снком. 4719

CHROMATOGRAPHIC ANALYSIS OF PRIMARY LIGHT GASOLINE AND Pbi-H FRACTIONS OF ROMASHKINO CRUDE OIL

J. ŠIMEKOVÁ, N. PRONAYOVÁ, R. PIEŠ AND M. ČIHA Slovnaft, Vlčie Hrdlo, Bratislava (Czechoslovakia)

SUMMARY

Both qualitative and quantitative analyses of the hydrocarbon components of straight-run and hydrogenated pyrolysis gasolines have been made by gas-liquid chromatography. Several different open tubular (capillary or Golay) columns with squalane, Ucon LB-550, or mannit-hexakis-cyanoethyl ether as stationary phase were used for the analysis. Chromatograms of the samples analysed on squalane are given, using temperature programming. The chromatographic peaks have been identified by the use of pure standards, prepared by different chemical methods, before the sample input and by purification of some components on a preparative gas chromatograph. The purified compounds were identified by infrared spectroscopy.

INTRODUCTION

In the beginning of our chromatographic analyses of light straight-run gasoline and hydrogenated pyrolysis gasoline, we had neither capillary columns, nor a sufficient assortment of pure standard compounds. We therefore decided to proceed in the following manner.

Samples were taken from the production plant, in our case the straight-run light gasoline as feed stock and the hydrogenated gasoline as a product of the ethylene plant, and separated by distillation into fractions according to the number of carbon atoms. Some pure compounds, used as standards, were obtained from these fractions by separation on a preparative gas-liquid chromatograph; they were then identified by their IR spectra.

In the case of the straight-run gasoline, we could assume the presence of saturated and cyclic hydrocarbons. On the other hand the presence of unsaturated, *i.e.* olefinic and polyolefinic or acetylenic compounds, could be excluded. Nevertheless, we tested our samples by shaking with concentrated sulphuric acid to confirm this. Since the composition of the straight-run gasoline is relatively simple, we resolved to identify the components of this mixture first. The chromatographic analysis of the hydrogenated pyrolysis gasoline was more complicated due to the presence of a lot of olefinic and diolefinic, as well as aromatic, hydrocarbons.

EXPERIMENTAL

The instruments used for the analyses were:

CHROM II, an instrument of the National Interprise Laboratorní přístroje, Prague, equipped with a flame-ionisation detector and an open tubular column.

Model GT, Series 200 from Carlo Erba, a dual column, research class instrument, equipped with FID, temperature and gas-flow programming, both being used in the analyses described simultaneously.

TABLE I

ANALYTICAL CONDITIONS

A .	Apparatus Detector Sample size Splitting ratio Column Temperature programming Carrier gas flow programming	GT Series 200, Carlo Erba FID (flame ionisation detector) 1 μ l 1:220 capillary, stainless steel 45 m × 0.2 mm I.D. with 8% by wt. of squalane in <i>n</i> -hexane from -5° at the start up to <i>iso</i> C ₅ , then at 25° to benzene elution, then programming up to 90° at a rate of 2°/min at - 5° at the start I.4 ml/min flow rate at 1.5 kp/cm ² to <i>o</i> -xylene elution, then with an increment of 0.2 ml per min up to a flow rate of 4.26 ml/min at 2.6 kp/cm ² pressure. The column was cooled according the method of SANDERS AND MAYNARD ¹⁰ .
в.	Apparatus Detector Sample size Column Splitting ratio Carrier gas	GT Series 200, Carlo Erba FID 0.5 μ l capillary, stainless steel 45 × 0.2 mm I.D. with 10% by wt. of polypropylene glycol (Ucon LB-550 X) 1:100 1 ml N ₂ /min at 1.5 kp/cm ²

Fractovap Model P, Carlo Erba, Milan, a preparative gas chromatograph. Unicam SP-200, a routine double-beam IR spectrophotometer.

