CHARACTERIZATION OF CRUDE OILS AND PETROLEUM PRODUCTS: (I) ELUTION LIQUID CHROMATOGRAPHIC SEPARATION AND GAS CHROMATOGRAPHIC ANALYSIS OF CRUDE OILS AND PETROLEUM PRODUCTS

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ABSTRACT. Some physical and chemical properties of samples of light, medium and heavy Nigerian crude oils and petroleum products including gasoline, kerosene and engine oil have been measured and are reported in this paper. The crude oils and petroleum products have also been characterized by fractional distillation and elution liquid chromatography. The fractions obtained from elution liquid chromatography were analyzed using gas chromatography (GC). The GC fractions were identified by comparing the retention time of peaks in the unknown samples with those of components of calibration standard mixtures. The importance of the physico-chemical properties and the significance of the fractional distillation and chromatographic separation methods to industrial process operations have been discussed.

KEY WORDS: Crude oils, Petroleum products, Gasoline, Kerosene, Engine oil, Elution liquid chromatography, Gas chromatography

INTRODUCTION

Crude oil or petroleum is a naturally occurring mixture, consisting predominantly of hydrocarbons with other elements such as sulphur, nitrogen, oxygen, etc. appearing in the form of organic compounds which in some cases form complexes with metals [1]. Elemental analysis of crude oil shows that it contains mainly carbon and hydrogen in the approximate ratio of six to one by weight [2]. The mixture of hydrocarbons is highly complex, and the complexity increases with boiling range. The instrumental techniques of chromatography, ultraviolet and infrared spectroscopy together with mass spectrometry facilitate knowledge of the detailed hydrocarbon type composition of crude oil [2-4].

Over the years the chemical utilisation of crude oil and its refined products has been on the increase globally. As a result concerted efforts are being made to understand its composition, structure and properties. This understanding will result in improved process conditions for improved yield and quality of products. The net effect is further enhancement of the utilisation of petroleum and its products [5]. Thus studies on crude oil or petroleum and petroleum products have utilised several analytical techniques after fractionalization to aromatics and nonaromatics [5-11]. Mair and Rossini [6] carried out the isolation of hydrocarbons in petroleum by distillation, crystallization, and solvent extraction, as well as the analysis of the fractions. Karr and co-workers [7] used the technique of elution chromatography for the fractionation of virgin crude oils while Crowley et al. [8] reported hydrocarbon class separation schemes for petroleum using liquid chromatography and glass capillary chromatography.

Some literature reports [9-13] have utilised different chromatographic techniques for separation of crude oil, crude oil fractions or petroleum products into acid, base, neutral, saturates and aromatic fractions. In other reports, further fractionation of the aromatics into

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monoaromatics, diaromatics and polyaromatics were carried out [9, 10]. The fractions were characterized using ultraviolet and infrared spectrophotometry [3, 5], X-ray flourescence spectrometry [3], as well as proton and carbon-13 nuclear magnetic resonance spectrometry [4, 14-15]. The metal contents of crude oils and petroleum products have also been determined using methods such as UV-visible spectrophotometry [16-19] and atomic absorption spectroscopy (AAS) methods [19, 20]. The non-metal constituents of petroleum and petroleum products, and especially sulphur, nitrogen and their derivatives have also been studied [21-23]. These non-metals as well as the metals constitute the impurities in petroleum.

In this paper, we present the results of physical and chemical characterization of Nigerian crude oils and petroleum products. We also report the elution liquid chromatography of the samples followed by the analysis of the fractions on a gas chromatograph equipped with a flame ionization detector. The crude oil samples analysed include Lagoma crude oil from Venezuella (heavy base crude) supplied by NNPC Kaduna, Nigeria, Bonny terminal (medium crude oil) obtained from the NNPC Portharcourt, Nigeria, and the Qua Iboe crude oil (light crude) supplied by Mobil Producing Nigeria Unlimited. The petroleum products analysed including gasoline (PMS), engine oil (SAE 40), and lube oil were obtained from NNPC Warri Refinery and Shell Company and kerosene is the commercial product obtained in the market.

EXPERIMENTAL

Materials and chemical reagents. All the chemical reagents used in this study were of analytical reagent grade and the glassware were washed with soap solution, rinsed with distilled and deionised water, and then kept in the oven to dry. Elution liquid chromatography was carried out using standard laboratory burrette (50 cm^3). It was cleaned with a mixture of chromic acid and sulphuric acid. This was followed by rinsing it with distilled water and acetone and then it was allowed to dry in air [24, 25].

Physicochemical properties. The following physical and chemical properties of samples of crude oils were determined following well established procedures [26]: specific gravity, API gravity, kinematic viscosity, pour point, water content, total acid content and sulphur content. For the petroleum products the tests carried out also include flash point for kerosene, aniline point, diesel index and lead content [25-28].

