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Aromatic Content in Medium Range Distillate Products—Part I: An Examination of Various Liquids*

ABSTRACT: Classification of ignitable liquids in accordance with voluntary consensus-based standards published by ASTM International has become increasingly specific, relying upon both the chemical composition and the boiling point range of submitted ignitable liquids. This classification system includes among others, specific classes for distillates and dearomatized distillates. In this study, a variety of medium-range ignitable liquids were analyzed by gas chromatography-mass spectrometry. Several methods of data analysis were utilized to examine the relative aliphatic and aromatic contents in these liquids. Results show that commercially available products in the medium range exhibit a broad range of compositions with respect to the relative proportion of aliphatic and aromatic compounds and that some liquids may not be easily classified. This study demonstrates the importance of examining the proportion of aliphatics:aromatics for classifying such liquids and suggests guidelines for differentiating medium range distillates, dearomatized distillates, and blended products.

KEYWORDS: forensic science, fire debris analysis, medium petroleum distillates, ignitable liquids, classification, aromatic content, aliphatic content

The forensic analysis of fire debris for the presence of ignitable liquids involves many steps, including extraction, instrumental analysis, data interpretation, and classification of any products detected. Voluntary consensus standards published by ASTM International suggest a classification system to aid analysts in this endeavor (1,2). This system for classifying ignitable liquids has been expanded since its initial development in an effort to be more inclusive and more descriptive of the variety of ignitable liquid products that are commercially available, and that could potentially be used as accelerants.

Historically, ignitable liquids were primarily classified according to boiling point range, although some distinctions based on chemical composition were included (3–5). In the most recent edition of ASTM E 1618, however, ignitable liquids are classified based on both chemical composition and boiling point range (2). This classification scheme is designed such that an ignitable liquid can be initially described based upon its chemical composition, and fitted into one of the following classes: gasoline, petroleum distillates, isoparaffinic products, aromatic products, naphthenic/paraffinic products, n-alkane products, dearomatized distillates, oxygenated solvents or others—miscellaneous. Then, it can be further described based upon its boiling point range, or peak spread, as light (C₄–C₉), medium (C₈–C₁₃) or heavy (C₈–C₂₀₊). Further explanation of the modifications that were made to the classification schemes are available in the literature (6,7).

Classification of a product as an isoparaffinic, naphthenic/paraffinic or normal-alkane product requires, in addition to other

specific criteria, that there be virtually no aromatic compounds present. Conversely, classification of a liquid as an aromatic product requires that there be virtually no aliphatic components; the liquid is entirely comprised of aromatic compounds. These types of products are also distinctive in pattern, leading to a relatively straightforward and unambiguous classification.

The presence and relative amount of aromatic components becomes critical in differentiating the classes of petroleum distillates and dearomatized distillates. These are products that are generally prepared by distillation, and may also be treated to remove aromatic hydrocarbons (8). Both medium petroleum distillates (MPDs) and medium-range dearomatized distillates will appear similar to one another in a total ion chromatogram (TIC) or a chromatogram generated with a flame ionization detector (FID). Accordingly, gas chromatography-flame ionization detection (GC-FID) is generally not sufficient to make this distinction, and when gas chromatography-mass spectrometry (GC-MS) is used, the examiner relies upon the extracted ion profiles to assist in making the determination. The criteria for identification of a distillate, as stated in ASTM 1618 include “AROMATICS: Always present in medium and heavy distillates; less abundant than alkane;” whereas the criteria for identification as a dearomatized distillate state “AROMATICS: Not present in significant amounts (2).” Adding to the significance of the amount of aromatics in medium range products is the existence of commercially manufactured blends of aromatics with medium range distillate-type products. We therefore have three general categories into which distillate-type products in the medium boiling point range may be placed; they are: dearomatized distillates, petroleum distillates, and blended products consisting of distillate-type products with an added aromatic component. Figure 1 shows the TIC and the summed extracted ion profiles for ions 57, 71, 85, and 99 (aliphatic profile) and for ions 91, 105, 119, and 133 (aromatic profile) for an MPD. Figures 2 and 3 show the corresponding data for a dearomatized distillate and a blended product, respectively.

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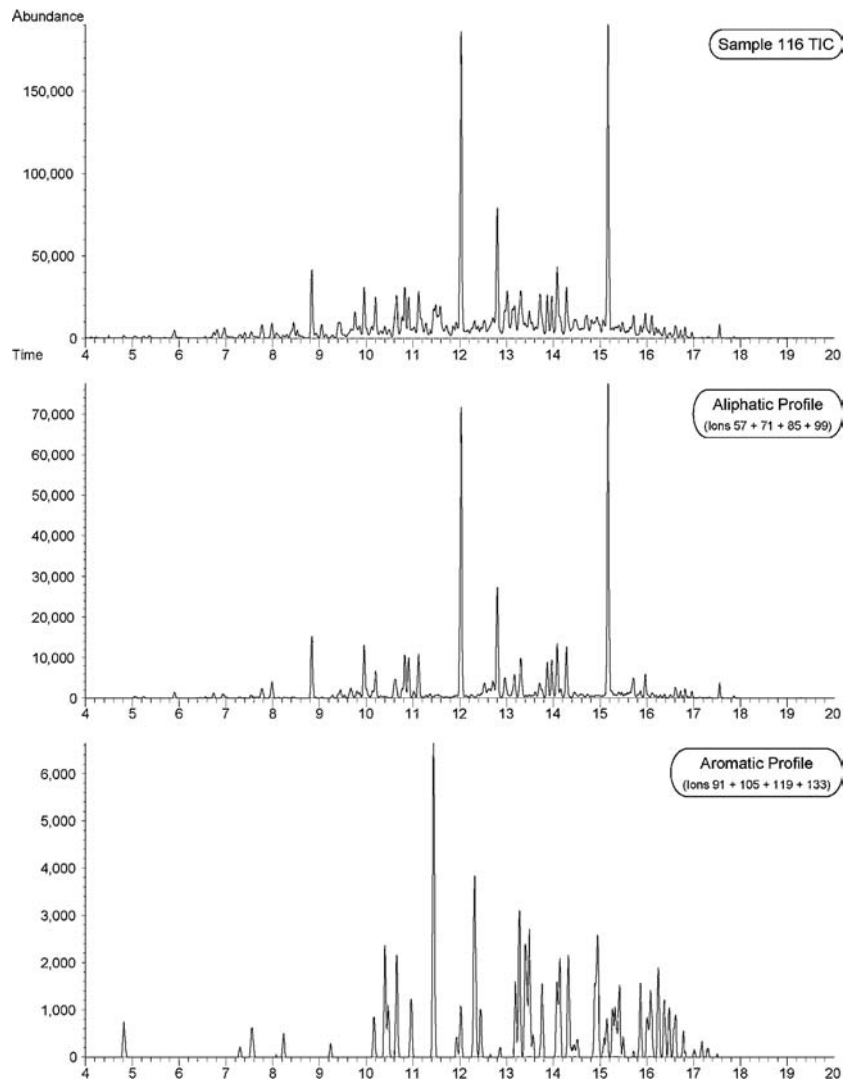


FIG. 1—Data for a typical medium petroleum distillate (Liquid 116).

The relative proportion of aromatic compounds is therefore a significant factor in determining into which category a product will be placed. In comparison with a straight run distillate, a dearomatized distillate will have a lower concentration of aromatics and a blended product will have a higher proportion of aromatics. In theory, there are three distinct ways of classifying these types of products, and three distinct ranges of aromatic contribution. The challenge for the fire debris analyst is that there are no guidelines for determining the limits for each of these groups of liquids. This paper will focus upon petroleum products in the medium boiling range (C_8 – C_{13}) and the effect that aromatic content has on their classification.

