

# TECHNICAL NOTE

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## Analysis of Fire Debris Samples by Gas Chromatography/Mass Spectrometry (GC-MS): Case Studies

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**ABSTRACT:** Gas chromatography/mass spectrometry (GC-MS) is a powerful tool for analyzing fire debris samples for the presence of accelerants. By generating ion profiles for specific chemical classes, GC-MS allows conclusive determinations to be made in some situations where GC-FID is insufficient. Three case examples that illustrate the use of GC-MS and ion profile techniques are presented here.

**KEYWORDS:** forensic science, arson, accelerants, chemical analysis

In a previous paper, the author discussed how gas chromatography/mass spectrometry (GC-MS) can be used to analyze fire debris samples for the presence of accelerants [1]. In it, I described how the analyst can determine whether a petroleum distillate or petroleum-based product is present. If such an accelerant is present, the analyst can classify it utilizing a set of guidelines that are based not only on the total ion chromatogram of the sample but also on four sets of ion profiles that correspond to the alkane, cycloalkane, aromatic, and naphthalene chemical classes [1].

In this paper, I will demonstrate how this technique is applied to real fire debris samples. Three examples are presented which illustrate how GC-MS can be used to make conclusive determinations in situations where conventional gas chromatography/flame ionization detection (GC-FID) analysis may not be sufficient.

### Experimental Procedure

Samples are routinely submitted to this laboratory in sealed metal paint cans and processed using the dynamic headspace sampling technique [2]. Charcoal tubes are prepared by placing approximately 30 mm of 50/200 mesh activated charcoal between cotton plugs in a 5¼ in. glass disposable pipet. Two holes are punched into the top of the can and a charcoal tube is inserted into each hole. One of the tubes is connected to a vacuum

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pump. The sample container is placed in a 90°C oven and vacuum is applied for 20 min. The sample charcoal tube is removed and extracted with 0.5 mL of carbon disulfide. Approximately 0.5  $\mu$ L of the eluent is injected into a Hewlett-Packard 5890 gas chromatograph equipped with a Model 5970 mass selective detector. The gas chromatographic conditions are as follows:

Column: 25 m by 0.2 mm HP-1 (methyl silicone), 0.5- $\mu$ m film thickness  
Injector temperature: 250°C  
Transfer line temperature: 280°C  
Oven temperature: 50°C initially for 2 min,  
ramp 10°/min to 250°C,  
hold at 250°C for 10 min  
Carrier gas: helium at 12 psi (85 kPa)  
Split ratio: 35:1

Data were collected on a Hewlett-Packard Chem Station running revision 3.2 software and macro programs written by this author.

### Case 1

Sample 1 contained charred brown paper and cardboard from the scene of a warehouse fire. The total ion chromatogram of Sample 1 shows a strong gasoline pattern (Fig. 1). The sample pattern and a gasoline standard differ mainly in the presence of a large peak at 7.5 min (marked with a star). With conventional GC-FID, the analyst can do little more than report gasoline present.

Figure 2 gives the alkane and aromatic ion profiles for a gasoline standard and Fig. 3 shows the alkane and aromatic profiles of Sample 1. When comparing these figures, it becomes apparent that, while the aromatic ion profiles of the sample and gasoline standard correspond, the alkane ion profiles differ substantially—both qualitatively and quantitatively. In the gasoline standard, the maximum abundance of the aromatic ion profile is about three times the maximum abundance of the alkane ion profile. However, in the case sample the alkane ion profile is about 1.2 times more abundant than the aromatic ion profile. Clearly, Sample 1 contains alkane compounds beyond what is normally expected to be present in gasoline.

The alkane ion profile of Sample 1 corresponds very well to that of a "Isopar" type of product, such as Gulflite brand charcoal starter fluid (Fig. 4). Isopars are catalytically synthesized products containing only isoparaffinic compounds. They are frequently used in such products as charcoal starter fluids and odorless mineral spirits because of their stability and lack of odor.

The presence of an Isopar in Sample 1 was confirmed by comparing the mass spectra of the individual peaks at 6.2, 6.4, 6.6, and 7.5 min with corresponding peaks in a Gulflite charcoal starter standard. Based on this GC-MS analysis that utilized ion profiling and peak identifications, Sample 1 was reported as containing a mixture of gasoline and an Isopar product.

