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Hydrocarbon-Type Analysis of Base Oils and Other Heavy Distillates by Thin-Layer Chromatography with Flame-Ionization Detection and by the Clay–Gel Method

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Abstract

Thin-layer chromatography (TLC) using quartz rods coated with sintered silica particles and flame-ionization detection (FID) is applied for the rapid determination of saturates, aromatics, and polar compounds in petroleum heavy distillates. A procedure is described by which hydrocarbon classes are baseline separated, and quantitative results are obtained by the measurement of peak areas. As different hydrocarbon groups are fully resolved from each other and replicate analysis can be carried out rapidly, accurate determination of hydrocarbon types is possible by the TLC–FID technique. TLC–FID results are compared with those obtained by the ASTM D2007 method, the latter being based on gravimetric determination of separated components by open-column clay–gel chromatography. It is found that the ASTM method does not always yield pure fractions of each hydrocarbon type, and cross-contamination introduces considerable inaccuracies in the results. Besides superior hydrocarbon type results, TLC–FID is shown to offer several operational advantages such as short analysis time, small sample requirement, simultaneous analysis of multiple samples, less solvent consumption, and reusable sorbent.

Introduction

Hydrocarbon-type analysis of petroleum-derived lubricant base stocks and other heavy distillates is useful for monitoring the quality of these products and determining their suitability for specific use or compatibility with lubricant additives. Moreover, such analysis provides data that are important for upgrading and processing high-boiling refining intermediates and products.

A handful of methods are available for the group-type or compositional analysis of heavy distillates using both chromatographic and nonchromatographic techniques (1–4). Perhaps two of the most commonly used methods are ASTM Method D2007, which is based on clay–gel adsorption chromatography (5), and thin-layer chromatography with flame-ionization detection (TLC–FID) (6–11). The ASTM method has been in use since the late 1960s. Since its initial development in the late 1960s and early 1970s (12–14), TLC–FID has been applied extensively for the analysis of numerous samples including lipids, natural fats and oils, drugs, surfactants, lubricant additives, crude oils, heavy petroleum fractions, coal-derived liquids, asphalts, and environmental samples (6–20).

The TLC–FID technique employs reusable thin quartz rods coated with micrometer-sized adsorbent particles. A layer of an adsorbent, such as silica or alumina, is sintered to each quartz rod. A set of 10 such "chromarods" (6,7) are generally mounted in a metal rack. Usually, 1 μL of a sample solution (equivalent to a few micrograms of sample) is spotted on each rod. These rods are developed simultaneously with appropriate solvents to achieve the separation of saturates, aromatics, and polars. Then the rack is placed on a moving frame within a scanner unit where each chromarod is individually passed lengthwise through a stationary oxygen–hydrogen flame. The ions produced due to the pyrolysis of separated sample components are collected by a metal cylinder, and the current is amplified and recorded.

ASTM D2007 is based on preferential adsorption of polar compounds on clay and of aromatic compounds on silica gel. As saturated hydrocarbons (naphthenes and paraffins) have low affinity for both clay and silica, they are eluted when the columns containing both adsorbents are percolated with *n*-pentane. Polars and aromatics are then recovered from clay and silica adsorbents using appropriate solvents. The separated components are determined gravimetrically after the evaporation of solvents.

The amounts of saturates, aromatics, and polars in the sample can be determined by both the ASTM and TLC–FID methods. Thus it should be of interest to compare results obtained by these two methods for a series of base stocks and other heavy distillates in order to evaluate the usefulness of the results and to identify bias inherent to each method. To our knowledge, such a study with a wide variety of heavy distillates is not available in the literature.

Experimental

Samples and solvents

The oil samples were of different viscosity grades according to Saybolt Universal Seconds (SUS) measured by the ASTM

Method D88 (21). A number of aromatic extracts that are used in commerce as rubber extender oils were also analyzed. Heptadecylbenzene was purchased from Lancaster Synthesis (Windham, NH), and Nujol was obtained from Aldrich Chemical (Milwaukee, WI). Toluene and *n*-heptane were used as received as developing solvents (EM Science; Gibbstown, NJ).

TLC–FID procedure

A Mark V Iatroscan instrument (Iatron Laboratories; Tokyo, Japan) equipped with an FID was used. The instrument is an improved model which, according to a recent report (11), has shown better FID sensitivity and wider detector linear range as compared with earlier models (Mark III and Mark IV). The silica-coated quartz rods used were chromarod-type S-III manufactured by Iatron Laboratories (supplied by RSS Inc.; Bemis, TN). These chromarods were 15.2-cm long and 1.0-mm in diameter with a uniform coating of 5.0-µm silica particles. As previously mentioned, the TLC–FID procedure involved spotting samples on a series of 10 chromarods in a metallic rack, developing of the chromarods sequentially with toluene and heptane, and finally, scanning of each chromarod with an oxygen–hydrogen flame for detection of the separated components.