Methods

Samples of 50 medium range distillate-type products were obtained and prepared for analysis by dilution (1%) in carbon disulfide. All samples were analyzed on a Hewlett-Packard (HP) 6890 Series Gas Chromatograph equipped with an HP 5972A Mass Selective Detector using the parameters shown in Table 1.

Data analysis was accomplished using the HP ChemStation software. The process for determining the ratio of aliphatic to aromatic

TABLE 1—Analytical conditions.

Column Type	Hewlett-Packard HP-1 (1% diphenyl methyl siloxane)
Dimensions	25 m × 0.2 mm × 0.5 μm
Mobile phase	
Carrier gas	Helium
Flow rate	1 mL/min (flow electronically controlled)
Injection Type	Liquid/Autosampler Split (ratio 20:1)
Volume injected	1 μL
Temperatures	
Injector	250°C
Column	60°C for 3 min 5°C/min to 120°C 12°C/min to 300°C for 5 min Total run 35 min
Transfer line	280°C
Quadrupole Source	150°C 230°C
Mass Spec.	
Scanning range	33–300 amu
Solvent delay	2.75 min
Sampling	3.92 scans/sec

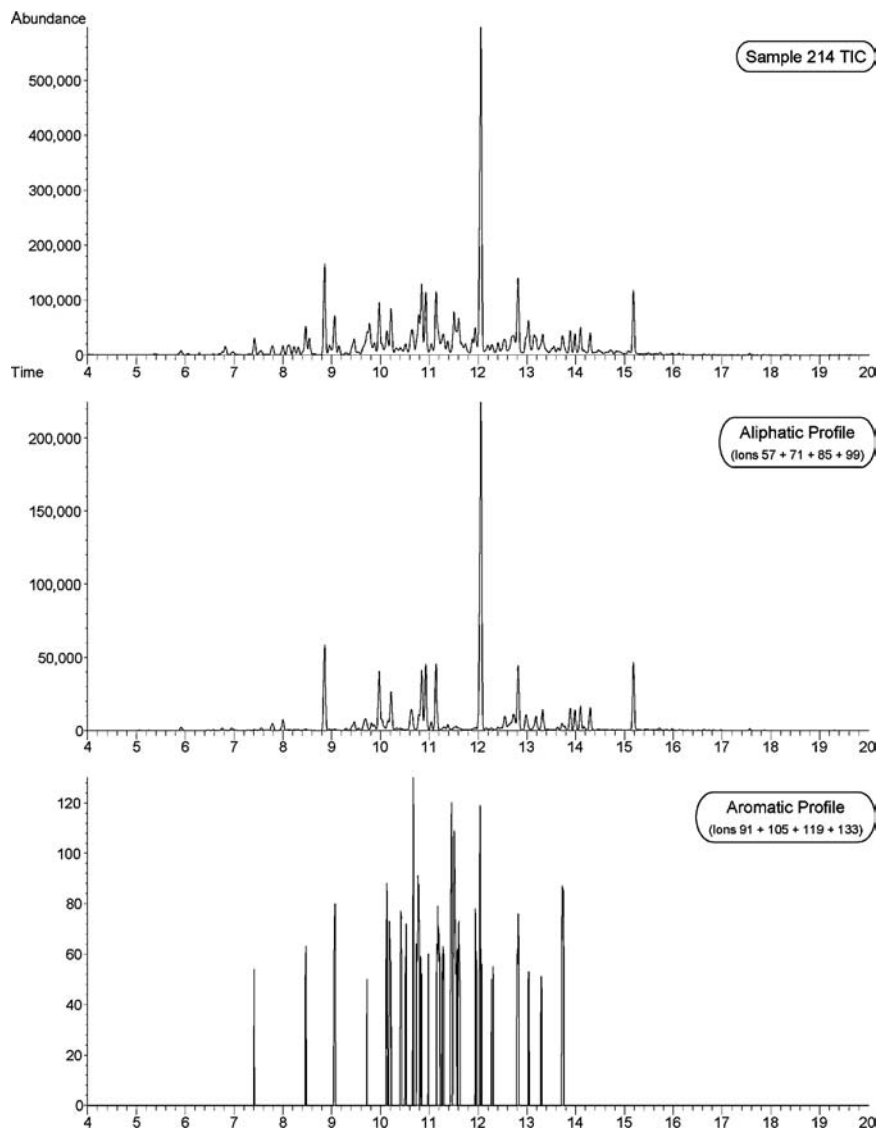


FIG. 2—Data for a typical medium-range dearomatized distillate (Liquid 214).

compounds was done by four different methods, and the results of the various methods were compared with one another.

Method 1

The first method (shown in Fig. 4a) was to calculate the ratio of the height for one selected peak in the aliphatic profile to that of one in the aromatic profile. This was done based on the peak height of n-decane in the aliphatic profile and the peak height of 1,2,4-trimethylbenzene in the aromatic profile. These compounds were selected because they are of similar boiling point and are well represented in most medium range products. This method of approximating content based on peak height is widely used in the field of forensic fire debris analysis. Because the ranges for ratios reported are dependent upon the ions used in profiling, it is necessary to specify the ions used. For this method, the aliphatic profile consists of the sum of the 57, 71, 85, and 99 extracted ion profiles, and the aromatic profile consists of the sum of the 91, 105, 119, and 133 extracted ion profiles.

Method 2

The same compounds were used for Method 2; however, this method is based upon the target ion peak area rather than peak height. The ions used were 57 for n-decane and 105 for 1,2,4-trimethylbenzene. This method (shown in Fig. 4b) uses objective parameters and is independent of an examiner's subjective measurements.

Method 3

Method 3 is similar to Method 1 in that both methods rely upon the subjective determination of peak height by the examiner; however, it differs from Method 1 in two ways. The first difference is that the ratio is calculated from the most abundant peak in the profile rather than the two specific compounds used in Method 1. The other difference is that this method utilizes abundance information from single extracted ion profiles rather than the summed profiles used in Method 1. The 57 ion was used to represent the aliphatic contribution, and 105 was used to represent the aromatic content. Figure 4c illustrates an example of this method.