### Case 2

Sample 2 consisted of slightly burned wood. The total ion chromatogram of Sample 2 (Fig. 5) does not correspond to any standard accelerant. There are some indications of gasoline present, namely, a peak around toluene (4 min) and peaks in the xylene region (6 min). However, the higher aromatics characteristic of gasoline in the 8-min region are

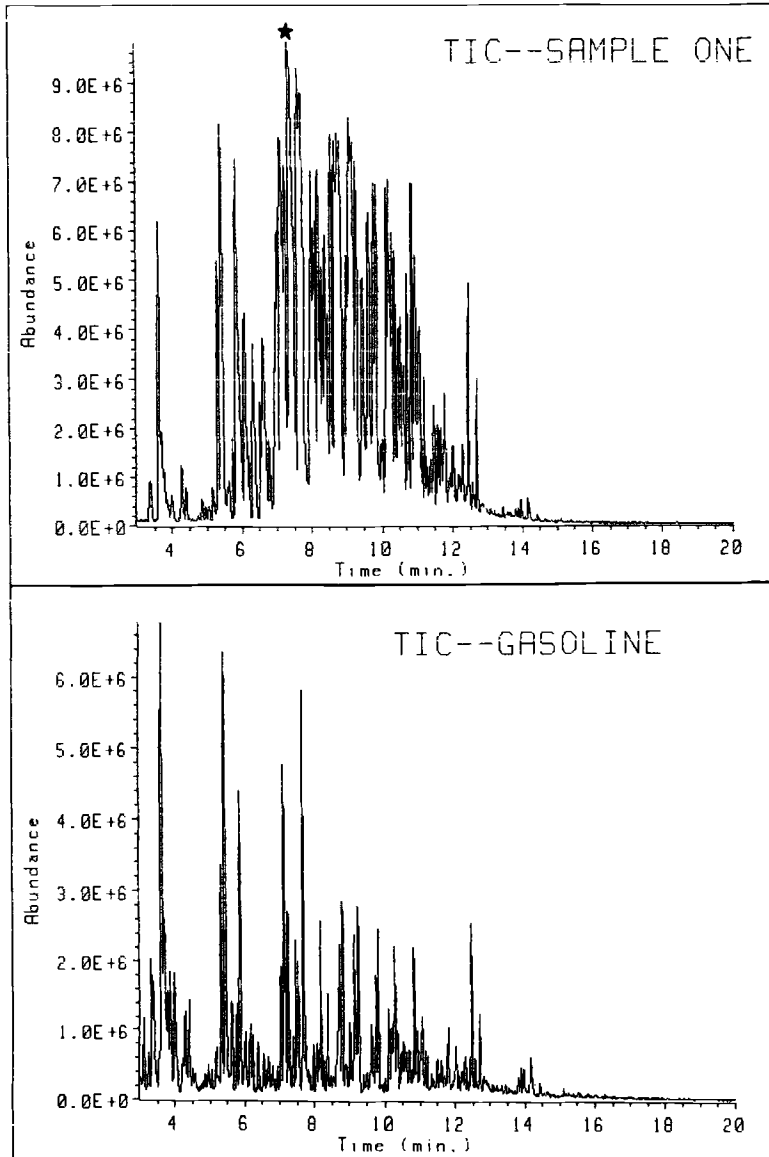


FIG. 1—Total ion chromatograms of Sample 1 (top) and a gasoline standard (bottom).