Fractional distillation. The fractional distillation of each sample of the six different crude oils was carried out in the Hempel distillation apparatus. This consists of the distillation flask, column packing, column lump, thermometers, graduated cylinder, cooling bath and heat source. After carefully introducing about 300 cm^3 of the crude oil into the distillation flask and making all necessary connections, heat was applied to distill the oil at a rate of about 2.5 cm^3 per min until it ceased to foam. Thereafter the rate of distillation was kept constant at 2-2.5 cm^3 per min (approximately one drop per second). The volume of distillate fraction collected at each 25 °C increase in temperature was recorded until 300 °C was reached. Then the heating of the flask was stopped to allow the condenser to drain into the receiver and the volume of distillate collected was recorded [26-28]. The same procedure was followed in an apparatus similar to that used above for the distillation of gasoline, kerosene and diesel oil and under prescribed conditions which are appropriate for each product. Volumes of condensate and corresponding boiling temperatures were recorded [26-28].
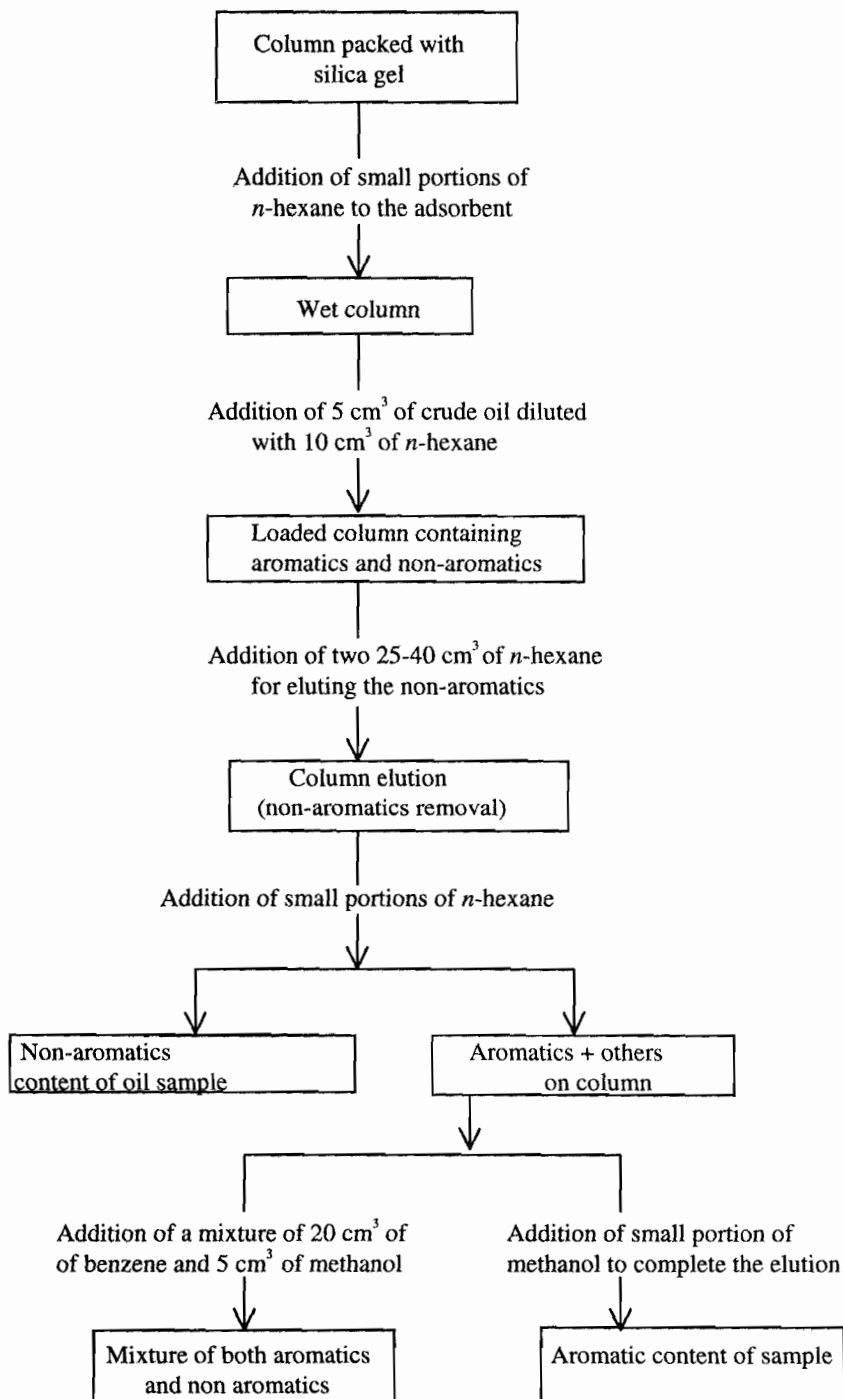
Figure 1. A schematic diagram of the separation procedure by column chromatography.

Elution liquid chromatography. Elution liquid chromatography for the fractionation of crude oils and petroleum products into aromatics and nonaromatics was carried out in a standard laboratory burrette. After cleaning and drying, the column was carefully packed with silica gel and moistoned with some quantity (30 cm^3) of *n*-hexane. The oil sample (5 cm^3) dissolved in 10 cm^3 of *n*-hexane was then introduced into the column. The nonaromatic component was obtained by adding *n*-hexane in small portions to the column to flush it. Thereafter a mixture of benzene and methanol in the ratio of 2:1 was added to the column to remove a mixture of aromatics and non-aromatics [13, 25]. The elution of the aromatic component was continued and completed by using only methanol to flush the column. Figure 1 gives the separation scheme used for the elution liquid chromatography. It is a modification of other schemes reported in the literature [3, 13, 29]. The three fractions obtained (namely nonaromatics, mixture of aromatics and nonaromatics, and aromatics only) were stripped off the eluting solvents by atmospheric distillation [1, 24-25]. These fractions were then analysed using gas chromatography.

Gas chromatography. The Philip's Pye-Unicam PU 4500 Gas Chromatograph equipped with a flame ionization detector was used for the analysis. The column was packed with polyethylene glycol (EG 200) on chromosorb P(80 – 100 mesh) solid support. The GC conditions include initial and final column temperatures of 65 °C and 245 °C, respectively. The detector temperature was 260 °C and nitrogen gas was used as the carrier gas.

In a typical analysis 1 μL of the sample was injected into the injection port of the GC using a microsyringe. The sample was immediately vapourised and swept down the column by the carrier gas. Following separation in the column, the components were identified and quantitatively analysed by the flame ionization detector whose output was transmitted to a recorder which produced a chromatogram. The output of the recorder interfaced with a computer with a suitable chromatographic program also includes retention time, peak area, peak height, area percent and height percent [30-33].