Sample solutions (typically 1% w/v) were prepared in toluene. About 1 µL of sample solution was spotted on freshly activated (prescanned) chromarods in four aliquots using a 10-µL repeating syringe (Hamilton Co.; Reno, NV). Solvent was evaporated completely between applications to minimize sample spreading. About 5 to 10 µg of sample was found to be optimum for the analysis, as determined by the signal-to-noise ratio and sample overloading.

A two-step chromarod development was found to be suitable for our work. Two developing tanks lined with filter paper, one containing toluene and the other containing *n*-heptane, were used. Each tank was allowed to saturate with the solvent vapor for about 20 min. First, the chromarods were developed with toluene for 5 min (to 4.0 cm from the origin). The second development was carried out with *n*-heptane for a period of 30 min (to 10.5 cm). The chromarods were dried in an oven at 70°C for about 2 min after each development. Usually one to four racks of chromarods were developed simultaneously.

For peak detection, a scan rate of 30 s per chromarod was used. The hydrogen and air flow rates were 165 mL/min and 2000 mL/min, respectively. A Beckman PeakPro system (Beckman Instruments; Allendale, NJ) was used to record the signal from the scanner and to integrate component peak areas for quantitative analysis.

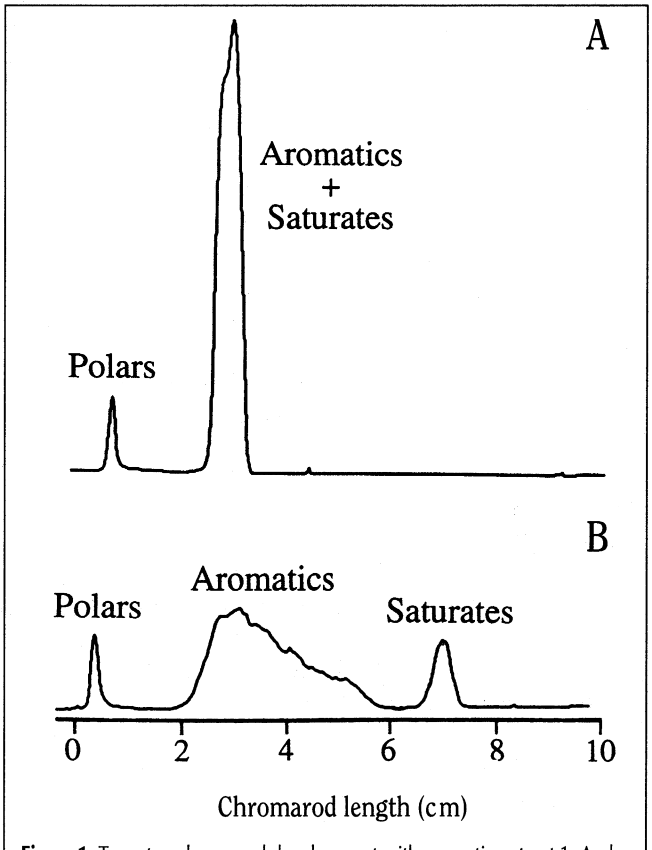

Figure 1. Two-step chromarod development with aromatic extract 1: A, development with toluene for 5 min; B, development with *n*-heptane for 30 min following the toluene development.

Clay–gel analysis

A detailed description of this open-column chromatographic method can be found elsewhere (5). In summary, two glass percolation columns were connected in series; the upper section contained clay (100 g), and the lower section contained clay (50 g) at the top and silica gel (200 g) at the bottom. The sample solution (5–10 g of sample in *n*-pentane) was added to the top of the upper section. Sufficient *n*-pentane (approximately 300 mL) was used to elute saturates from clay and silica. Polar compounds were adsorbed strongly on clay, and aromatics were retained by silica. The upper section was removed and washed further with *n*-pentane (approximately 200 mL) before charging a 1:1 (v/v) toluene–acetone mixture (approximately 300 mL)

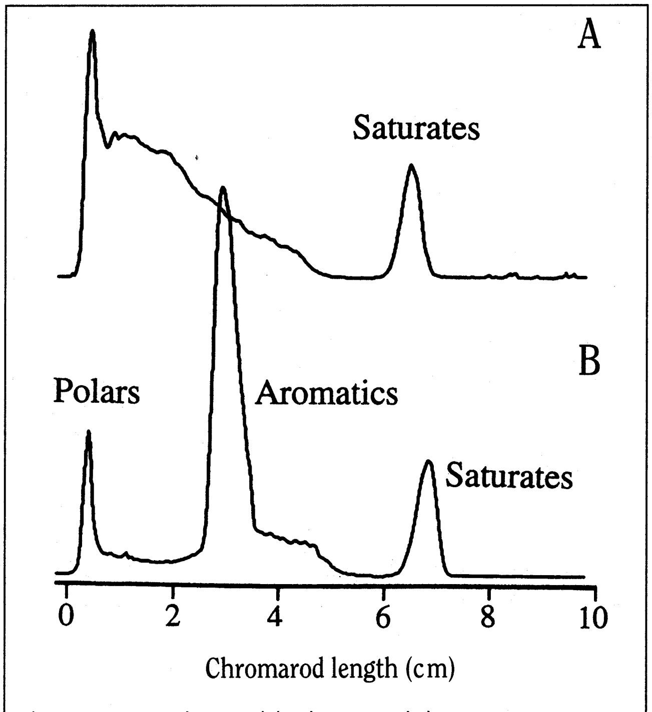

Figure 2. Two-step chromarod development with the same aromatic extract as in Figure 1: A, development with *n*-heptane for 30 min; B, development with toluene for 5 min following the *n*-heptane development.