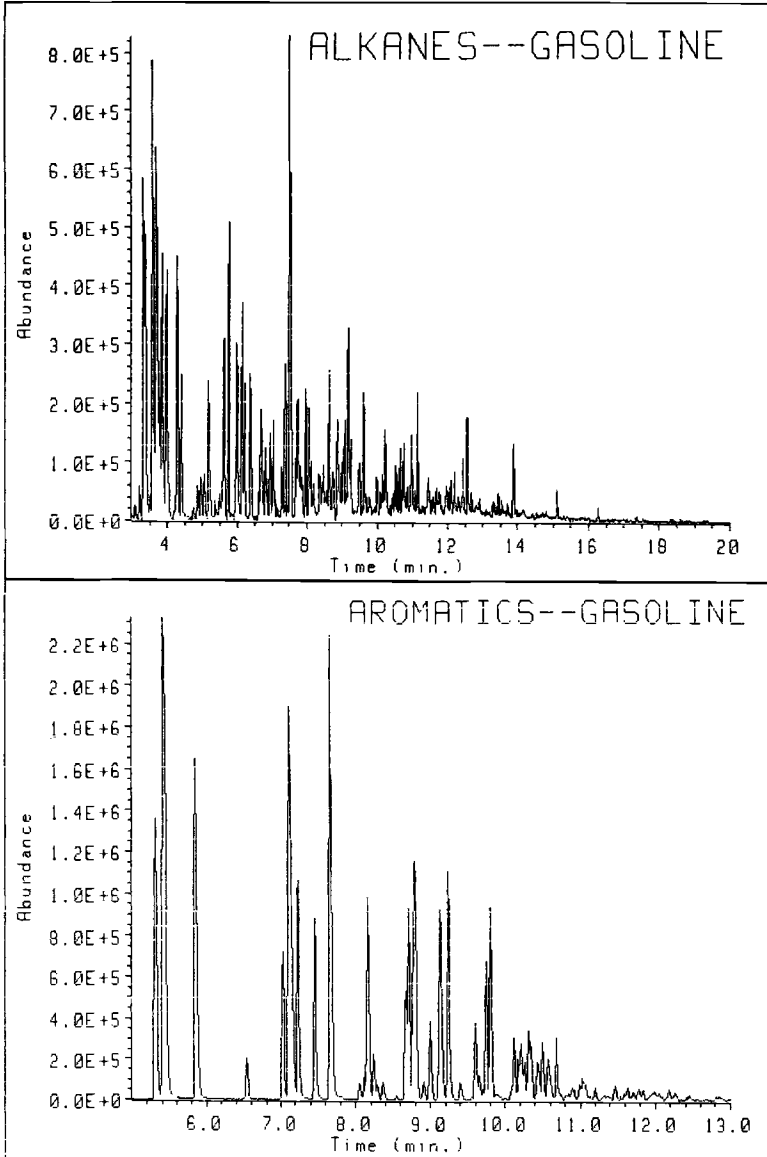


FIG. 2—Alkane ion profile (top) and aromatic ion profile (bottom) of a gasoline standard.