Three calibration standard mixtures were prepared for easy identification of the components of the fractions. The first calibration standard mixture contained 0.1 mole per dm^3 of *n*-hexane, *n*-heptane, cyclohexane, *n*-octadecane (C$_{18}$H$_{38}$), *n*-nanodecane (C$_{19}$H$_{40}$) in ethyl benzene (C$_8$H$_{10}$). The second calibration standard mixture contained 0.1 mole per dm^3 of *o*-xylene, *m*-xylene, *p*-xylene (C$_8$H$_{10}$), *p*-cymene (C$_{10}$H$_{14}$), toluene (C$_7$H$_8$) and benzene dissolved in ethyl benzene. The third calibration standard mixture contained 0.1 mole per dm^3 of all the compounds contained in the first two calibration standard mixtures.

RESULTS AND DISCUSSION

Physicochemical properties. The physicochemical properties of the crude oils are summarised in Table 1, while Table 2 contains similar data for the petroleum products. The data in Table 1 show that the API gravity of light crudes is above 30° while that of heavy crude is less than 30°. These API gravity values agree with published data [13]. Furthermore, as expected, the pour point of light crude oil is higher than that of heavy crude oil. Forcados blend crude oil, which is a mixture of light, medium and heavy crude oils, has physical parameter values which range between those for light and heavy crude oils. The data in Table 2 show that SAE 40 and lube oil have different physicochemical properties despite the fact that both are engine oils. This is due to the fact that the lube oil was a freshly obtained distillation fraction from the Gulf crude oil whereas the SAE 40 (obtained from the Forcados blend) has been treated to meet certain

characteristics. Thus the final properties of the finished products depend on the properties of the source crude oil as well as the process conditions and final treatments. The data for the diesel oil and kerosene (Table 2) confirm this point.

Table 1. Physico-chemical properties of crude oils.

Parameters	Forcados blend	Bonny light	Bonny medium	Gulf crude	Lagomar crude	Qua lboe
Specific gravity @ 60/60 °F	0.8808	0.8576	0.8845	0.8690	0.8960	0.8706
API gravity @ 60/60 °F	39.1	33.4	29.3	31.4	26.4	30.9
Kinematic viscousity (cst) @ 100 °F	7.00	7.16	7.01	5.69	4.49	9.86
Sulphur content (w/w %)	0.11	0.06	0.16	0.09	1.36	0.04
Pour point (°F)	+25	+85	+5	+35	-5	+35
Total acid content (mg KOH/g)	0.33	-	-	-	-	-
Water content (v/v %)	0.08	-	-	0.05	-	-

Table 2. Physico-chemical properties of petroleum products.

Test	SAE R40 (Shell Micella)	Lube oil fresh	Kerosene	Diesel oil (AGO)	Gasoline (PMS)
Specific gravity @ 60/60 °F	0.9531	0.8711	0.8195	0.8504	0.7501
API gravity @ 60/60 °F	16.96	30.9	41.17	32.95	57.1
Pour point (°C)	-	-18	-	+3	-
Flash point (°C)	180	198	35.8	89.8	-
Smoke point (mm)	-	-	24	-	-
Aniline point (°C)	-	100	-	54.7	-
Kinematic viscosity @100 °C (cst)	12.25	5.10	-	3.14	-
Diesel index	-	30.9	-	51.4	-
Total acid content (mg KOH/g)	0.50	0.099	0.008	0.008	0.03
Water content (v/v %)	0.09	0.01	Trace	Trace	<0.05
Colour	Dark brown	Clear yellow	Colourless	Dark	Golden yellow
Lead content (g/L)	-	-	-	-	0.13
Copper corrosion	-	-	-	-	16
Sulphur content (w/w %)	-	-	0.005	0.11	-

Fractional distillation. The experimental data from fractional distillation of various crude oils are presented in Table 3, and similar data for petroleum products are in Table 4. The data in Table 3 show clearly that light crudes yielded a greater proportion of light fractions (naphtha)

than heavy crudes during distillation. Futhermore, in general, the heavier the crude oil the higher the kerosene, gas oil and other higher-boiling fractions obtainable from it. The data in Table 3 are expressed graphically in Figure 2 which gives a plot of the true boiling points (TBP) of the distillate fractions versus the percentage cummulative volume for the various crudes. Figure 3 presents the plots of the volume fractions as a function of the true boiling points. The distribution of products of atmospheric distillation of crude oil in the Hempel distillation apparatus is similar to what is obtained in industrial operations [1, 6, 34].

Table 3. Products of distillation of crude oils using Hempel distillation apparatus.

Crude oil type	True boiling point (TBP) (°C)	Bonny light	Bonny medium	Gulf crude	Lagomar crude	Qua Iboe	Forcados blend
Light naphtha	90	8.4	6.2	8.8	5.6	12.9	6.5
Heavy naphtha	170	22.0	12.6	17.2	10.8	17.5	15.5
Kerosene	240	14.4	17.7	16.6	19.2	15.0	15.5
Light gas oil	340	24.2	25.8	24.6	27.9	23.8	26.2
Heavy gas oil	425	13.7	22.4	15.0	25.3	11.3	21.6
Residue	425+	16.7	14.8	17.1	10.8	18.8	13.8
	Total	99.4	99.5	99.3	99.6	99.3	99.1
	Loss	0.6	0.5	0.7	0.4	0.7	0.9

Table 4. Distillation profiles of different petroleum products.

