

GC/MS Data Interpretation for Petroleum Distillate Identification in Contaminated Arson Debris

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ABSTRACT: Extracted ion profiling ("mass chromatography") and target compound analysis are compared as complementary techniques for the identification of petroleum distillates in fire debris. The positive and negative aspects of both techniques are discussed, including common pitfalls and interpretation suggestions. Neither technique is superior to the other; each has strong qualities where the other has deficiencies. It is recommended that both techniques be applied to GC/MS data for difficult high-background samples.

KEYWORDS: forensic science, fire debris, chromatographic analysis, petroleum distillates, mass spectrometry

GC/MS analysis can be applied to fire debris samples too contaminated for the identification of ignitable liquids by GC alone [1-4]. Contamination is usually the result of pyrolysis of organic materials (wood, carpet, padding, floor tile, etc.) at the fire scene, and adds unwanted peaks to the gas chromatographic pattern [4-8]. When these interfering peaks become of sufficient number and intensity to obscure an accelerant pattern and make petroleum distillate identification impossible, the sample may be referred to the GC/MS laboratory for further analysis. The mass spectrometer's data system is used to "filter out" contaminating species in the chromatogram and produce data that is petroleum distillate related.

Two methods of mass spectral data analysis are commonly used for the identification of petroleum distillate residues in fire debris. The first method is extracted ion profiling, or "mass chromatography" [1]. In mass chromatography, intensity profiles for characteristic ions are displayed and visually compared against profiles for the same ions in known petroleum distillates. The comparison is carried out in much the same manner as with gas chromatography [9]. Characteristic ions consist of the base ion or other strong daughter ion for each of various types of hydrocarbons present in petroleum. Individual characteristic ion profiles may be added to create a multiple ion mass chromatogram for the compound type being sought [1]. In this laboratory, the addition of less intense ion profiles to the base ion profile for a particular compound type has been necessary only on rare occasions. Accordingly, the mass chromatograms illustrated in this paper are base ion profiles. Mass chromatography increases the signal-to-background ratio for the

pattern of interest by reducing contributions from compounds that are not of the same type, and therefore do not have the same major ions. Petroleum hydrocarbon types and their strong ions are listed in Table 1.

The second method of data evaluation is target compound analysis [4,7,10]. By this method, the data system searches specified retention time windows for the mass spectra of specific compounds expected to elute within each window. The mass spectra for these compounds can be defined by up to five ions, but for contaminated arson samples it has been found best to limit these to the base ion and one or two other strong daughter ions. Upon verification of the target compounds, the data system quantitates and prints out a list of those found and their relative peak areas. Comparison of unknowns and standards is facilitated by a graphics program that presents the data as reconstructed chromatograms, or "target compound chromatograms," which are basically stick plots of peak area versus retention time for each compound identified [4,7].

Mass Chromatography

A major hazard of mass chromatography is that each petroleum hydrocarbon type in Table 1 might be visualized and compared with the same hydrocarbon type in the petroleum distillate standard without regarding its relationship to other hydrocarbon types within the sample. Figure 1 shows the mass chromatograms for the trimethylbenzene/ethyltoluene/propylbenzene (C₃-alkylbenzene) family of hydrocarbons (extracted ion mass [m/z] 105) present in

TABLE 1—Hydrocarbon types and ions useful for mass chromatography of petroleum distillates.

Hydrocarbon type	Major ions
Aliphatic:	
Alkanes	43,57,71,85
Alkylcyclohexanes	82,83,55
Decalins	138,96,81
Aromatics:	
Toluene	91,92
Ethylbenzene, xylenes	91,106
C3 Alkylbenzenes	105,120
Indane	117,118,115
Methylindanes	117,132
C4 Alkylbenzenes	119,134
Naphthalene	128
Dimethylindanes	131,146
C5 Alkylbenzenes	133,148
Methylnaphthalenes	142,141
C2 Alkylindaphthalenes	156,141
C3 Alkylindaphthalenes	170,155

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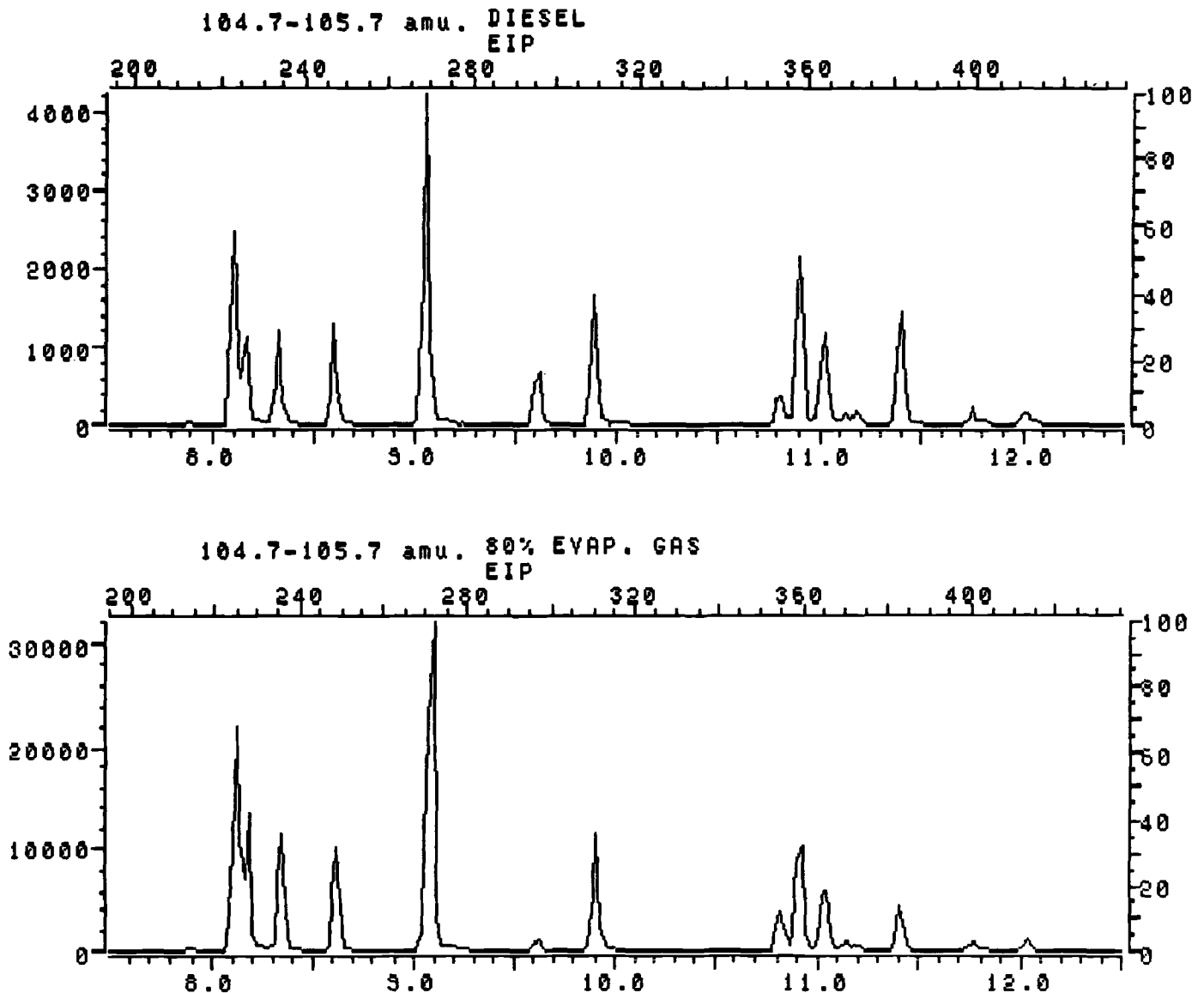


FIG. 1— C_3 -alkylbenzene (m/z 105) mass chromatograms for diesel fuel (top) and weathered gasoline (bottom).

diesel fuel and gasoline. Except for minor differences in the peak heights, the two patterns are equivalent. Mass chromatograms such as these can be used to support the presence of a petroleum distillate in fire debris, but they cannot be used to classify the petroleum distillate until their relationship to other mass chromatograms (such as the alkane m/z 57 pattern) is determined.