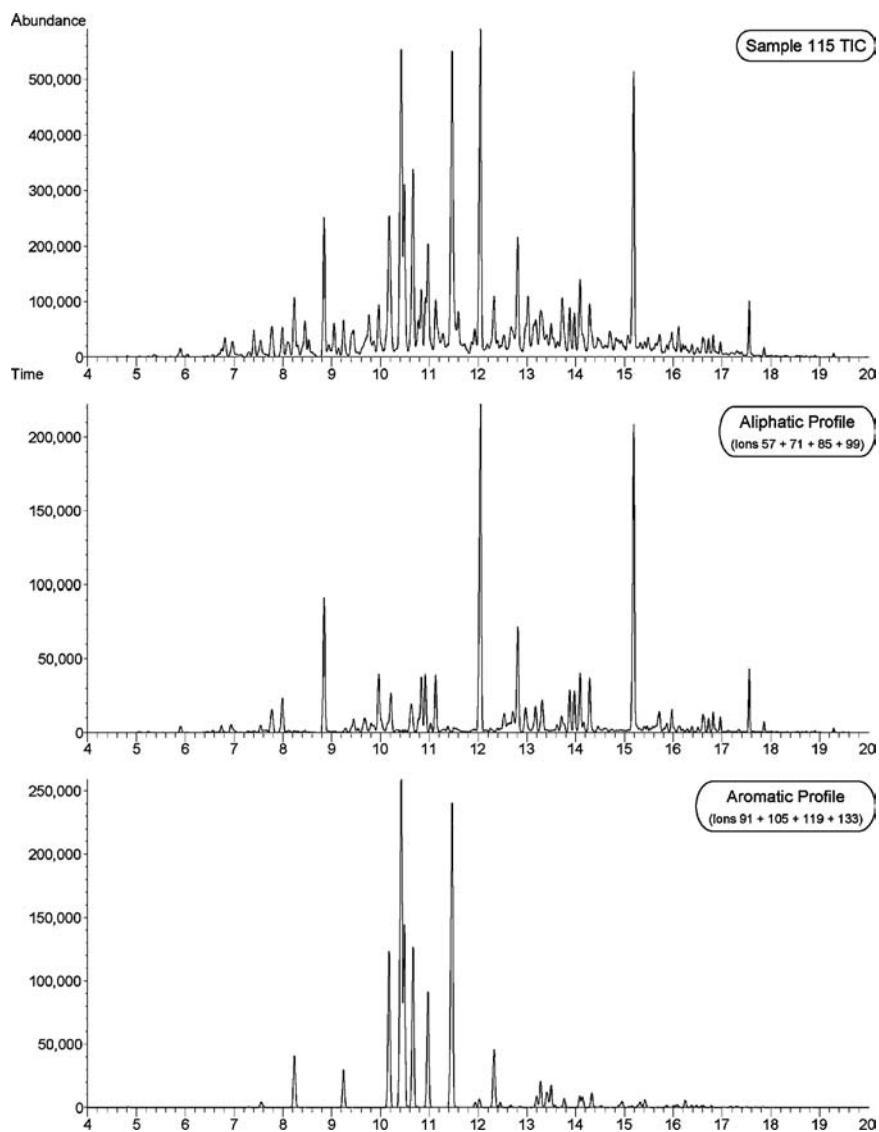


FIG. 3—Data for a blended product, consisting of a medium aromatic product combined with a medium petroleum distillate (Liquid 115).

Method 4

The fourth method for representing the relative aliphatic:aromatic content was done by creating a single mass spectrum that shows the average of all scans within a constant time range that included C₇ through C₁₄. A time range of 4 to 20 min was selected to meet these conditions. From this spectrum average, the ratio of ion 57 to ion 105 was calculated. Due to the scanning characteristics of the mass spectrometer, and the fact that the average spectrum represents thousands of individual spectra, the 57 ion did not always appear at a single m/z ion value of 57.00. Rather, the 57 ion was in some instances, split among several values, including 57.00, 57.05, 57.10, or 57.15 for this dataset. This phenomenon is illustrated in Fig. 4d. When calculating the ratios, the abundance for ion 57 was determined as the sum of all abundances with a nominal value of 57.

Results

Data obtained by each of the methods are shown in Table 2. The numerical values that represent the relative proportion of aliphatic compounds to aromatic compounds differs depending upon the

method used. The liquids in Table 2 are sorted by ascending ratios for Method 1. A comparison of the values obtained by each of the different methods is shown in Fig. 5a. Figure 5a shows a good correlation between Methods 1 and 2. In contrast, Methods 3 and 4 show significant discrepancies from the trend given by Methods 1 and 2, as well as from each other. Where greater aromatic content (lower ratios) are exhibited, as shown in Fig. 5b, a better correlation among all four methods is observed. Discrepancies will be examined in the discussion section.

The liquids examined represent at least three different classifications, as described by ASTM: medium dearomatized distillates, medium petroleum distillates, and a blend of a medium aromatic product with a medium distillate, which is classified as miscellaneous. Additionally, some products did not fall neatly into these classes. Using values obtained by Method 1, the range of ratios of aliphatic:aromatic content for the liquids studied spanned 3 orders of magnitude (from less than 1 to over 1800). Initial examination of the distribution of calculated ratios indicates that there is not a clearly defined natural break between these categories. However, additional methods of data analysis were applied in order to establish a logical distinction between the classes.

