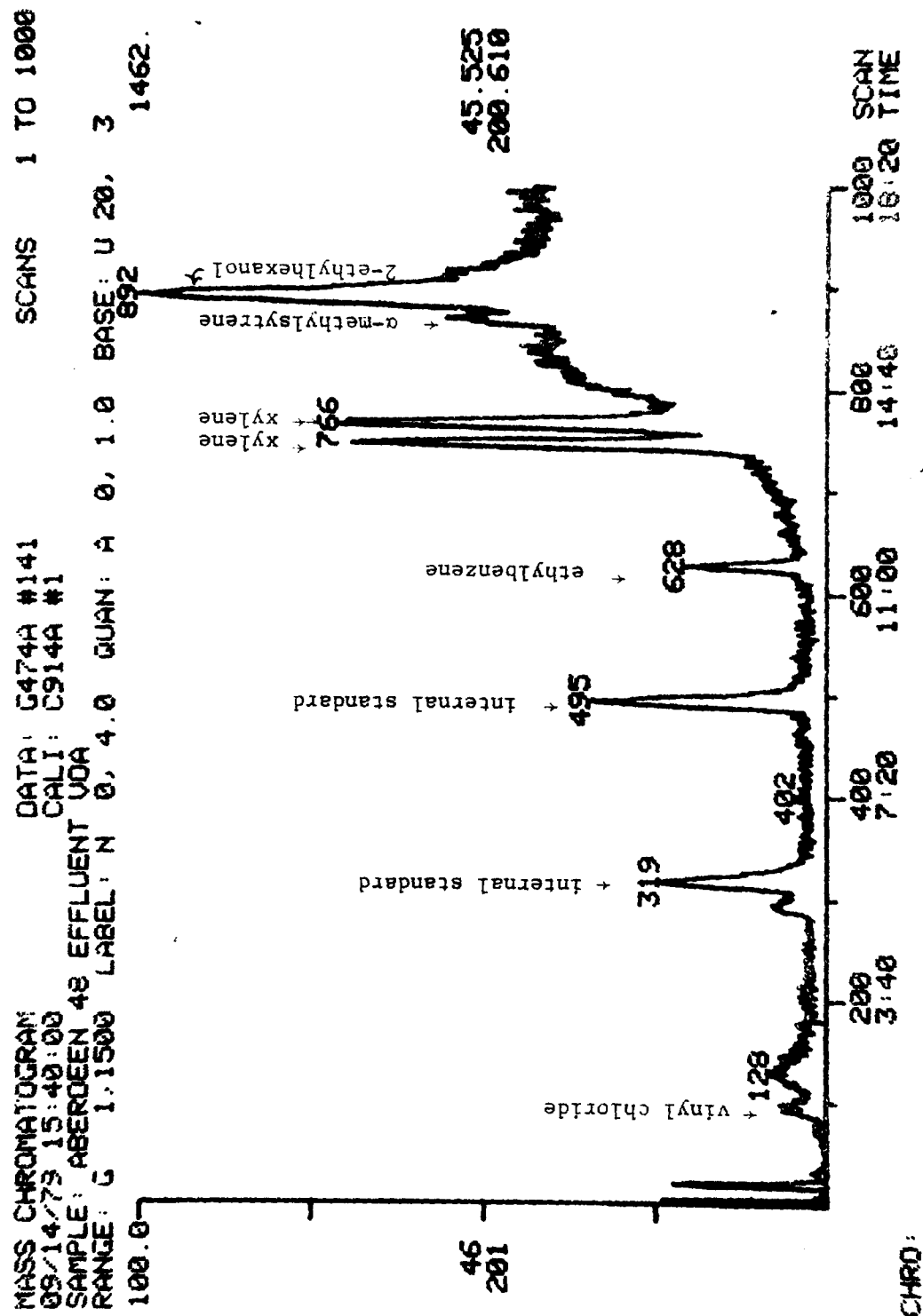
Influent to Treatment Volatile Organics



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Figure 2

Effluent Volatile Organics

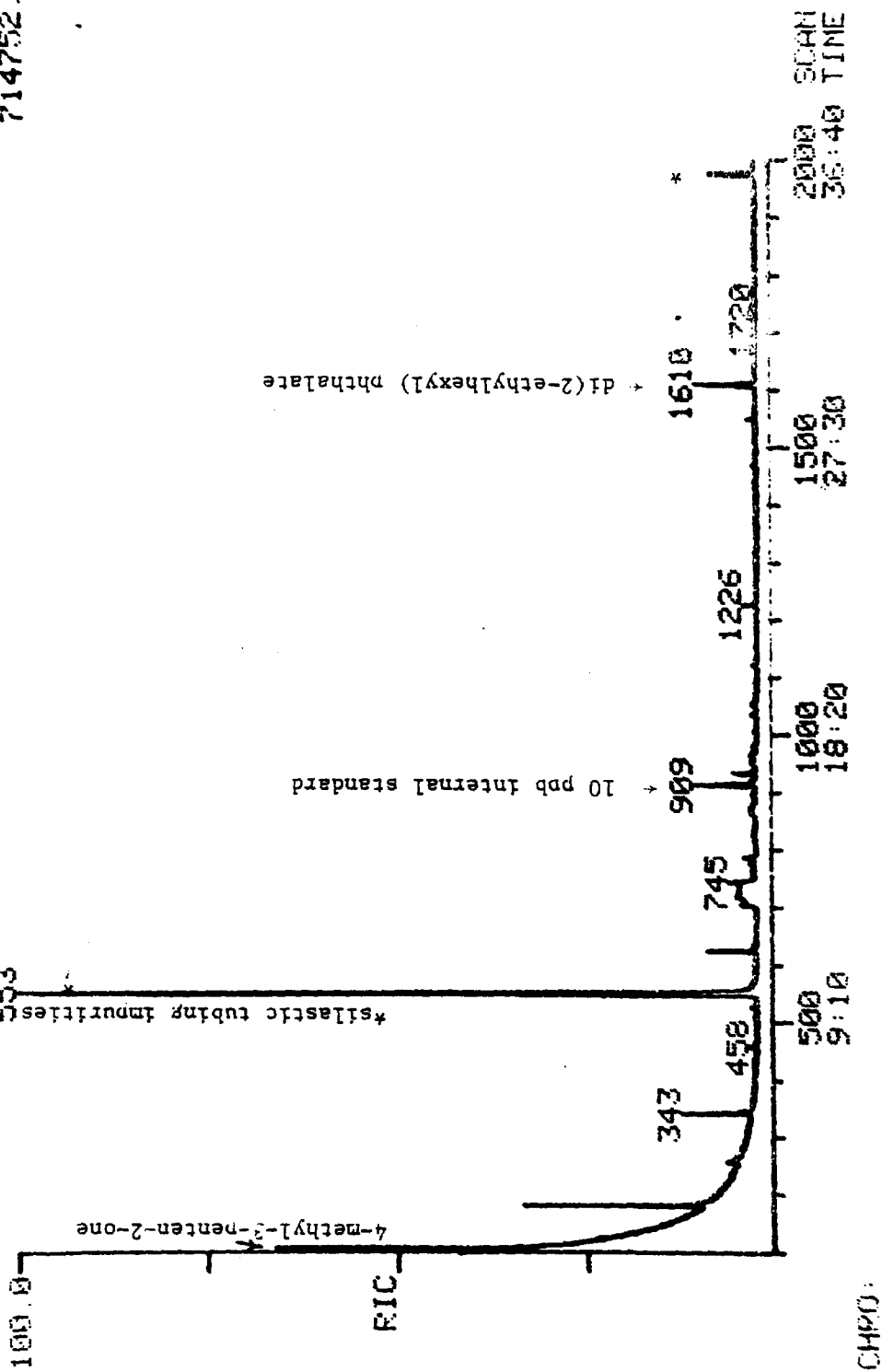


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Figure 2

Pump Blank Base/Neutrals

92118779 12 05 00
 DATA: 548504 #1
 CALL: C911F #1
 SAMPLE: ABERDEEN B/N PUMP BLANK 9/79
 RANGE: G 1.3500
 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3
 SCANS 100 TO 2000
 714752