for the desorption of polars from clay. Solvents are completely evaporated from the n-pentane and toluene–acetone fractions to determine the amounts of saturates and polars, respectively. Aromatics are usually calculated by difference. However, aromatics adsorbed in the silica gel can be desorbed with hot toluene (using Soxhlet extraction), and can be measured gravimetrically after the evaporation of toluene.

Results and Discussion

Separation and resolution of hydrocarbon types by TLC–FID

Figure 1B shows a typical separation of polars, aromatics, and saturates from an extract that is highly aromatic. It was obtained from solvent refining of paraffinic distillates and will be referred to as aromatic extract 1. A two-step development scheme (scheme I) was used where the chromarods were developed first with toluene and then with n-heptane as outlined in the Experimental section. Figure 1A shows that polars were separated from an aromatics plus saturates fraction when the chromarod was developed with toluene for 5 min. In the second development with n-heptane (30 min), saturates were displaced much more than aromatics, resulting in complete resolution of the two group types. In Figure 1B, each component class is fully resolved. A broad distribution of aromatics is also observed in this Figure. Presumably, this is due to an incremental displacement of aromatic components as their polarity decreases.

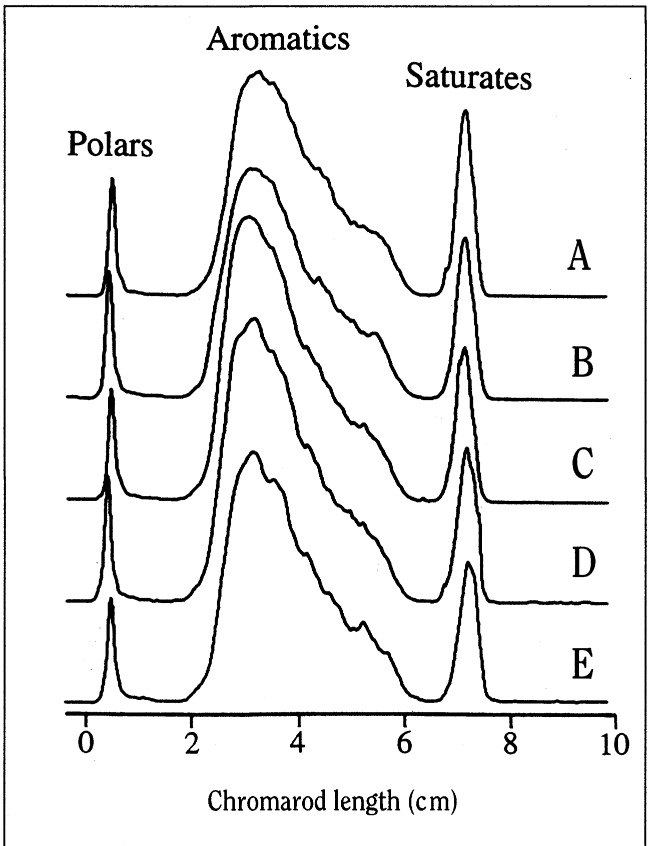

Figure 3. Reproducibility of the separation of component types by TLC–FID. Chromatograms were obtained with aromatic extract 1 on five different chromarods.

In TLC, the displacement of solutes is a function of solvent polarity. We note that solvent polarity parameters for toluene and n-heptane are P' = 2.4 and P' = 0.2, respectively, compared with water which has a polarity parameter of P' = 10.2 (22). As expected, in Figure 1B, the position and distribution of polars on the chromarod remained unchanged when nonpolar n-heptane was used in the second development. Thus, in this TLC–FID procedure, polars are defined as compounds that are essentially immobile on silica when the chromarods are developed with toluene.

Earlier workers used development schemes in which the chromarods were developed first with an n-alkane and then with a solvent or solvent mixture of higher polarity (6,7,10,11). A similar development scheme (scheme II) was tried here. Aromatic extract 1 was analyzed by developing the chromarod first with n-heptane for 30 min and subsequently with toluene for 5 min. Figure 2A shows that saturates were separated from polars plus aromatics using n-heptane. The subsequent development of the chromarod with toluene yielded Figure 2B. The marked difference between Figures 1B and 2B is due only to the reversed solvent order for the double developments.