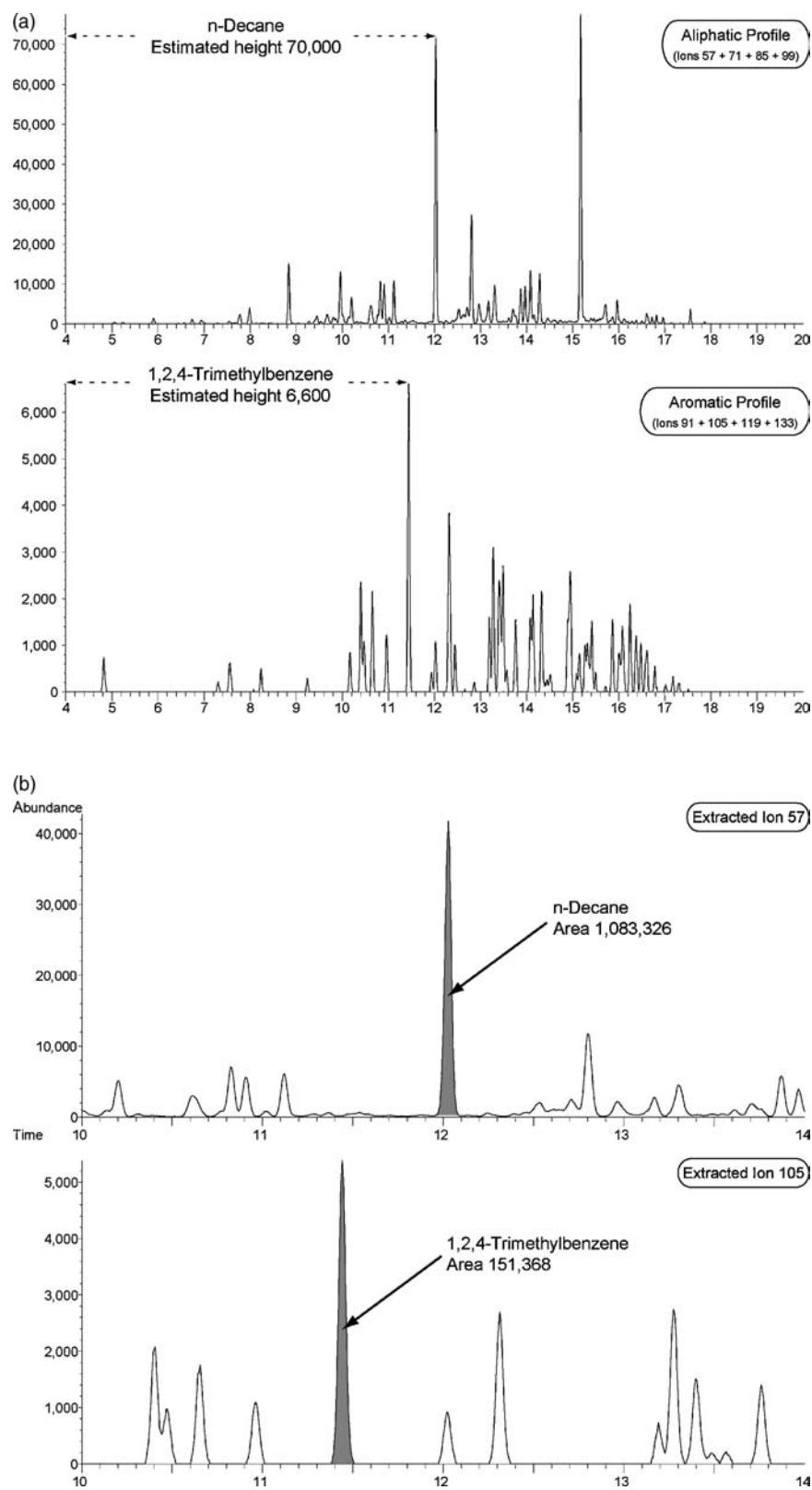


FIG. 4—Graphical representation of the four methods used for determining aliphatic and aromatic content to be used in determining ratios: (a) Method 1: Aliphatic content is represented by the peak height of n-decane in the aliphatic profile, and aromatic content is represented by the peak height of 1,2,4-trimethylbenzene in the aromatic profile. (b) Method 2: Aliphatic content is represented by the peak area of ion 57 for n-decane, and aromatic content is represented by the peak area of ion 105 for 1,2,4-trimethylbenzene. (c) Method 3: Aliphatic content is represented by the peak height of the tallest peak in the 57 profile, and aromatic content is represented by the peak height of the tallest peak in the 105 profile. (d) Method 4: An average mass spectrum is obtained for the area of interest and tabulated. The ratio is calculated from the abundance of 57 ions to the abundance of 105 ions.

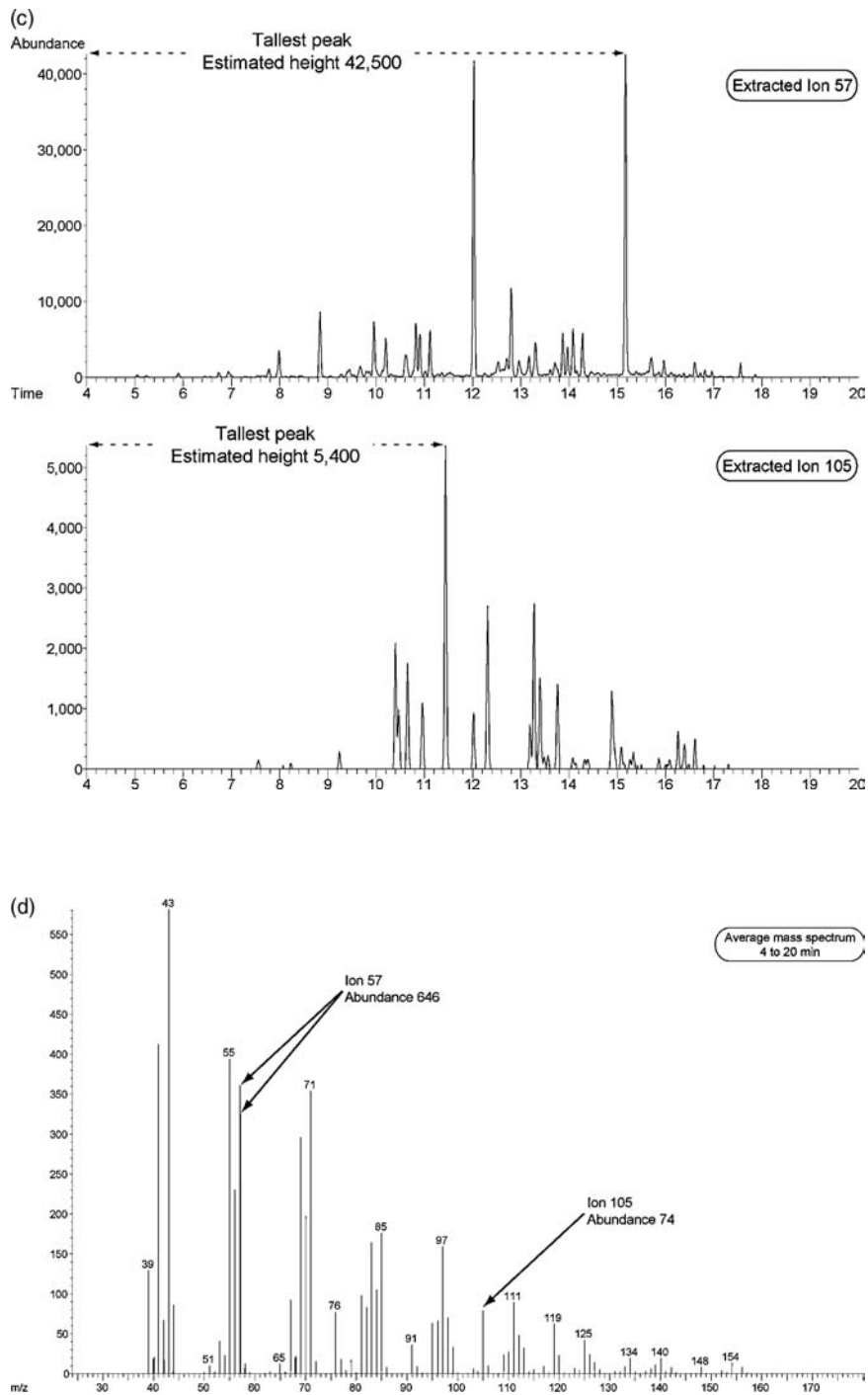


FIG. 4—Continued.

Discussion

Choice of a Method

There are many different ways to determine the aliphatic:aromatic ratio in a medium range distillate. While this study does not purport to find the best way, it does include four different methods, each of which has advantages and disadvantages.

Method 1 is probably the method most commonly used among fire debris analysts. While it is not technically correct to estimate concentration based on peak height rather than area from a chromatogram generated with a GC-MS or GC-FID, it is widely done

among practitioners. Method 2 requires a slightly more complicated data processing, but offers the advantage over Method 1 of eliminating the inaccuracy of the height estimation by taking into account the area.

It is interesting to observe that although these two methods based their calculations on two different sets of values, the curves in Figs. 5a and 5b are very closely related, with the exception of liquids 212 and 224. These discrepancies are due to the fact that the two liquids exhibiting poor agreement are both products that lie at extreme values. Liquid 224 is a narrower cut than most of the liquids studied, ranging from C₁₀ to C₁₂. Because both n-decane

TABLE 2—List of liquids analyzed and their aliphatic:aromatic ratios obtained by each of the four different methods. The list is sorted by ascending ratios for Method 1.