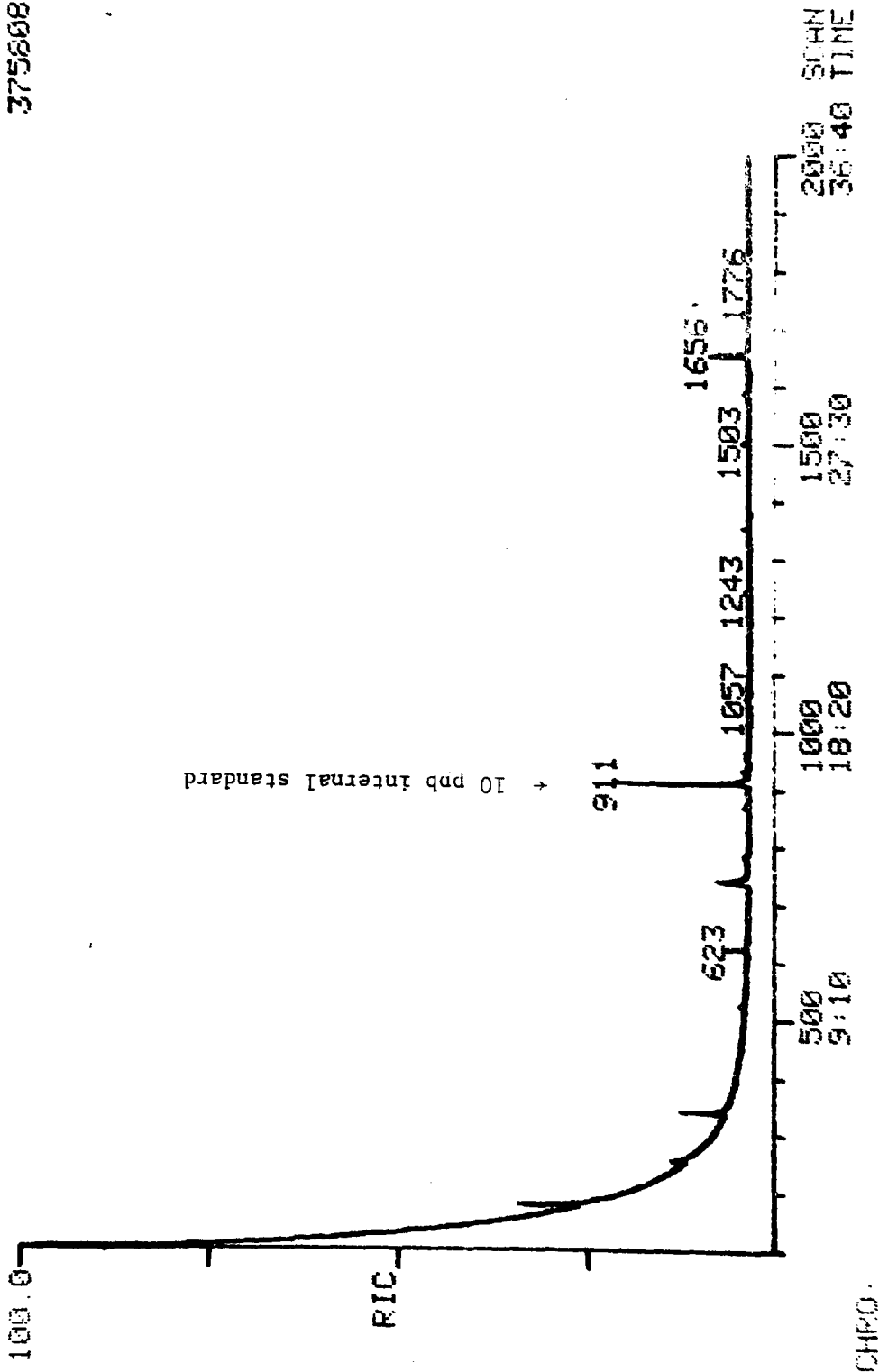


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Figure 4

Pump Blank Acids

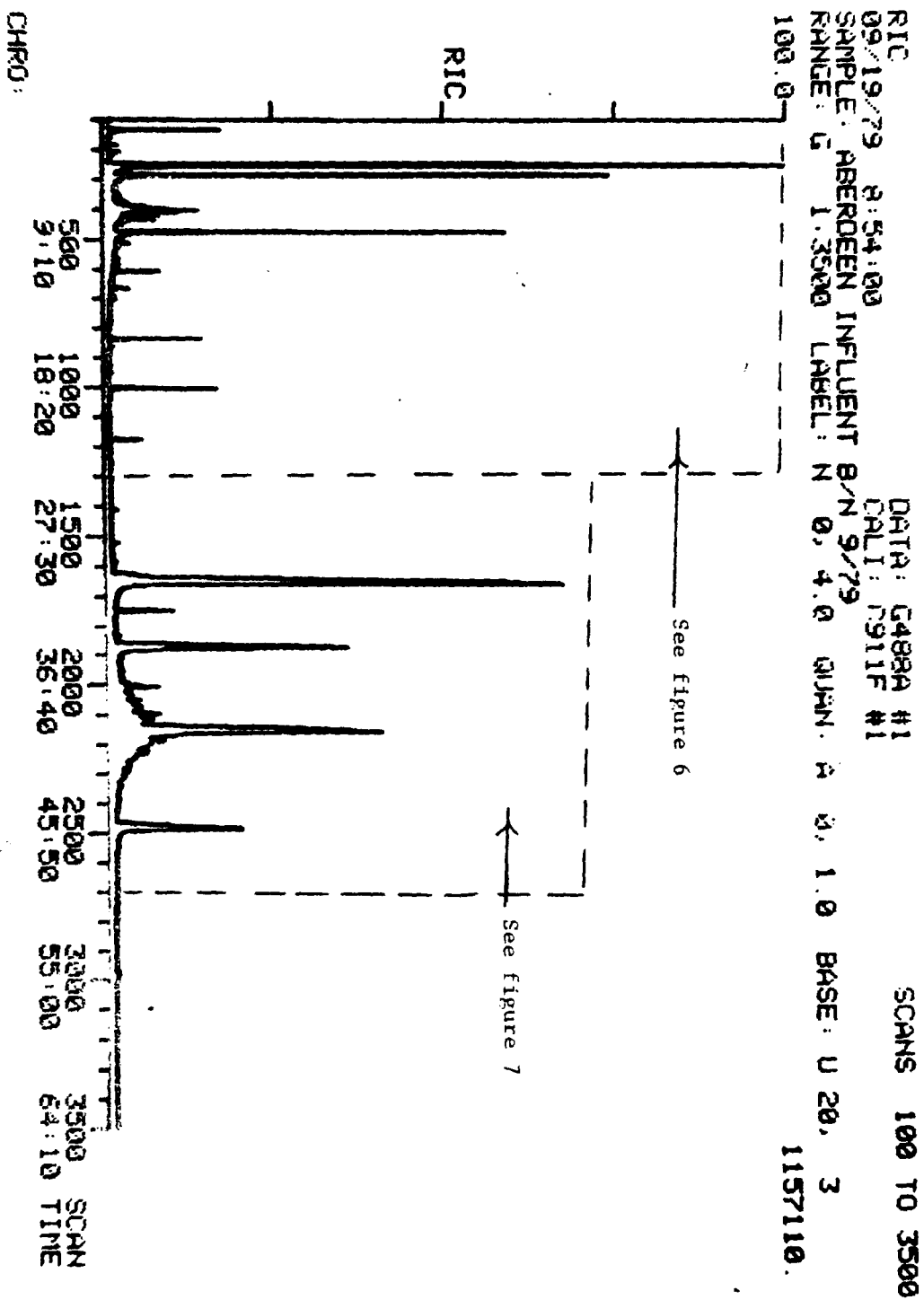
RIC 04/18/19 13:26:00 SCANS 100 TO 2000
 DATA: G4805A #1
 DATA: C911F #1
 SAMPLE: ABERRIEEN ACIDS PUMP BLANK 9/79
 RANGE: G 1.3500 LABEL: N 0.4.0 QUANT: A 0.1.0 BASE: U 20. 3
 375808.



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Figure 5

Influent to Treatment Base/Neutrals



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Figure 6
Influent to Treatment Case/Neutrals