The analytical conditions used in these instruments are listed in Table I.

Some of the standards used for identification were obtained from Lachema N.I., Brno, others were obtained by purification on the Fractovap P from synthetic samples prepared in our laboratories or from distillation fractions of hydrogenated or nonhydrogenated pyrolysis gasoline and straight-run light gasoline.

Squalane and Ucon LB-550 X — as liquid stationary phases for the capillary column — were obtained as gas chromatographic pure chemicals from Carlo Erba.

Analysis and identification

First the C_1-C_6 hydrocarbons present in the straight-run light gasoline were identified on classical columns packed with a carrier and stationary phase. In further work, we continued with capillary columns with squalane as stationary phase. The conditions can be seen in Table I.

The standards available were used for plotting the correlation between elution times and boiling points, for the calculation of the Kováts indices and relative retention times. The latter were compared with literature date of TOURRES¹ and DESTY et al.². The straight-run light gasoline so analysed was then used as a mixture of known saturated hydrocarbons for further identifications in the analysis of the hydrogenated pyrolysis gasoline, containing olefinic, diolefinic, and higher aromatic hydrocarbons. The identification of such a wide range of heterogenous hydrocarbons in hydrogenated pyrolysis gasoline in such a manner was not unambiguous over the entire range. It was necessary to separate the distillation fractions further by chemical methods, removing some components from the chromatographic spectrum by chemical reaction. Several fractions of hydrogenated pyrolysis gasoline were modified by the following operations:

- (a) hydrogenation (for removing olefins, diolefins, acetylenes and styrenes);
- (b) reaction with conc. sulphuric acid (for removing unsaturated and aromatic hydrocarbons);
- (c) reaction with Ilosvay reagent (removal of acetylenes);
- (d) reaction with maleic anhydride (removal of conjugated diolefins);
- (e) adsorption on silica gel (removal of olefins and aromatics). This last mentioned method was not satisfactory with the silica gel used.

For the separations, known classical columns were used:

- (I) n-Butyric ester of triethylene glycol on Chromosorb P, 60-80 mesh (EKM).
- (2) Tricresylphosphate on Chromosorb P, 60-80 mesh (TKF).
- (3) Squalane on Chromosorb P, 60-80 mesh.

The C_1-C_4 hydrocarbons were identified on the EKM column by comparison with injected pure gaseous hydrocarbons or calibration gases of known composition from the rich gas separation and pyrolysis gas separation plants (ethylene plant).

In further work, we continued on capillary columns and gradually used further pure compounds, which had not been available at the beginning of our investigations. For the identification of the non-aromatic components, a 45 m long stainless steel open tubular column was used wetted with squalane. The operational temperature was 40°. The method of identification of the hydrogenated pyrolysis gasoline components on the capillary columns was the same as in the case of the straight-run gasoline. We could make full use here of our analytical results on packed columns and from literature data published by MATSUKUMA⁴ and HIVELY AND HINTON⁵, containing Kováts indices of olefins and paraffins.

For the identification of aromatics in the straight-run light gasoline and in the hydrogenated pyrolysis gasoline, it was necessary to use several columns with stationary phases having different polarities. Squalane was used as a basic liquid phase, separating the analysed components of the mixtures approximately according to their boiling points. We used a 45 m long capillary column at the temp. 90°. The corresponding retention times were compared with the relative retention times given in the paper by MIYAKE *et al.*⁶. Using a more polar solvent, Ucon LB-550 X, and linear temperature programming from 60 to 130° at a rate of 1°/min the elution of alkenyl-aromatic hydrocarbons was retarded relative to alkylaromatic hydrocarbons having the same or similar boiling points. The relative retention times and the Kováts indices were compared with data given by BAUMANN AND CZICSERY⁸ and McTAGGART AND MORTIMER⁹.