Liquid No.	Description	Ratios			
		Method 1	Method 2	Method 3	Method 4
115	Nasco Turpex	0.92	0.61	0.57	0.91
212	Chevron Techron Concentrate	1.95	5.66	1.79	2.77
219	Klean Strip 100% Miner	2.50	1.73	1.82	2.56
208	Pro-Gard Clean Up	2.52	1.70	2.03	3.38
72	Deodorized Mineral Spirits-Paint Thinner	2.64	1.73	1.87	2.28
207	Pro-Gard Fuel Injector Plus Intake	2.81	1.86	2.72	3.94
77	Hechinger Paint Thinner	2.90	1.88	2.02	3.40
83	Dart Drug Paint Thinner	3.38	2.24	2.38	2.78
47	Martin Senour Surface Prep	3.40	2.33	2.51	4.55
195	Varsol 1	3.53	2.31	2.77	3.66
145	Ashland Mineral Spirits NE	3.54	2.55	2.59	3.24
225	Exxon Varsol 1 (duplicate)	3.70	2.37	3.86	2.91
9	STP Lead Substitute	4.00	2.42	2.55	3.39
50	R.M. Hollingshead Penetrating Type Oil	4.30	3.17	3.12	3.38
221	E-Z Paint Thinner	4.63	3.16	3.48	5.59
217	Flood Penetrol Quality	4.68	2.93	12.7	25.0
37	Sears Tirpolene Thinner	4.86	3.16	3.39	4.63
69	Boron Garden Lite	5.25	3.46	3.70	3.82
213	SCCC Mineral Spirits 145EC	5.57	3.75	4.07	7.76
92	Hi-KB Kwik-Dri	5.94	4.19	2.24	2.97
109	Montgomery Wards Paint & Lacquer Thinner	5.95	3.77	5.58	5.92
216	Flood ESP (Easy Surface Protectant)	6.63	4.37	4.73	7.97
196	Varsol 18	6.67	4.49	5.15	7.32
209	Pro-Gard Fuel Injector Cleaner	6.67	4.71	5.76	7.80
7	Gumout Carburetor & Fuel Injector Cleaner	6.73	4.36	4.74	5.93
81	Barbeque Charcoal Lighting Fluid	7.78	5.19	10.5	8.56
97	Varsol Mineral Spirits	8.13	5.27	5.67	7.99
210	Pro-Gard Gas Treatment	8.41	5.66	6.55	8.11
113	MinWax #2108 Goldenrod	10.3	6.78	7.20	7.05
116	Nasco Paint Thinner	10.6	7.16	7.87	8.73
65	Marvel Diesel Fuel Conditioner	11.9	8.46	8.86	8.97
66	Ashland 140 Solvent	13.3	8.10	1260	2000
122	Wood Floor	14.7	10.1	0.54	7.92
5	Turtle Wax Bug and Tar Remover	14.8	9.93	10.5	9.35
80	Oriole Paint Thinner	14.9	9.69	10.6	8.17
53	Tru-Burn Charcoal Lighter Fluid	38.8	27.9	27.6	26.8
206	Exxsol D-60 Solvent	45.5	26.5	595	509
218	Klean Strip Odorless M	52.8	36.4	37.4	92.6
52	Kingsford Charcoal Lighter Fuel	82.5	57.1	61.8	93.8
211	Kingsford Odorless Charcoal	94.1	65.0	68.8	138
11	TRAK Carburetor & Fuel Injector Cleaner	152	89.4	86.3	315
220	Whitaker Rule 66 Miner	204	153	167	271
222	Shellsol D38	300	205	298	219
224	Shellsol D60	317	1400	1710	1390
95	Kwik-Dri	319	197	206	358
8	Super g Odorless Charcoal Lighter Fluid	348	212	20.8	532
215	Publix Charcoal Lighter	428	295	247	244
223	Shellsol D43	504	297	208	467
146	Ashland Mineral Spirits Rule 66	956	593	630	2430
214	Royal Oak Premium Odorless	1830	1619	942	1410

and 1,2,4-trimethylbenzene are at the extreme periphery of the Gaussian pattern, they are present in relatively low concentrations. In addition, Liquid 224 has a very low aromatic content. The combination of these two factors results in an extremely low level of 1,2,4-trimethylbenzene in this product. Consequently, there will be an inherent error in calculating ratios for this product because when one of the values is extremely small, a slight difference in value will result in a relatively large difference in calculated ratio. In contrast, Liquid 212 has a very high aromatic content. Errors may be expected to be larger for larger numerical values, resulting in greater variation in the calculated ratios. Although these noted exceptions do exist, the overall trend for ratios is in very good agreement for

Methods 1 and 2. This demonstrates that peak height is a valid approximation when comparing the signal abundance of components within a chromatogram.

The main disadvantage of both of these methods is that they are based on the comparison of n-decane and 1,2,4-trimethylbenzene. While this is acceptable for most of the liquids, serious discrepancies are observed when Methods 1 and 2 are compared with Methods 3 and 4 when n-decane is not the most abundant peak in the aliphatic profile. For example, liquids 66, 206 and 224 are products with narrow boiling point ranges, with n-decane as a minor peak in the aliphatic profile. This explains why these liquids present very low ratios in Methods 1 and 2, while they present much

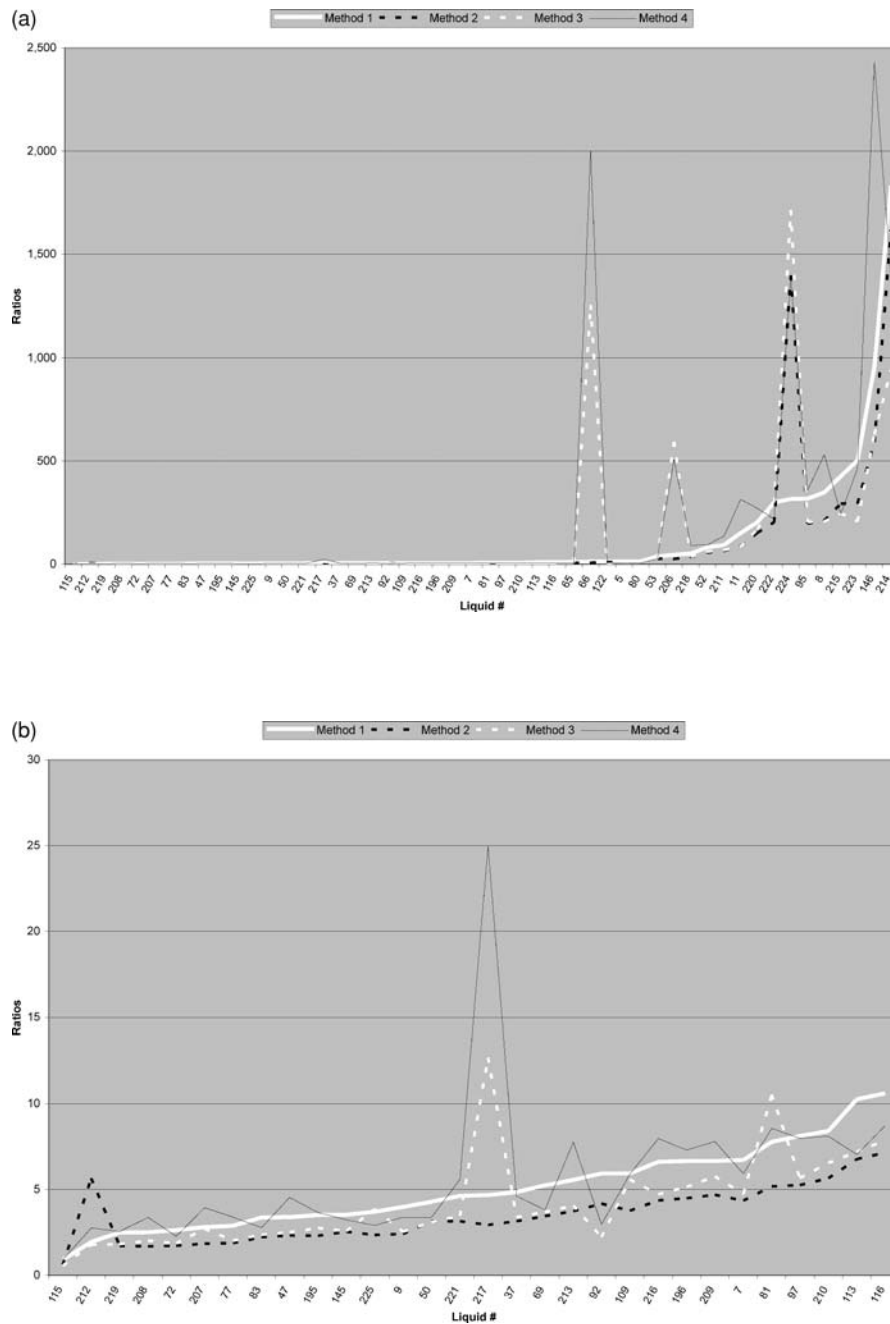


FIG. 5—Graph of the ratio values obtained for each liquid using the four different methods sorted by ascending ratios for Method 1: (a) All 50 liquids. (b) Expanded view of the 30 first liquids.

higher ratios in Methods 3 and 4. Figure 6 shows an example of this occurrence with liquid 66. As seen, n-decane is barely visible in the 57 extracted ion profile.