Another hazard is that petroleum like isomer profiles may not originate from petroleum distillate at all. Figure 2 shows the indane/methylindane pattern (m/z 117) for a carpet padding pyrolyzate and a comparison petroleum distillate (gasoline). Although the pyrolyzate has major components not present in gasoline, the indane and methylindane isomers are clearly present in ratios similar to those for petroleum distillate. Figure 3 shows the C_4 -alkylbenzene patterns (m/z 119) for the same sample and standard. Again, the pyrolyzate pattern, although weak, has many characteristics in common with petroleum distillate. These patterns illustrate the hazard of basing an identification on just one or two extracted ion profiles. Petroleum distillate pattern matches of this type occur frequently in polymer pyrolyzates, and make it evident that these isomer patterns are not characteristic of petroleum distillate alone.

Isomer ratios are apparently established by factors that go into the compound's production, and may be a characteristic of the compound itself and not its source.

The hazards inherent in mass chromatography can be overcome by increasing the number of ion masses profiled. The greater the number of different mass chromatograms that match between an arson sample and a petroleum distillate, the greater the confidence that the petroleum distillate is present. Because of the possibility that background isomer profiles will produce petroleum distillate-like patterns, the relative abundances for *unrelated* hydrocarbons (eluting in the same retention time region) should be evaluated. This can be done by dividing the ion count intensity (Y-axis) numbers for mass chromatogram "A" by that for mass chromatogram "B" and seeing if this ratio is the same for the sample and standard. In the above example, the "indane" to " C_4 -alkylbenzene" ratio is $1400/300 = 4.6$ for the pyrolyzate and $7000/12000 = .58$ for gasoline. The unknown is clearly shown not to be consistent with gasoline. Another way of doing this is by adding the two mass chromatograms to produce a composite profile that contains peaks of both hydrocarbon types. This process creates a new pattern

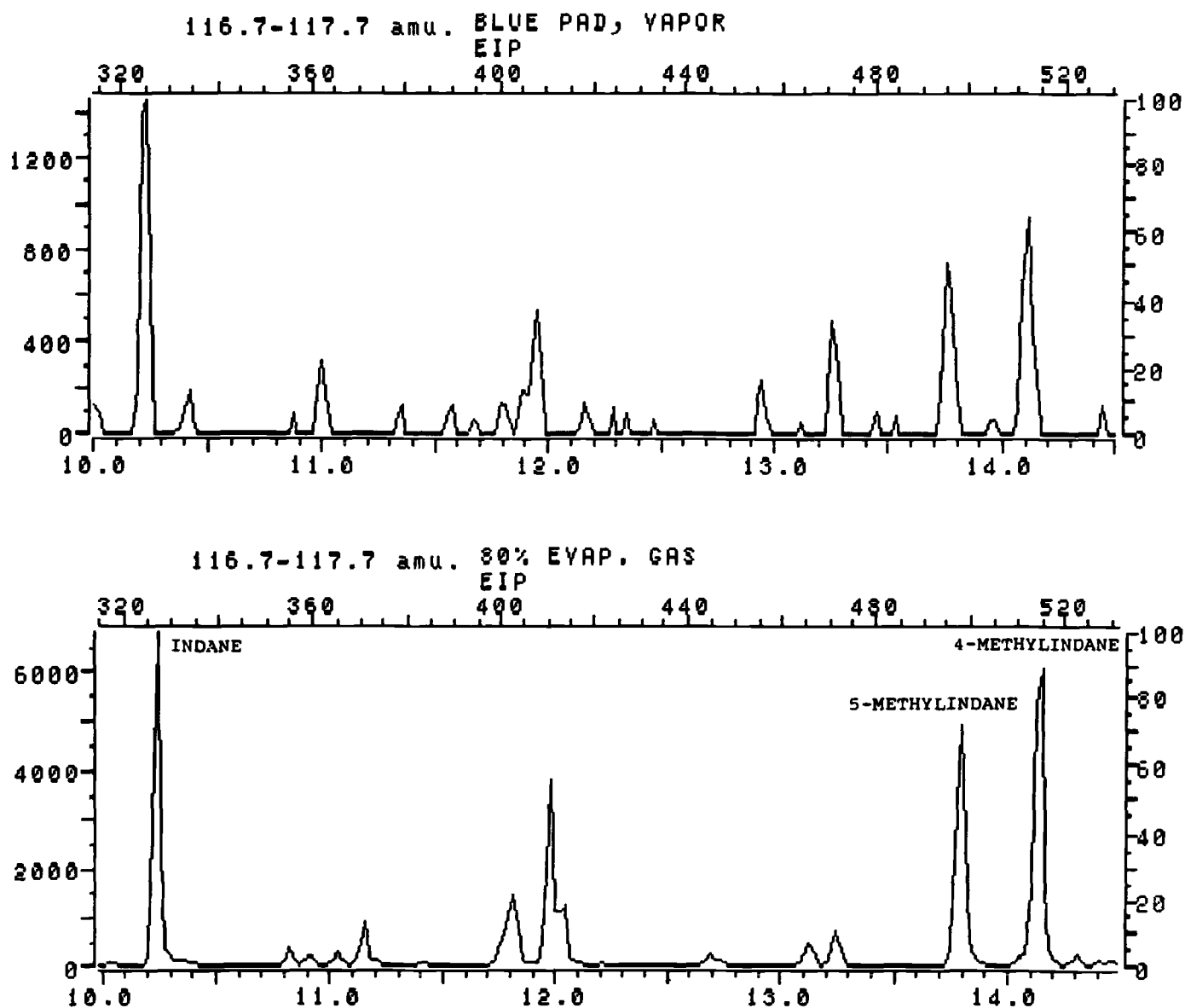


FIG. 2—Indane (m/z 117) mass chromatograms for carpet padding pyrolyzate (top) and weathered gasoline (bottom).

that shows relative abundances for the unrelated compounds and provides another means of visually comparing sample with standard. Figure 4 shows the result of adding the indane and C_4 -alkylbenzene mass chromatograms for the carpet padding pyrolyzate and gasoline standard, respectively. These composite mass chromatograms show obvious differences that would prevent misidentification of the pyrolyzate. In situations where the resulting patterns are still compatible, the procedure can be repeated by co-adding mass chromatograms for additional hydrocarbon types, until the sample no longer matches the standard or until sufficient hydrocarbon types are patterned to support an identification of the unknown. The disadvantage to this procedure is that extracted ion profiling does not distinguish between a designated mass ion arising from petroleum hydrocarbons and the same mass ion arising from non-petroleum sources. All of the contaminant compounds that contain the extracted ion are profiled along with the compounds of interest, resulting in mass chromatograms containing extraneous peaks that may mask the pattern of interest. An improvement over mass chromatography would be a method that extracts ion profiles for only the compounds of interest, and rejects ion contributions

from other sources. Target compound analysis does not completely fulfill this ideal, but it can help.

