Appendix A

Fischer-Tropsch Wax Characterization Raw Data

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Appendix

FT Wax Solvent Extraction Procedure

1. Melt about 4 grams of wax and disperse into 1 liter of warm reagent grade acetone.
2. Agitate using ultrasonic vibration for about 15 minutes at 50°C.
3. Let solution stand for 16 hours and decant solubles through a standard 3 micron fluoropore membrane filtration unit.
4. Wash filter and insoluble wax again with an additional 500 cc of acetone and allow solution to stand for about 4 hours.
5. Decant solubles through filter, rewash filter with acetone and add to acetone solubles.
6. Remove acetone with a nitrogen stream while gently warming the wax on the hot plate from both fractions (using a vacuum enhances the procedure).
7. Weigh solubles and insolubles.
8. Melt insolubles and disperse into 250-800 cc of cyclohexane dependent on the amount remaining after the removal of the acetone.
9. Repeat steps 2-7 above for separating the cyclohexane into soluble and insoluble portions.
10. Continue weighing procedure until no weight loss is observed, recovery is between 97-100%.

Standard Analytical Methods

Method Title	ASTM Method No.*
API Gravity	D-1298
Specific Gravity	D-4052
IBP, °F	D-1160
Carbon and Hydrogen	
Oxygen, Total, in Organic Materials	
Trace Sulfur (Micro Coulometric Titration)	
Trace Nitrogen (Micro Coulometric Method)	D-3431
Aniline and Mixed Aniline Point	D-611-82
Melting Point of Petroleum Wax	D-87-77
Viscosity, Kinematic	D-445
Conradson Carbon Residue of Petroleum Products	D-189
Heptane Insolubles - Membrane Filtration	
Metals by Wet Ash Emmision	

* "1987 Annual Book ASTM Standards," American Society for Testing and Material, Philadelphia, PA 1987.

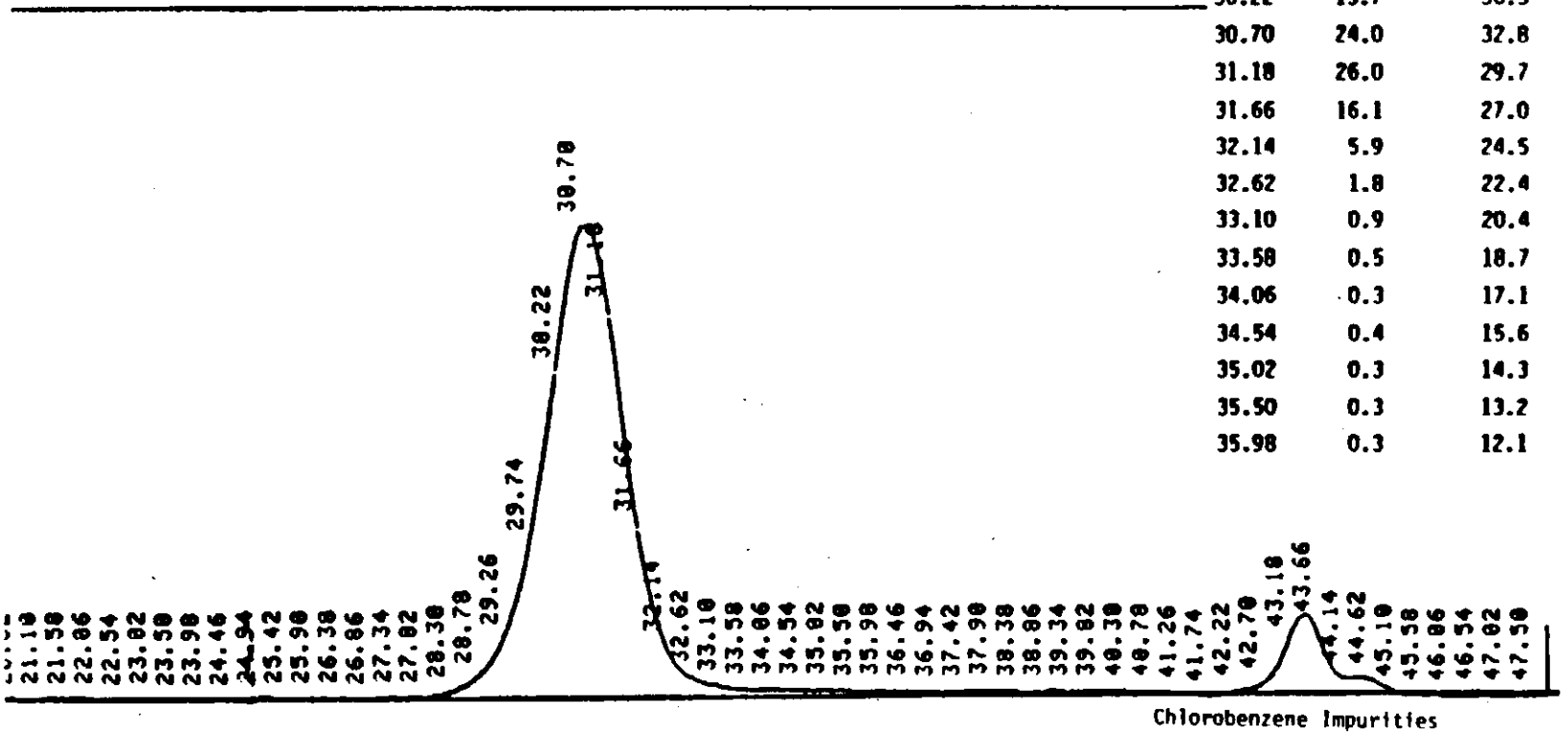
Gas Chromatography Analyses

	Distillation Cut 1	Cut 2
C_{11}	0.1	
C_{12}	0.7	
C_{13}	1.5	
C_{14}	2.4	
C_{15}	3.5	
C_{16}	4.8	
C_{17}	6.4	0.1
C_{18}	8.3	0.4
C_{19}	10.2	1.0
C_{20}	11.7	2.4
C_{21}	12.3	4.7
C_{22}	11.2	7.6
C_{23}	9.3	10.8
C_{24}	6.8	12.5
C_{25}	4.5	13.0
C_{26}	2.8	11.8
C_{27}	1.6	9.8
C_{28}	0.9	7.5
C_{29}	0.5	5.5
C_{30}	0.3	3.9
C_{31}	0.1	2.8
C_{32}	0.1	1.9
C_{33}		1.3
C_{34}		0.9
C_{35}		0.6
C_{36}		0.5
C_{37}		0.3
C_{38}		0.2
C_{39}		0.2
C_{40+}	___	0.3
Total	100.0 Area %	100.0 Area %

GPC Analysis

FT Wax
UOP 115-5563
Distillation Cut 3

RT	Wt-%	Carbon No.
28.30	.2	55.9
28.78	.8	49.9
29.26	2.2	44.8
29.74	5.9	40.2
30.22	13.7	36.3
30.70	24.0	32.8
31.18	26.0	29.7
31.66	16.1	27.0
32.14	5.9	24.5
32.62	1.8	22.4
33.10	0.9	20.4
33.58	0.5	18.7
34.06	0.3	17.1
34.54	0.4	15.6
35.02	0.3	14.3
35.50	0.3	13.2
35.98	0.3	12.1

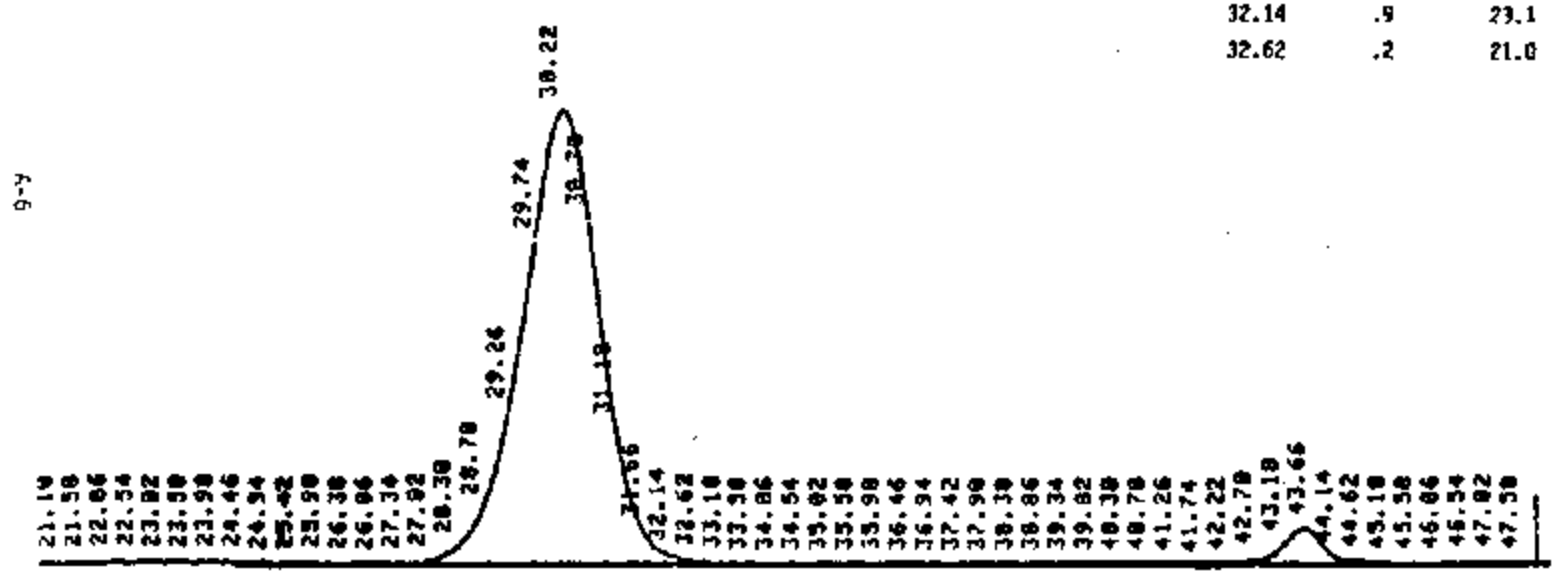


Retention Time (RT)

GPC Analysis

FT Wax
(DOE [IS-5563
Distillation Cut 4

RT	Wt-%	Carbon No.
28.30	.5	52.1
28.78	2.2	46.7
29.26	6.7	41.9
29.74	16.0	37.7
30.22	27.1	34.1
30.70	27.4	30.8
31.18	14.7	27.9
31.66	4.3	25.4
32.14	.9	23.1
32.62	.2	21.0



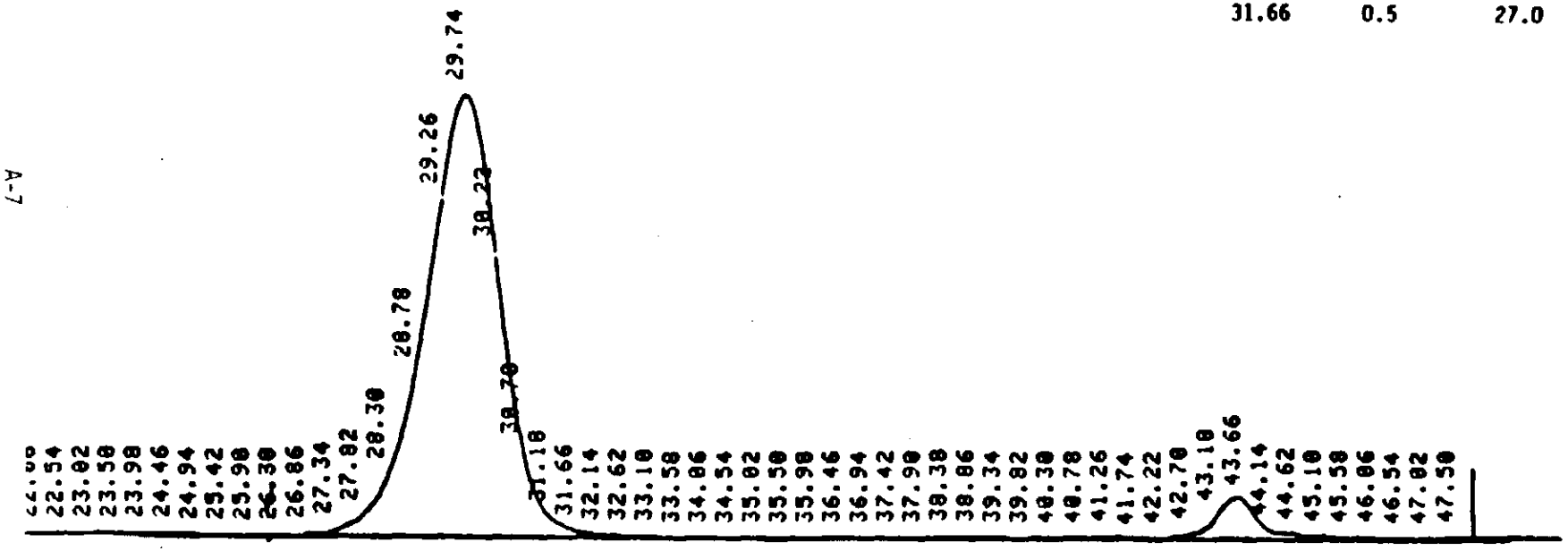
Retention Time (RT)