The elution data using Ucon LB-550 X were evaluated on the basis of the conclusions of WILLIS⁷. The di-tridecylphthalate used in this paper as stationary phase and Ucon LB-550 X have similar polarities. For confirmation of our opinion that the

hydrogenated pyrolysis gasoline under investigation contains also styrenes and indene, we used the strongly polar stationary phase mannit-hexakis-cyanoethyl ether, prepared in the Main Laboratory of Leuna-Werke, G.D.R. On this phase, *n*-decane is eluted before benzene and the elution times of styrenes and indene are appreciably retarded compared with those of the alkylaromatics.

We tried also *m*-bis-*m*-phenoxyphenoxybenzene as a stationary phase, suggested originally by WALKER AND AHLBERG¹¹ and recently used in several papers. Our results were good, but different from the results published.

In conclusion, we applied the chromatographic method of SANDERS AND MAYNARD¹⁰ to our problems for the determination of C_3-C_{12} hydrocarbons in full-range motor gasoline.

We analysed the straight-run light gasoline and hydrogenated pyrolysis gasoline as a whole on a 45 m long stainless steel capillary column with squalane as liquid phase, with temperature programming from -5° to $+90^{\circ}$ and simultaneous carrier



Fig. 1. Chromatogram of straight-run light gasoline on a capillary column with squalane. For the operational conditions see Table I. The identification of the peaks is given in Table II.

94

J. Chromatog., 51 (1970) 91–101



Fig. 2. Chromatogram of a hydrogenated pyrolysis gasoline on a capillary column with squalane. For the operational conditions see Table I. The identification of the peaks is given in Table_III.

...

. 1

<u>9</u>6

TABLE II

IDENTIFICATION OF CHROMATOGRAPHIC PEAKS SHOWN IN FIG. I

Pee	ak number	Boiling point (°C)	% wt.
I	Isobutane	- 11.73	0.15
2	n-Butane	- 0.5	9.45
3	Isopentane	27.85	11.04
4	n-Pentane	36.07	16.42
5	2,2-Dimethylbutane	49.74	0.14
ŏ	Cyclopentane	49.26	1.04
7	2.3-Dimethylbutane	57.99	1.30
8	2-Methylpentane	60.27	5.09
9	3-Methylpentane	63.28	5.51
IÓ	n-Hexane	68.74	10.72
II	Methylcyclopentane	71.81	
	2,2-Dimethylpentane	79.20	3.00
12	Benzene	80.10	- c ⁰
	2,4-Dimethylpentane	80.50	1.20
13	Cyclohexane	80.74	1.83
14	2-Methylhexane	90.05	1.93
15	2,3-Dimethylpentane	89.78	
-	I,I-Dimethylcyclopentane	87.85	1.03
16	3-Methylhexane	91.85	3.09
17	1-cis-3-Dimethylcyclopentane	91.73	0.89
18	1-trans-3-Dimethylcyclopentane	90.77	0.81
19	1-trans-2-Dimethylcyclopentane	91.87	
	3-Ethylpentane	93.48	1.71
20	<i>n</i> -Heptane	98.43	4.99
21	1-cis-2-Dimethylcyclopentane	99.57	0.18
22	Methylcyclohexane	100.93	
	2,2-Dimethylhexane	106.84	2.47
	1,1,3-Trimethylcyclopentane	106.89	
23	2,5-Dimethylhexa n e	109.10	0.20
	Ethylcyclopentane	103.47	0.20
24	2,4-Dimethylhexane	109.43	0.74
25	2,2,3-Trimethylpentane	109.84	0.14
26	1,2,4- <i>cis</i> -Trimethylcyclopentane	109.29	0.43
27	Toluene	110.63	т 28
	3,3-Dimethylhexane	111.97	1.50
28	2,3,4-Trimethylpentane	113.47	0.51
29	2,3,3-Trimethylpentane	114.76	0.42
	1,1,2-Trimethylcyclopentane	113.73	0145
30	2-Methylheptane	117.65	1.31
31	4-Methylheptane	117.71	0.45
32	3,4-Dimethylhexane	117.73	0.13
	1-cis-2-trans-4-Irimethylcyclopentane	116.73	J
33	3-Ethylhexane	118.53	
	3-Methylheptane	118.93	0.92
	3-Methyl-3-ethylpentane	118.26	
34	1,1,3- <i>trans</i> -4-1 etramethylcyclopentane	121.6	
	2,2,5-1rimethylnexane	124.08	0.12
	1-cis-2-cis-4-1rimethylcyclopentane	118	
35	· 1-cis-3-Dimethylcyclohexane	120.09	0.73
_	I, I-Dimethylcyclonexane	119.54	75
36	I-Methyl-trans-3-ethylcyclopentane	120.8	0.19
37	2,2,4-Trimethylhexane	126.54	0.43
38	I-trans-2-Dimethylcyclohexane	123.42	0.21
	I-cis-2-cis-3-Trimethylcyclopentane	123.0	