	Gasoline (PMS)	Kerosene	Diesel oil (D60)
IBP (°C)	45	153	228
BP at 5% recovery (°C)	56	-	248
BP at 10% recovery (°C)	68	176	260
BP at 20% recovery (°C)	-	185	-
BP at 30% recovery (°C)	85	198	274
BP at 70% recovery (°C)	124	210	284
BP at 90% recovery (°C)	158	230	298
BP at 95% recovery (°C)	173	238	324
EBP (°C)	194	245	338

In a petroleum refinery the physical and chemical operations are integrated, and both atmospheric and vacuum distillation processes are involved. Thus the products are expected to be more in number and to show a wider range than obtained in our study [6]. However, our present results show that the product distribution of atmospheric fractional distillation of crude oils is a function of the nature of the crude oil [34]. The distillation profiles (Table 4 and Figure 4) of the petroleum products investigated also show the same trend. The boiling point of the distillate fractions increases as the volume percent of the fraction increases. Moreover, as expected, diesel oil (AGO) has a higher boiling point than kerosene which in turn has a higher boiling point than gasoline (PMS) for all the fractions collected.
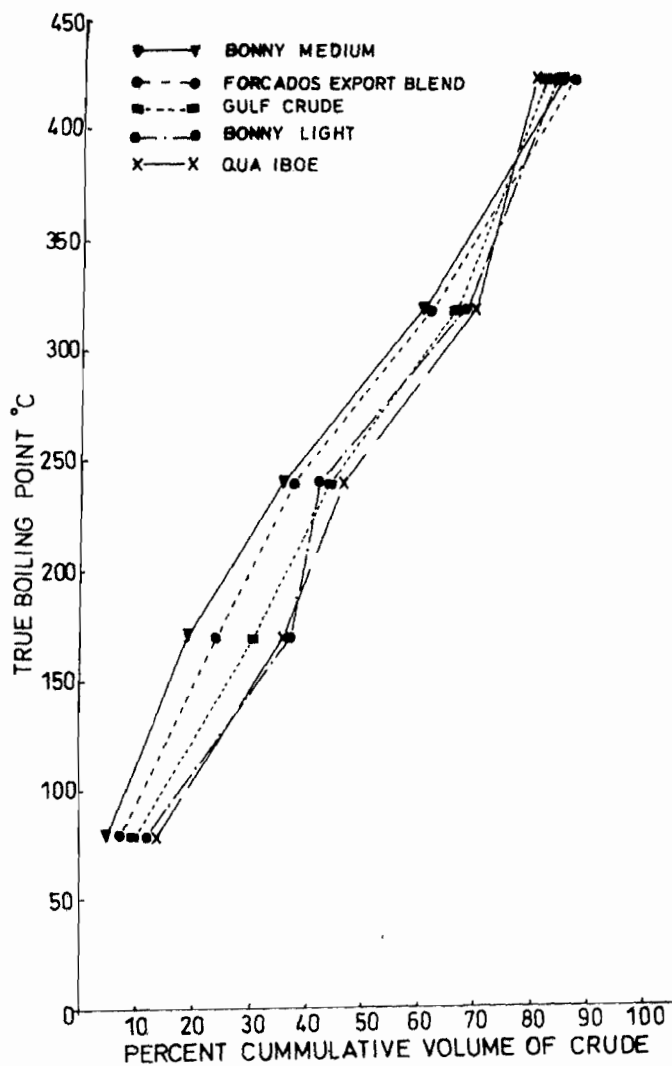
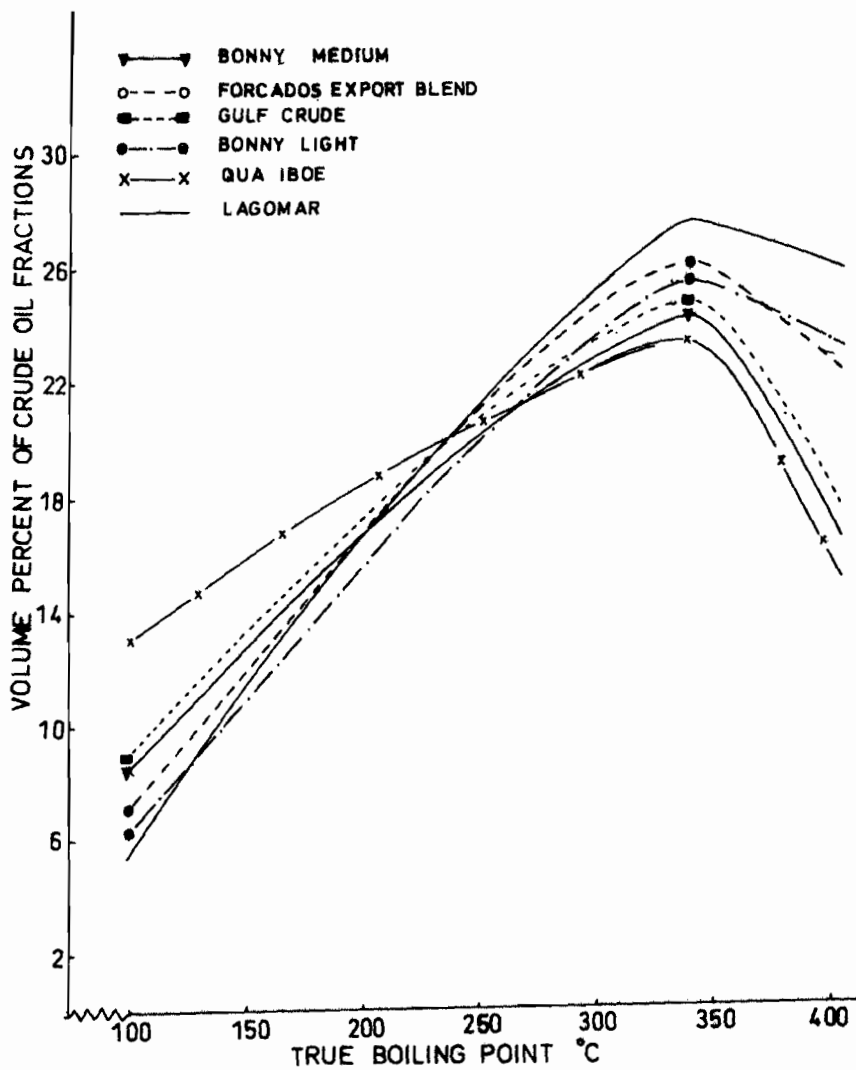Figure 2. True boiling point versus percentage cummulative volume of fractions.