GPC Analysis

FT Wax
UOP 115-5563

DISTILLATION CUT 5

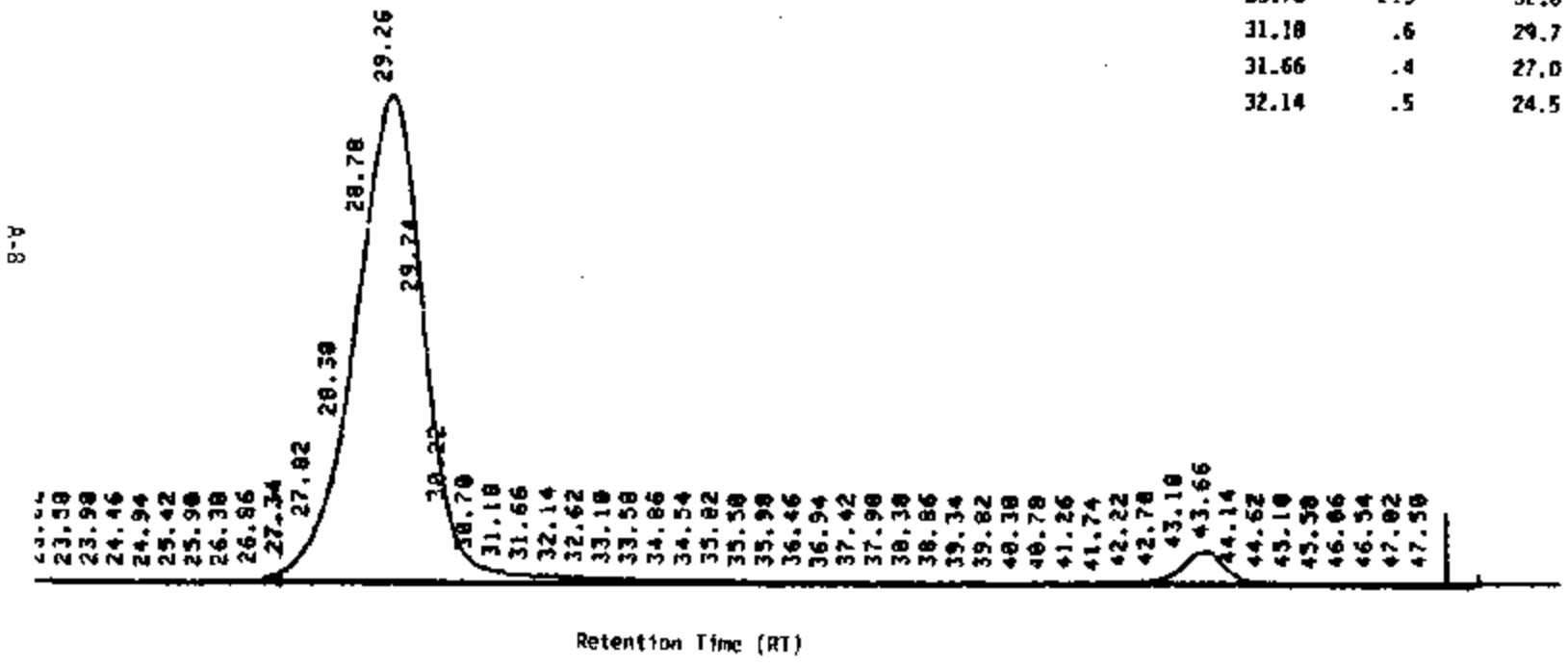
RT	Wt-%	Carbon No.
27.82	.4	62.8
28.30	2.5	55.9
28.78	7.3	49.9
29.26	17.6	44.8
29.74	29.3	40.2
30.22	27.4	36.3
30.70	12.2	32.8
31.18	2.8	29.7
31.66	0.5	27.0



GPC Analysis

FT Wax
UOP 115-5563
Distillation Cut 6

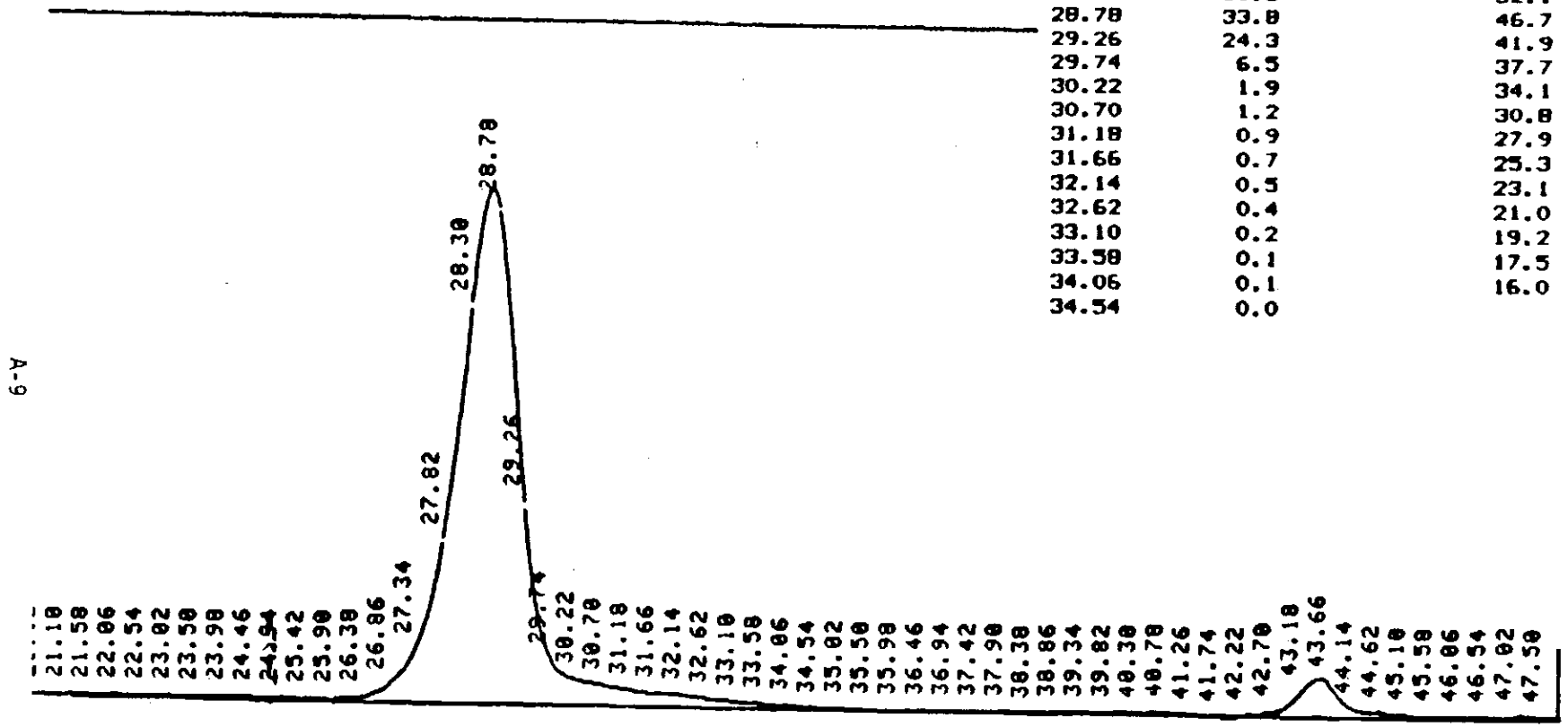
RT	Wt-%	Carbon No.
27.34	.4	70.8
27.82	1.9	62.8
28.30	6.4	55.9
28.78	17.2	49.9
29.26	31.4	44.7
29.74	28.2	40.2
30.22	10.6	36.3
30.70	2.3	32.8
31.18	.6	29.7
31.66	.4	27.0
32.14	.5	24.5



GCP Analysis

FT Wax
UOP 115-5563
Distillation Cut 7

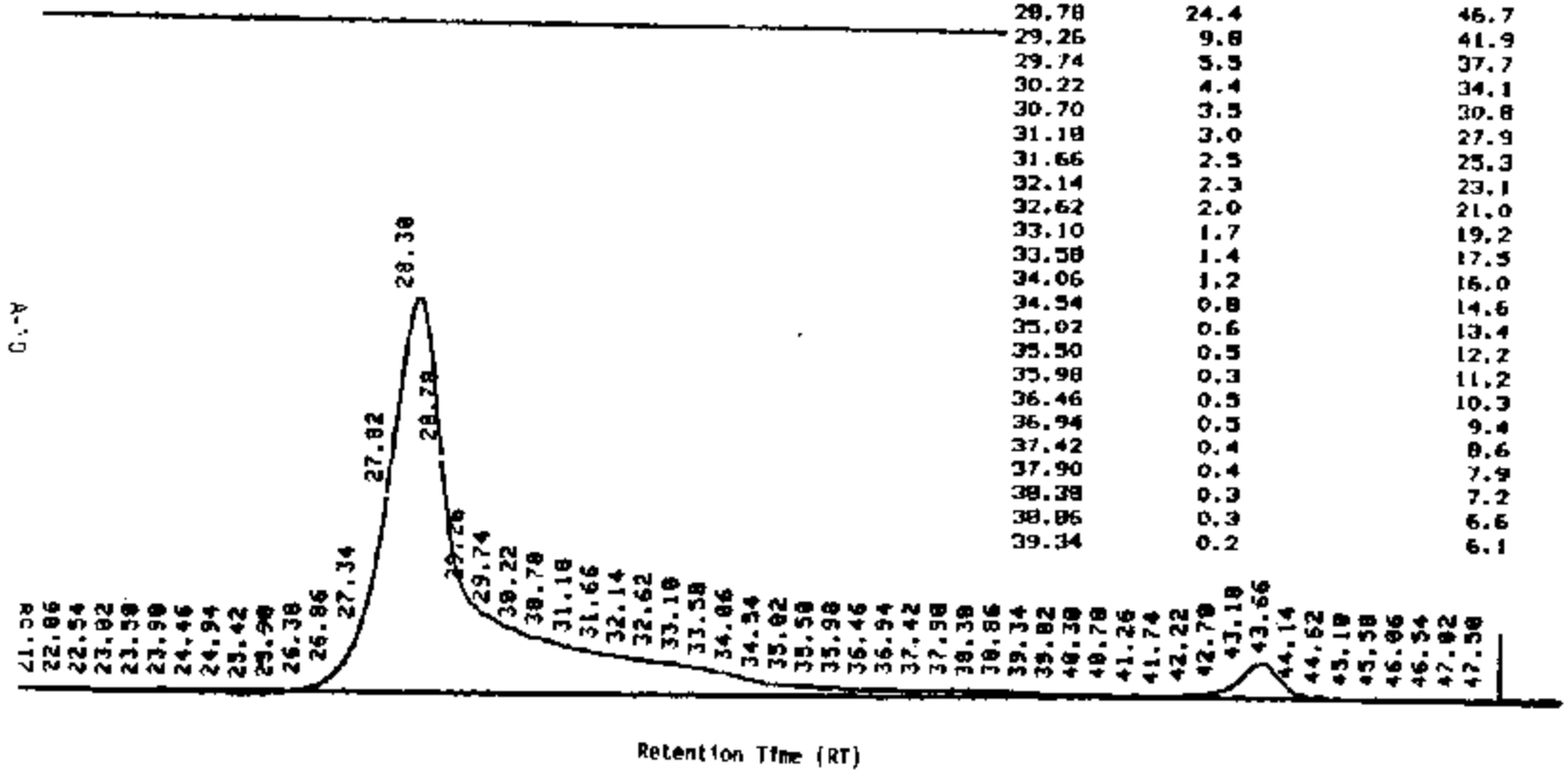
RT	WT-%	CARBON NO.
26.86	0.7	73.9
27.34	2.3	65.6
27.82	7.1	58.4
28.30	19.3	52.1
28.78	33.8	46.7
29.26	24.3	41.9
29.74	6.5	37.7
30.22	1.9	34.1
30.70	1.2	30.8
31.18	0.9	27.9
31.66	0.7	25.3
32.14	0.5	23.1
32.62	0.4	21.0
33.10	0.2	19.2
33.58	0.1	17.5
34.06	0.1	16.0
34.54	0.0	



Retention Time (RT)