It is apparent that development scheme II has adverse influence on the distribution of aromatics and resolution between polars and aromatics. When toluene is used to separate aromatics from polars following development with n-heptane, toluene (a stronger solvent) displaces more polar aromatics (retained near

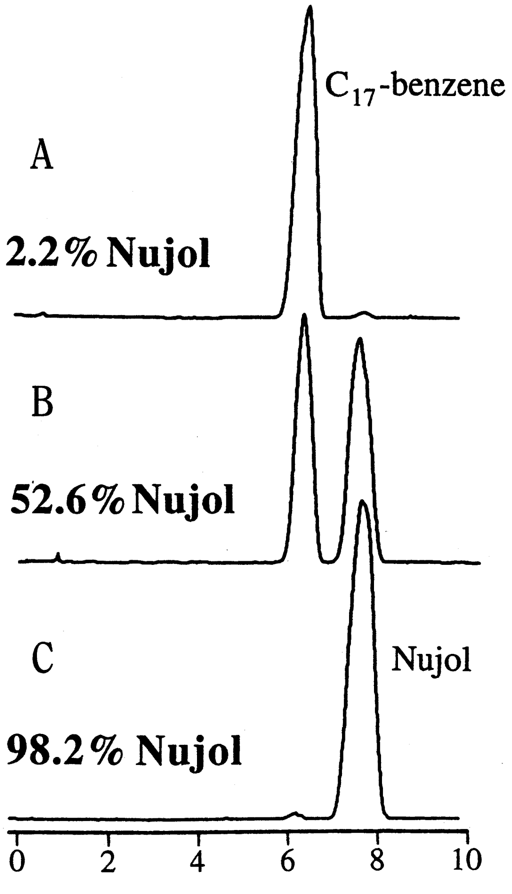

Figure 4. Separation of Nujol and heptadecylbenzene mixtures by TLC–FID.

the origin during *n*-heptane development) and superimposes these on already displaced less polar aromatics, resulting in a large frontal peak as observed in Figure 2B. The baseline between polars and aromatics is also found to be somewhat elevated. This may result from minor changes in the silica adsorbent after the chromarod has been developed with *n*-heptane, causing incomplete resolution between polars and aromatics.

As superior baseline, complete resolution of hydrocarbon classes, and polarity-based distribution of aromatics could be achieved, development scheme I was preferred. In the subsequent experiments, chromarods were developed using toluene first and then *n*-heptane. In this work, we have avoided the use of any solvent mixture to retain better control on the composition of the eluting solvent. This should provide better reproducibility in the TLC–FID method.

Highly reproducible results are obtained with TLC–FID. The five chromatograms shown in Figure 3 are for aromatic extract 1. In each case, the three hydrocarbon classes are cleanly resolved. However, there are some minor differences in the peak profile corresponding to each class. These differences can be attributed to minor nonhomogeneity in the silica adsorbent layer on the chromarod. They may also result from the variability inherent in manual spotting of sample to the chromarod.

The reproducibility of results can be evaluated best from normalized peak areas of the three group types. For simplicity, it is assumed here that mass percent results can be derived from area percent data using an identical response factor for each hydrocarbon class. This assumption is based on an earlier report where the packed-column response factors for the gas chromatographic–flame-ionization analysis for high molecular weight alkanes and aromatics were shown to be similar (23). Recently, it was also shown that plots of TLC–FID response versus mass are linear for saturates, alkylaromatics, aromatics, and polars and that the data points for various hydrocarbon classes can be represented by a single straight line passing through the origin (11). Accordingly, the mass percent (with standard deviations) of polars, aromatics, and saturates in aromatic extract 1 are 4.0 ± 0.4, 85.2 ± 1.3, and 10.8 ± 1.2, respectively.

The sensitivity and resolving capability of TLC–FID are illustrated in Figure 4. The chromatograms (Figures 4A–4C) are for 2.2%, 52.6% and 98.2% Nujol in a Nujol–heptadecylbenzene (C_{17}-benzene) mixture, respectively. Nujol, a medicinal white oil containing no aromatics, was easily resolved from C_{17}-benzene. Chromatograms A and C in Figure 4 indicate that 1–2% of either material can easily be detected and determined.

The compounds belonging to the aromatic class, as in Figure 3, vary in the number of aromatic rings. The components nearest to the saturates contain a single aromatic ring (as in Figure 4) and those displaced least from the polars contain multiple aromatic rings; aromatics with greater numbers of rings are more strongly retained on the chromarod (9,10,15,17). Thus, the distribution of aromatics based on the number of aromatic rings is reflected in a TLC–FID chromatogram.