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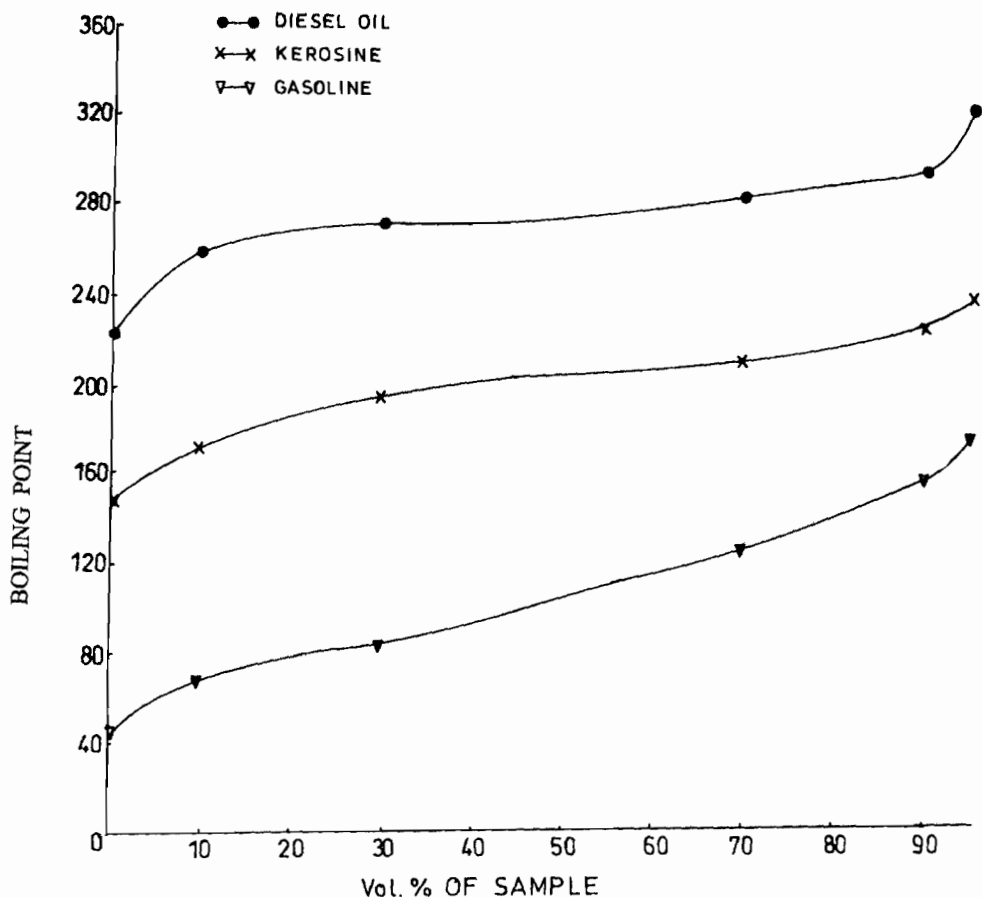Figure 3. Volume percent of crude oil fractions versus true boiling point.

Figure 4. Distillation profiles of petroleum products.

Fractions of elution liquid chromatography. The colours of the three fractions obtained from elution liquid chromatography of light, medium and heavy crude oils as well as those from gasoline, kerosene and engine oil are presented in Table 5. In general for each crude oil the colours of the fractions deepen on proceeding from nonaromatics to aromatics. Thus the colour of fraction III (aromatics) is black for medium and heavy crude oils but yellowish-brown for the light crude oil. Furthermore the colour of each fraction deepens on proceeding from light to heavy crude oils. For example, for fraction I (nonaromatics) the colour is golden yellow, yellow and dark yellow for light, medium and heavy crude oils, respectively. Similar general trends are observed for the aromatic and nonaromatic fractions of the petroleum products studied. For example, for gasoline the observed colours are pale yellow, golden yellow and yellowish-brown for fractions I, II and III, respectively. Thus colour can be used to characterize the fractions collected during elution liquid chromatography of crude oils and petroleum products. The colour variation also indicates that the elution liquid chromatography has been successful in fractionating the samples into aromatics and nonaromatics.

Table 5. Colours of fractions of elution column chromatography of crude oil and petroleum products.

Sample	Fraction I non-aromatics	Fraction II mixture of aromatics and non-aromatics	Fraction III aromatics
Light crude oil	Golden yellow	Brown	Yellowish brown
Medium crude oil	Yellow	Deep brown	Black tar
Heavy crude oil	Dark yellow	Dark brown	Black tar
Gasoline	Pale yellow	Golden yellow	Yellowish brown
Kerosene	Colourless	Light yellow	Deep yellow
Engine oil	Deep yellow	Golden yellow	Yellow

Characterization by gas chromatography. The GC analysis results are summarised in Tables 6-9 and the chromatograms are given as Figures 5-7 for heavy, medium and light crude oils. The data show that the elution column chromatograghy successfully fractionated the samples into aromatics and nonaromatics. The chromatograms also show that the components of both the aromatic and nonaromatic fractions were well resolved in the GC columns. The only exception is the aromatic fraction of heavy crude oil (Figure 5B) which was not well resolved. The retention times of the peaks of the calibration standard mixtures were used to identify the fractions obtained from elution liquid chromatography.