J. Chromatog., 51 (1970) 91–101

TABLE II (continued)

Peak number		Boiling point (°C)	% wt.
39	<i>n</i> -Octane	125.67	2.32
40	1-cis-4-Dimethylcyclohexane	124.32	0.14
41	2,4,4-Trimethylhexane	130.65	0.05
42	2,3,5-Trimethylhexane	131.34	0.07
43	2,6-Dimethylheptane	135.21	
• -	1-cis-2-Dimethylcyclohexane	129.73	0.31
44	<i>n</i> -propylcyclopentane	130.95	0.09
45	Ethylcyclohexane	131.78	
• -	Ethylbenzene	136.19	0.01
46	3,3-Dimethylheptane	137.3	traces
47	I, I, 3-Methylcyclonexane	136.63	0.39
48	2,2,3-Trimethylhexane	137.68	0.07
49	p-Xylene	138.35	0.21
50	<i>m</i> -Xylene	139.1	0.42
5 I	X ₁		0.13
52	\mathbf{X}_{2}^{-}		0.13
53	X ₃		0.12
54	X ₄		0.14
55	X ₅		0.34
56	o-Xylene	144.4 I	0.29
57	X ₆		0.18
58	X ₇		0.03
59	Isopropylbenzene	152.39	0.66
	<i>n</i> -Nonane	150.80	0.00
60	<i>n</i> -Propylbenzene	159.22	0.04
61	X ₈		0.02
62	X ₉		0.06
63	X ₁₀		0.12
64	I-Metnyl-3-ethylbenzene	161.3 1	0.06
65	I-Methyl-4-ethylbenzene	161.9 9	0.09
66	N ₁₁		0.02
67	I,3,5-1 rimethylbenzene	164.72	0.06
68	X ₁₂		0.04
69	X ₁₃	-	0.03
70	1,2,4-Irimethylbenzene	169.35	0.09
71	1,2,3-Trimethylbenzene	176.08	0.09

gas flow programming. In this way, all the components were determined quantitatively.

An analysis of the straight-run light gasoline, made in the manner described, is shown in Figs. 1 and 2. In Tables II and III the separated components in the chromatograms mentioned and their amounts in the mixtures analysed are shown.

In the analysed straight-run light gasoline, 71 chromatographic peaks were found, 57 of which have been identified. The non-identified components are present in very low concentrations and they consist mainly of non-aromatic hydrocarbons boiling over 130°; the total amount of non-identified hydrocarbons is only 1.36%.