In Figure 5, chromatograms for three aromatic extracts are shown. These samples show significant variations in the dis-

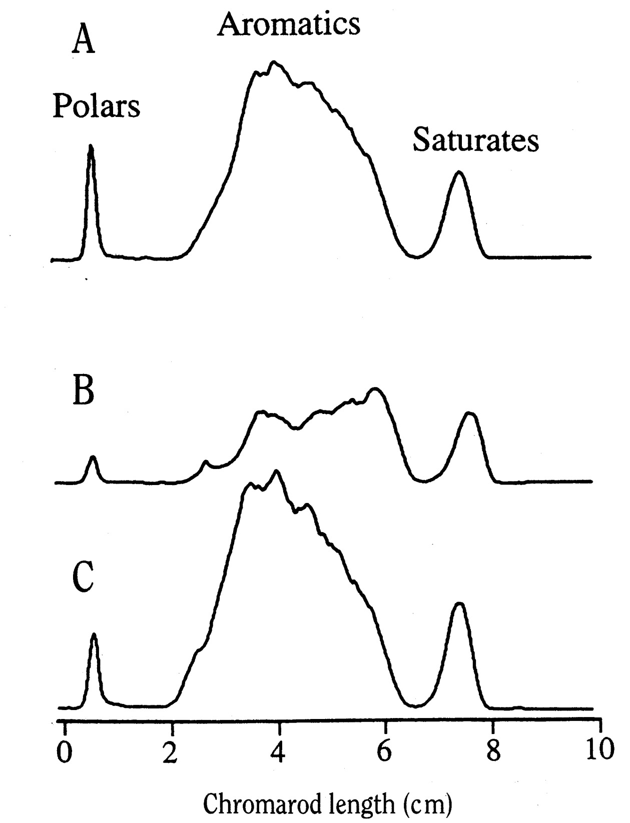

Figure 5. Compositional analysis of three aromatic extracts: A, semisolid aromatic extract 2; B, liquid aromatic extract 3; C, semisolid aromatic extract 4.

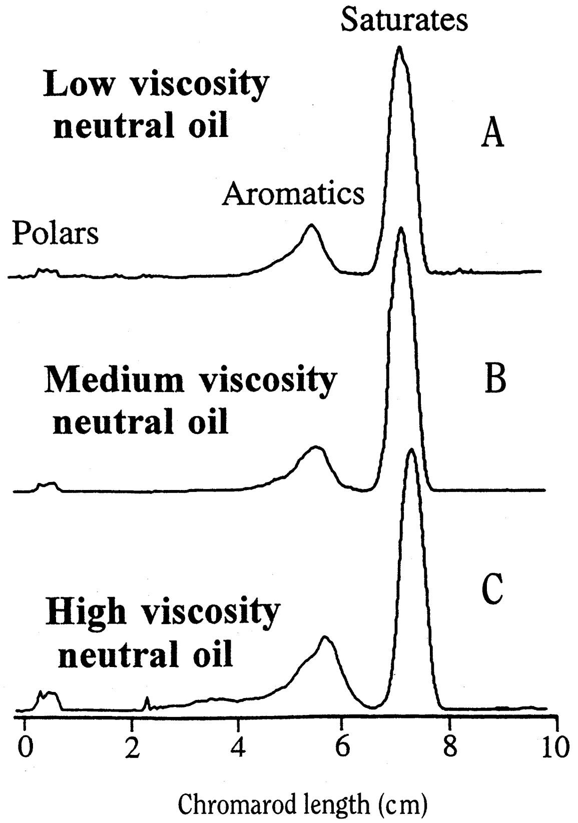

Figure 6. TLC–FID chromatograms for three solvent neutral oils belonging to different viscosity grades.

tribution of aromatics with respect to their polarity or ring numbers. More polar aromatics, presumably with multiple rings, are retained in the 2–4-cm range of the chromarod, whereas alkylbenzenes are displaced close to the saturates. Aromatic extracts 2 and 4 (chromatograms A and C, respectively) are semisolids at room temperature. Chromatograms A and C show that both samples contain high concentrations of aromatic compounds with multiple rings that are retained strongly on the chromarod. Aromatic extract 3 (chromatogram B) is a liquid sample. A larger proportion of aromatic compounds in this sample have fewer aromatic rings.

Analysis of base oils by TLC–FID

The chromatograms of three typical solvent neutral oils are shown in Figure 6. These oils are paraffinic base stocks and are used for manufacturing numerous formulated lubricants and products (24). These neutral oils differ in viscosity measured in Saybolt Universal Seconds at 100°F (37.8°C) (21). Here, a low viscosity neutral oil represents a base stock providing less than 200 SUS. For a medium viscosity neutral oil, SUS ranges between 200 and 500. A high viscosity neutral oil will have an SUS of over 500.