GPC Analysis

FT Wax
UOP 115-5563
Distillation Cut 8

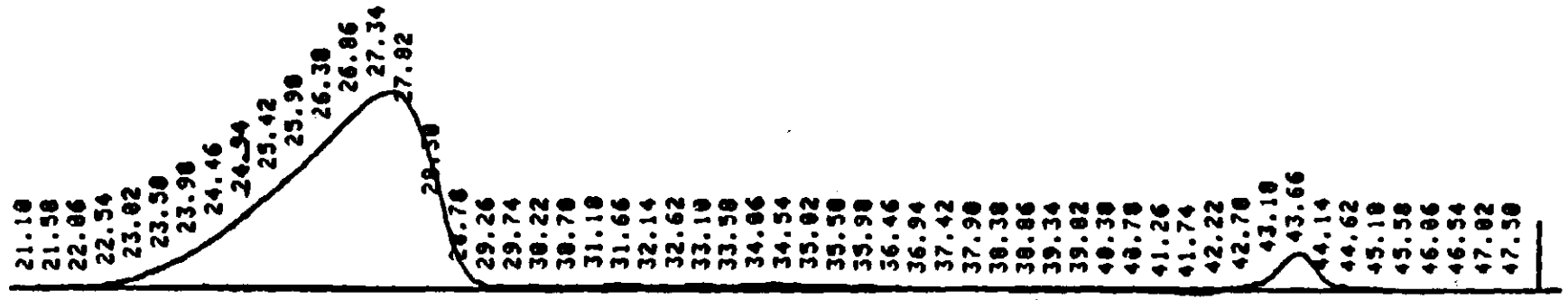


RT	WT-%	CARBON NO.
26.86	0.7	73.9
27.34	2.6	65.6
27.82	8.6	58.4
28.30	21.6	52.1
28.78	24.4	46.7
29.26	9.8	41.9
29.74	5.5	37.7
30.22	4.4	34.1
30.70	3.5	30.8
31.18	3.0	27.9
31.66	2.5	25.3
32.14	2.3	23.1
32.62	2.0	21.0
33.10	1.7	19.2
33.58	1.4	17.5
34.06	1.2	16.0
34.54	0.8	14.6
35.02	0.6	13.4
35.50	0.5	12.2
35.98	0.3	11.2
36.46	0.5	10.3
36.94	0.5	9.4
37.42	0.4	8.6
37.90	0.4	7.9
38.38	0.3	7.2
38.86	0.3	6.6
39.34	0.2	6.1

GPC Analysis

FT Wax
UOP 115-5563
Distillation BOTTS (579C+)

RT	Wt-%	Carbon No.
23.02	.4	221
23.50	1.2	190
23.98	2.3	164
24.46	3.6	142
24.94	5.4	124
25.42	7.2	108
25.90	9.0	95
26.38	11.3	84
26.86	13.5	74
27.34	15.2	66
27.82	15.1	58
28.30	10.2	52
28.78	2.8	47
29.26	.3	42
31.66	.5	27
33.10	.2	20
34.06	.3	17
35.02	.4	14
35.50	.3	13
35.98	.2	12
36.94	.4	10
37.42	.2	9

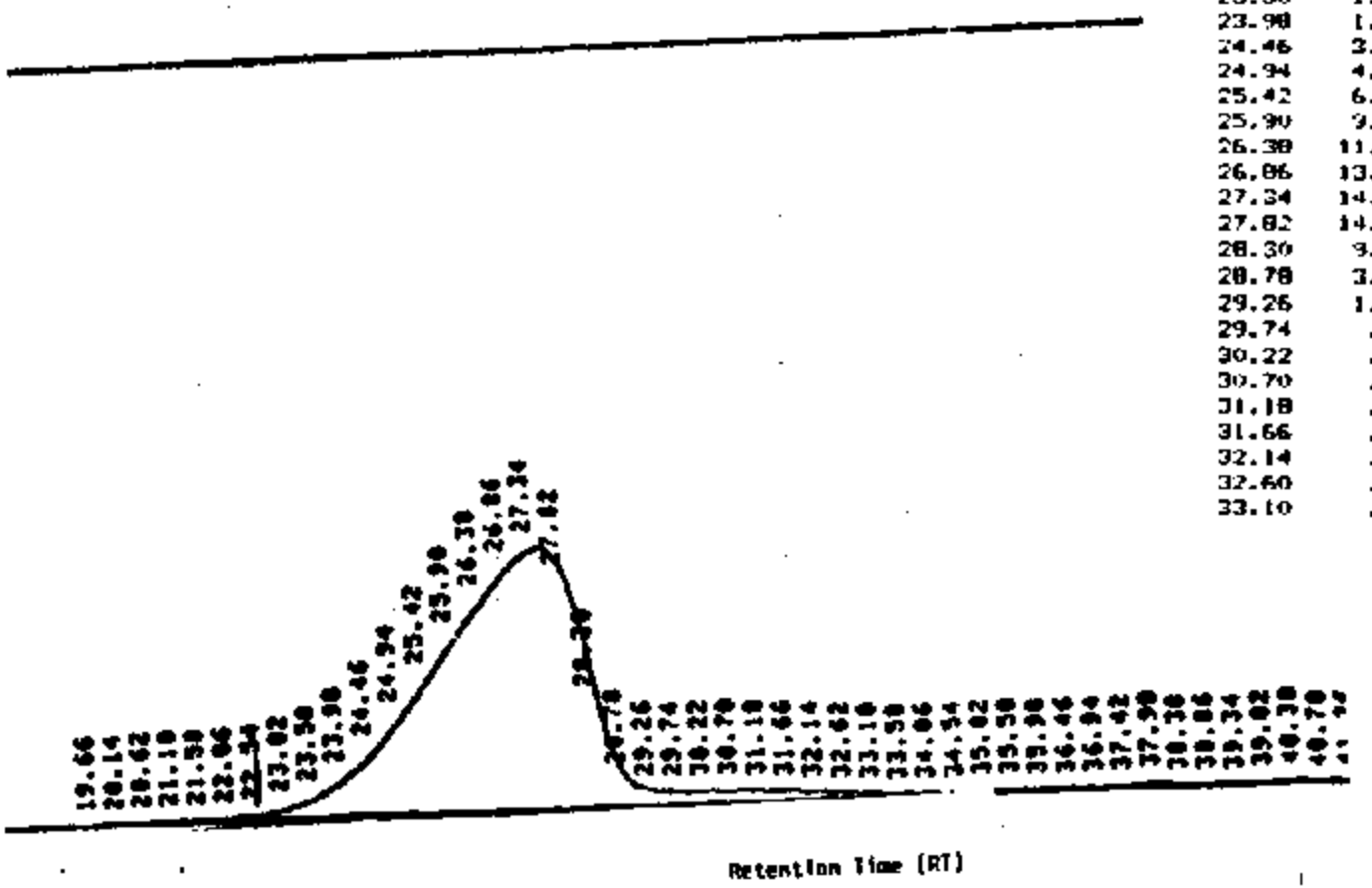


Retention Time (RT)

GPC Analysis

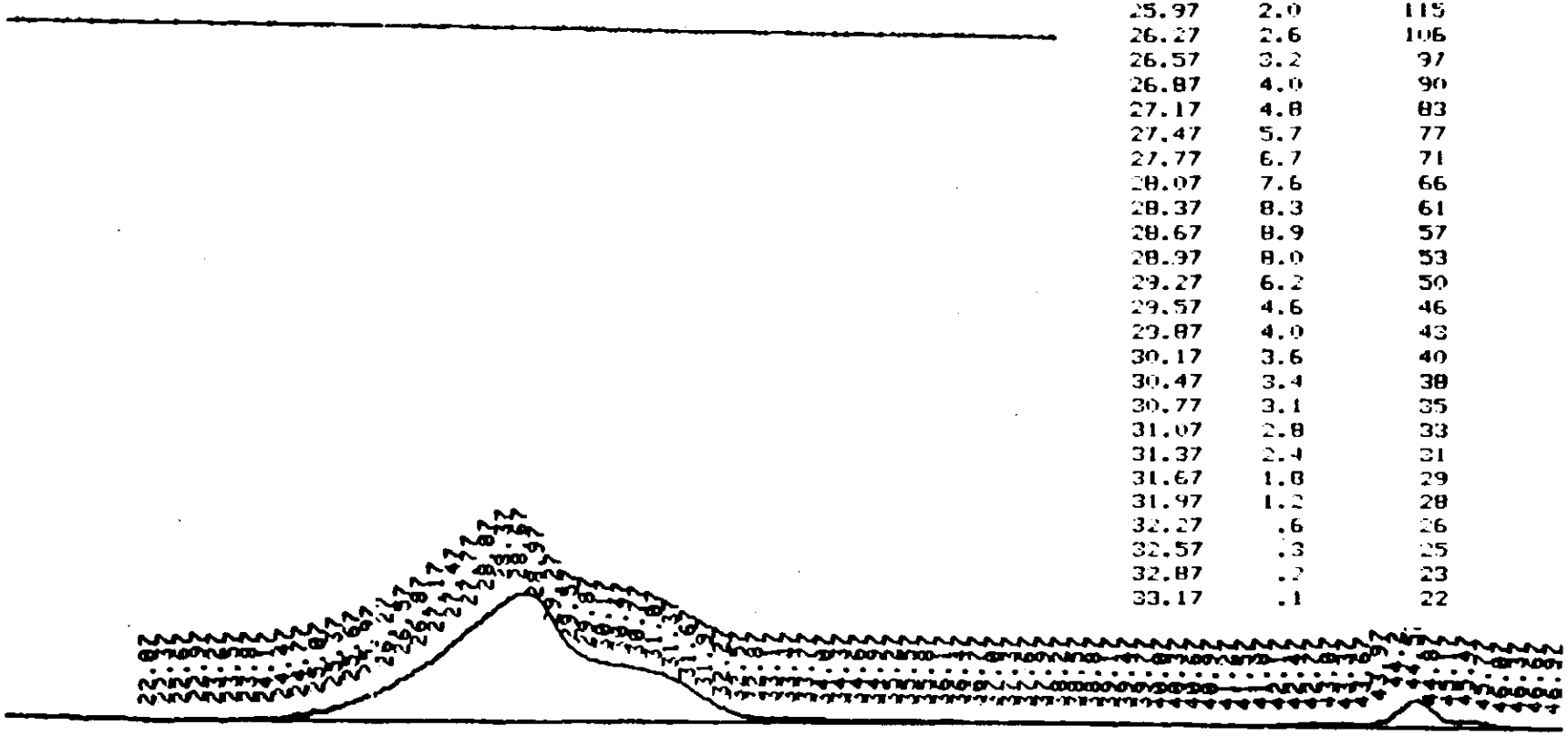
Commercial Arge Wax
Cyclohexane Insolubles

RT	Wt-%	Carbon No.
22.54	.3	340
23.02	.4	283
23.50	1.0	236
23.98	1.8	199
24.46	3.0	168
24.94	4.7	143
25.42	6.8	123
25.90	9.0	106
26.38	11.1	92
26.86	13.1	81
27.34	14.6	71
27.82	14.4	63
28.30	9.9	55
28.78	3.3	49
29.26	1.1	44
29.74	.8	40
30.22	.8	36
30.70	.9	32
31.18	.7	29
31.66	.6	26
32.14	.6	24
32.60	.6	22
33.10	.4	20



GPC Analysis

Union Carbide Wax
Cyclohexane Insolubles



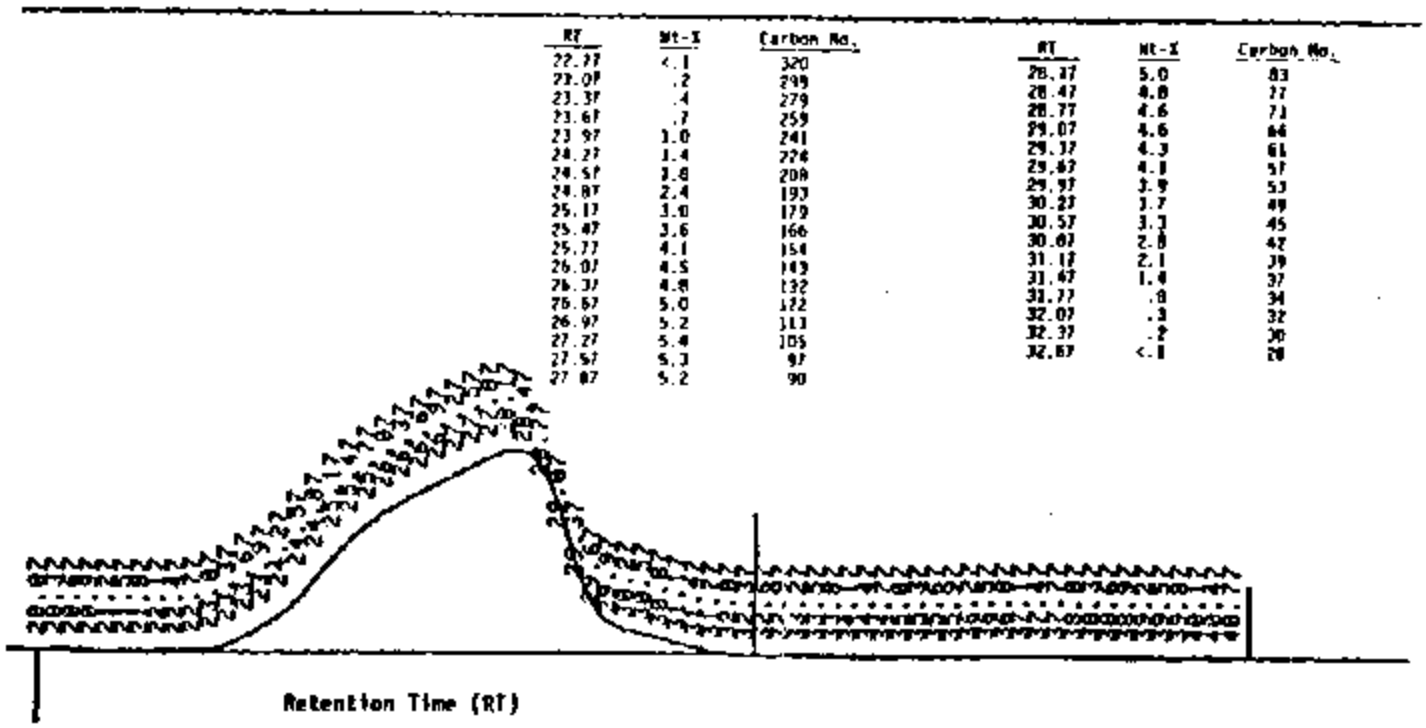
Retention Time (RT)