Target Compound Analysis

Target compound analysis cleans up the profiles to be compared by including only relevant information. The method is more selective than mass chromatography because the requirements for compound identification are more stringent. Not only must the major ion for the compound elute at the proper retention time, but other daughter ions must also be present at the same retention and have the correct intensities relative to the major ion. For mass chromatography to have comparable specificity, multiple ion profiles would have to be extracted and displayed for each compound and checked visually for ion intensity and retention agreement. Target compound software does this qualitative checking internally. Identified compounds are then quantitated and displayed as a reconstructed target compound chromatogram (TCC) that can be visually compared with petroleum distillate standards treated in the same manner. Target compound chromatograms simplify the comparison

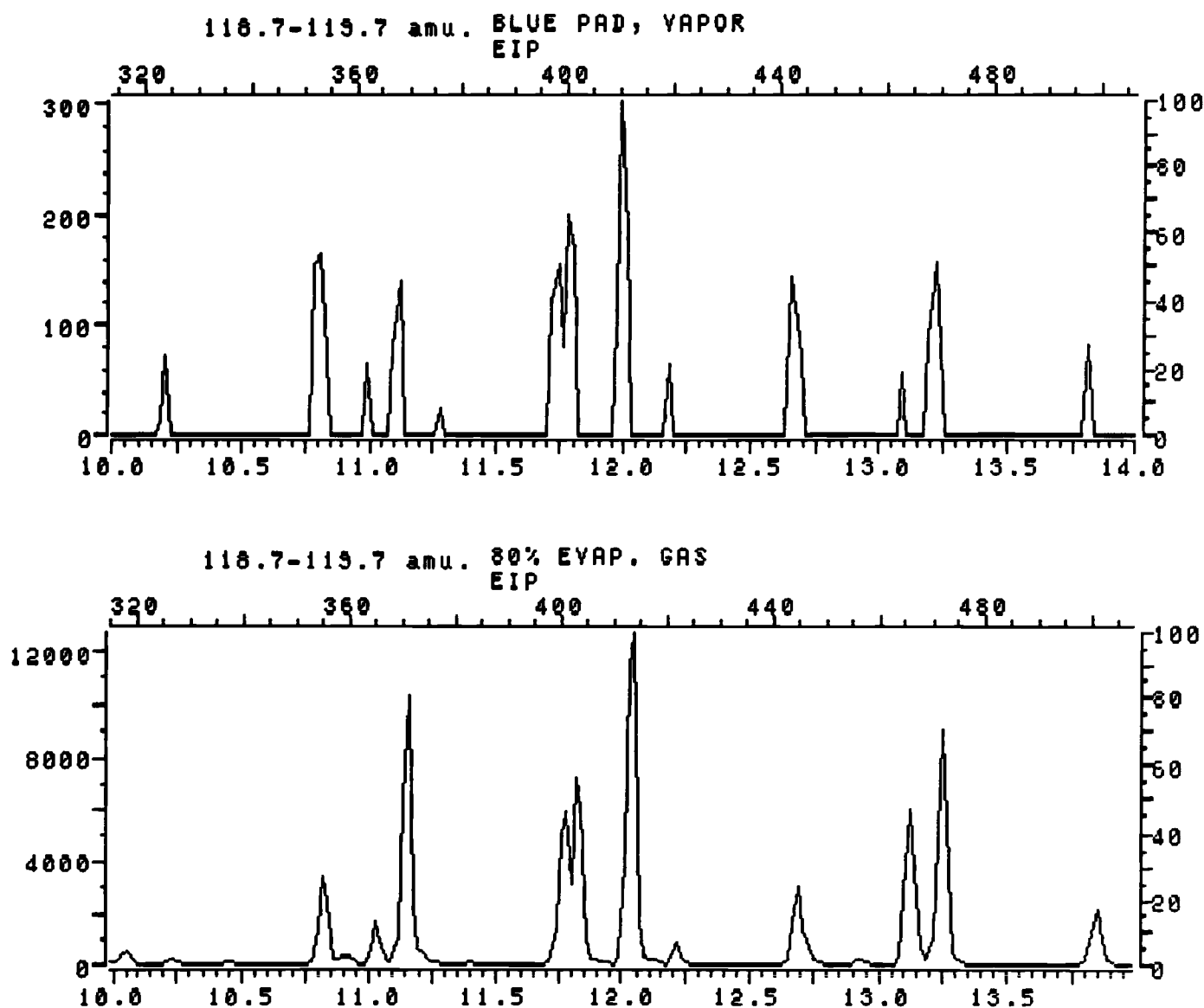


FIG. 3— C_1 -alkylbenzene (m/z 119) mass chromatograms for carpet padding pyrolyzate (top) and weathered gasoline (bottom).

of sample with standard by including all fifteen hydrocarbon types listed in Table 1 in a single pattern. The relative abundance for each hydrocarbon type, as well as the abundance ratios for selected isomers within each hydrocarbon type, are graphically represented. Target compound chromatograms for typical petroleum distillate standards (weathered gasoline, weathered mineral spirits, and diesel fuel) are shown in Fig. 5. All of the target compounds and their identification criteria are listed in Table 2. For high-background arson samples, these patterns are free from contamination by compounds that are not of interest. The only possible background interference is from target compounds that are themselves generated by pyrolysis at the fire scene, a frequent problem that cannot be avoided by any type of data manipulation.

There are three disadvantages to target compound analysis. The first is that the number of pattern comparison points is limited. This laboratory's current petroleum hydrocarbon list for arson analysis contains 43 entries, consisting of those compounds for which we had known standards. In contrast, a typical series of mass chromatograms may contain hundreds of points of comparison. More points of comparison can be added to the TCC by

empirically defining unidentified petroleum compounds by retention time and mass spectra. This was done previously for the MPD target compound list [4]. The second disadvantage is lower sensitivity. The base ion for each target is by definition the most intense (sensitive) ion fragment for that compound. Other daughter ions useful for target compound analysis generally range from 20 to 99 percent of the base ion intensity. If one requires the presence of an ion having an intensity of 20 percent relative to the base ion for target compound identification, sensitivity is automatically reduced by eighty percent relative to the base ion. The third disadvantage is a possible increase in false negative findings due to the greater specificity. Because two (or three) ions are required for compound identification, the random probability that a co-eluting compound would have one of these ions is greater than it is with the base ion alone. This increases the likelihood that the compound will not be recognized by the data system due to distortion of the mass spectral data.

The retention times and relative ion intensities listed in Table 2 were obtained using the GC/MS conditions shown in Table 3. Table 2 (with a few additions) is a composite of the three separate