Table I. Comparison of Results Obtained by TLC–FID and ASTM D2007

Sample	Hydrocarbon type	TLC–FID (mass %)	D2007 (mass %)	Difference (%)
Low viscosity neutral oil	Saturates	74.3	73.8	+0.5
	Aromatics	23.7	25.9	-2.2
	Polars	2.0	0.3	+1.7
Medium viscosity neutral oil	Saturates	74.2	75.5	-1.3
	Aromatics	23.9	24.2	-0.3
	Polars	1.9	0.3	+1.6
High viscosity neutral oil	Saturates	57.1	67.9	-10.8
	Aromatics	39.5	31.0	+8.5
	Polars	3.4	1.1	+2.3
Aromatic extract 1	Saturates	10.8	17.4	-6.4
	Aromatic	85.2	72.6	+12.6
	Polars	4.0	10.0	-6.0
Aromatic extract 2	Saturates	8.7	16.7	-8.0
	Aromatic	88.6	73.2	+15.4
	Polars	2.7	10.1	-7.4
Aromatic extract 3	Saturates	14.9	22.0	-7.1
	Aromatic	83.2	70.0	+13.2
	Polars	1.9	8.0	-6.1
Aromatic	Saturates	9.2	15.6	-6.4
	Aromatics	86.6	71.0	+15.6
	Polars	4.1	13.4	-9.3
Alkylbenzene -150	Saturates	<1.0	49.2	–
	Aromatics	>99.0	50.5	~48.5
	Polars	–	0.3	–
Alkylbenzene -300	Saturates	<1.0	59.2	–
	Aromatics	>99.0	40.7	~58.3
	Polars	–	0.1	–

A cursory examination of Figure 6 reveals that there are some variations in the amounts of each hydrocarbon type as well as in the distribution of aromatics in these oils. The hydrocarbon type results obtained by the ASTM D2007 and TLC–FID methods on these solvent neutral oils, as well as on other samples, are listed in Table I.

The TLC–FID method is more appropriate for oils and residues boiling above 500°F (260°C). For these materials, sample loss due to exposure to heat of the FID flame before the separated materials enter the FID flame zone should be minimal (15). Since most base oil stocks have initial boiling points above 500°F, these are ideally suited to the TLC–FID technique.

Comparison of TLC–FID and ASTM D2007 methods

As previously mentioned, in the ASTM D2007 method, a sufficient amount (5–10 g) of sample is used so that enough materials can be collected for a reasonably precise gravimetric determination of hydrocarbon classes. Such high sample loads can overload the adsorbents, causing incomplete separation of group types, and therefore, introduce bias in the hydrocarbon type determination. Similarly, complete desorption of polars and aromatics from clay and silica, respectively, may not always occur. Thus problems of sample overloading and incomplete recovery of materials from adsorbents were examined for ASTM D2007.

Figure 7 shows a number of chromatograms obtained for aromatic extract 1 (chromatogram A) and for a number of its fractions obtained by the ASTM method (chromatograms B–D).

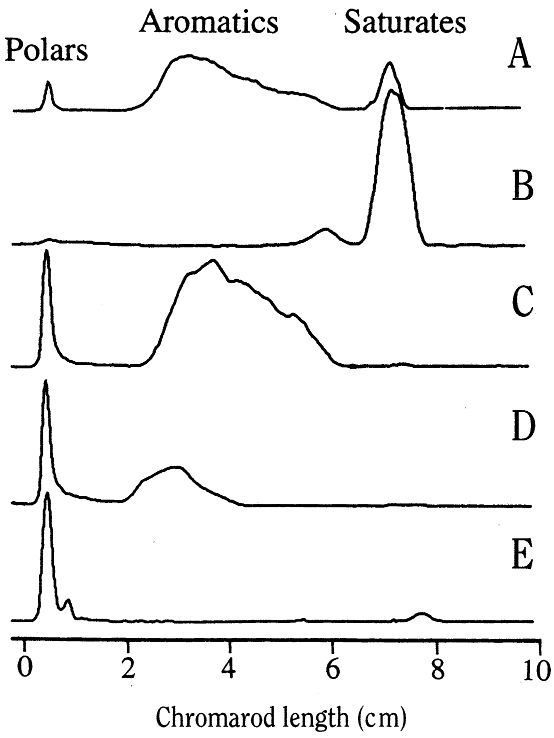

Figure 7. Cross-contamination and recovery problems in ASTM D2007 exemplified with aromatic extract 1 and its fractions. Chromatograms: A, aromatic extract 1; B, saturates by ASTM D2007; C, aromatics by ASTM D2007; D, polars by ASTM D2007; E, residual polars from clay.

Chromatogram E shows the residual polars extracted from used clay (from which polars had been desorbed) with hot toluene.

Figure 7B shows that saturates obtained by the ASTM method were contaminated significantly with single-ring aromatics. The aromatic fraction in Figure 7C was contaminated with high amounts of polars. The polar fraction (Figure 7D) also contained substantial amounts of aromatics, presumably with multiple rings. The recovery of polars from clay was not complete when these materials were desorbed with the toluene–acetone mixture, as evident from Figure 7E. It was estimated that at least 1% (w/w) of the sample was strongly retained by clay and could not be recovered using the ASTM procedure. As shown in Figure 7E, in addition to polars, even some saturates (most likely waxy materials) were left on the clay. Similar observations were made with two more aromatic extracts and their fractions (not shown).