Method 3 is relatively simple to perform. While it still presents some inaccuracies inherent to peak height estimation, it takes into account the tallest peak in each profile rather than two predetermined compounds. Thus, Method 3 is not influenced by important shifts in the boiling point range, as are Methods 1 and 2. By examining Figs. 5a and 5b, one realizes that Method 3 ratios follow Methods 1 and 2 ratios pretty closely, except for the liquids described above and for the liquids with ratios above 30 as calculated

by Method 1. This is particularly true for the liquids with a low ratio as shown in Fig. 5b.

Of the four methods, Method 4 probably provides the most accurate ratio since it takes into account the whole chromatogram rather than a selected number of compounds as in the other methods. In this manner, it is the best representation of the entire aromatic and aliphatic contents. However, it still takes into account only one ion to represent the aliphatic content (ion 57) and one ion for the aromatic content (ion 105), which were deemed representative. One major disadvantage of this method is the fact that it will include the contributions from interfering products in real samples.

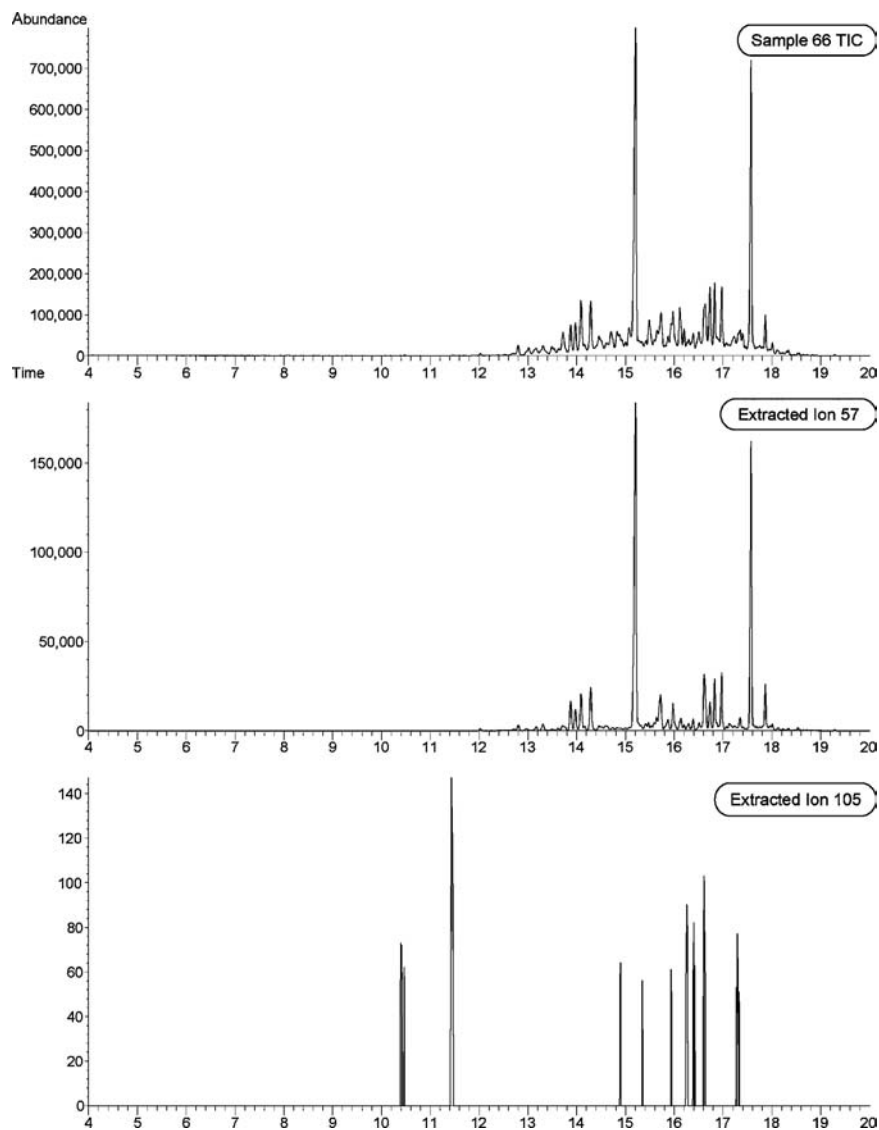


FIG. 6—Data for an MPD of higher *n*-hydrocarbon (boiling point) range (Liquid 66). This illustrates the difference in ratios between Methods 1 and 2, which are dependent upon *n*-decane and 1,2,4-trimethylbenzene, and Methods 3 and 4, which are not.

In this study, diluted liquids were used, so this method is applicable; however, it would be difficult to obtain reproducible results with a real sample in which contributions to the 57 and 105 ions are not necessarily limited to originating from components of the ignitable liquid. This method does present a very close correlation with Methods 1 and 2, except for the liquids discussed above and for the liquid with ratios above 30.

Differentiation of the Liquids

When comparing the amount of aliphatics to the amount of aromatics using Method 1, the value for a typical MPD is generally in the range of 2 to 15. This means that the abundance of *n*-decane in the aliphatic profile is roughly 2 to 15 times that of 1,2,4-trimethylbenzene in the aromatic profile. For a dearomatized distillate, the ratio is generally greater than 100, and for a blended product, the ratio will generally be less than one. Blended products can also often be recognized by the presence of 1,2,4-trimethylbenzene and the co-eluting 3- and 4-ethyltoluenes

clearly visible in the TIC rising above the profile of the typical minor peaks. It is also not uncommon for there to be a greater abundance of the co-eluting 3- and 4-ethyltoluenes relative to the 1,2,4-trimethylbenzene abundance which can be seen clearly in the aromatic profile.

For many of the products examined, such as those shown in Figs. 1, 2 and 3, the process of classifying them is straightforward. The aliphatic:aromatic ratio (Method 1) for the MPD shown is 10.6, greater than 1800 for the dearomatized distillate, and 0.92 for the blended product. In addition, the major aromatic components in the blended product are clearly visible in the TIC (Fig. 3). For other products, however, the classification is much more ambiguous. Figures 7 and 8 show examples of mid-range products that do not fall neatly within the defined classes. Figure 7 shows a product with stronger than expected aromatic content, having a calculated ratio of 1.95 (Method 1). Conversely, Fig. 8 shows a product with lower than expected aromatic content, although aromatic compounds are still present, with a recognizable profile. The calculated ratio for the product depicted in Fig. 8 is 38.8 (Method 1).