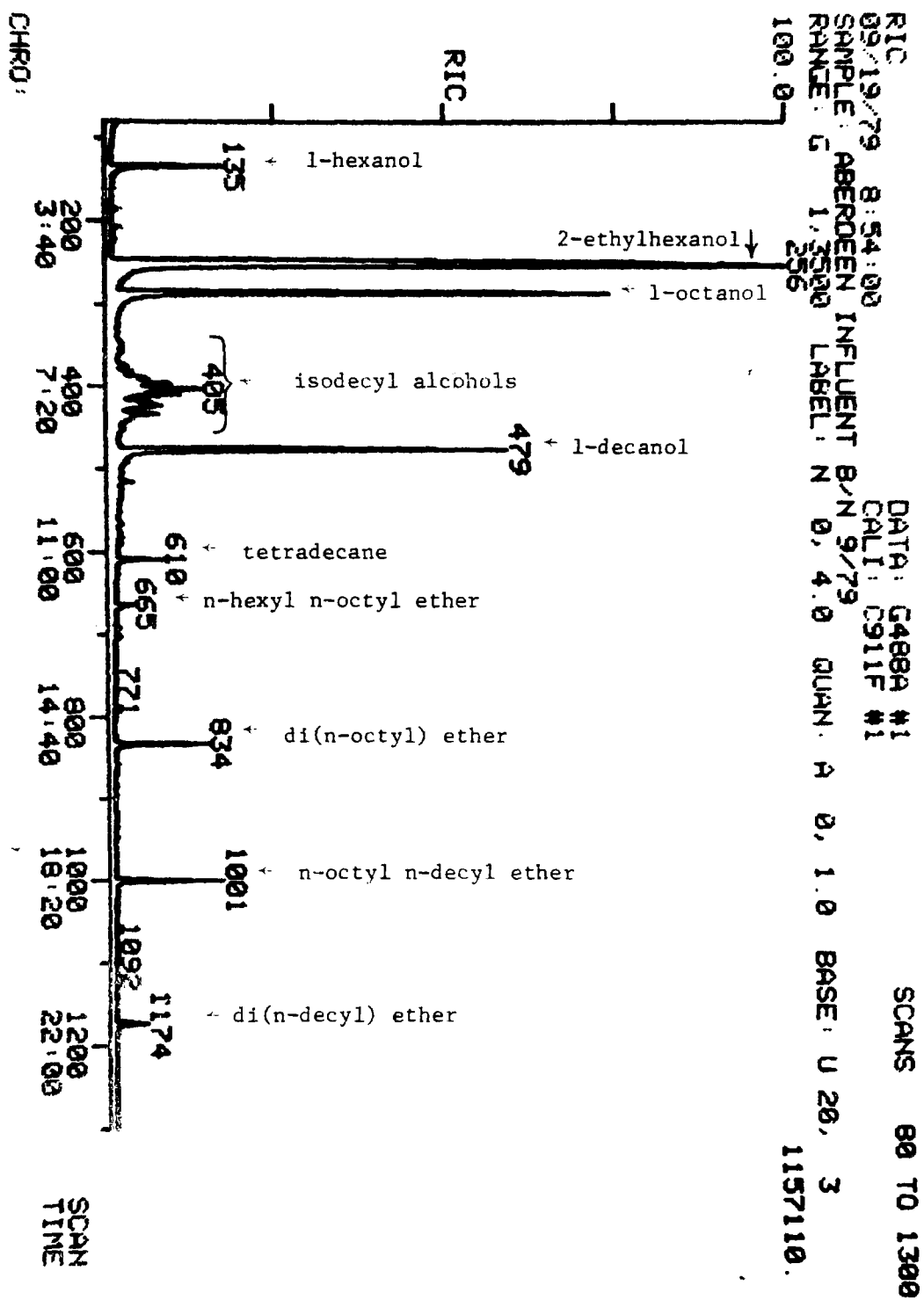


Figure 7

Influent to Treatment Base/Neutrals