These results suggest that the ASTM method does not always provide pure fractions of polars, aromatics, and saturates, and the results obtained are subject to serious inaccuracies arising from cross-contamination and incomplete recovery problems. Although the definitions (and thus the amounts) of polars and aromatics are dependent on the method used, the amounts of saturates should be identical by either method. Therefore, the error in saturates should not be overlooked when many single-ring aromatics are mixed with the saturate fraction obtained by the ASTM method.

TLC–FID chromatograms of two alkylbenzene base stocks of different viscosity grades are shown in Figure 8. The numbers associated with alkylbenzene-150 and alkylbenzene-300 refer to viscosity grades in terms of SUS. The chromatograms in Figure 8 indicate that these oils contain negligible amounts of saturates. However, as shown in Table I, the ASTM method provided data indicating that these base stocks contain as much as 50–60% saturates. Obviously, an error of this magnitude is consistent with the elution profile shown in Figure 7B where it was found that some single ring aromatic compounds can be misidentified as saturates in the ASTM method.

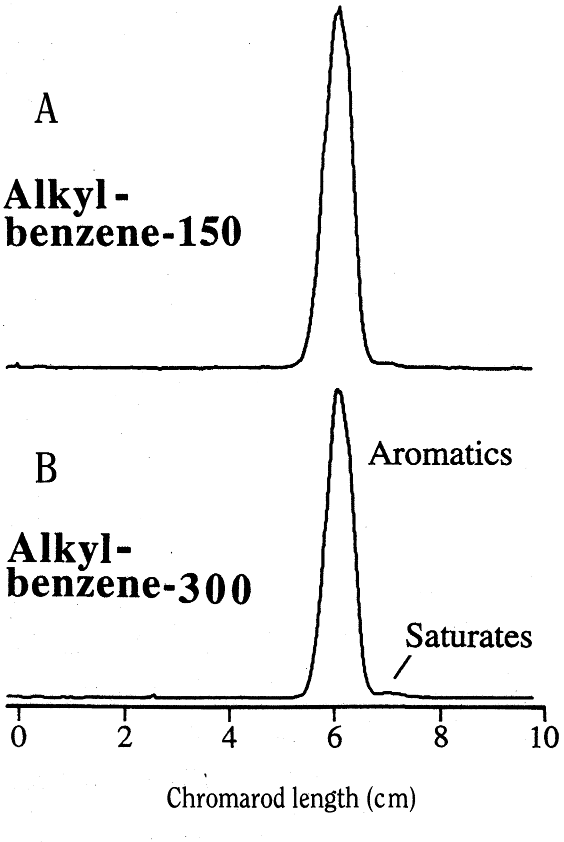

Figure 8. Analysis of two alkylbenzene base stocks.

It is difficult to carry out a meaningful comparison of results from the ASTM method with those from TLC–FID as in Table I. Although the cross-contamination and recovery problems exist in the ASTM method, some of the errors may offset each other. Thus the good agreement (within 3 mass %) between hydrocarbon-type results for low viscosity neutral and medium viscosity neutral oils could be purely fortuitous. For relatively high viscosity oils and aromatic extracts, saturates and polars are commonly overestimated, and consequently, aromatics are underestimated in the ASTM D2007 method.

Conclusion

Although similar hydrocarbon-type data are obtained by both TLC–FID and ASTM D2007 methods, TLC–FID results are superior to those obtained by the ASTM method. Baseline resolution of the components in the hydrocarbon classes is achieved with TLC–FID. As shown in Figure 7, the ASTM method may not always provide pure fractions of each hydrocarbon type. Additional errors may also occur due to incomplete recovery of samples from the adsorbents used in the ASTM method.

TLC–FID possesses a number of operational advantages including small sample size (few micrograms), less solvent consumption, reusable chromarods, rapid analysis time, accurate results, and high throughput. (In our present setup with solvent tanks capable of accommodating up to four metallic racks, 12 samples can easily be analyzed in 2 h using the TLC–FID method.) The small sample size is particularly attractive for the characterization of heavy distillates extracted from environmental samples or obtained from small-scale laboratory experiments. Only about 150 mL each of toluene and *n*-heptane are required for the TLC–FID method, and the solvents can be used repeatedly. The chromarods are reusable up to about 100 developments per scan cycle. By contrast, the ASTM method requires about 5–10 g of sample, 800 mL of solvent, and 350 g of adsorbents per analysis. The clay–gel analysis is labor intensive, requiring 6–8 h. The solvents are evaporated (and cannot be recovered) in this method. The method generates spent adsorbents that require proper handling and disposal. A method like ASTM D2007 does not lend itself to replicate sample testing because of the consumption of solvents and adsorbents and the lengthy analysis time.

The distribution of aromatics with respect to the number of rings or amount of polarity is obtained in TLC–FID. This additional information, available only from TLC–FID, should provide valuable insights into the suitability of heavy distillates for numerous applications.