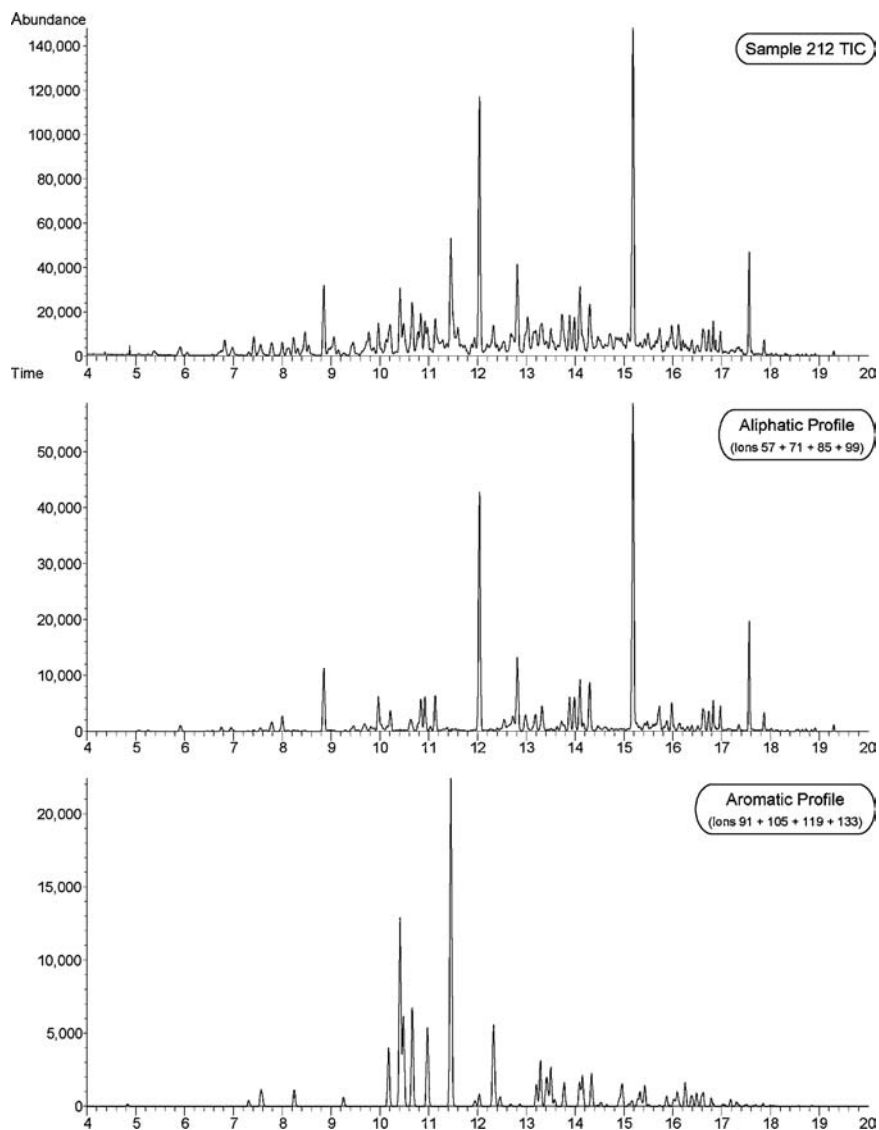


FIG. 7—Data for a distillate with a relatively high aromatic content (Liquid 212).

Another unusual product is Liquid 92, which is shown in Fig. 9. Information from the manufacturer indicates that this product is a blend of an aromatic product with a medium dearomatized distillate. Examination of the data shows that while the C_3 -alkylbenzene grouping in the aromatic profile is relatively strong, the later aromatics such as the C_4 -alkylbenzenes are much lower than expected. Consequently, ratio values will be deceptive. A careful examination of the aromatic profile should indicate to the analyst that the pattern of the aromatic profile is different from that of a typical distillate (compare the aromatic profile in Fig. 9 with the aromatic profile in Fig. 1). The aromatic profile is a tighter fraction than expected, and also exhibits a greater abundance of the co-eluting 3- and 4-ethyltoluenes relative to the normally more abundant 1,2,4-trimethylbenzene. It is important that the fire debris analyst be aware of the presence of unusual blends, such as this “re-aromatized dearomatized” product, and that the data be carefully considered. In cases such as this, the utility of ratios may be severely limited, regardless of the method used.

After analysis and calculations of each liquid's ratio, a neat delineation between the three classes did not appear obvious in the graph as shown in Figs. 5a and 5b. Occasionally, examination of the natural logarithm (\ln) of the values rather than the values themselves provides more information regarding shifts and trends of a curve. Thus, the natural logarithm of each liquid's ratio was calculated and plotted along with the Method 1 ratios and is shown in Fig. 10.

The natural logarithm helps to point out shifts that are not very apparent in a curve. In this instance, the natural logarithm plot reveals an important shift after liquid 80 (ratio of 14.9). The ratio more than doubles and the following liquid (53) presents a ratio of 38.8. This approximately coincides with a marked change in the slope of the curve and, therefore, can be used as a limit between regular petroleum distillates and dearomatized distillates.

Also, a value below 1 will give a negative natural logarithm. There is only one liquid (115) with a ratio less than 1 and therefore a negative natural logarithm, which is clearly a blended product as shown in Fig. 3. This appears to be a good break between the blended

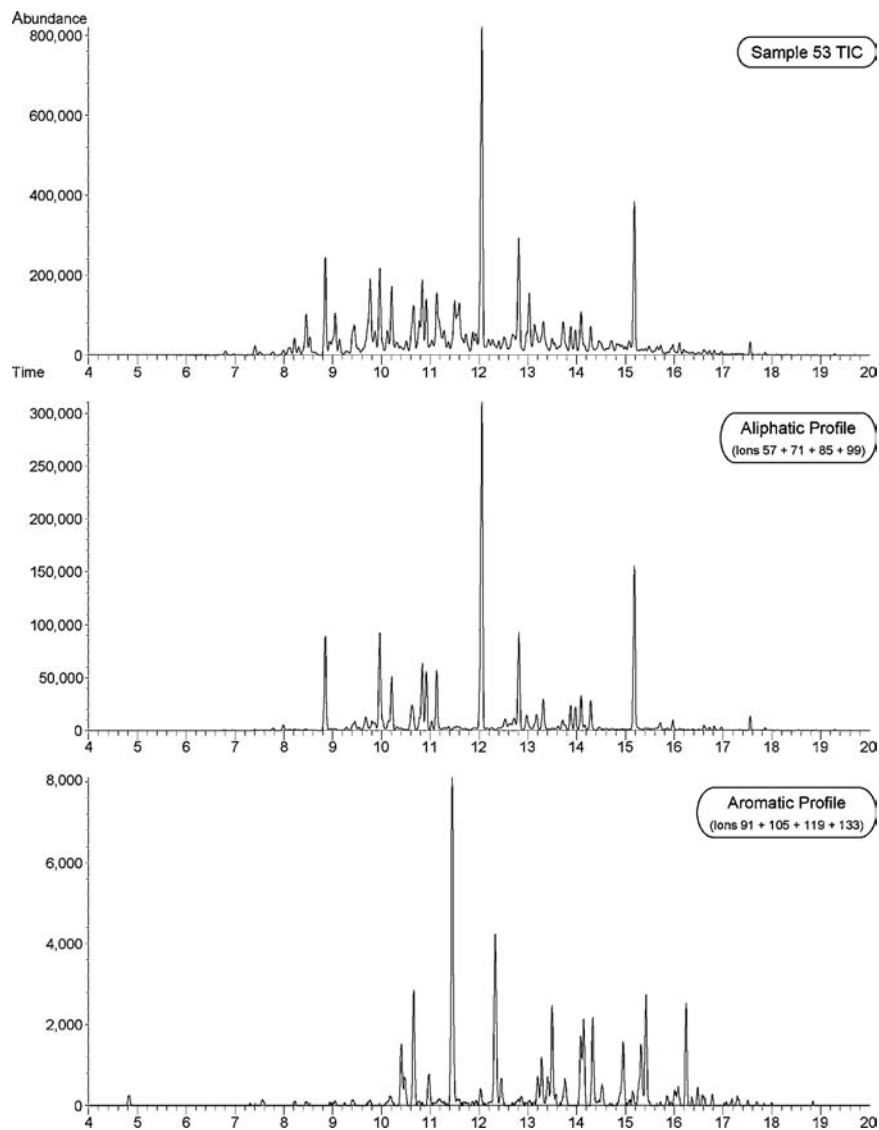


FIG. 8—Data for a distillate with a relatively low aromatic content (Liquid 53).