In the hydrogenated pyrolysis gasoline, 126 substances were determined and 91 identified, the amount of non-identified hydrocarbons being 3.05%. Taking into account the fact that some aromatics elute together with some aliphatic components on squalane, we repeated these analyses of hydrogenated pyrolysis and straight-run

TABLE III

IDENTIFICATION OF CHROMATOGRAPHIC PEAKS SHOWN IN FIG. 2

I Isobutane -11.7 2 Isobutylene -6.9 Butene-1 -6.2 1,3-Butadiene -4.4 3 n-Butane -0.5	73 0.76 6 0.48 11 5 0.75 58 0.45 5 0.03 72 0.19 96 0.48
2 Isobutylene	6 0.48 1 5 0.75 5 0.75 5 0.75 5 0.75 5 0.63 72 0.19 96 0.48
Butene-1	26 0.48 1 5 0.75 58 0.45 5 0.03 72 0.19 26 0.48
I, 3-Butadiene $-$ 4.43 n -Butane $-$ 0.5	1 0.75 3 0.45 5 0.63 7 0.19 6 0.48
3 n-Butane -0.5	5 0.75 58 0.45 5 0.03 72 0.19
5	88 0.45 0.03 2 0.19 06 0.48
A trans-2-Butene 0.8	5 0.03 72 0.19 06 0.48
5 Neopentane 0.5	0.19 06 0.48
6 cis-2-Butene 3.7	of 0.48
7 2-Methyl-r-butene 20.0	
8 Isopentane 27.8	5 2.23
o Pentene-1 20.0	7 0.64
To a Methyl-L-butene 31.1	б <u>т</u> т8
TT 2-Methyl-1-butche 34.0	07 0.01
$\frac{11}{2} = \frac{1}{2} = 1$	7 0.91
trans-2-Pentene 26.2	5.47
ta cis-a-Pentene 36.0	00 0.81
$\frac{15}{2} \frac{15}{2} \frac{15}{2} \frac{10}{2} \frac$	7 2.06
re r 2 Pontadiene 42 (17 5.00
To 1,3-1 entatione 420	'S Na 216
1,3- <i>Wuns</i> -Cyclopentatione 42 .	23 20.10
76 a Dimethylbutane 40.2	·4 74 001
To 2,2-Dimethylbutane 49.7	
rg a Mothul-r-pentene 541	·4 0.17
Mothul r pontone 54	0.12
4-Methyl-1-pencene 53.c	6
19 Cyclopentane 49.2	
4-Methyl-cis-2-pentene 50.3	50 0.54
2,3-Dimethyl-1-Dutene 55.	
20 2,3-Dimetryibutane 5/5	
21 2-Methylpentane 60.2	2,20 22 016
22 2-Methyl-r-pentene 00.7	8
a Ethul a hutana 646	56 T 65
Verena I 62	1.05
$\frac{1}{10000000000000000000000000000000000$	19
24 00-3-110X000 67 (1, O.I
ar a Mothyl-cis-a-pentene 67 f	7
a Mothyl-c-pentene 67	, 10 021
2-Methylevelopentene 65	.9 0.51
c6 m-Herane 68.	74
20 <i>n</i> -fickance 00.	3.78
4,4 10 10 10 10 10 10 10 10	17 37 0.07
$28 cic_2 Herene 688$	β ₄ Ο.τ2
$20 \text{ M} = 2^{-110} \text{ Achieves} = 2^{-110} Achie$	75
2-Methyl-trans-2-pentene 70.	0.49
20 Methylcyclopentane 71	r T R T
2 2-Dimethyl-r-pentene 774	I.33
j, j-Dimonitati pointono //i	77 10 21.02
2 A-Dimethylpentane 80	=======================================
A A-Dimethyl-cit-2-pentene 80	3.73
a, 4-Dimonity-003-2-pentione 60/2	** 88
2 4 Dimethyl_t_nentene 81	54 I.78
2,4-Dimethyl-1-pentene 01.0	-4 26
2. Tthul. 1. nentene	
3-Euryr-r-pencene 04.	0.41
3-methyl-1-nexene 04	

.

· .