RT	Wt-%	Carbon No.
24.17	.1	200
24.47	.2	181
24.77	.4	165
25.07	.7	150
25.37	1.0	137
25.67	1.5	125
25.97	2.0	115
26.27	2.6	106
26.57	3.2	97
26.87	4.0	90
27.17	4.8	83
27.47	5.7	77
27.77	6.7	71
28.07	7.6	66
28.37	8.3	61
28.67	8.9	57
28.97	8.0	53
29.27	6.2	50
29.57	4.6	46
29.87	4.0	43
30.17	3.6	40
30.47	3.4	38
30.77	3.1	35
31.07	2.8	33
31.37	2.4	31
31.67	1.8	29
31.97	1.2	28
32.27	.6	26
32.57	.3	25
32.87	.2	23
33.17	.1	22

GPC Analysis

Mobil Wax
Cyclohexane Insolubles

RT	Wt-%	Carbon No.
22.77	<.1	320
23.07	.2	299
23.37	.4	279
23.67	.7	259
23.97	1.0	241
24.27	1.4	224
24.57	1.8	208
24.87	2.4	193
25.17	3.0	179
25.47	3.6	166
25.77	4.1	154
26.07	4.5	143
26.37	4.8	132
26.67	5.0	122
26.97	5.2	113
27.27	5.4	105
27.57	5.3	97
27.87	5.2	90
28.17	5.0	83
28.47	4.8	77
28.77	4.6	71
29.07	4.6	66
29.37	4.3	61
29.67	4.1	57
29.97	3.9	53
30.27	3.7	49
30.57	3.3	45
30.87	2.8	42
31.17	2.1	39
31.47	1.4	37
31.77	.8	34
32.07	.3	32
32.37	.2	30
32.67	<.1	28

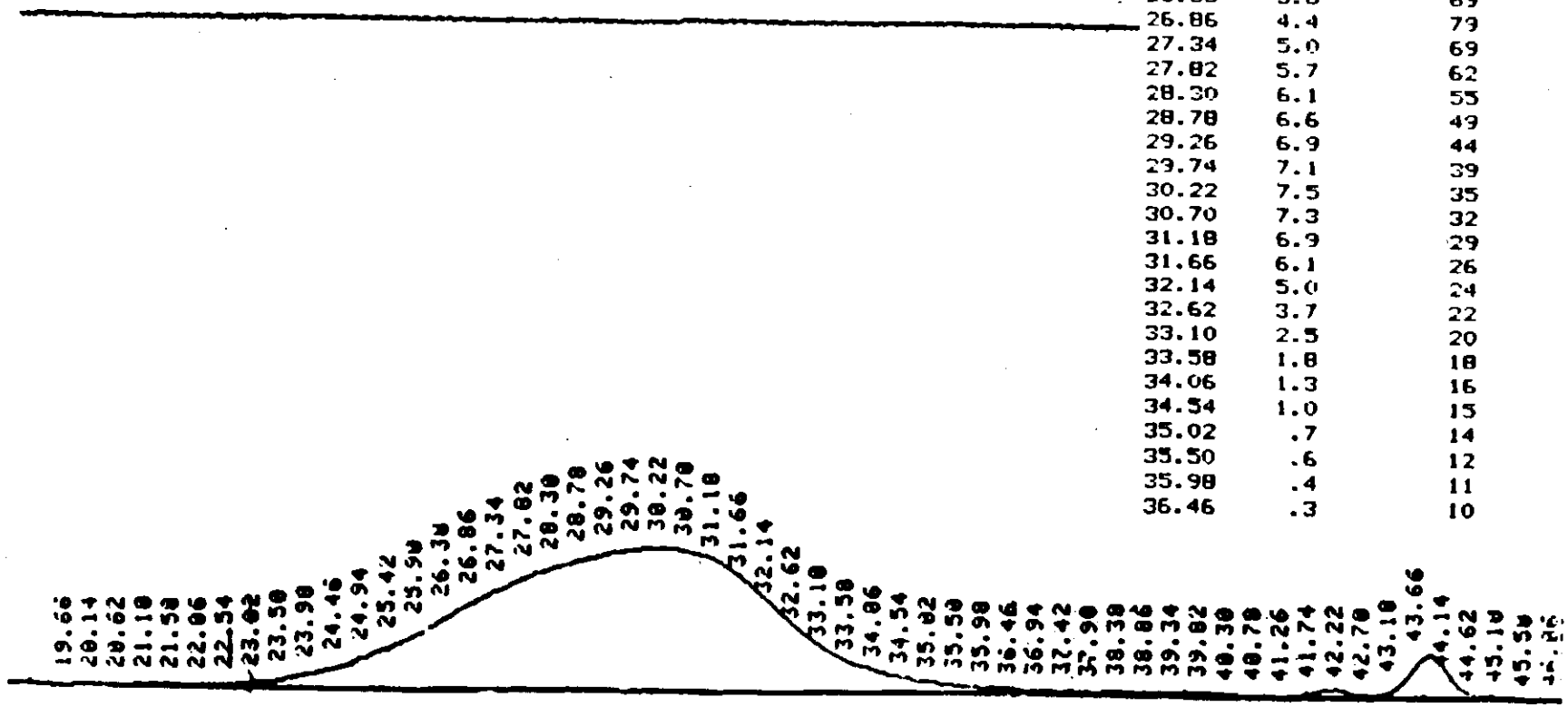


GPC Analysis

Total

Commercial Arge Wax
UOP 115-5563

RT	Wt-%	Carbon No.
23.02.	.5	250
23.50	.6	212
23.98	.8	182
24.46	1.2	156
24.94	1.5	135
25.42	2.2	117
25.90	2.8	102
26.38	3.6	89
26.86	4.4	79
27.34	5.0	69
27.82	5.7	62
28.30	6.1	55
28.78	6.6	49
29.26	6.9	44
29.74	7.1	39
30.22	7.5	35
30.70	7.3	32
31.18	6.9	29
31.66	6.1	26
32.14	5.0	24
32.62	3.7	22
33.10	2.5	20
33.58	1.8	18
34.06	1.3	16
34.54	1.0	15
35.02	.7	14
35.50	.6	12
35.98	.4	11
36.46	.3	10



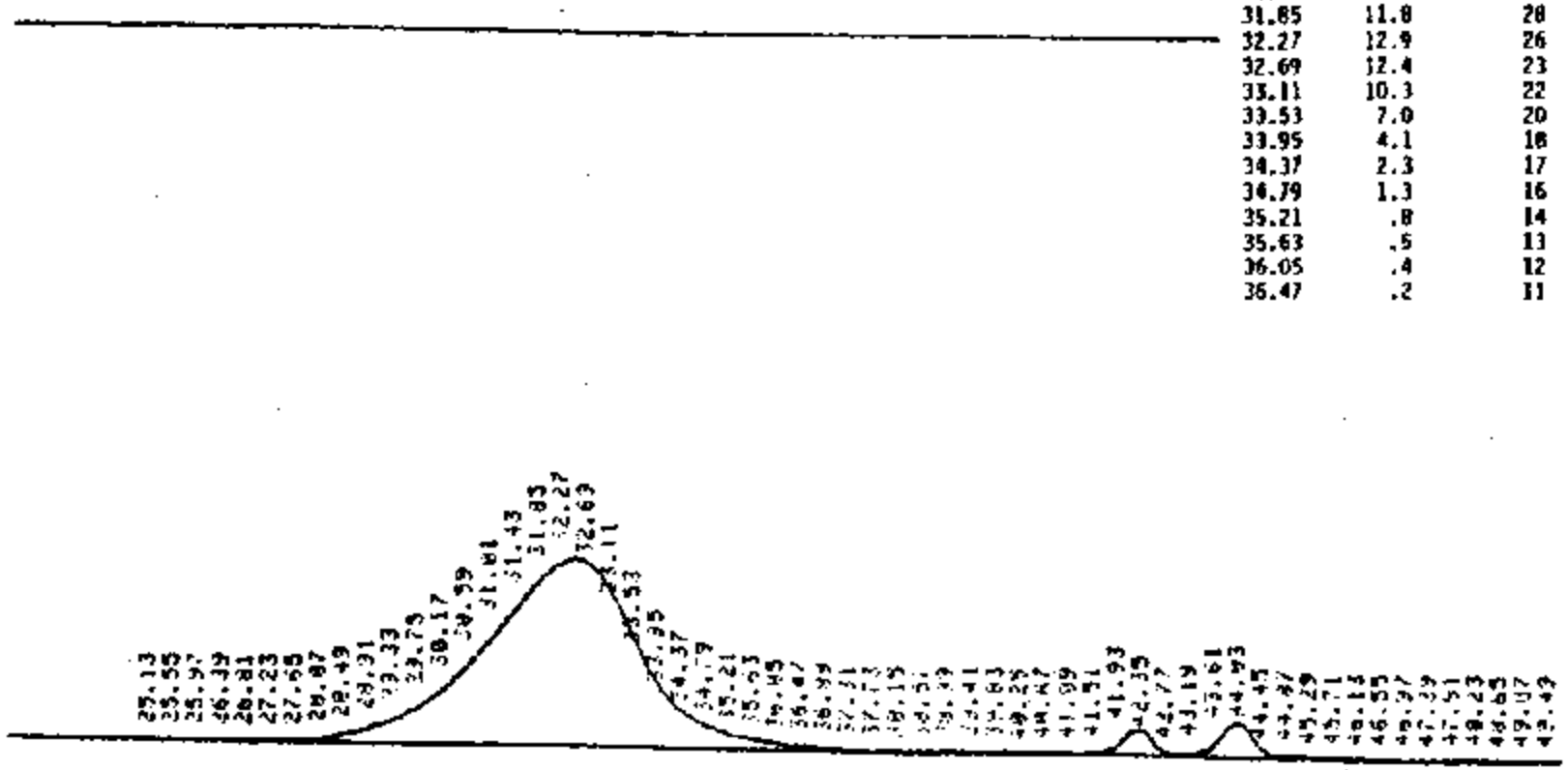
Retention Time (RT)

GPC Analysis

Total

Air Products Wax
UOP 49-2920

RT	Wt-%	Carbon No
28.07	0.4	64
28.49	0.8	58
28.91	1.3	52
29.33	2.0	48
29.75	3.1	43
30.17	4.4	40
30.59	6.1	36
31.01	7.9	33
31.43	10.0	30
31.85	11.8	28
32.27	12.9	26
32.69	12.4	23
33.11	10.3	22
33.53	7.0	20
33.95	4.1	18
34.37	2.3	17
34.79	1.3	16
35.21	.8	14
35.63	.5	13
36.05	.4	12
36.47	.2	11