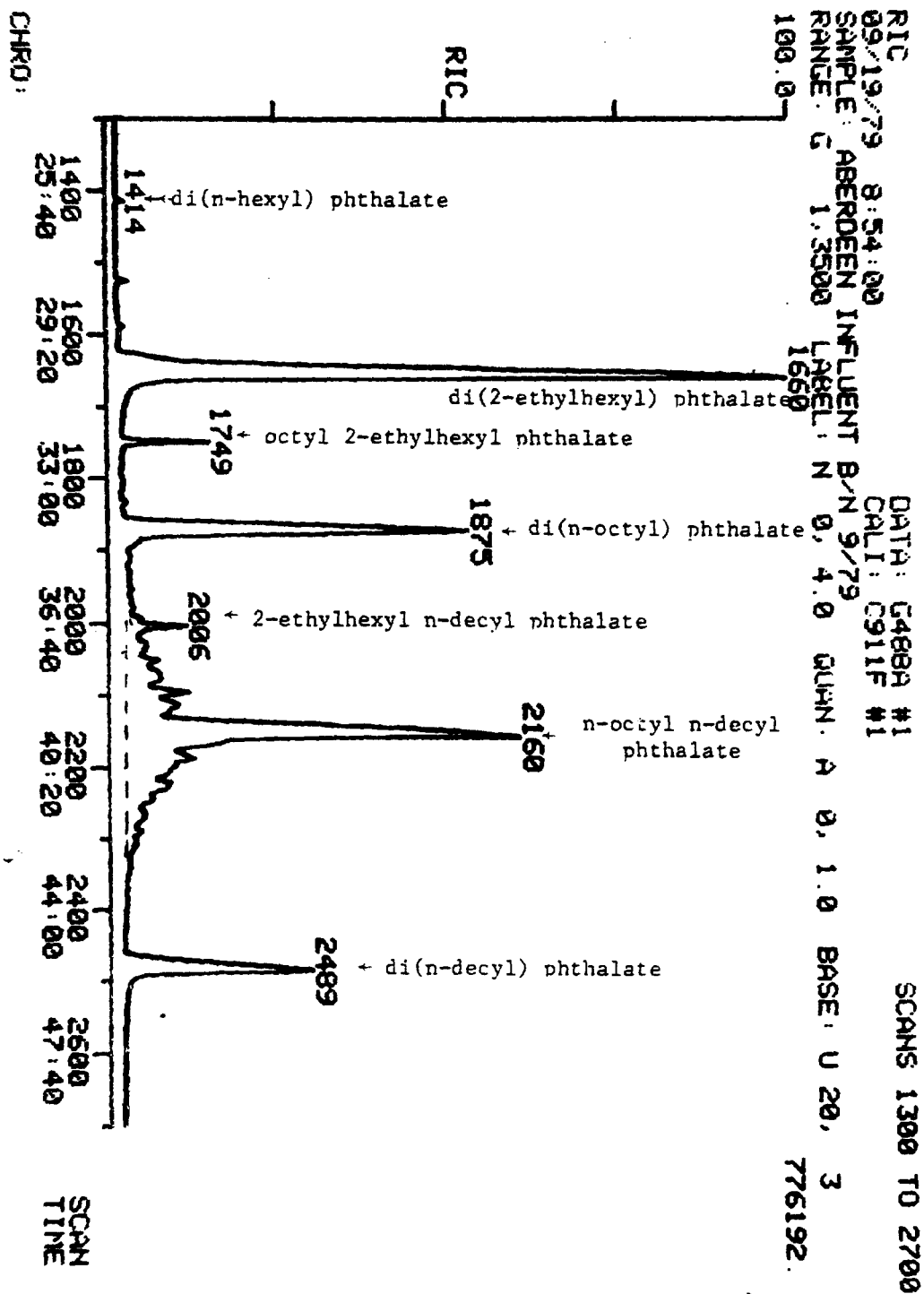
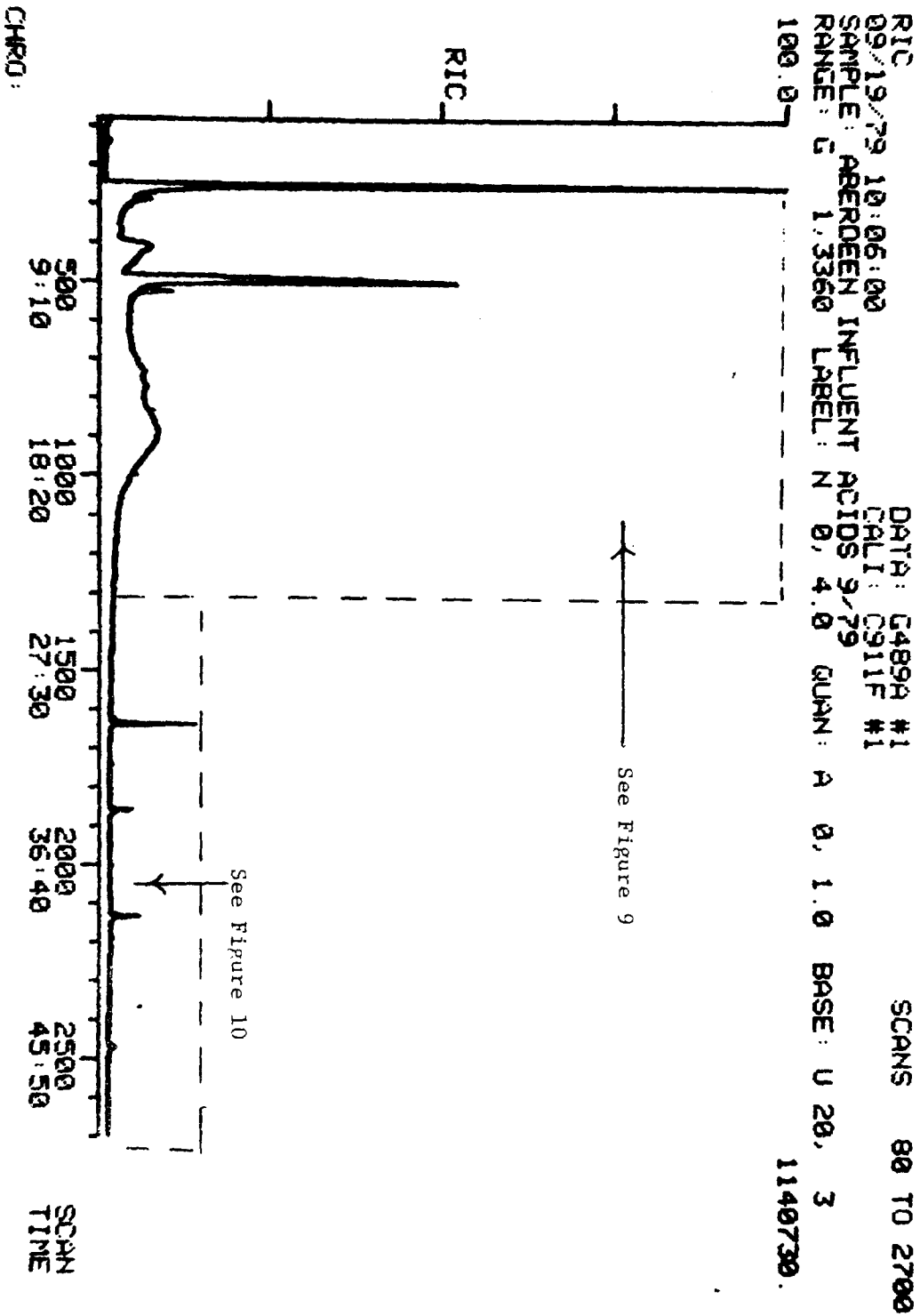