J. Chromatog., 51 (1970) 91-101

,

GC of gasoline fractions of Romashkino crude oil

TABLE III (continued)

Pea	k number	Boiling point (°C)	% wl.
34	2-Methyl-trans-3-hexene	86	0.04
	5-Methyl-1-hexene	85.31	
35	Cyclohexane	80.74	0.61
- 1	4-Metnyl-cis-2-nexene	86.71	
30	4-Methyl-1-nexene	80.73	0.08
	4-Methyl-wans-2-nexche	82.08	0.52
37	2-Methylbergue	02.90	0.53
30	z-Methylacis-z-hexene	80.5	0.89
30	2. 3-Dimethylpentane	89.78	
39	3.4-Dimethyl- <i>cis</i> -2-pentene	87.99	0.26
40	3-Methylhexane	91.85	1.14
41	2-Methyl-1-hexene	91.95	
•	3,4-Dimethyl-trans-2-pentene	90.5	0.32
42	Heptene-r	93.64	0.26
	2-Ethyl-1-pentene	94	0.30
43	3-Ethylpentane	93.48	0.81
	3-Methyl-trans-2-hexene	94	0.01
44	2,2,4-Trimethylpentane	99.24	0.13
	trans-3-Heptene	95.67	
45	cis-3-Heptene	95.75	
	3-Methyl-cis-3-hexene	95.33	0.17
	2-Methyl-2-nexene	95.44	•
.6	3-Methyl-trans-3-nexche	93.53	
40	3,5-Diffethyl-1-nexche		0.07
47	<i>u</i> -Hentane	08.43	
47	traws-2-Heptone	97.05	2.37
48	3-Methyl-cis-2-hexene	97.95	
- -	2. 3-Dimethyl-2-pentene	97.40	0.05
	cis-2-Heptene	98.5	•
49	4,4-Dimethyl-1-hexene	107.2	0.00
	3,4,4-Trimethyl-1-pentene	104	0.09
50	Methylcyclohexane	100.93	_
	cis-2,4-Dimethyl-3-hexene	109	1.28
	2,2-Dimethylhexane	106.84	-
51	2,4-Dimethyl-2-hexene	110.6	0.06
52	2,5-Dimethylnexane	109.1	0.15
	Wans-2,4-Dimetnyl-3-nexene	107.0	-
53	2,4-Dimethylicxane	109.43	0.28
5.4	3,3-Diffethyl-z-ethyl-1-Dutene	110.3	0.25
54	Toluene	110.63	16.28
55	3.3-Dimethylhexane	111.97	0
	2,4-Dimethyl-1-hexene	111.2	0.18
56	3, 3, 4-Trimethylpentane	113.47	0.07
0	trans-4,4-Dimethyl-2-hexene	106	0.01
57	2,3,3-Trimethylpentane	114.76	0.34
58	2-Methylheptane	117.65	0.61
	2,3,4-Trimethyl-2-pentene	116.26	0.01
59	4-Methylheptane	117.71	
	trans-6-Methyl-2-heptene	118	0.19
-	2- <i>n</i> -Propyl-1-pentene	117.7	
60	3-Ethylnexane	118.53	
	3-methylneptane	110.93	0.46
	3-15thyl-3-nexcne	110	
	cis-o-metnyi-2-neptene	, 110	

99

(continued on page 100)

.

TABLE III (continued)