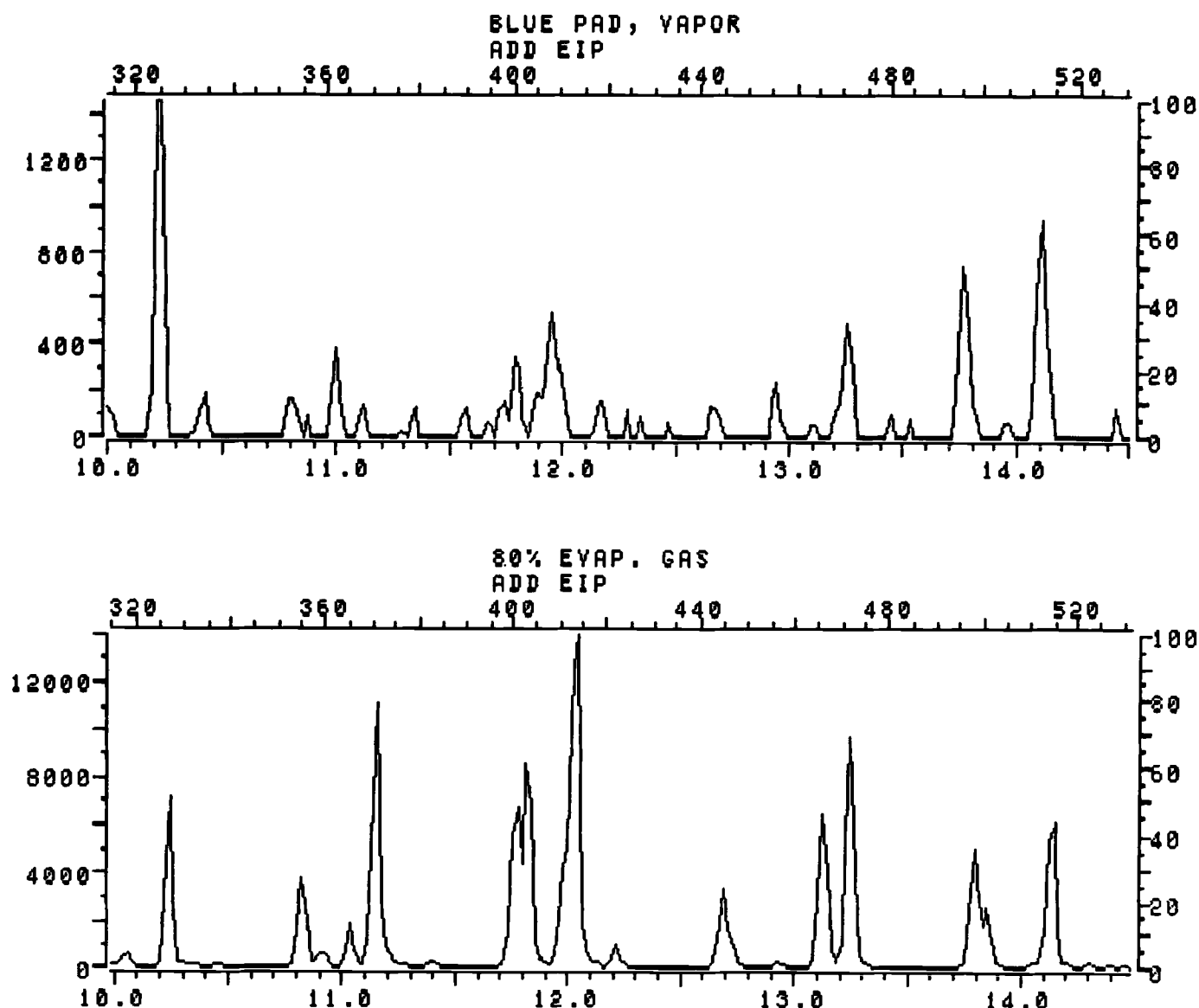


FIG. 4—Summed mass chromatograms (m/z 117 + 119) for carpet padding pyrolyzate (top) and weathered gasoline (bottom).

compound identification files (I.D. files) previously used for the identification of gasoline, medium petroleum distillate (MPD), and heavy petroleum distillate (HPD), respectively [4,7]. Since this process was first published, the chromatographic temperature program for the analysis of arson debris was modified to encompass all three accelerant classes in a shortened run. In addition, a single user program (procedure file) was adopted for the generation of target compound chromatograms, instead of separate procedure files used previously for the three classes. Combining the targets into a single "petroleum distillate" file simplified the process of distillate identification. With all three distillate classes on the same TCC, the relative presence of each is readily apparent. In certain situations, it is even possible to recognize the presence of mixtures of classes in weathered and contaminated fire debris.

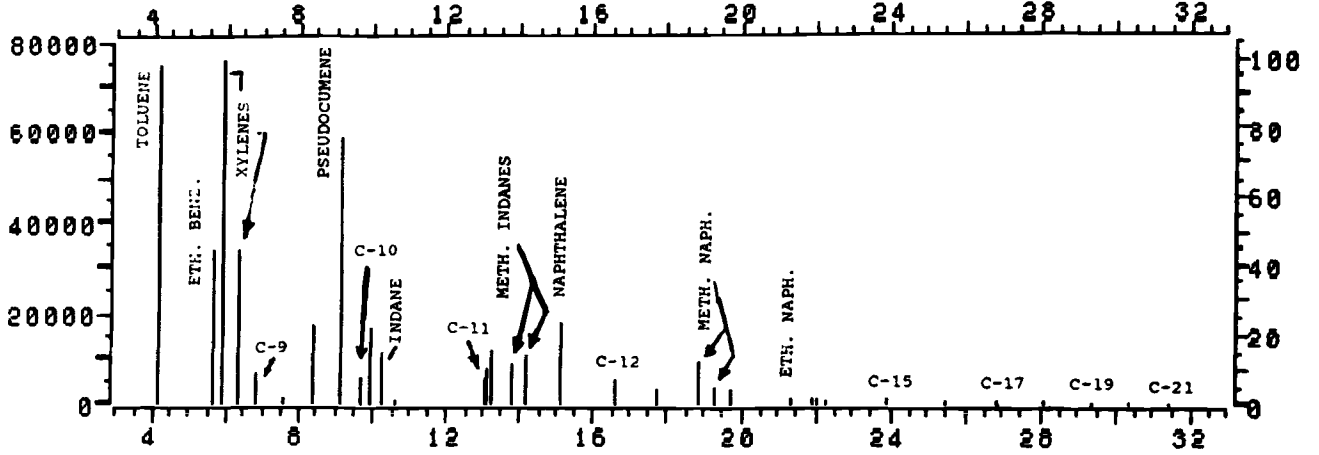
Applications

All 43 target compounds are present in both gasoline and heavy petroleum distillates. Although medium petroleum distillates have a wide range of product variation, they usually consist of tighter

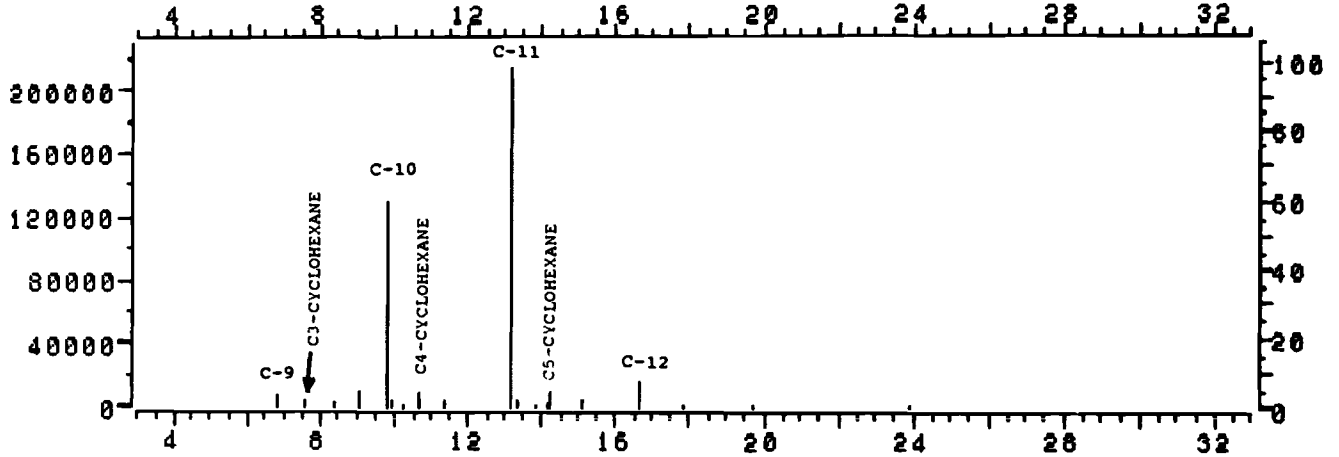
"cuts" than gasoline or HPDs, and may not contain all of the compounds used for petroleum distillate identification. The three classes can be distinguished by the relative amounts and distributions for the aliphatic and aromatic hydrocarbons present. MPDs and HPDs are characterized by their normal alkane distributions, whereas gasoline is identified by its aromatic compound profile.