Table 6. Retention time and peak areas of peaks in non-aromatic fraction of heavy crude oil.

t_R (min)	Area	Area percent	t_R (min)	Area	Area percent
0.945	216991472	15.1	15.05	26557424	1.8
1.394	136687632	9.5	15.45	14957188	1.0
1.624	64222748	4.5	16.04	34133288	2.4
2.202	6912551	0.5	16.73	8927533	0.6
2.86	1308943	0.1	17.02	24440028	1.7
3.82	5590017	0.3	17.60	9374823	0.6
4.54	2362057	0.2	17.93	19004178	1.3
4.99	757769	0.1	18.23	4018367	0.3
5.50	4583836	0.3	18.42	10544190	0.7
6.20	11504722	0.8	18.81	22918732	1.6
7.11	6441312	0.4	19.66	40441540	2.8
7.74	15994494	1.1	20.47	31917378	2.2
8.66	10214665	0.7	21.25	24969050	1.7
9.20	22330326	1.6	22.01	36326164	2.5
9.94	20940022	1.5	22.71	56673232	3.9
10.52	26294590	1.8	23.55	41934508	2.9
11.28	19064220	1.3	24.49	45430132	3.2
11.74	24075888	1.7	25.61	48769160	3.4
12.33	19216734	1.3	26.97	57436310	3.9
12.91	22609074	1.6	28.63	61682328	4.3
13.50	15366933	1.1	30.72	35757064	2.5
14.01	19238052	1.3	31.61	26011484	1.8
14.39	17733510	1.2	32.88	66103332	4.6

t_R = retention time. Total area = 1438769030.

Table 7. Retention time and peak areas of peaks in nonaromatic fraction of medium crude oil.

t_R (min)	Area	Area percent	t_R (min)	Area	Area percent
0.918	210860896	30.9	11.82	186851	0.0
1.330	115976744	17.0	12.06	57507	0.0
1.628	148181680	21.7	12.97	75315	0.0
2.218	41580228	6.1	21.34	344629	0.0
2.93	7582500	1.1	22.76	11457539	1.7
3.86	17251418	2.5	23.65	12710773	1.9
4.63	3609914	0.5	24.58	13828514	2.0
5.17	306622	0.0	25.74	24116958	3.5
5.58	16362336	2.4	27.08	9791500	1.4
6.25	20913482	3.1	28.74	8464872	1.2
7.16	11468483	1.7	29.80	1339282	0.1
7.70	4963319	0.7			

t_R = retention time. Total area = 681431362.

Table 8. Retention time and peak areas of peaks in non-aromatic fraction of
         light crude oil.

t_R (min)	Area	Area percent	t_R (min)	Area	Area percent
0.960	264741040	20.1	13.74	16549474	1.3
1.492	123557440	9.4	14.39	44679136	3.4
1.826	156951248	11.9	15.42	22152916	1.7
2.326	35611348	2.7	16.10	3367093	0.3
2.934	3899552	0.3	16.39	14076195	1.1
3.73	7097438	0.5	16.95	1896590	0.1
4.37	5677314	0.4	17.38	11141020	0.8
4.81	4194304	0.3	17.81	1273418	0.1
5.17	15243943	1.1	18.17	9541829	0.7
5.79	51923224	3.9	19.01	9332264	0.7
6.61	37390304	2.8	19.47	199701	0.0
7.16	29514432	2.2	19.82	8314231	0.6
8.13	21437846	1.6	20.25	441256	0.0
8.33	6695424	0.5	20.60	9543132	0.7
8.57	23655620	1.8	21.36	15084786	1.1
9.05	2936676	0.2	22.08	23575066	1.8
9.35	16942522	1.3	22.90	17948818	1.3
9.90	19107264	1.4	23.84	16909988	1.3
10.67	20399442	1.5	24.95	14834400	1.1
11.12	16197201	1.2	26.32	7672292	0.6
11.41	11934468	0.9	27.94	7322728	0.6
11.69	25995964	1.9	29.58	33711912	2.6
12.29	27334298	2.1	32.11	33741404	2.7
12.77	18712674	1.4	33.66	30638120	2.3
13.38	16707337	1.3			

t_R = retention time. Total area = 1317806092.

Table 9.   Retention time and peak areas of peaks in aromatic fraction.

(A) Heavy crude oil.

t_R (Min)	Area	Area percent
1.701	17707144	4.8
2.122	695063	0.2
3.21	351776160	95.0

t_R = retention time. Total Area = 370178367.

(B) Medium crude oil.

t_R (min)	Area	Area percent
1.760	268296096	17.8
2.197	107262592	7.1
2.686	207139456	13.7
3.460	193509984	12.8
4.306	159569200	10.6
5.458	136661456	9.1
6.334	95298952	6.3
7.062	29865334	1.9
7.565	122334808	8.1 ·
8.434	116111736	7.7
9.656	40956904	2.7
10.374	24054918	1.6
11.24	2942815	0.2

t_R = retention time. Total Area = 1504004251.

(C) Light crude oil.

t_R (min)	Area	Area percent
1.509	118269168	9.2
1.977	72695888	5.6
2.374	166657328	12.9
3.122	195083152	15.1
3.962	167711408	13.0
5.170	142558752	11.1
6.088	98480368	7.6
6.856	30968872	2.4
7.388	125546480	9.7
8.290	112704952	8.7
9.554	36160732	2.8
10.289	20981996	1.6
11.7	1308130	0.1