Peal	k number	Boiling point (°C)	% wt.
61	2,2,5-Trimethylhexane cis-4-Methyl-3-heptene	124.08 122	0.05
62	2-Ethyl-1-hexene	120	0.02
63	trans-3-Ethyl-2-hexene	121	0.51
64	Octene-I	121.28	0.07
65	2. 3-Dimethyl-2-hexene	122	0.07
-0	2,2,4-Trimethylhexane	126.54	0.26
66	cis-4-Octene	122.54	0.06
67	trans-2-Octene	125	0.15
00	(<i>n</i> -Octane) cis-2-Octene	125.07	1.21
69	X,	123.04	0.22
70	\mathbf{X}_{2}^{1}		0.02
71	\mathbf{X}_{3}		0.05
72	X ₄		0.03
73	X ₅ X		0.02
74	2 6-Dimethylheptene	T 2 5 2 T	0.01
75	<i>n</i> -Propylcyclopentane	130.05	0.05
77	Ethylbenzene	136.19	0.69
	Ethylcyclohexane	131.78	0.65
78	X ₇		0.32
79	A ₈ A Vulono	T 28 25	0.25
81	<i>m</i> -Xylene	130.35	0.80
82	X	139.10	0.06
83	Styrene	145.14	1.11
84	X ₁₀		0.25
85	o-Xylene	I44.4I	0.88
80	\mathbf{X}_{11}		0.05
88	X12 X12		0.02
89	X_{14}^{13}		0.02
90	X ₁₅		0.04
91	Isopropylbenzene	152.39	0.10
~~	(n-Nonane)	150.80	0.05
92	X ₁₀ X ₁₀		0.05
93	X ₁₂		traces
95	X ₁₉		0.02
9 6	<i>n</i> -Propylbenzene	159.22	0.08
97	X ₂₀		0.04
90	-A21 I-Methyl-2-ethylbenzene	161.21	0.03
100	I-Methyl-4-ethylbenzene	161.99	0.22
101	α-Methylstyrene	165.5	0.04
102	I-Methyl-2-ethylbenzene	165.15	0.07
103	1,3,5 Trimethylbenzene	164.71	0.08
104	<i>tert.</i> -Butylbenzene	109.12	0.02
105	<i>p</i> -Methylstyrene	171.0	traces
107	1,2,4-Trimethylbenzene	169.35	0.14
108	X ₂₂		0.67
109	X_{23}		traces

.

TABLE III (continued)

Peak number		Boiling point (°C)	% wl.
110	1,2,3-Trimethylbenzene 1-Methyl-4-isopropylbenzene	176.08 177.10	0.01
111	1-Methyl-2-isopropylbenzene Indane Indene	178.15 177.82 182.44	0.04
112	Xa		0.01
113	X 05		0.07
114	X 20		traces
115	X		traces
тıб	X [*] _{ap}		0.22
117	X		0.09
118	X ₃₀		0.27
119	Xan		0.01
120	X ₃₂		0.01
121	X _{aa}		0.02
122	Xat		0.04
123	\mathbf{X}_{35}^{**}		0.03
124	$\mathbf{X}_{\mathbf{ab}}$		0.08
125	X ₉₇		0,01
126	X ₃₈		0.01

gasolines on Ucon LB-550 X for quantitative evaluation; under these conditions these components were separated.

REFERENCES

- I D. A. TOURRES, J. Chromatog., 30 (1967) 357. 2 D. H. DESTY, A. GOLDUP AND W. J. SWANTON, Nature, 183 (1959) 107.
- 3 ASTM Designation D 2268-64 T, Issued 1964. 4 T. MATSUKUMA, Intern. Symp. Gas Chromatog. and its Exploitation, 1968, Institute of Petroleum, 1968.
- 5 R. A. HIVELY AND R. E. HINTON, J. Gas Chromatog., 6 (1968) 203. 6 H. MIYAKE, M. MITOOKA AND T. MATSUMOTO, Bull. Chem. Soc. Japan, 38 (1965) 1062.
- D. E. WILLIS, Anal. Chem., 39 (1967) 1624. 7
- 8 F. BAUMANN AND S. M. CSICSERY, J. Chromatog., 26 (1967) 262. 9 N. G. MCTAGGART AND J. V. MORTIMER, J. Inst. Petrol., 50 (1964) 257.
- 10 W. N. SANDERS AND J. B. MAYNARD, Anal. Chem., 40 (1968) 522.
- 11 J. Q. WALKER AND D. L. AHLBERG, Anal. Chem., 35 (1963) 2022.

J. Chromatog., 51 (1970) 91-101