A comparison of the relative peak heights for closely eluting aromatic and aliphatic compounds can help in ascertaining which class of petroleum distillate is present in high background samples. For example, the *o*-xylene/nonane, pseudocumene/decane, and isodurene/undecane ratios are characteristic. These ratios in gasoline are greater than 1:1, whereas in HPD they run less than 1:1. The actual ratios can vary significantly among producers as well as with the season, complicating the identification of mixtures. Typical values, calculated on the basis of base ion peak area, are shown in Table 4. Figure 6 illustrates this comparison by mass chromatography. A primary consideration in this type of analysis is that the peaks to be compared have close retention times (within 1 minute of each other) to minimize the effects of weathering on

75% EVAP. GAS



75% EVAP. NASCO



DIESEL

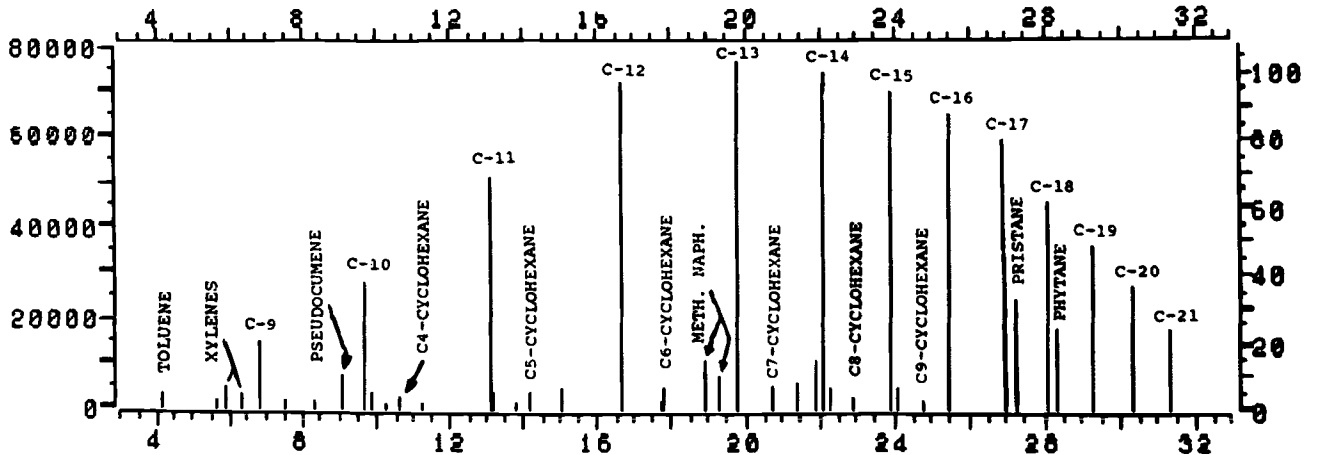


FIG. 5—Target compound chromatograms (TCCs) for 75 percent evaporated gasoline, 75 percent evaporated mineral spirits, and fresh diesel fuel. Unlabelled compounds may be found in Table 2.

TABLE 2—Petroleum distillate target compounds.

Compound	Retention time (min.)	m/z Ion	Relative abundance
Toluene	4.12	91	100
		92	60
Ethylbenzene	5.65	91	100
		106	25
M-, P-Xylenes	5.86	91	100
		106	50
		105	25
O-Xylene	6.30	91	100
		106	45
		105	20
Nonane	6.81	57	100
		85	50
		71	35
Propylcyclohexane	7.53	83	100
		82	60
1,3,5-Trimethylbenzene	8.32	105	100
		120	50
1,2,4-Trimethylbenzene	9.08	105	100
		120	45
Decane	9.64	57	100
		71	40
		85	30
1,2,3-Trimethylbenzene	9.90	105	100
		120	45
Indane	10.25	117	100
		118	55
		115	35
Butylcyclohexane	10.63	83	100
		82	70
Trans-Decalin	11.30	138	100
		96	65
		81	55
Undecane	13.09	57	100
		71	55
		85	35
1,2,4,5-Tetramethylbenzene	13.10	119	100
		134	50
1,2,3,5-Tetramethylbenzene	13.23	119	100
		134	50
5-Methylindane	13.80	117	100
		132	40
4-Methylindane	14.12	117	100
		132	40
Pentylcyclohexane	14.18	83	100
		82	80
		55	70
Naphthalene	15.05	128	100
Dodecane	16.64	57	100
		71	65
		85	50
4-7-Dimethylindane	17.70	131	100
		146	40
Hexylcyclohexane	17.86	83	100
		82	80
		55	60
2-Methylnaphthalene	18.87	142	100
		141	80
1-Methylnaphthalene	19.29	142	100
		141	85
Tridecane	19.73	57	100
		71	65
		85	40
Heptylcyclohexane	20.71	83	100
		82	85
Ethyl-naphthalene	21.38	141	100
		156	45
1,3-Dimethylnaphthalene	21.92	156	100
		141	90
Tetradecane	22.02	57	100
		71	65
		85	50

TABLE 2—Continued

Compound	Retention time (min.)	m/z Ion	Relative abundance
2,3-Dimethylnaphthalene	22.32	156	100
		141	90
Octylcyclohexane	22.91	83	100
		82	90
Pentadecane	23.92	57	100
		71	70
		85	50
2,3,5-Trimethylnaphthalene	24.03	170	100
		155	90
Nonylcyclohexane	24.68	83	100
		82	80
Hexadecane	25.45	57	100
		71	70
		85	50
Heptadecane	26.84	57	100
		71	80
		85	55
Pristane	26.99	57	100
		71	80
		85	40
Octadecane	28.05	57	100
		71	80
		85	55
Phytane	28.22	57	100
		71	75
		85	60
Nonadecane	29.22	57	100
		71	75
		85	60
Eicosane	30.30	57	100
		71	80
		85	55
Heneicosane	31.35	57	100
		71	80
		85	55

the compound ratio. Another important consideration is that neither compound arise from sources other than petroleum distillate, specifically, contaminants from the debris.

Aliphatic Patterns

When the data is being evaluated for the presence of MPD or HPD, interference usually comes in the form of polyolefin pyrolyzates, which can form "Gaussian" distributions of normal alkanes in patterns very similar to petroleum distillate (Fig. 7). However, polyolefins also produce 1,2-unsaturates (normal alkenes and alkynes) [11] that can be seen in Fig. 7a as major peaks preceding each normal alkane peak. If these unsaturate peaks are present, and there are no branched alkane patterns between the normal alkanes (as seen in Fig. 7b), the presence of MPD or HPD cannot be confirmed. If the branched alkane pattern can be seen, a mixture of polyolefin pyrolyzates and petroleum distillate may exist.