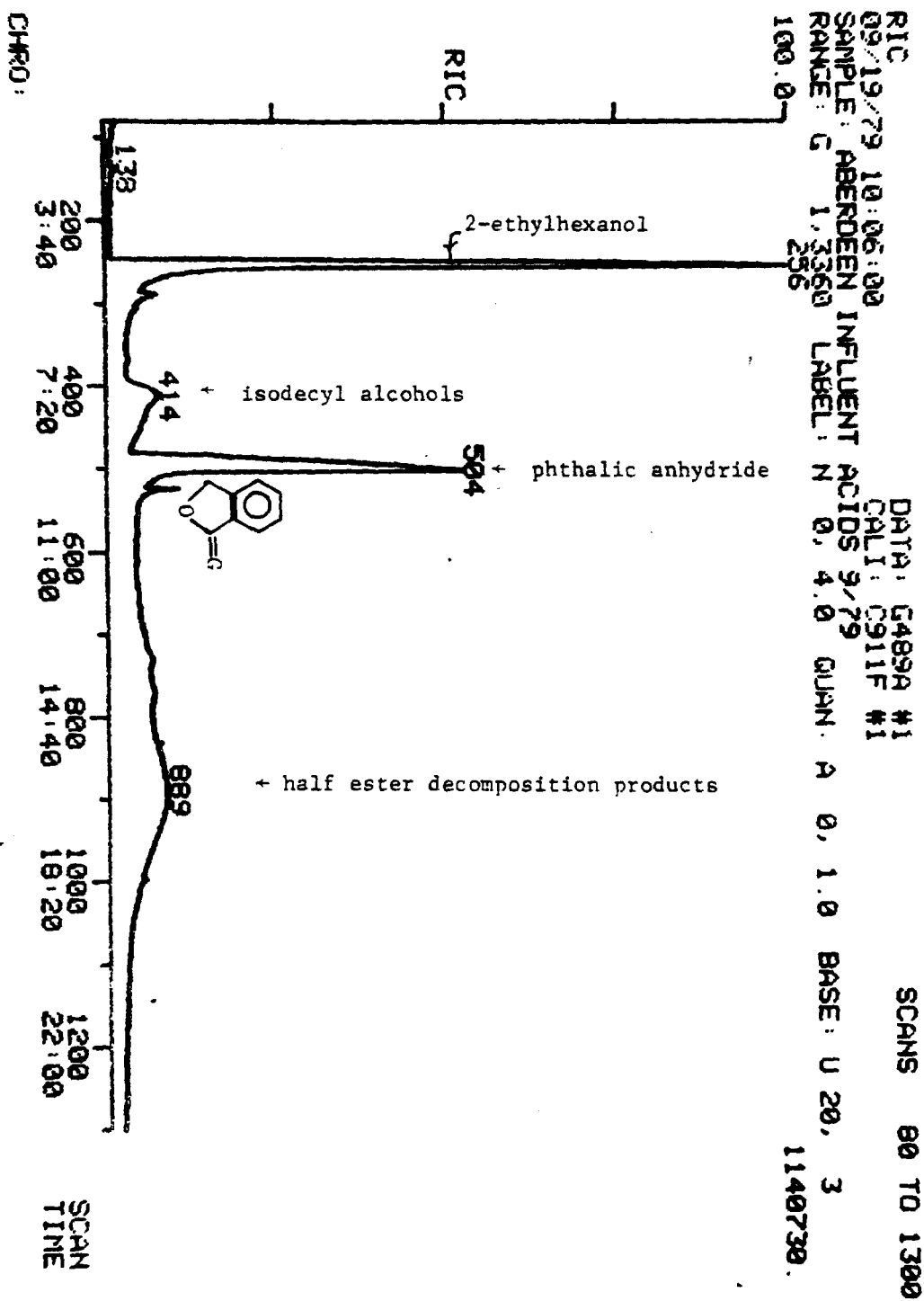
Figure 2
Influent to Treatment Acids



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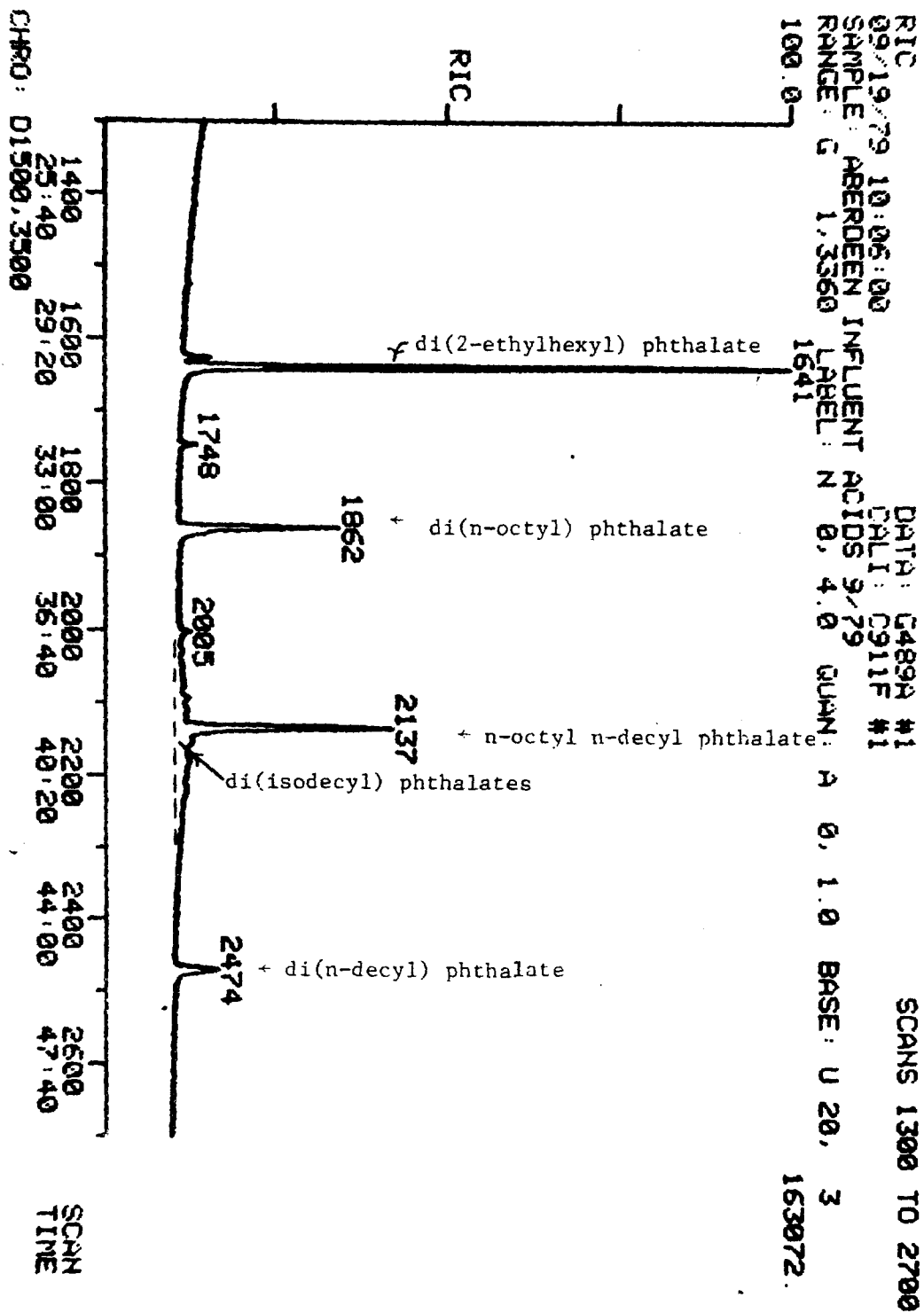