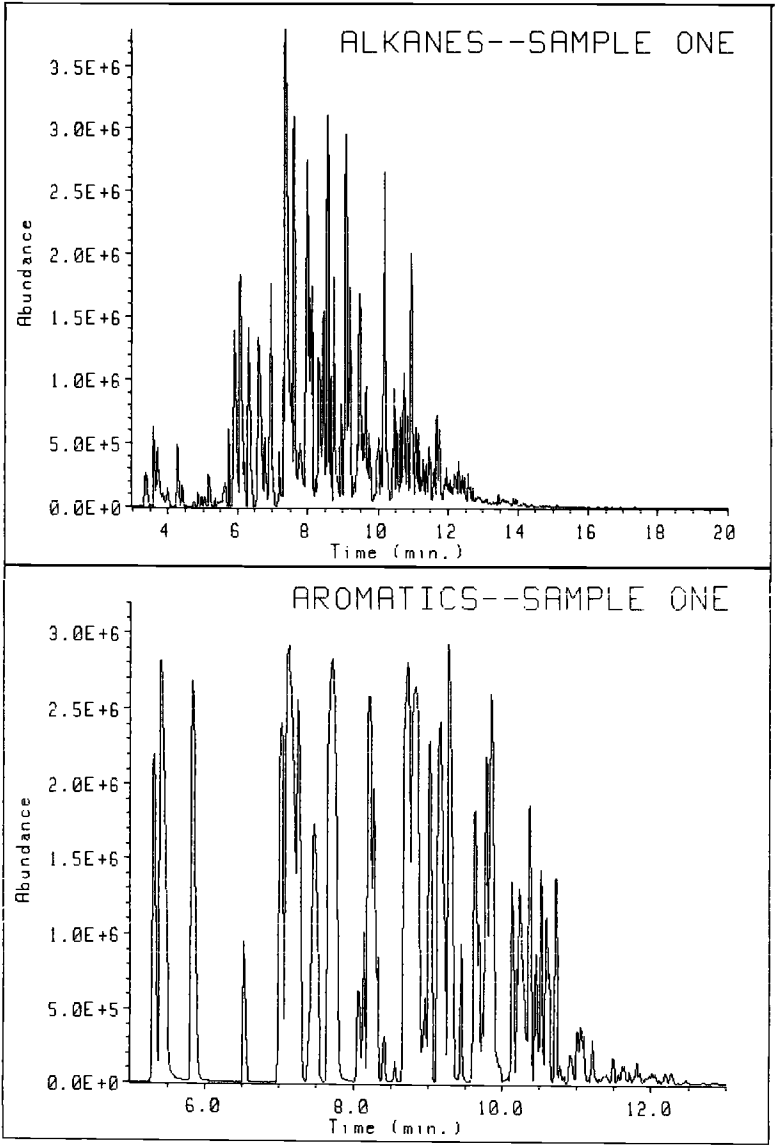


FIG. 3—Alkane ion profile (top) and aromatic ion profile (bottom) of Sample 1.

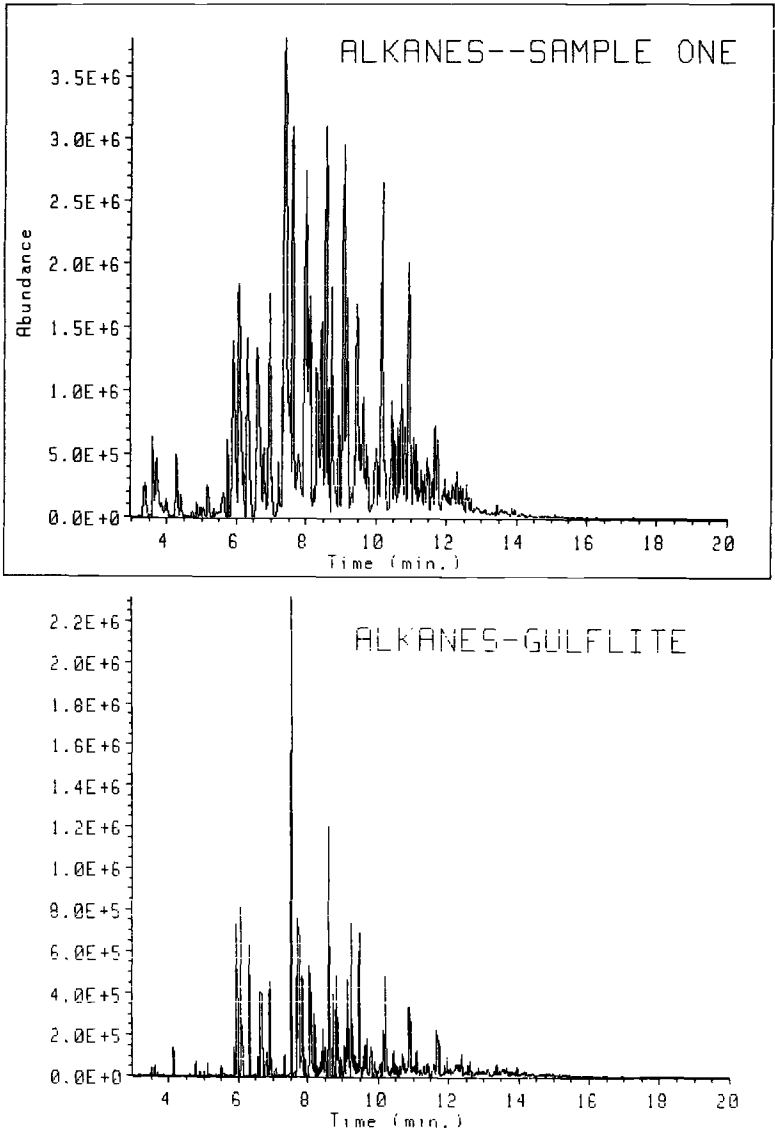


FIG. 4—Alkane ion profiles of Sample 1 (top) and Gulflite charcoal starter fluid (bottom).