The homologous normal alkylcyclohexanes also form a "Gaussian" distribution that parallels the normal alkane distribution in petroleum distillates. For example, a weathered kerosine whose alkane pattern maximizes at C₁₃ (tridecane) will have an alkylcyclohexane pattern that also maximizes at C₁₃ (that is, at heptylcyclohexane) (compare Figures 7b and 8b). All of the alkylcyclohexanes elute after their normal alkane counterparts, and have ion intensities ranging from ten to twenty percent of the normal alkane intensities (that is, the alkylcyclohexane mass 83: normal alkane mass 57

TABLE 3—GC/MS operating parameters.

Instrument	: Hewlett-Packard Model 5988A Mass Spectrometer with Model 5890 Gas Chromatograph (Hewlett-Packard, Avondale, PA)
Column	: DB-1 nonpolar capillary, 0.32 mm i.d. × 60 M, 0.25 μM methylsilicone bonded phase (J&W Scientific, Folsom, CA)
Carrier	: Helium @ 20 psig (138 kPa), 2.0 cm ³ /min @ 70°C 42 cm/sec linear velocity
Injector	: Split/splitless in splitless mode, splitless time 0.6 min, temp. 260°C
Transfer line	: 250°C
<i>Temperature program</i>	
Initial temperature	: 60°C
Initial hold	: 1 min
First ramp rate	: 4.0°C/min
Intermediate temp.	: 124°C
Intermediate hold	: 0.1 min
Second ramp rate	: 10°C/min
Final temperature	: 260°C
Final hold	: 4.0 min
Total run time	: 34.7 min
<i>Mass spectrometer</i>	
Scan range	: 50–200 amu
Start time	: 3.50 min
Scan cycle	: 1.25 s (16 A/D samples)
Source temp.	: 200°C
Ionization	: Electron impact
Electron energy	: 70 eV
Emission current	: 300 μA
Threshold	: 20 counts

TABLE 4—Typical aromatic/aliphatic ratios for gasoline and diesel fuel.

	O-xylene: Nonane	Pseudocumene: Decane	Isodurene: Undecane
Ions profiled	91/57	105/57	119/57
Diesel fuel	.42	.50	.11
Gasoline	5.5	11.9	2.4
10% Gasoline in Diesel	1.6	1.3	.17

ratio = .1 to .2). These criteria can be used as another way of verifying the presence of petroleum distillate in the presence of interfering normal alkanes arising from pyrolysis. The 1,2-alkenes also produce a mass 83 pattern (Fig. 8a), but these are chromatographically separated from the alkylcyclohexanes and pose no risk of overlap.

Aromatic Patterns

The aliphatic/aromatic ratios can also be affected by pyrolyzates that contain aromatic compounds. Toluene, ethylbenzene, the xylenes, and naphthalene were originally excluded from the target compound list because of the high frequency with which they were found in polymer pyrolyzates [4,7]. They have since been found useful in the identification of mixtures of distillate classes when pyrolysis is not overburdening. Other aromatic target compounds can be produced by pyrolysis as well. Figure 9 shows the target compound chromatogram for the carpet padding pyrolyzate used for Figs. 2, 3, and 4. Thirty-seven of the forty-three petroleum distillate target compounds are present in this sample, including

all but two of the aromatics. Even though individual mass chromatograms for several of the compound types showed similarities with petroleum distillate (as previously illustrated), the complete picture presented by the TCC has no possibility of being misinterpreted as a petroleum product.

Interpretation of TCCs

The two strongest peaks in Fig. 9 are due to naphthalene and ethylbenzene. These have intensities, relative to their neighbors, that are considerably greater than expected for petroleum distillate (compare Fig. 5). These two compounds are obviously of non-petroleum distillate origin, and can be ignored as arising from contamination. The strongest peaks then become the methylnaphthalenes, which, except for their intensity reversal, would indicate the presence of highly weathered gasoline (>95% evaporated). Intensity ratios for the heavier aromatics (ethyl- and dimethylnaphthalenes) are inconsistent with petroleum distillate, and eliminate any possible match with highly weathered gasoline. The lighter aromatics (toluene, xylenes, trimethylbenzenes, and indane) also have intensity ratios that are inconsistent with petroleum distillate. For example, there is far too much indane in relation to the trimethylbenzene pseudocumene, and the ratio for the xylene peaks is reversed. This part of the pattern does not support the presence of either lightly or moderately weathered gasoline. The normal alkane profile contains a Gaussian-like distribution ranging from dodecane through hexadecane, which rules out an identification of MPD. Pristane and two of the alkylcyclohexanes are absent, making an identification of HPD indefensible. A look at the alkane mass chromatogram (m/z 57) showed a pattern equivalent to Fig. 7a, having normal unsaturates and an absence of branched alkanes. The source for the alkanes could be concluded not to be a petroleum distillate, and to be consistent with pyrolysis products.

Figure 10 shows the target compound chromatogram for a 1:10 (v/v) mixture of weathered gasoline with diesel fuel. Diesel fuel is easily identified by its normal alkane profile (including pristane and phytane) and concomitant alkylcyclohexane profile. The alkane mass chromatogram (not shown) is consistent with Fig. 7b, although broader. The aromatic profile also matches that for petroleum distillate. The relatively high intensities for the early eluting aromatics, however, is inconsistent with HPD or MPD, and indicates the addition of gasoline (compare Fig. 5). This conclusion is supported by the aromatic/aliphatic ratios for this sample, shown in Table 4.

In actual casework, the target compound chromatogram will generally fall somewhere between the extremes represented by the previous two examples (Fig. 9 and 10). In positive samples, there is usually some degree of contamination, resulting in the overlap of the petroleum distillate pattern with an unknown background pattern. Because the background can contribute more to some target compound intensities and less to others, the petroleum distillate pattern may be skewed. This deviation will also be seen in corresponding mass chromatograms. The degree of contamination can affect the degree of certainty with which the analyst reaches a conclusion. For high background samples, analysts should draw on as much data as they can reasonably obtain to support a finding. In this regard, mass chromatography and target compound chromatography should be used as complementary techniques, with mass chromatograms providing detailed information and target compound chromatograms providing a better overall picture. When GC/MS analysis is deemed necessary for fire debris samples too contaminated for identification by GC alone, both methods of data analysis should be applied.