Acknowledgments

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References

1. E. Lundanes and T. Greibrokk. Separation of fuels, heavy fractions, and crude oils into compound classes: a review. *J. High Resol. Chromatogr.* **17:** 197–202 (1994).
2. K.H. Altgelt and M.M. Boduszynski. *Composition and Analysis of Heavy Petroleum Fractions.* Chemical Industries, Vol. 54. Marcel Dekker, New York, NY, 1994.
3. K.A. Lintelmann. Heavy oils (natural and refined). *Anal. Chem.* **65:** 180R–85R (1993).
4. M. Radke, H. Willsch, and D.H. Welte. Preparative hydrocarbon group type determination by automated medium pressure liquid chromatography. *Anal. Chem.* **52:** 406–11 (1980).
5. ASTM Standard Test Method D2007-91. Characteristic groups in rubber extender and processing oils and other petroleum-derived oils by the Clay–Gel absorption chromatographic method. *1993 Annual Book of ASTM Standards.* American Society for Testing and Materials, Philadelphia, PA, 1993, Vol. 5.01 pp 693–99.
6. M. Ranny. *Thin-Layer Chromatography with Flame Ionization Detection.* D. Reidel Publishing Co., Dordrecht, Holland, 1987.
7. R.G. Ackman, C.A. McLeod, and A.K. Banerjee. An overview of analysis by chromarod-Iatroscan TLC–FID. *J. Planar Chromatogr.* **3:** 450–90 (1990).
8. J.E. Ray, K.M. Oliver, and J.C. Wainwright. The application of the Iatroscan TLC technique to the analysis of fossil fuels. Petroanalyis, '81: Advances in Analytical Chemistry in the Petroleum Industry. Proc. Inst. Pet., Wiley, New York, NY, 1982, chap 33.
9. M.A. Poirier and A.E. George. A rapid method for hydrocarbon-type analysis of heavy oils and synthetic fuels by pyrolysis thin layer chromatography. ACS Div. Pet. Chem. Prepr. **27:** 973–80 (1982).
10. D.A. Karlsen and S.R. Larter. Analysis of petroleum fractions by TLC–FID: applications to petroleum reservoir description. *Org. Geochem* **17:** 603–17 (1991).
11. J. Vela, V.L. Cebolla, L. Membrado, and J.M. Andrés. Quantitative hydrocarbon group type analysis of petroleum hydroconversion products using an improved TLC–FID system. *J. Chromatogr. Sci.* **33:** 417–25 (1995).
12. F.B. Padley. Use of a flame-ionization detector to detect components separated by thin-layer chromatography. *J. Chromatogr.* **39:** 37–46 (1969).
13. J.J. Szakasits, P.V. Peurifoy, and L.A. Woods. Quantitative thin-layer chromatography using a flame ionization detector. *Anal. Chem.* **42:** 351–54 (1970).
14. T. Okumura, T. Kadono, and A. Isoo. Sintered thin-layer chromatography with flame ionization detector scanning. *J. Chromatogr.* **108:** 329–36 (1975).
15. A. Obuchi, H. Aoyama, A. Ohi, and H. Ohuchi. Application of thin-layer chromatography with flame ionization detection to the characterization of organic extracts from diesel exhaust particulates. *J. Chromatogr.* **288:** 187–94 (1984).
16. L.L. Leazar. Quantitative analysis of petroleum residues and heavy oils by TLC/FID. *J. Chromatogr. Sci.* **24:** 340–43 (1986).
17. B. Jurkiewicz, M. Nosek, and Z. Kubica. Thin layer chromatography with flame ionization detection in the analysis of liquid coal products. *J. Planar Chromatogr.* **4:** 89–90 (1991).
18. C.C. Wan, T.H. Waters, and R.D. Wolever. Development of a reproducible Iatroscan method to chemically characterize asphalt. *ACS Div. Fuel Chem. Prepr.* **37:** 1350–59 (1992).
19. S.J. Pollard, S.E. Hrudey, B.J. Fuhr, R.F. Alex, L.R. Holloway, and F. Tosto. Hydrocarbon wastes at petroleum and creosote-contaminated sites: rapid characterization of component classes by thin-layer chromatography with flame ionization detection. *Environ. Sci. Technol.* **26:** 2528–34 (1992).
20. N.C. Shantha. Thin-layer chromatography–flame ionization detection Iatroscan system. *J. Chromatogr.* **624:** 21–35 (1992).
21. ASTM Standard Test Method D 88-81. Standard test method for Saybolt viscosity. 1991 Annual Book of ASTM Standards. Vol. 10.03. American Society for Testing and Materials, Philadelphia, PA, pp 1–6.
22. L.R. Snyder and J.J. Kirkland. *Introduction to Modern Liquid Chromatography.* Wiley-Interscience, New York, NY, 1979, chap 6.
23. H.Y. Tong and F.W. Karasek. Flame ionization detector response factors for compound classes in quantitative analysis of complex organic mixtures. *Anal. Chem.* **56:** 2124–28 (1984).
24. A. Sequeira, Jr. Lubricant Base Oil and Wax Processing. Chemical Industries, V.60, Marcel Dekker, New York, NY, 1994.

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