TABLE 3—Summary of the ratio values used to classify the liquids.

Ratio (R)	ln	Product Type
$0 < R \leq 1$	negative or 0	blended product
$1 < R < 30$	between 0 and 3.4	petroleum distillate
$R > 30$	above 3.4	dearomatized distillate

products and regular distillates. By taking the second derivative of the natural logarithms of the calculated ratios, the change in the slope of the curve can be determined. This more objective approach also shows that the greatest change in slopes does in fact occur near the calculated ratio of 1.

Conclusions

While there was no real demarcation observed between the three categories (MPD, dearomatized products, and blended products), the results obtained permit some boundaries to be set according to the ratio, which are summarized in Table 3. It is recognized that this

classification system is not rigid and is far from perfect; however, it may provide some guidance.

Part 1 of this study focused on the analysis of diluted liquids. They were not weathered, degraded, nor contaminated with interfering products, nor were they subjected to an extraction process. As one would expect, the contribution of the substrate in a real case sample may affect the ratios and limit the applicability of the proposed guidelines with real casework. Similarly, extraction conditions will also be expected to affect the relative recovery of aromatic and aliphatic compounds, and consequently the calculated ratios. These effects will be presented in greater detail in Part 2 of this study.

While the ASTM standards leave the interpretation of the aliphatic:aromatic ratio unclear and make the classification between MPD and dearomatized distillates very difficult in some instances, it is recommended that each laboratory have a written protocol that will ensure that an analyst's determination will be consistent within the laboratory. These protocols or standard procedures may be based upon a system such as that presented in Table 3. It is recognized that this process is relatively straightforward for neat

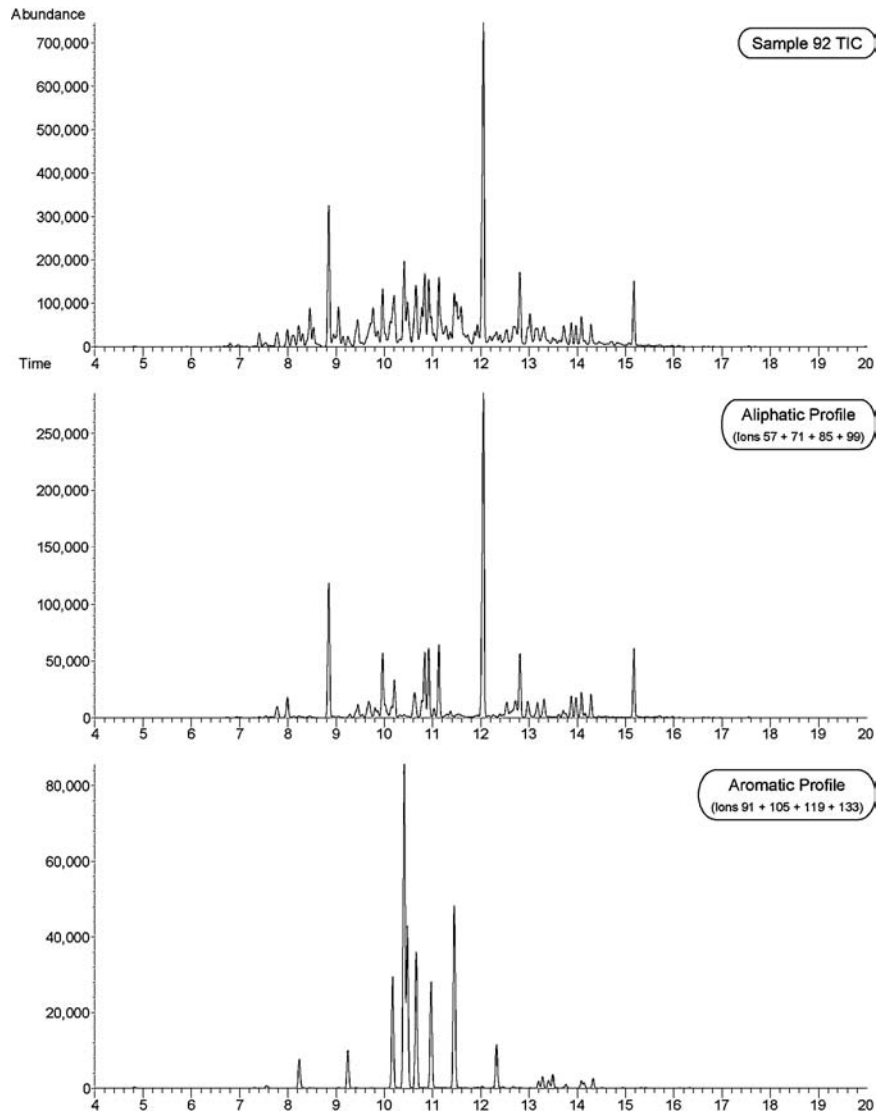


FIG. 9—Data for an unusual blended product (Liquid 92). This product represents a commercially manufactured blend of an aromatic product with a dearomatized distillate.

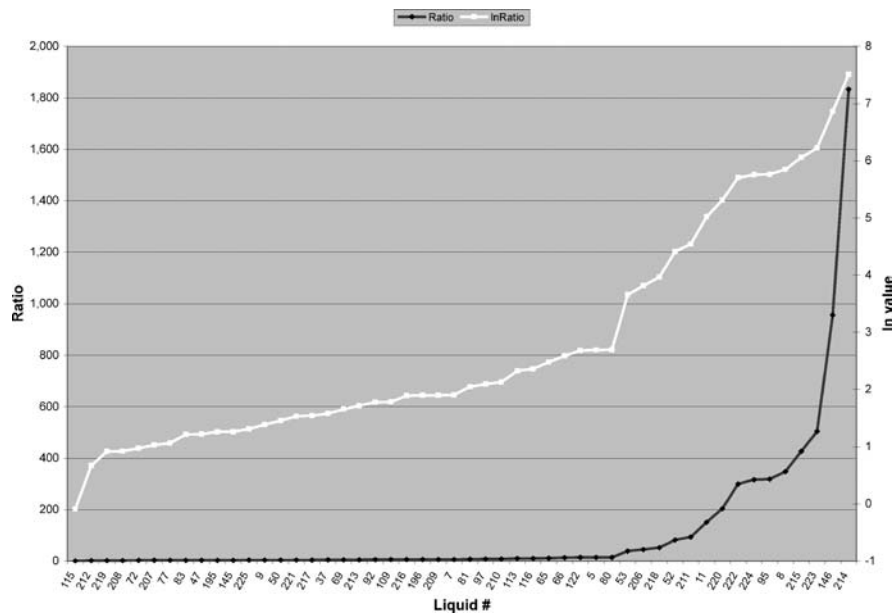


FIG. 10—Graph of the ratios obtained by Method 1 and the natural logarithms of the calculated ratios for all 50 liquids.

liquids, and that it will likely present a much greater challenge for real case samples.

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