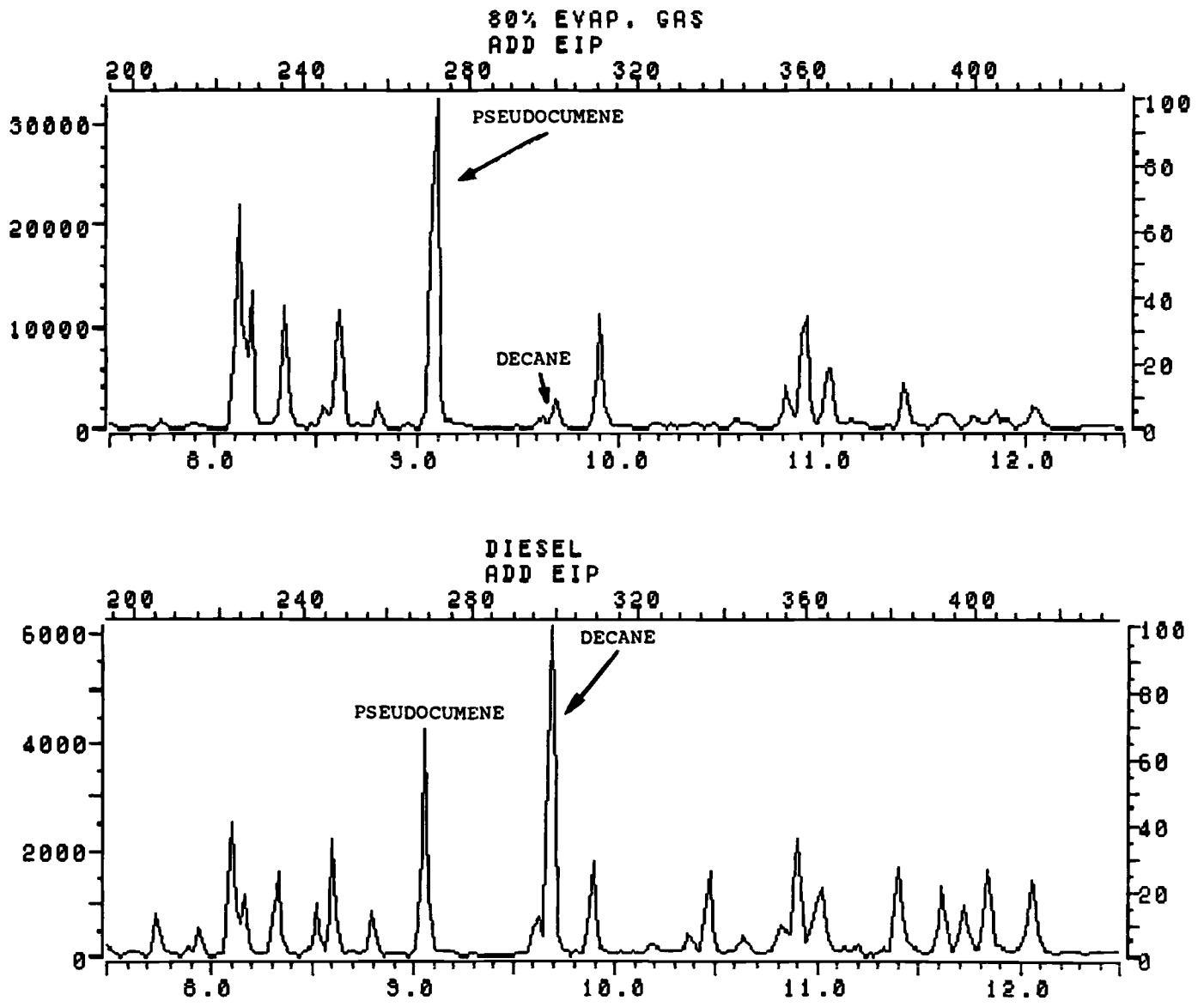
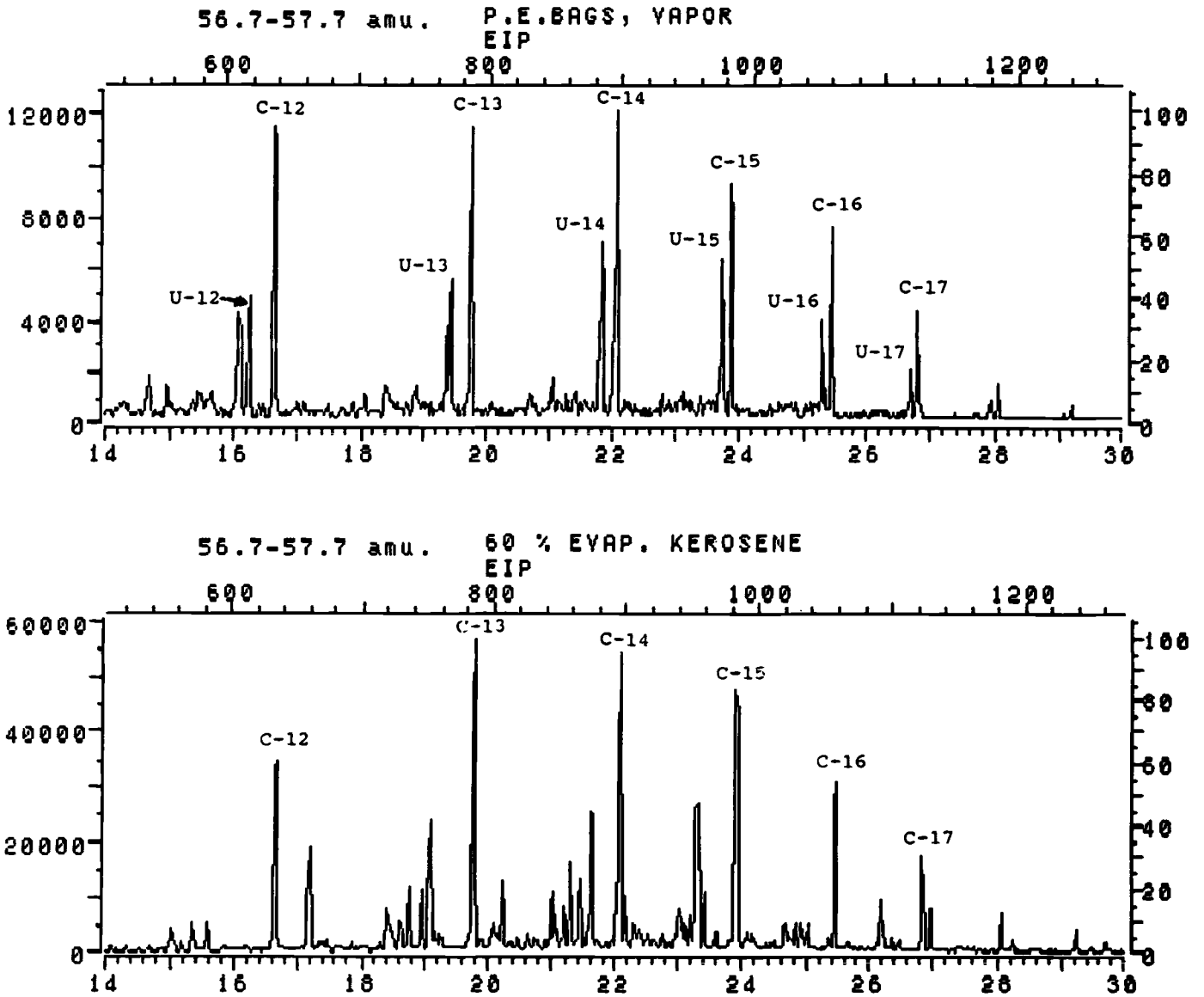


FIG. 6—Summed mass chromatograms (m/z 105 + 57) for weathered gasoline (top) and diesel fuel (bottom). Note aromatic/aliphatic (pseudocumene/decane) ratios.