Figure 9

Influent to Treatment Acids



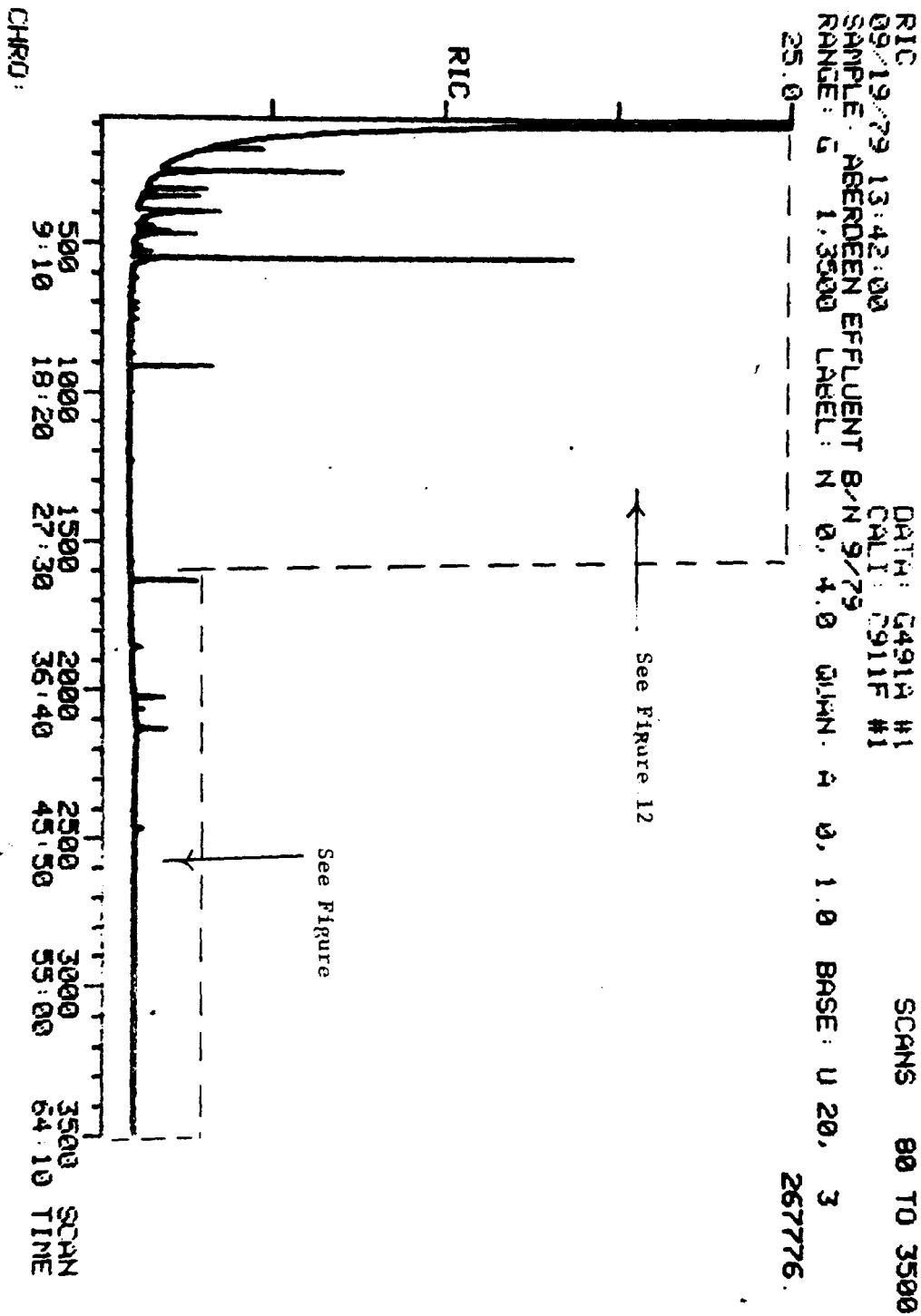
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Figure 17
Influent to Treatment Acids