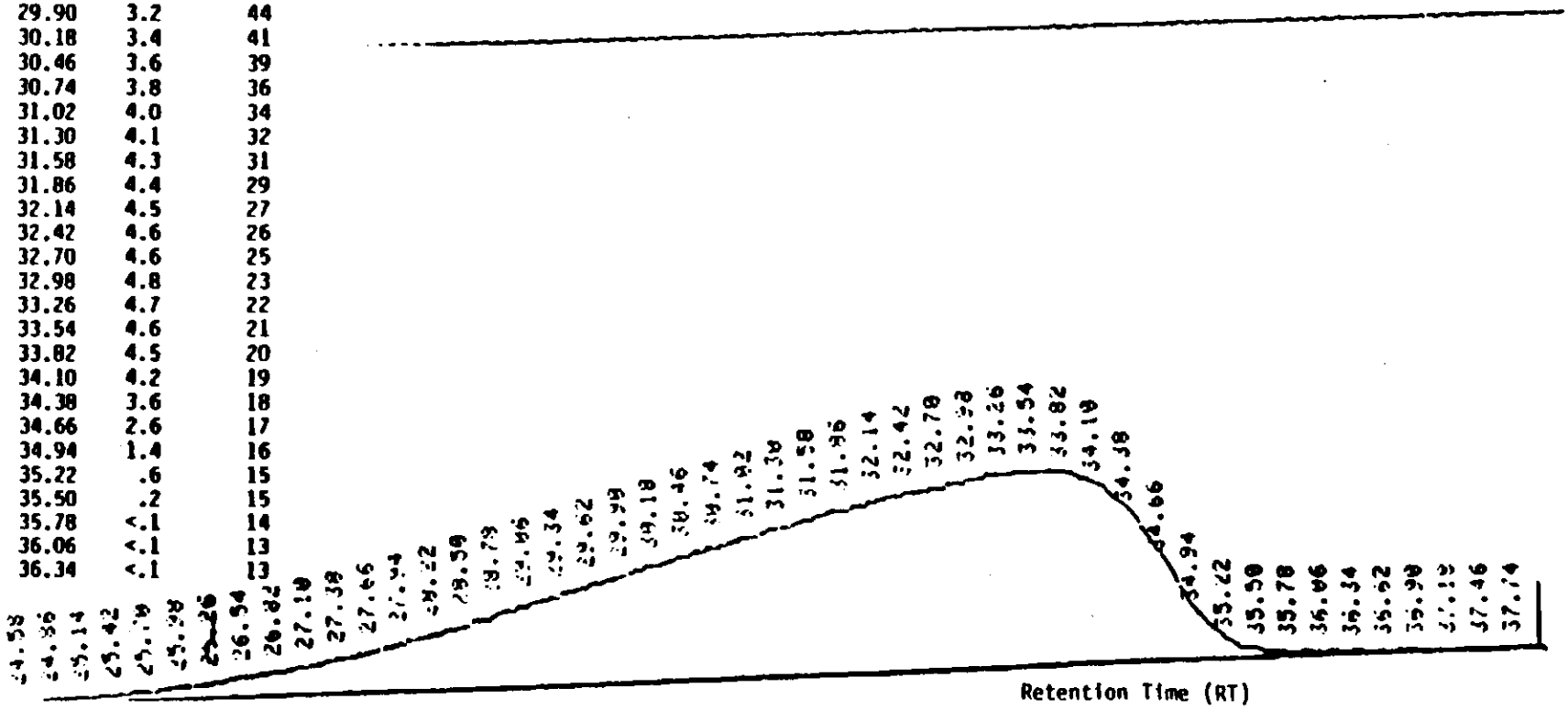


RT	Wt-%	Carbon No.
24.58	.2	196
24.86	.2	178
25.14	.3	162
25.42	.3	147
25.70	.4	134
25.98	.5	123
26.26	.6	113
26.54	.7	104
26.82	.9	96
27.10	1.1	88
27.38	1.2	82
27.66	1.4	76
27.94	1.6	70
28.22	1.9	65
28.50	2.1	61
28.78	2.3	57
29.06	2.6	53
29.34	2.7	50
29.62	3.0	46
29.90	3.2	44
30.18	3.4	41
30.46	3.6	39
30.74	3.8	36
31.02	4.0	34
31.30	4.1	32
31.58	4.3	31
31.86	4.4	29
32.14	4.5	27
32.42	4.6	26
32.70	4.6	25
32.98	4.8	23
33.26	4.7	22
33.54	4.6	21
33.82	4.5	20
34.10	4.2	19
34.38	3.6	18
34.66	2.6	17
34.94	1.4	16
35.22	.6	15
35.50	.2	15
35.78	A A.1	14
36.06	A A.1	13
36.34	A A.1	13

GPC Analysis

Total

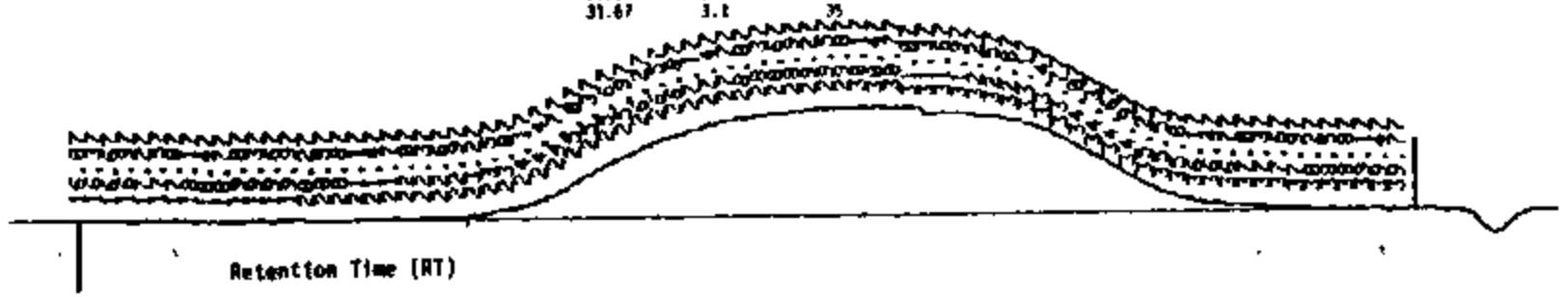
Union Carbide Wax
#86-0062222



GPC Analysis

Mobil Wax

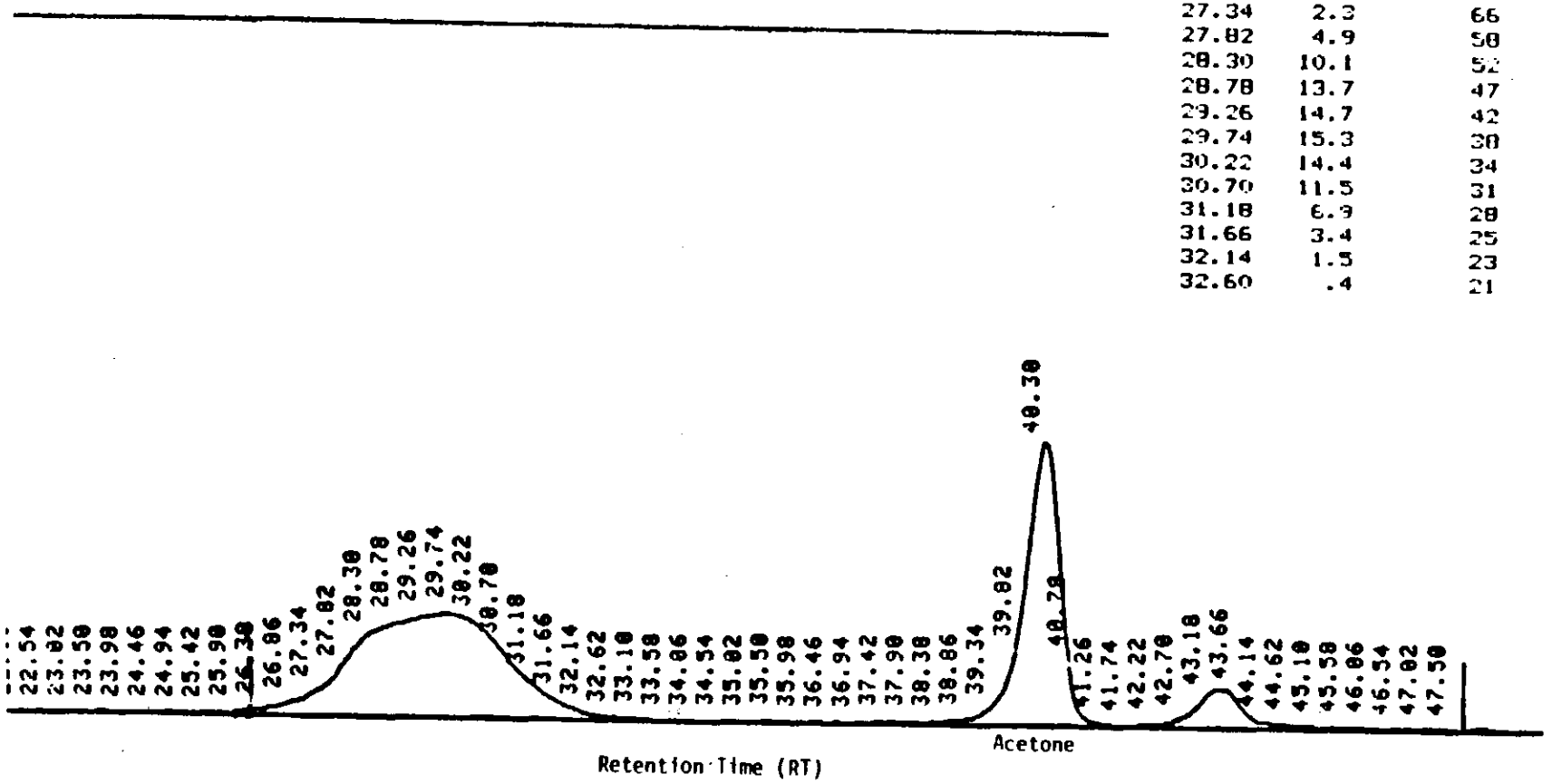
RT	Wt-%	Carbon No.	RT	Wt-%	Carbon No.
23.27	.1	285	31.97	2.9	33
23.57	.2	265	32.27	2.8	30
23.87	.3	247	32.53	2.7	29
24.17	.5	230	32.83	2.5	27
24.47	.7	213	33.17	2.3	25
24.77	1.0	198	33.47	2.0	24
25.07	1.6	184	33.77	1.8	22
25.37	1.9	170	34.07	1.6	21
25.67	2.2	158	34.37	1.2	20
25.97	2.5	146	34.67	1.0	19
26.27	2.8	135	34.97	.7	18
26.57	3.0	125	35.27	.6	17
26.87	3.2	116	35.57	.3	16
27.17	3.4	107	35.87	.2	15
27.47	3.5	100	36.17	.1	14
27.77	3.6	92	36.47	.1	14
28.07	3.6	85	36.77	.1	13
28.37	4.2	79	37.07	.1	13
28.67	3.8	73			
28.97	3.8	68			
29.27	3.8	63			
29.57	3.7	58			
29.87	3.6	54			
30.17	3.6	50			
30.47	3.5	47			
30.77	3.4	43			
31.07	3.4	40			
31.37	3.1	38			
31.67	3.1	35			



GPC Analysis

Commercial Arge Wax
Acetone Insolubles

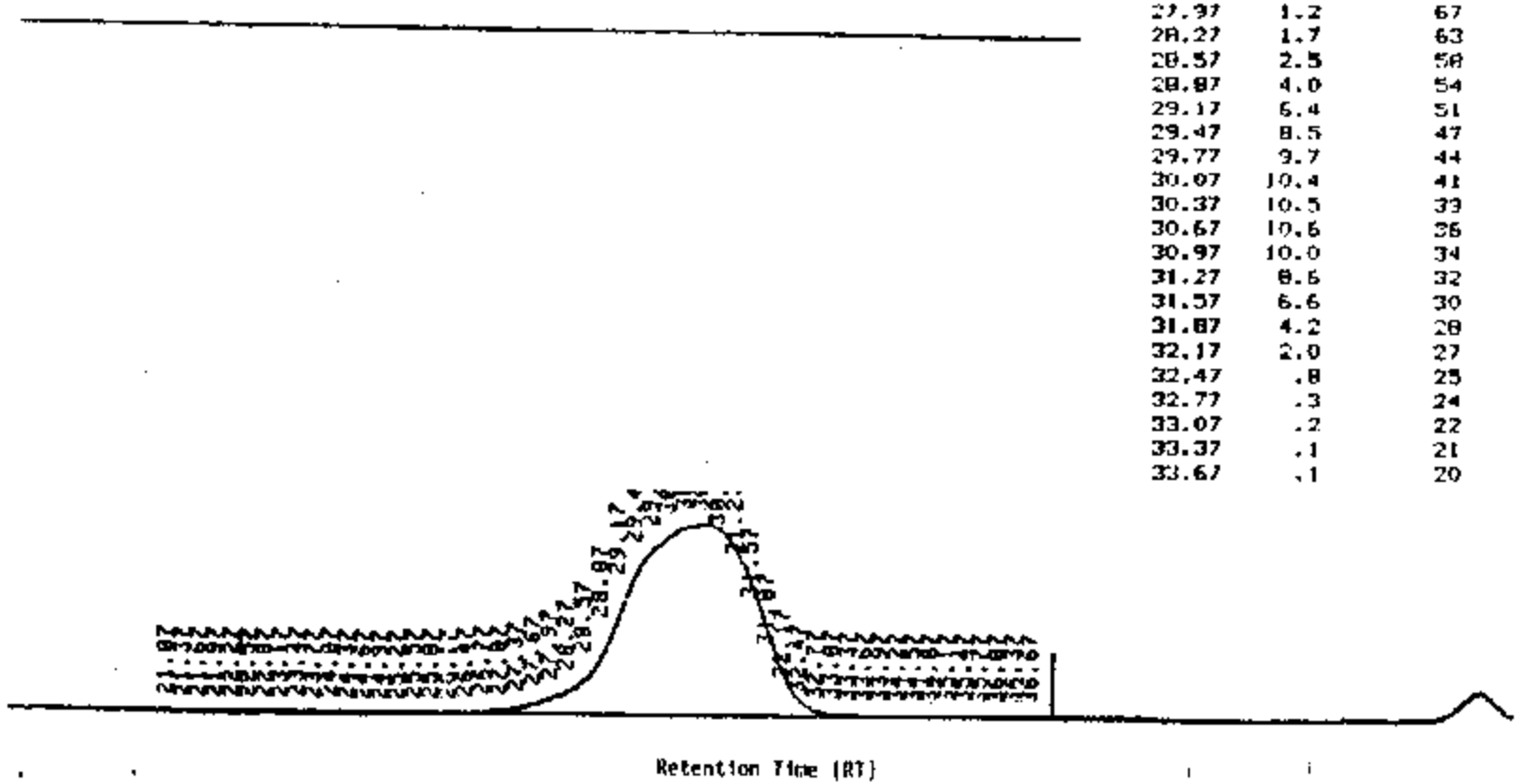
RT	Wt-%	Carbon No
26.86	.9	74
27.34	2.3	66
27.82	4.9	58
28.30	10.1	52
28.78	13.7	47
29.26	14.7	42
29.74	15.3	38
30.22	14.4	34
30.70	11.5	31
31.18	6.9	28
31.66	3.4	25
32.14	1.5	23
32.60	.4	21