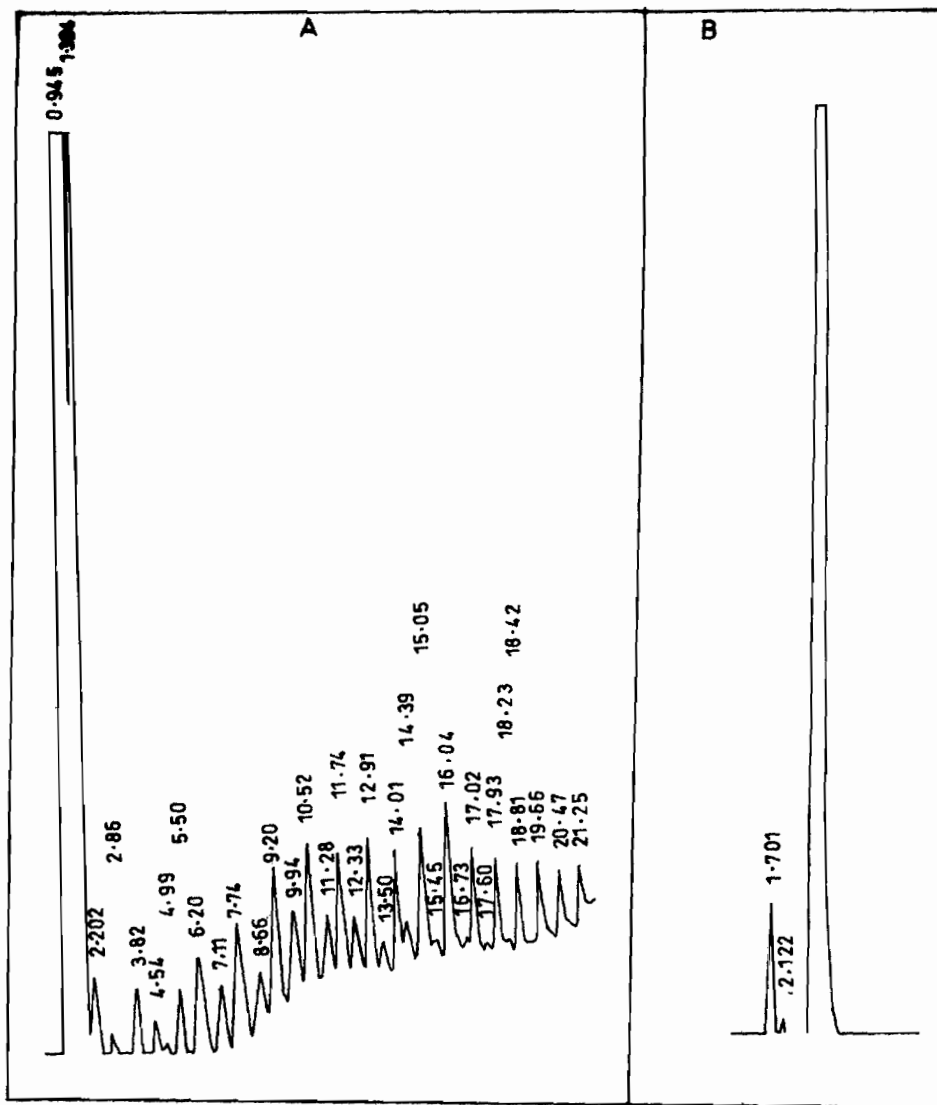t_R = retention time. Total area = 1289127226.