Effluent Base/Neutrals

Figure 11



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Figure 12
Effluent Base/Neutrals

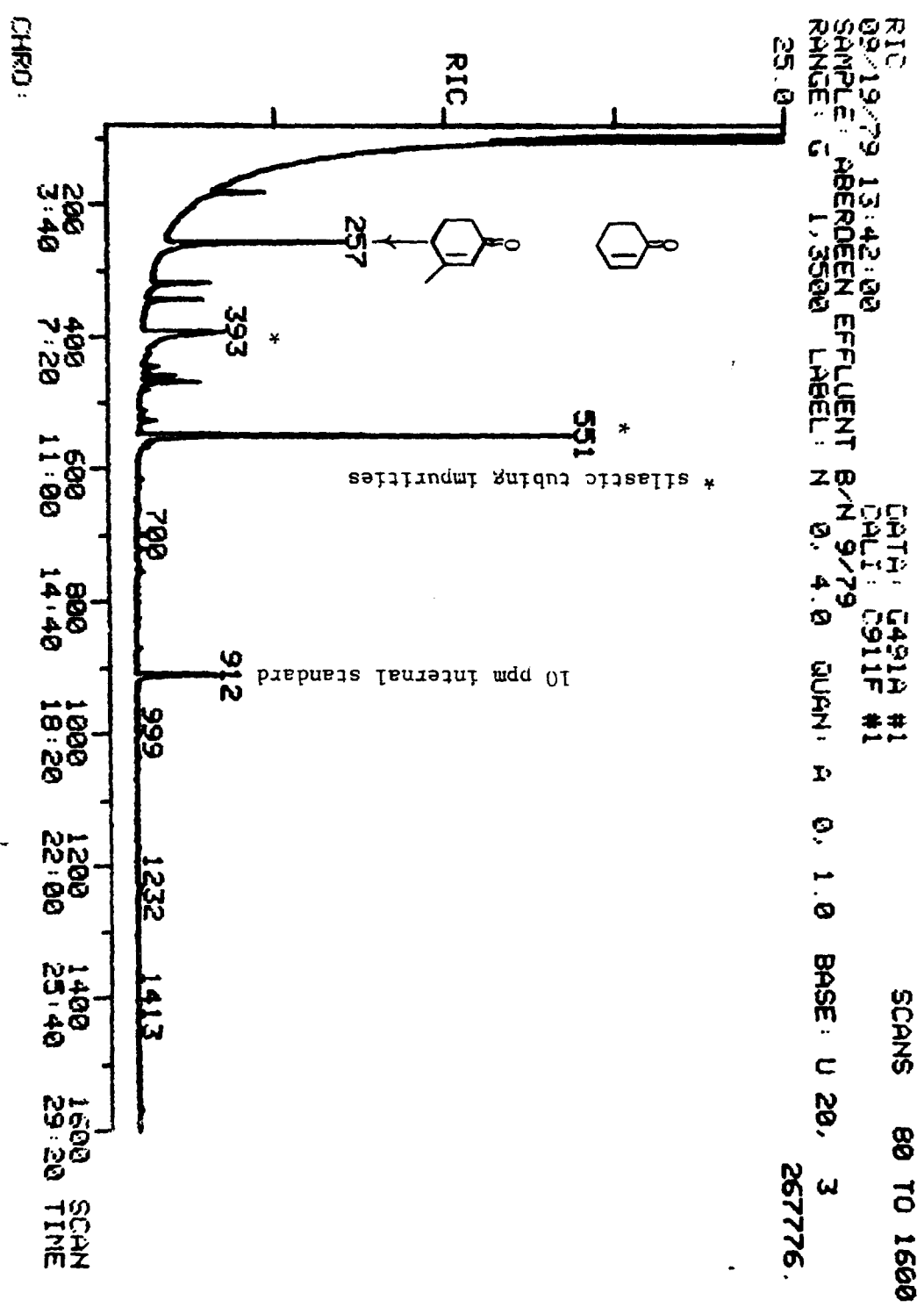
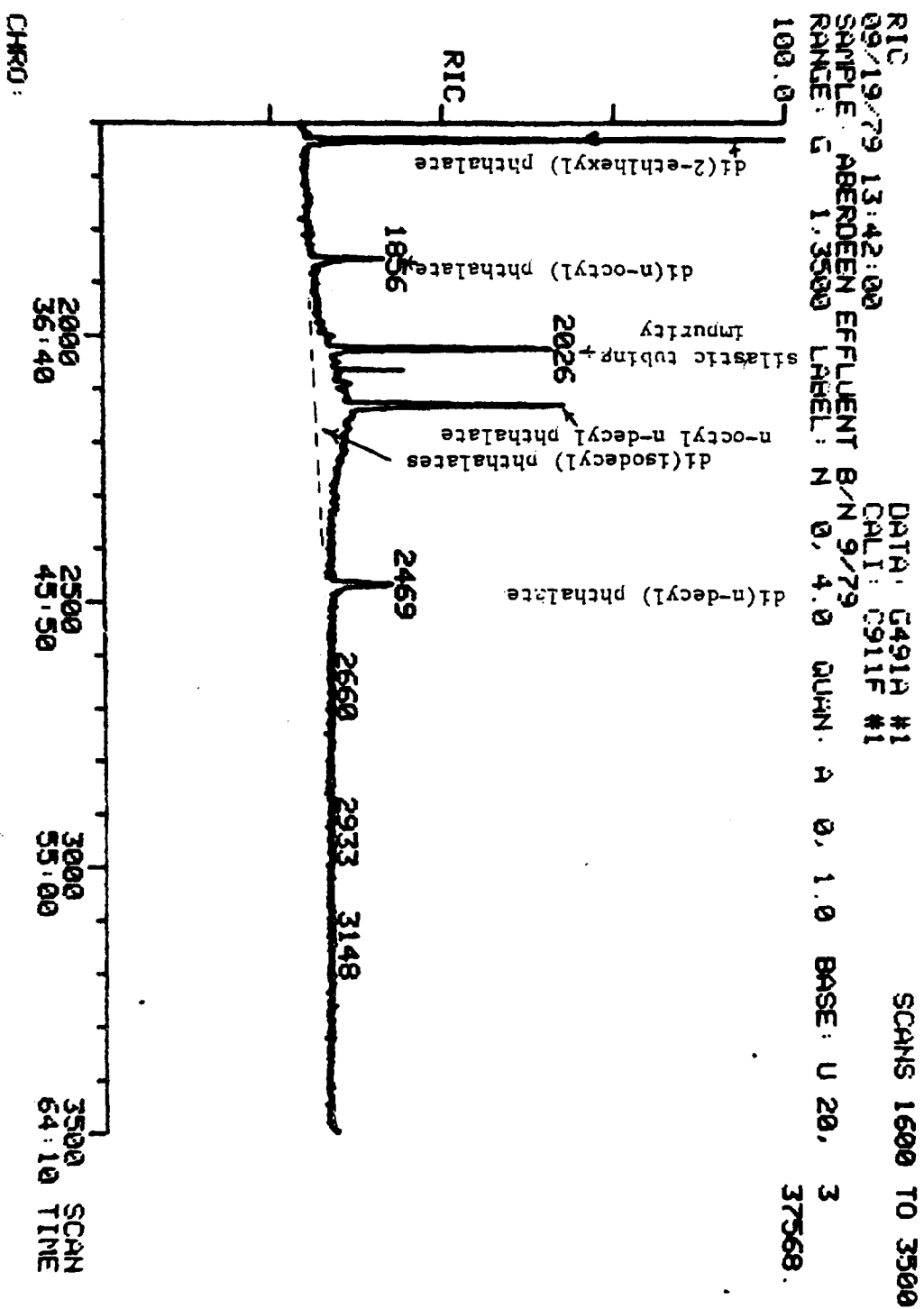