GPC Analysis

Union Carbide Wax
Acetone Insolubles

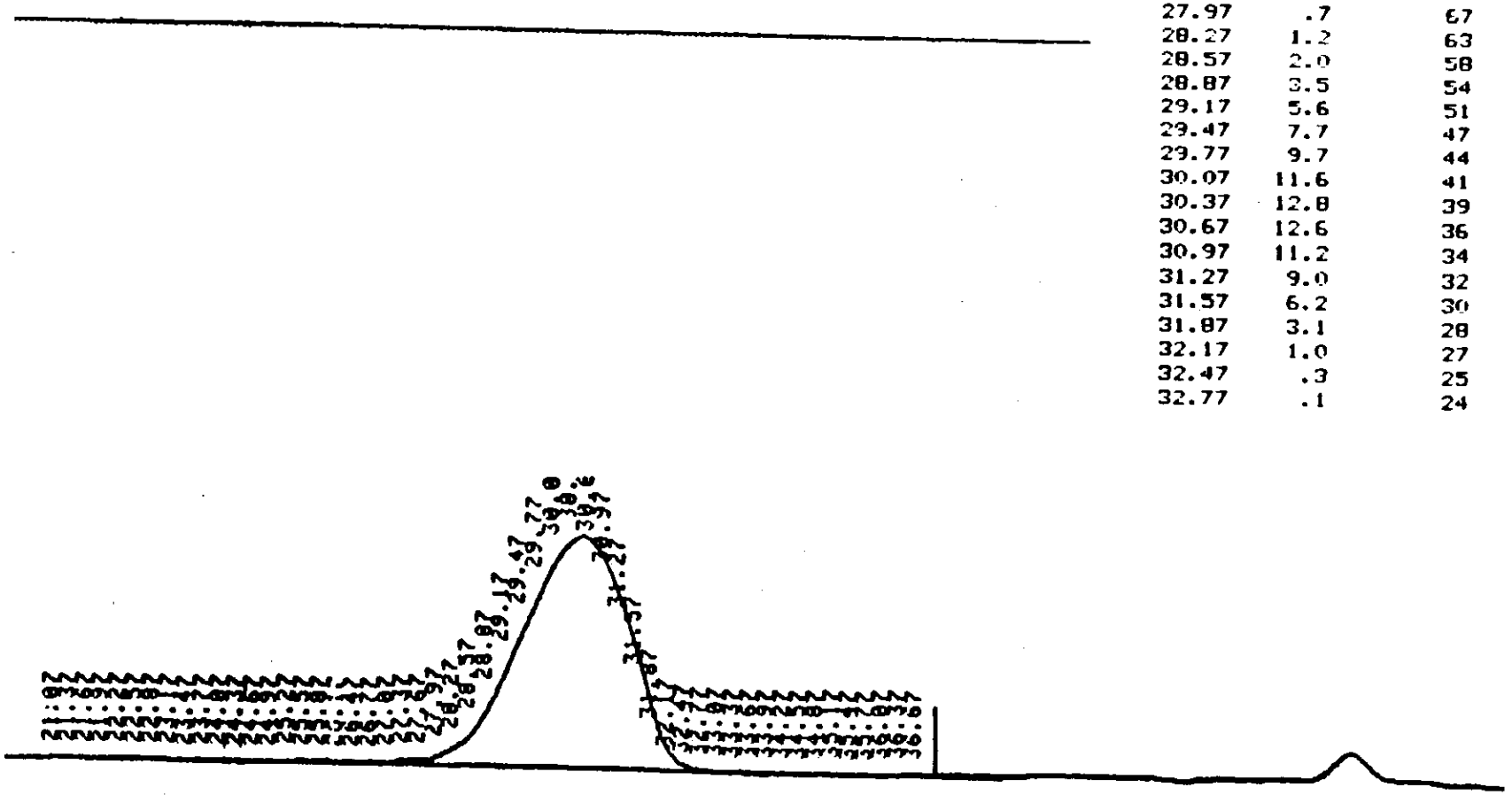
RT	Wt-%	Carbon No.
26.77	.1	92
27.07	.2	85
27.37	.5	79
27.67	.8	73
27.97	1.2	67
28.27	1.7	63
28.57	2.5	58
28.87	4.0	54
29.17	6.4	51
29.47	8.5	47
29.77	9.7	44
30.07	10.4	41
30.37	10.5	39
30.67	10.6	36
30.97	10.0	34
31.27	8.6	32
31.57	6.6	30
31.87	4.2	28
32.17	2.0	27
32.47	.8	25
32.77	.3	24
33.07	.2	22
33.37	.1	21
33.67	.1	20



GPC Analysis

Air Products Wax
Acetone Insolubles

RT	Wt-%	Carbon No
26.17	.2	109
26.47	.2	100
26.77	.2	92
27.07	.3	85
27.37	.3	79
27.67	.5	73
27.97	.7	67
28.27	1.2	63
28.57	2.0	58
28.87	3.5	54
29.17	5.6	51
29.47	7.7	47
29.77	9.7	44
30.07	11.6	41
30.37	12.8	39
30.67	12.6	36
30.97	11.2	34
31.27	9.0	32
31.57	6.2	30
31.87	3.1	28
32.17	1.0	27
32.47	.3	25
32.77	.1	24

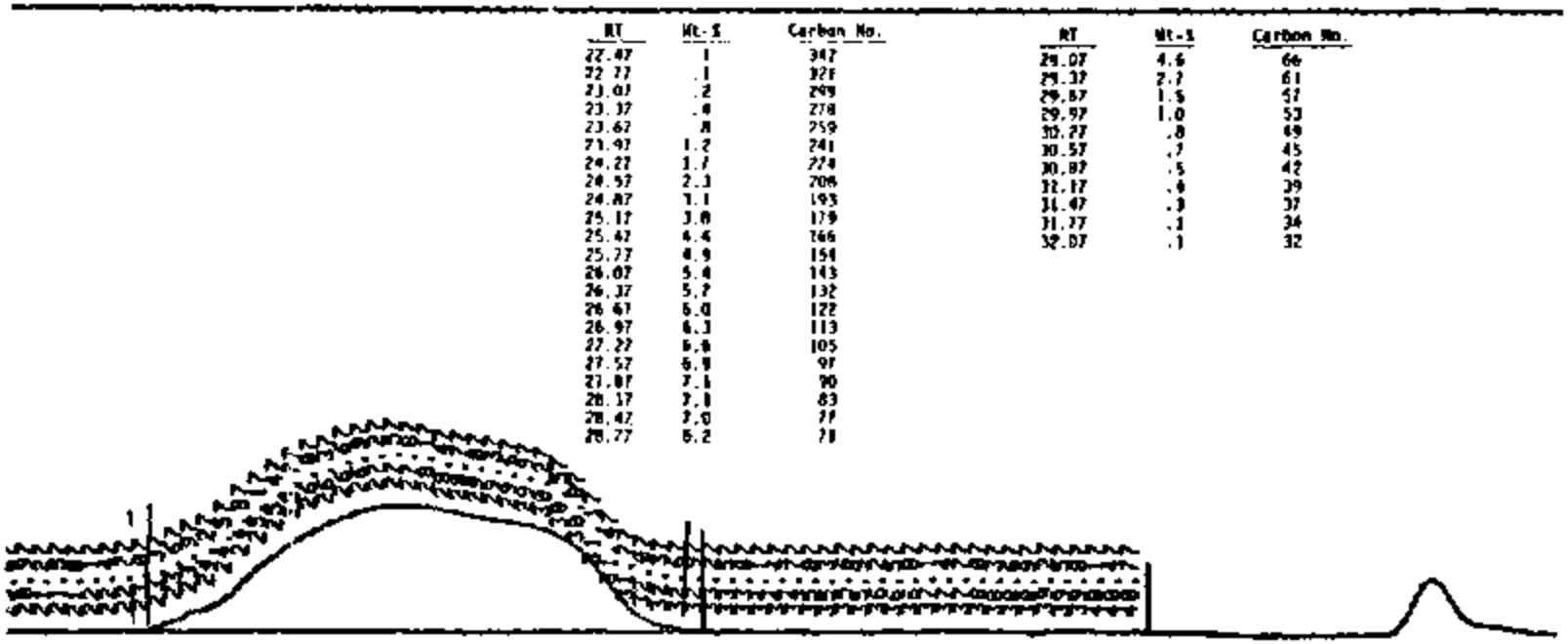


GPC Analysis

Mobil Wax
Acetone Insolubles

RT	Wt-%	Carbon No.
22.47	1	347
22.77	.1	328
23.07	.2	309
23.37	.4	278
23.67	.8	259
23.97	1.2	241
24.27	1.7	224
24.57	2.1	208
24.87	2.1	193
25.17	3.0	179
25.47	4.4	166
25.77	4.9	154
26.07	5.4	143
26.37	5.7	132
26.67	6.0	122
26.97	6.3	113
27.27	6.6	105
27.57	6.8	97
27.87	7.1	90
28.17	7.1	83
28.47	7.0	77
28.77	6.2	71

RT	Wt-%	Carbon No.
29.07	4.6	66
29.37	2.7	61
29.67	1.5	57
29.97	1.0	53
30.27	.8	49
30.57	.7	45
30.87	.5	42
31.17	.4	39
31.47	.3	37
31.77	.1	34
32.07	.1	32



Retention Time (RT)

Size Exclusion - Chromatographic Separation of Fischer-Tropsch Waxes (II)
(Correction of Retention Times for Calibrations)

INTRODUCTION

Since the release of the memo "Size Exclusion-Chromatographic Separation of Fischer-Tropsch Waxes" addressed to Hayim Abrevaya dated April 14, 1986, it was discovered that when calibration retention times were measured using the Waters model 730 Data Module in the calibration mode, faulty retention times were provided.

The following report contains corrected calibrations.

EXPERIMENTAL

It was found that when the Model 730 Waters Data Module was used in the calibration mode, with the chart speed set at 1 cm/min., a run time of 50 minutes had a pen trace of 50.5 cm. This was 0.99 minutes per centimeter. When the Data Module was placed in the analysis mode the pen trace for a 50 minute run time measured 50 centimeters.

Some of the calibrations in the April 14, 1986 report were made where the retention times had been measured in the calibration mode.

Results appearing in the following report were obtained using corrected retention times for the calibrations.

A 50 µl aliquot of a 0.25 percent solution of a Fischer-Tropsch wax in ODCB was separated on a set of ASI Ultragel 2-500Å,2-100 Å30 cm x 7.8 mm columns using a Waters ALC/GPC Model 150C Chromatograph.

Separations were made using ODCB as the eluent with the columns, differential refractometer and the injector maintained at 100°C. The flowrate was set at 4.8/5.0 mL/min. A Waters Model 730 Data Module was used to monitor the detector response. The chart speed was set at 1 cm/min.

The chromatograph of a Fischer-Tropsch wax is shown in Figure 1.

In order for the chromatogram to be of any value, the retention times had to be correlated with the carbon number. Individual samples with known carbon numbers were analyzed. Results are shown in Table 1. A plot of these values is found in Figure 2.

It was noted that for the carbon range of C_6 to C_{60}, the plot was not linear. The carbon numbers and their respective retention times were entered into a Water Model 730 Data Module, and the third order fit calibration coefficients were calculated using the following formula where:

$$\text{Log CN} = D_0 + D_1\ RT + D_2\ (RT)^2 + D_3\ (RT)^3$$

The calibration coefficients are shown in Table 2.

The individual retention times for the standards were manually reentered into the Data Module. The resulting carbon numbers obtained from the retention time input are shown in Table 3. The calculated carbon numbers agreed favorably with the actual carbon numbers.

In order to verify the Waters Data Module calibrations, a least squares fit plot of the retention times vs. carbon numbers was plotted using the Hewlett Packard Model 3357 laboratory automation system. The plot is shown in Figure 3. The calculated carbon numbers were determined using the formula where:

$$C = (1.6B352E+11) \times T^{-6.53713}$$

Results are shown in Table 4.

Like the results obtained using the Waters Data Module, the Hewlett Packard calculated values agreed favorably with the actual carbon numbers. The Waters Data Module calibration was used for all analyses.

An attempt was made to extend the range of the carbon number calibration. Aliquots of solutions of Apolane-87, a branched 87 carbon number and polyethylene with a number average molecular weight of 2015 and a calculated carbon number of 143.9 were analyzed in addition to the linear alkanes. The retention times were correlated with the carbon numbers as shown in Table 5.

A plot was made of the values presented in Table 5. The results are shown in Figure 4.

From the above plot it was shown that Apolane-87, a branched 87 carbon number eluted later than when a corresponding linear C_{87} alkane would elute. However the polyethylene with a number average molecular weight of 2015 and a calculated carbon number of 143.9 fit in the plot of the linear alkanes.