Figure 5. GC chromatograms of nonaromatic (A) and aromatic (B) fractions of heavy crude oil.
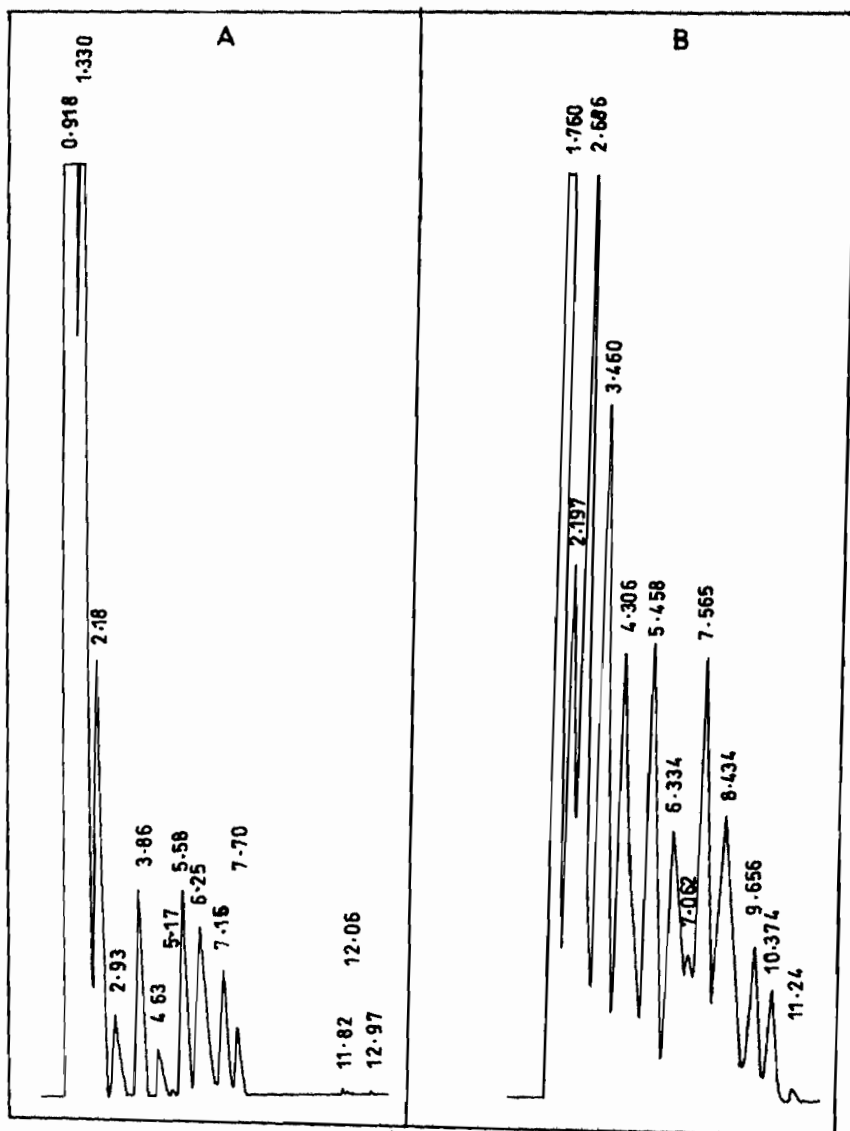
Figure 6. GC chromatograms of nonaromatic (A) and aromatic (B) fractions of medium crude oil.
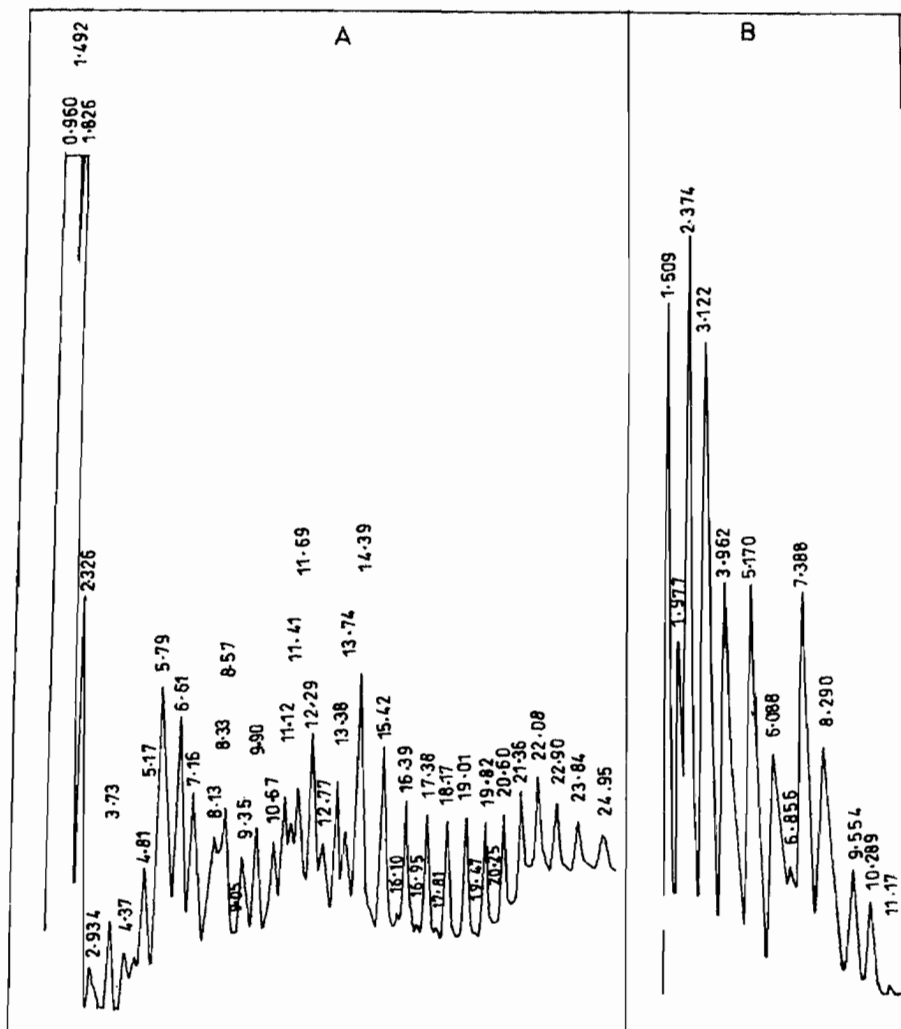
Figure 7. GC chromatograms of nonaromatic (A) and aromatic (B) fractions of light crude oil.

The data of Tables 7 and 9B and Figure 6 for medium crude oil are used to show the general trend. The aromatics were better resolved than the nonaromatics on the polar columns used. The hydrocarbon components (of the nonaromatic fraction) with low retention times were not well resolved. This suggests that a lower rate of heating the GC columns may improve resolution of the peaks. A far better resolution of the peaks could be obtained by using different columns for the GC analysis of the aromatics and nonaromatics [30-33]. All the components of our calibration standard mixtures could be found in the corresponding nonaromatic and aromatic fractions of the crude oil. This means that the nonaromatic fraction of the medium crude oil contains *n*-hexane, *n*-heptane, cyclohexane, *n*-octadecane, *n*-nonane, *n*-heneicosane and *n*-dodecane. However, there are many more peaks than these peaks. Similarly the aromatic

fraction of the medium crude oil contains benzene, toluene, *o*-xylene, *m*-xylene, *p*-xylene, *p*-cymene and ethyl benzene among others [30-33, 35, 36].

Although retention time of peaks in the GC column chromatogram serves as a means of qualitative analysis [33, 34, 39], confirmation of the individual peaks can be made by using mass spectrometry. This is partly because retention time is a function of carrier gas, column temperature, column pressure drop, the stationary phase, the void volume between the injection port and detector, etc. [37, 38]. Thus to confirm the identities of the above compounds and other peaks, GC-MS analysis would be most appropriate. In this analysis, the components of the sample will be separated in the GC into individual peaks while the mass spectrometer will produce the mass spectra of each peak which would facilitate the identification of the peak [22].

The area reported for each peak (Tables 6-9) is an appropriate measure of the concentration of each component of the mixture. For more accurate results, however, the response factors of each component should be used to determine the concentration of the component [26-34, 37].

The results obtained for the nonaromatic components compare favourably well with what is reported in some editions of the American Society for Testing and Materials (ASTM) book of standards [39, 40]. Similarly, for the aromatic components, the results compare favourably well with what was reported in the test for aromatics in light naphthas, reformates and gasoline by gas chromatography [12, 41]. These results also agree with other published data [16, 30-31].

ACKNOWLEDGEMENTS

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