Figure 13

Effluent Base/Neutrals



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Figure 14

Effluent "Acids"

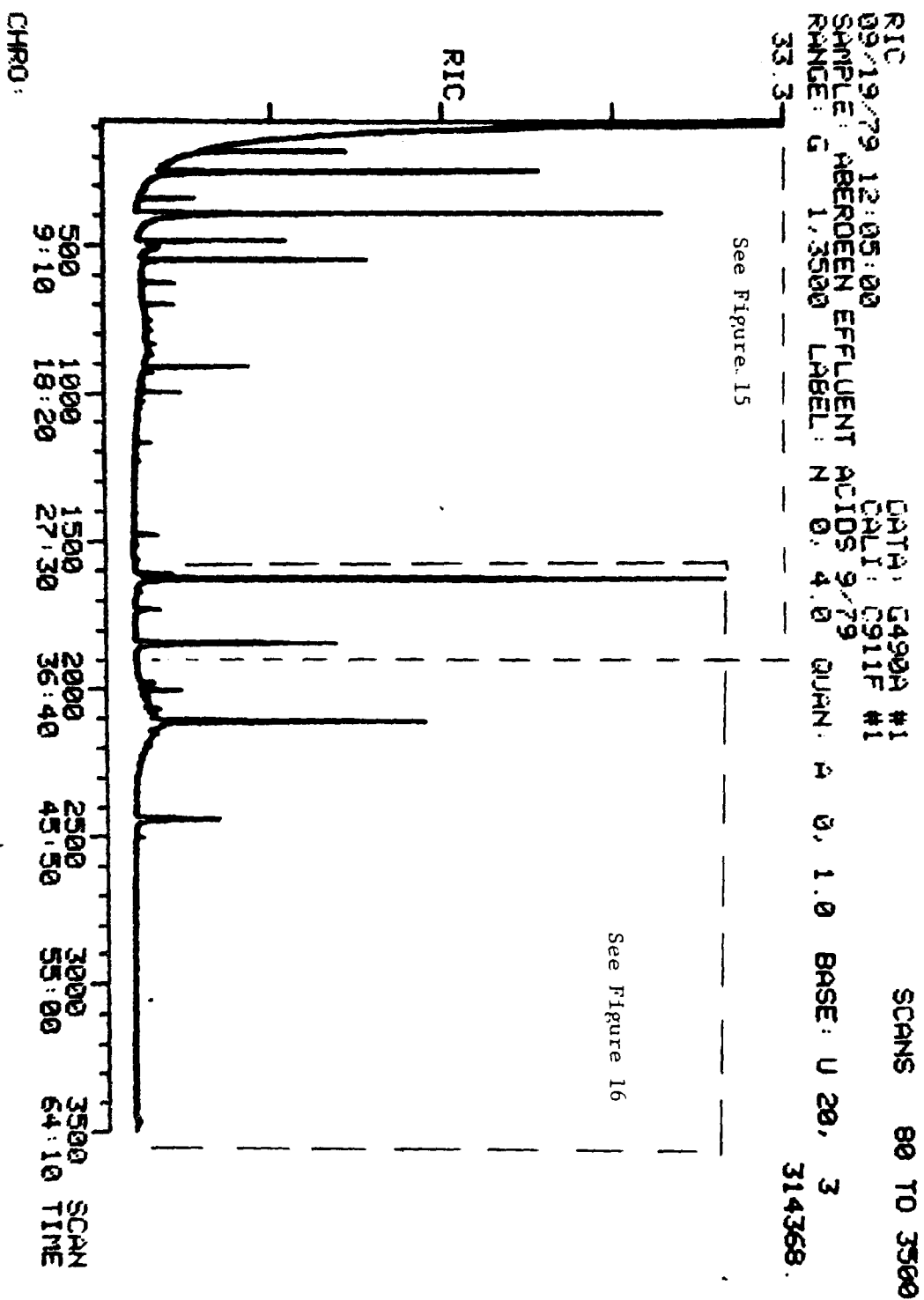


Figure 16
Effluent "Acids"