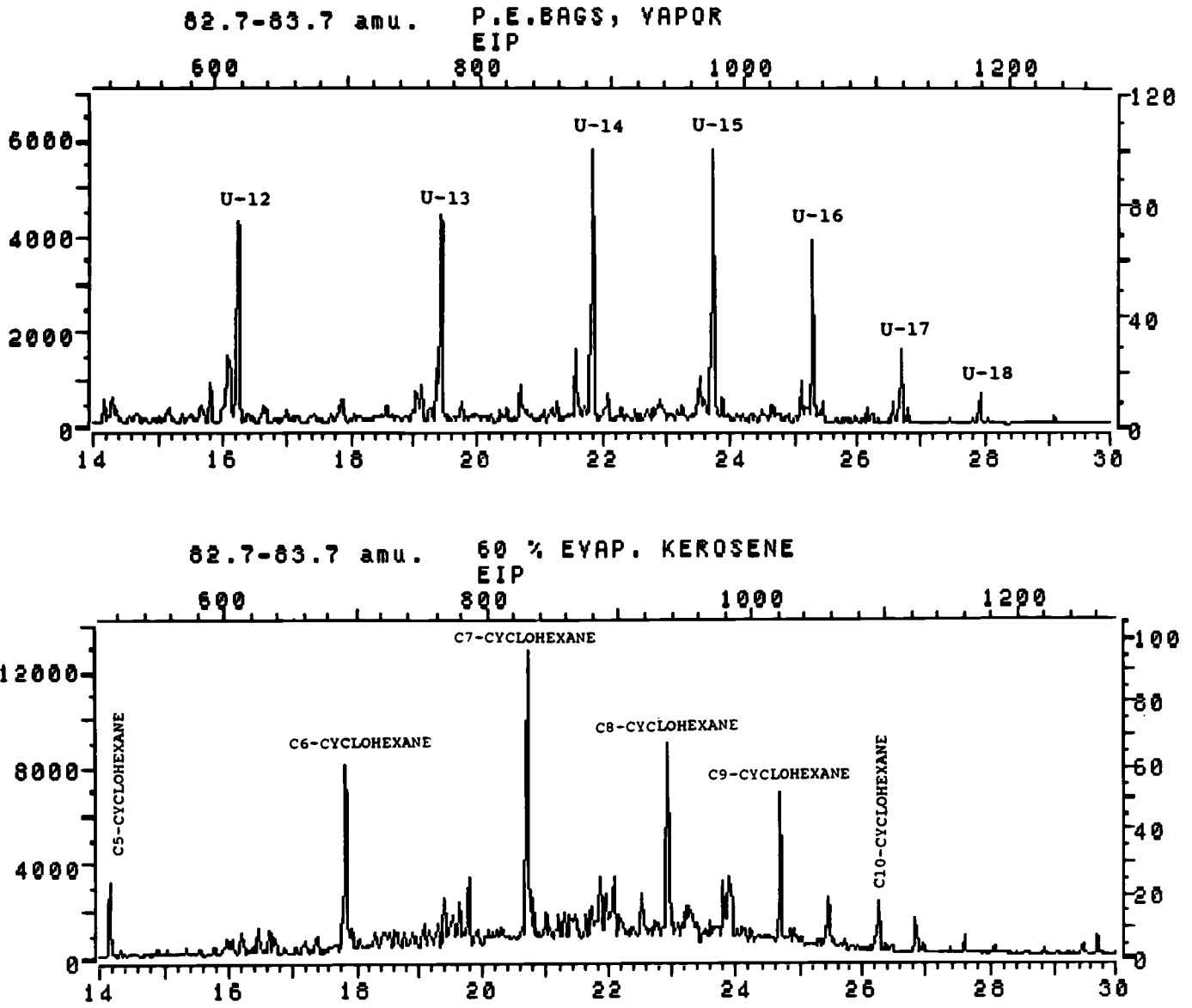


FIG. 8—Mass chromatograms (*m/z* 83) for polyethylene pyrolyzate (top) and weathered kerosene (bottom). U = normal 1,2-unsaturates in pyrolyzate.

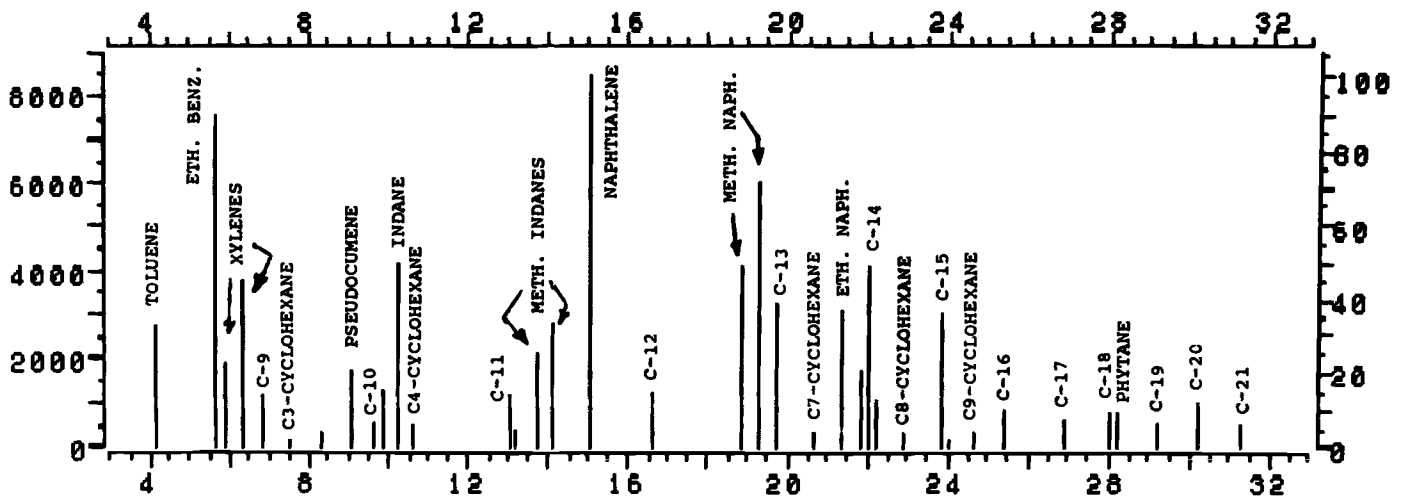


FIG. 9—TCC for carpet padding pyrolyzate.

1:10 88GAS:DIESEL

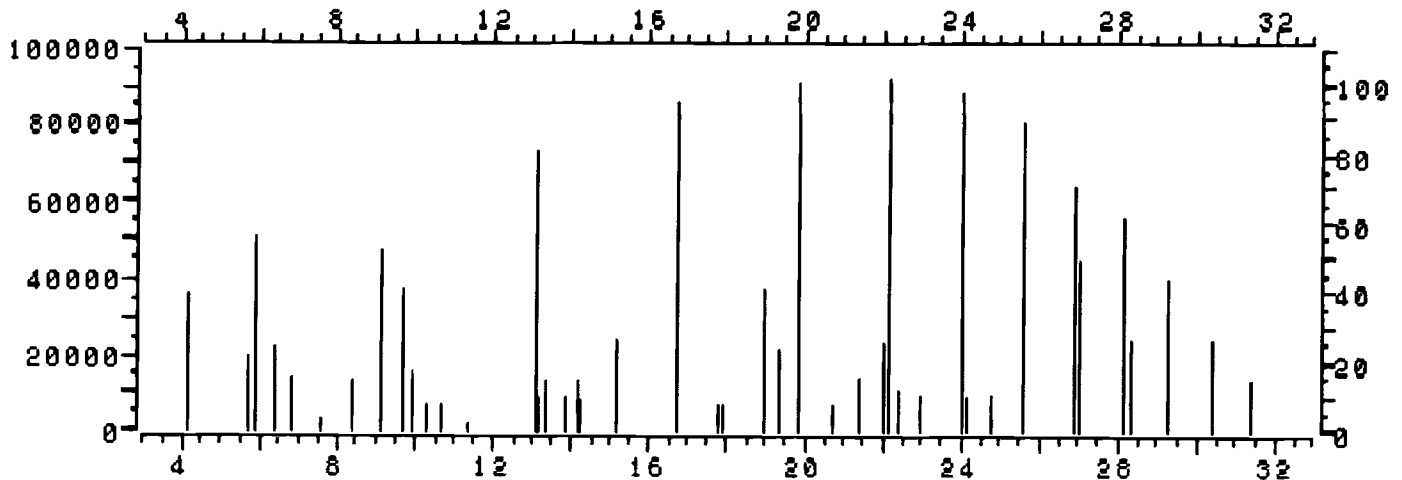


FIG. 10—TCC for a mixture of 10 percent weathered gasoline in diesel fuel.

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