The third order calibration coefficients were calculated for the extended range calibration. Results are shown in Table 6. The individual retention times for the standards were manually reentered into the Data Module. The resulting carbon numbers obtained from the retention time input are shown in Table 7. The calculated carbon numbers agreed favorably with the actual carbon numbers.

The Waters Data Module calibration coefficients for carbon number range of 6-60 shown in Table 8 were used to calculate the results for the was sample shown in Figure 1. Area percentages and carbon numbers were correlated by utilizing the area percents vs. retention times from Table 9 and the carbon numbers vs. retention times found in Table 10.

It has been known that the refractive indicies change from compound to compound. Therefore, in order to report concentrations of carbon number as weight percent, response factors for the carbon range relating area to weight had to be determined.

Aliquots with known weights of the individual alkane standards were analyzed. The weight per unit area for the standards were calculated. Results are shown in Table 11.

A plot of the above values in Figure 5 showed a sharp rise of the picogram per unit area from carbon number 12 to 30. From 30 on the curve started to level off.

Using the above correlation between picogram per area vs. carbon number, CHPfeiffer wrote a program for the Hewlett Packard Model 3357 Laboratory Automation system, whereby the area percentages could be converted to weight percent. The corrected area was calculated by using the following formula.

$$A_c = A \times \frac{C}{0.314 + (0.0198 \times C)}$$

The carbon numbers and their respective area percents for the sample shown in Figure 1 were entered into the program.

The resulting weight percents were calculated. Results are shown in Table 12.

CONCLUSIONS

From the work done, it was found that the carbon number range and weight percent for various Fischer-Tropsch wax fractions could be determined by size exclusion chromatography. Two calibrations were essential for the analysis. The first was the third order fit correlating the retention times vs. carbon number and the second one correlating the weight to area response over the carbon number range.

R. F. Swensen

DC/16

ODCB = ortho di-chloro benzene

TABLE I

ULTRAGEL

Calibration of Ultragel 2-500, 2-100 Å Columns Using Linear Alkanes as Standards

RT MIN	CN
27.90	60
28.60	50
29.20	44
30.10	36
30.60	32
31.30	28
32.10	24
32.60	22
33.00	20
33.60	18
34.30	16
34.90	14
35.70	12
39.40	6

TABLE 2

Calculation of Third Order Fit Calibration
Coefficients Using a Waters Model 740 Data Module

GPC CALIBRATION
NUMBER OF STANDARDS: 14

RT	CN x 10^3	LOG CN x 10^3
27.90	0.600000E5	4.778
28.60	0.500000E5	4.699
29.20	0.440000E5	4.643
30.10	0.360000E5	4.556
30.50	0.320000E5	4.505
31.30	0.280000E5	4.447
32.10	0.240000E5	4.380
32.50	0.220000E5	4.342
33.00	0.200000E5	4.301
33.60	0.180000E5	4.255
34.30	0.160000E5	4.204
34.90	0.140000E5	4.146
35.70	0.120000E5	4.079
39.40	0.600000E4	3.778

CALIBRATION COEFFICIENTS

D0	0.135140000000E2
D1	-0.572161000000E0
D2	0.167675000000E-1
D3	-0.156311000000E-3

STD ERR OF ESTIMATE		0.324363E-1
CORR	COEFFICIENT	0.392685E0

TABLE 3

Comparison of the Calculated Carbon Number from the Waters Model 730 Data Module 3rd Order Fit vs. the Actual Carbon Number

RT Min	ACTUAL CN	Calculated CN Via Entering RT
27.90	60	59.5
28.60	50	50.3
29.20	44	43.85
30.10	36	35.99
30.60	32	32.38
31.30	28	28.0
32.10	24	23.9
32.60	22	21.7
33.0	20	20.1
33.6	18	17.9
34.30	16	15.75
34.90	14	14.09
35.70	12	12.16
39.40	6	5.99

TABLE 4

Comparison of the Calculated Carbon Number from
The Hewlett Packard Model 3357 Laboratory Automation
System vs. the Actual Carbon Number

$$C = (1.68352\ E{+}11) \times T^{-6.53713}$$

```
Details for curve type (1-6/7-PLOT/0-END)? 3
   3. Y=A*(X^E) IS A POWER FUNCTION.  THE RESULTS
      OF A LEAST-SQUARE FIT OF ITS LINEAR TRANSFORM
      (SORTED IN ORDER OF ASCENDING VALUES OF X)
      ARE AS FOLLOWS:

X-ACTUAL        Y-ACTUAL        Y-CALC          PCT DIFFER

27.9            60              59.72               .4
28.6            50              50.7893           -1.5
29.2            44              44.3433           -.7
30.1            36              36.3616           -.9
30.6            32              32.649            -1.9
31.3            26              26.1316           -.5
32.1            24              23.8785
32.6            22              21.5636
                20              19.9296
33.6            18              17.7159
34.3            16              15.4812
34.9            14              13.8221
35.7            12              11.9198
39.4            6               6.25546           -4

14              34.9            14
15              35.7            12
16              39.4            6

Modifications (Y/N) ? N

Outdvc ? T4

                LEAST SQUARES CURVE FIT

CURVE TYPE      INDEX OF        A               E
                DETERMINATION

1. Y=A+(E*X)    .887819         177.466         -4.888888
2. Y=A*EXP(E*X).998254          14290.9         -.1888884
3. Y=A*(X^E)    .999987         1.683528E+11    -6.53713
4. Y=A+(E/X)    .921695         -133.412        5160.04
5. Y=1/(A+E*X)  .894687         -.922187        1.158888E-02
6. Y=X/(A+E*X)  .886616         -11.6888        .4222885
```

TABLE 5

ULTRAGEL

Extended Calibration of Ultragel 2-500, 2-100 Å Columns Using Polyethylene and Apolane-87 in Addition to the Linear Alkanes

RT MIN	CN	
24.6	143.9	Polyethylene
27.0	87	Apolane-87
27.7	60	
28.5	50	
29.1	44	
29.9	36	
30.5	32	
31.2	28	
32.0	24	
32.4	22	
32.9	20	
33.5	18	
34.1	16	
34.8	14	
35.6	12	
39.2	6	

TABLE 6

Calculation of Third Order Fit Calibration Coefficients for an Extended Carbon Number Range Using a Waters Model 730 Data Module

GPC CALIBRATION
NUMBER OF STANDARDS: 15

RT	MOL WT	LOG MW
24.60	0.143900E6	5.158
27.70	0.600000E5	4.778
28.50	0.500000E5	4.698
29.10	0.440000E5	4.643
29.90	0.360000E5	4.556
30.50	0.320000E5	4.505
31.20	0.280000E5	4.447
32.00	0.240000E5	4.380
32.40	0.220000E5	4.342
32.90	0.200000E5	4.301
33.50	0.180000E5	4.255
34.10	0.160000E5	4.204
34.80	0.140000E5	4.146
35.60	0.120000E5	4.079
39.20	0.600000E4	3.778

CALIBRATION COEFFICIENTS

D0	0.146134990000E2
D1	-0.743032300000E0
D2	0.190661700000E-1
D3	-0.183240960000E-3

STD ERR OF ESTIMATE 0.181333E-1
CORR COEFFICIENT 0.998485E0

TABLE 7

Comparison of the Calculated Carbon Number From the Waters Model 730 Data Module 3rd Order Fit Using an Extended Carbon Number Calibration vs. the Actual Carbon Number

RT Min	ACTUAL CN	Calculated CN Via Entering RT
24.60	143.9	143.5
27.70	60	60.4
28.50	50	49.8
29.10	44	43.4
29.90	36	36.4
30.50	32	32.1
31.20	28	27.8
32.00	24	23.8
32.4	22	21.99
32.90	20	19.99
33.50	18	17.86
34.10	16	15.97
34.80	14	14.03
35.6	12	12.1
39.20	6	5.98

TABLE 8

Third Order Fit Calibration Coefficients
Used for the Calculations for Sample Shown in Figure 1

GPC CALIBRATION
NUMBER OF STANDARDS: 7

RT	MOL WT	LOG MW
28.00	0.600000E5	4.778
28.70	0.500000E5	4.698
29.30	0.440000E5	4.643
30.20	0.360000E5	4.556
34.30	0.160000E5	4.204
35.90	0.120000E5	4.079
39.40	0.600000E4	3.778

CALIBRATION COEFFICIENTS

D0	0.157661100000E2
D1	-0.859148000000E0
D2	0.217886000000E-1
D3	-0.208451000000E-3

STD ERR OF ESTIMATE 0.448956E-1
CORR COEFFICIENT 0.992795E0

TABLE 5

Correlation of Area Percent vs. Retention Time
For Fischer-Tropsch Wax Shown in Figure 1

EXTERNAL STANDARD QUANTITATION

PEAK#	AMOUNT	RT	%
1	0.00300	20.14	0.00
3	0.00300	21.10	0.00
4	0.00300	21.50	0.00
5	38.15400	22.06	0.40
6	54.79200	22.54	0.56
7	111.87000	23.02	1.19
8	175.31100	23.50	1.86
9	238.83900	23.98	2.54
10	327.77700	24.46	3.49
11	437.29500	24.94	4.66
12	503.67300	25.42	5.36
13	531.23100	25.90	5.66
14	547.63200	26.38	5.83
15	549.63000	26.86	5.85
16	530.62800	27.34	5.65
17	514.86600	27.82	5.48
18	482.39400	28.30	5.14
19	454.33500	28.78	4.84
20	418.17300	29.26	4.45
21	390.96100	29.74	4.16
22	349.63200	30.22	3.72
23	323.07000	30.70	3.44
24	306.37800	31.18	3.26
25	308.94900	31.66	3.29
26	320.33700	32.14	3.41
27	348.10500	32.62	3.71
28	333.51600	33.10	3.55
29	299.99400	33.58	3.19
30	214.66500	34.06	2.28
31	130.11300	34.54	1.38
32	74.36400	35.02	0.79
33	35.86200	35.50	0.38
34	28.53300	35.98	0.30
TOTAL	9381.10000		100.00

TABLE 10

Correlation of Carbon Numbers Obtained from
The Waters Model 730 Data Module
Third Order Fit vs. Retention Time
For the Fischer-Tropsch Wax Shown in Figure 1

GPC QUANTITATION

RT	AREA	CN X 10^3	A/CN X10^3	
20.14	1	0.100160E7	0.998402E-6	L
21.10	1	0.634402E6	0.157688E-5	L
21.58	1	0.512037E6	0.195298E-5	L
22.06	12718	0.416869E6	0.305083E-1	L
22.54	18264	0.342235E6	0.533668E-1	L
23.02	37290	0.283289E6	0.131632E0	L
23.50	58437	0.236387E6	0.247209E0	L
23.98	79613	0.198679E6	0.400711E0	L
24.46	109259	0.168214E6	0.649523E0	L
24.94	145765	0.143430E6	0.101627E1	L
25.42	167891	0.123116E6	0.136368E1	L
25.90	177077	0.106302E6	0.166579E1	L
26.38	182544	0.923580E5	0.197640E1	L
26.86	183210	0.806731E5	0.227101E1	L
27.34	176876	0.708556E5	0.249626E1	L
27.82	171622	0.625261E5	0.274486E1	L
28.30	160798	0.554516E5	0.289979E1	L
28.78	151445	0.493729E5	0.306737E1	L
29.26	139391	0.441541E5	0.315692E1	L
29.74	130327	0.396282E5	0.328874E1	L
30.22	116544	0.356829E5	0.326610E1	L
30.70	107690	0.322399E5	0.334027E1	L
31.18	102126	0.292089E5	0.349735E1	L
31.66	102983	0.265200E5	0.388322E1	L
32.14	106779	0.241258E5	0.442592E1	L
32.62	116035	0.219874E5	0.527734E1	L
33.10	111172	0.200765E5	0.553741E1	L
33.58	99998	0.183511E5	0.544915E1	L
34.06	71555	0.167793E5	0.426448E1	L
34.54	43371	0.153560E5	0.282436E1	L
35.02	24788	0.140561E5	0.176350E1	L
35.50	11954	0.128676E5	0.928999E0	L
35.98	9511	0.117752E5	0.807714E0	L

NO-AVG 0.429977E5
WT-AVG 0.761628E5
Z-AVG 0.128865E6
VIS-AVG 0.761493E5

DISPERSITY 0.177132E1
INTRINSIC VISCOSITY 0.761341E-1

TABLE 11

Comparison of the Weight to Differential Refractoner Response for Carbon Number Range 12 to 60

5124-203
RI Weight to Area Response for Alkanes

C_N	PIC_{og}/A
60	39.8
50	38.8
44	37.7
36	34.9
32	34.2
28	31.5
24	30.4
22	29.4
20	27.3
18	26.7
16	25.4
14	23.8
12	21.9

TABLE 12

Conversion of Area Percent to Weight Percent
Using a Hewlett Packard Model 3357 Laboratory Automation System

Conversion of Area Percent to Weight Percent for Pit 700 R15 Wax From Catalyst Outlet 5226-42-3

CN	A%	Wt.%
416.9	.4	.5
342.2	.58	.7
283.3	1.19	1.5
236.4	1.86	2.3
198.6	2.54	3.1
168.2	3.49	4.2
143.4	4.66	5.6
123.1	5.36	6.3
106.3	5.66	6.5
92.4	5.83	6.6
80.7	5.85	6.5
70.9	5.65	6.1
62.5	5.48	5.8
55.5	5.14	5.3
49.4	4.84	4.9
44.2	4.45	4.3
39.6	4.16	3.9
35.7	3.72	3.4
32.2	3.44	3.1
29.2	3.26	2.8
26.5	3.29	2.7
24.1	3.41	2.7
22.0	3.71	2.9
20.1	3.55	2.6
18.4	3.19	2.3
16.8	2.28	1.6
15.4	1.38	.9
14.1	.79	.5
12.9	.38	.2
11.8	.3	.2

FIGURE 1

Size Separation of a Fischer-Tropsch Wax

COLUMNS: ASI Ultragel 2-500, 2-100A, 30cm x 7.8 mm
SOLVENT: Orthodichloro benzene, ODCB
TEMPERATURE: 100°C
DETECTOR: Diff. Refractometer
FLOW RATE: 4.8/5.0 mL/min
CHART SPEED: 1 cm/min

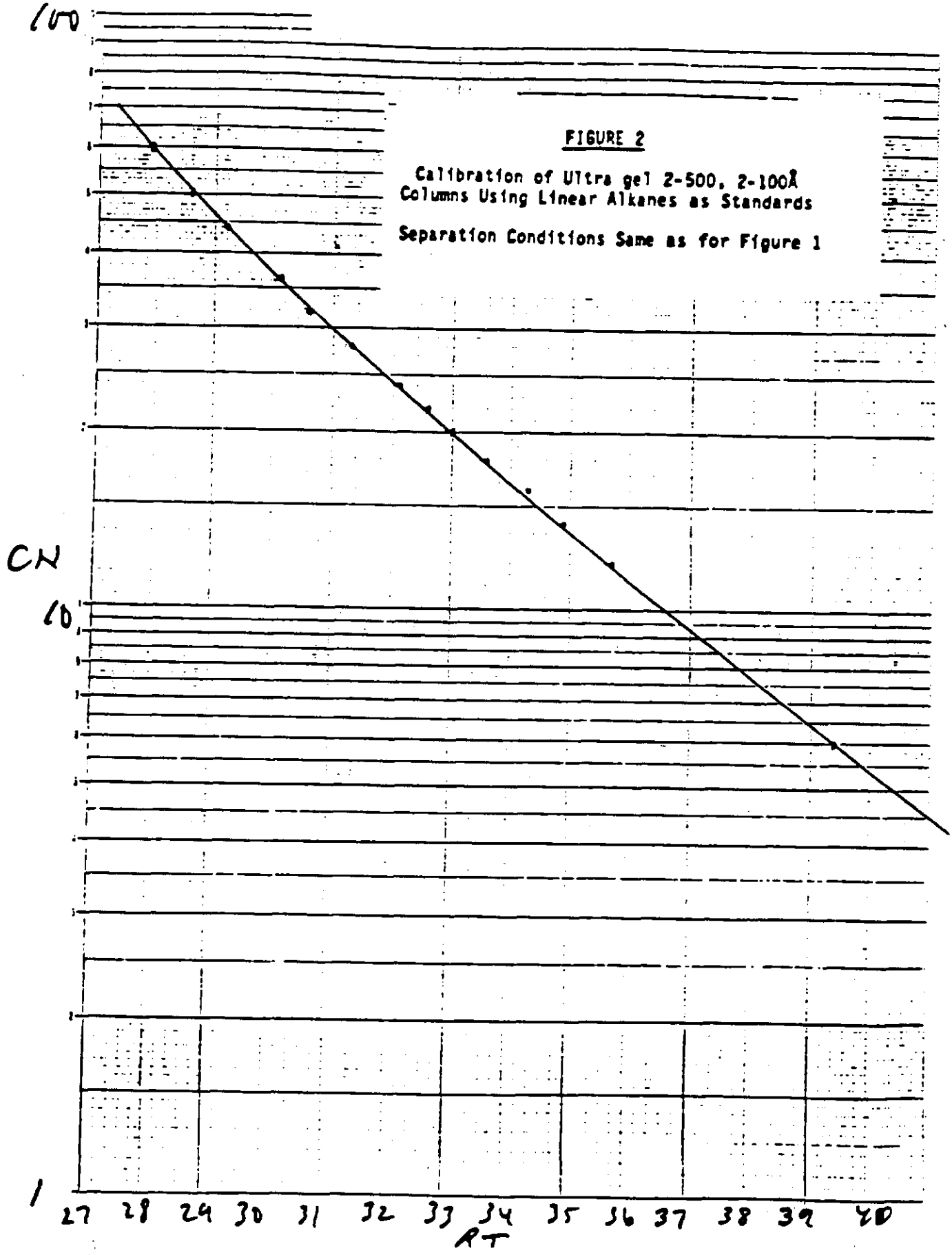


FIGURE 2

Calibration of Ultra gel 2-500, 2-100Å Columns Using Linear Alkanes as Standards

Separation Conditions Same as for Figure 1

FIGURE 3

Least Squares Curve Fit Plot of Carbon Number vs. Retention Time
Using the Hewlett Packard Model 3357 Laboratory Automation System

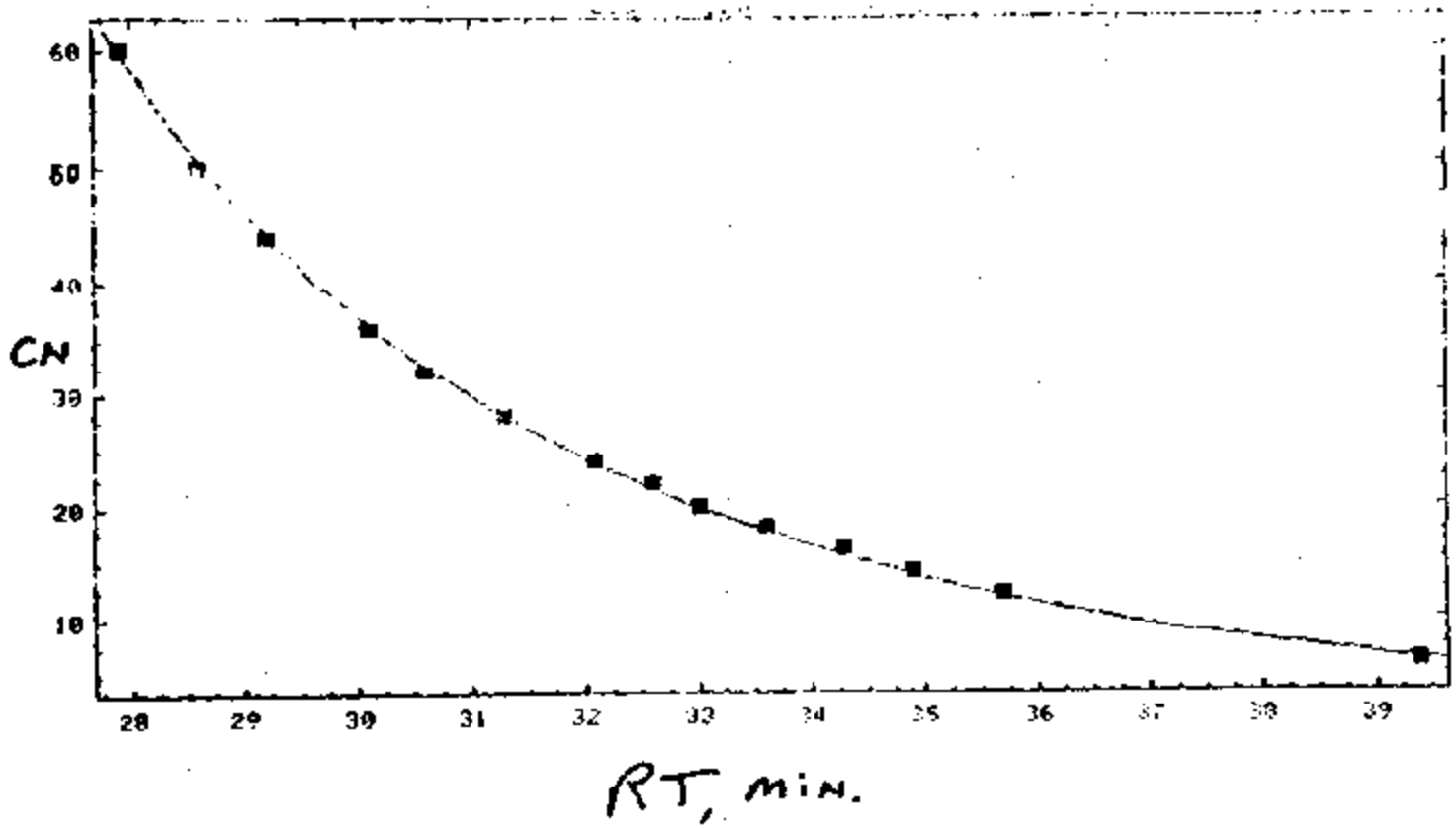


FIGURE 4

Attempt to Extend Carbon Number Calibration Range

Separation Conditions Same as in Figure 1

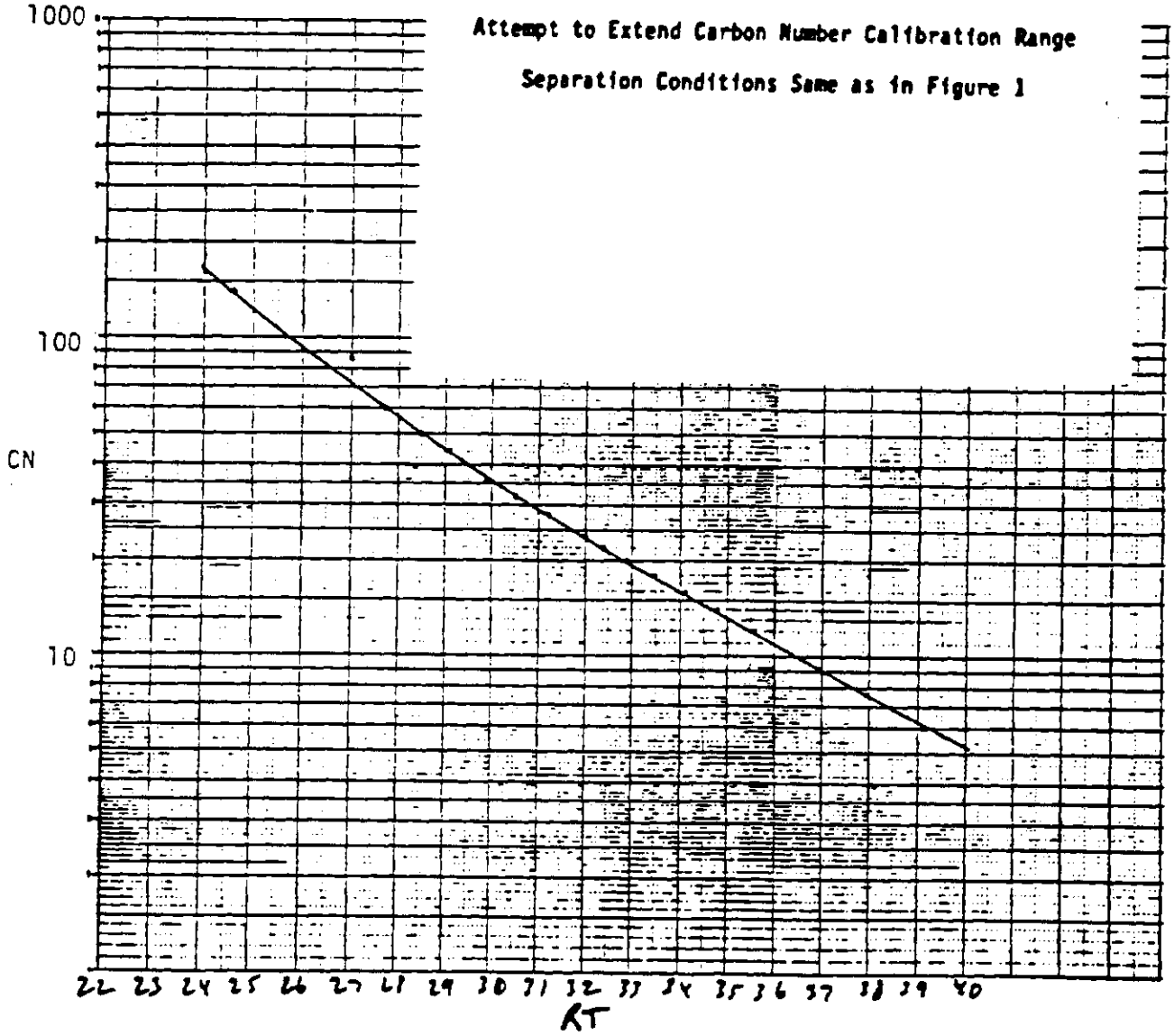


FIGURE 5

Plot of Weight to Differential Refractometer Area Ratio vs. Carbon Number