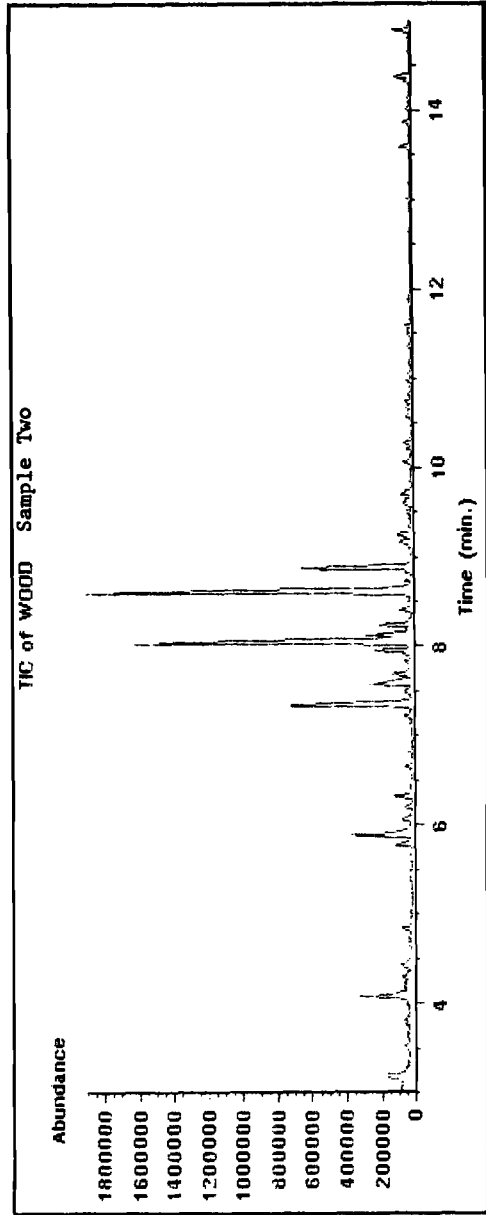


FIG. 5—Total ion chromatogram of Sample 2.

obscured by large peaks which are natural terpene products from the wood. An analyst using GC-FID would, at best, report inconclusive findings.

Whenever aromatic ion profiling is performed (that is, plotting the sum of ions 91, 105 and 119), problems will be encountered if terpenes are present. The mass spectrum of a typical terpene (Fig. 6) will contain significant mass 91 and 105 ions. Therefore, any terpenes present in the sample will appear on an aromatic ion profile and may interfere when the analyst attempts to match the aromatic pattern to an accelerant standard. There is a simple solution to this problem. While terpenes contain major mass 91 and 105 ion fragments, aromatic compounds exhibit essentially no mass 93 ion fragments. If the analyst, after summing extracted ion profiles for masses 91, 105, and 119, then subtracts the extracted ion profile of mass 93, he will subtract any peaks resulting from terpenes without eliminating any aromatic components. The result is an aromatic ion profile free of interference from most terpene compounds, as demonstrated in Fig. 7. The aromatic ion profile before subtracting mass 93 is inconclusive. After subtracting mass 93, the profile clearly indicates the presence of gasoline.

### Case 3

Sample 3 contained burnt debris from a desk drawer, including what appeared to be burnt plastic. The total ion chromatogram shows a regular series of triplets (Fig. 8). An analyst using only GC-FID may be tempted to call this pattern inconclusive for the presence of an accelerant, noting a regular series of peaks around the *n*-alkanes, such as in a heavy petroleum distillate, but being unable to specifically identify the components present.

Using GC-MS, the alkane ion profile does in fact exhibit the regular series of *n*-alkanes (Fig. 9), and the analyst may suspect some sort of heavy petroleum distillate. Sample 3 illustrates how first impressions may be deceiving. There are several indications that the sample did not contain a heavy petroleum distillate: the absence of the pristane and phytane peaks directly after the *n*-heptadecane (17.8 min) and *n*-octadecane (18.9) peaks, the relative absence of any branched alkanes, and the unusual triplet pattern in the total ion chromatogram (TIC). Examining the mass spectra of the triplet pattern shows that they consist of a diene, an alkene, and an *n*-alkane, with the alkene being the highest peak in most of the triplets (Figs. 10 and 11).

The TIC of Sample 3 corresponds to previously published chromatograms of polyethylene pyrolysis products [3]. That the individual components of this triplet pattern are characteristic of polyethylene was confirmed by analyzing burnt polyethylene bag standards by GC-MS. In this case, analysis by GC-MS has demonstrated that the peaks appearing in the chromatogram are due to pyrolysis of the debris and that no indications of an accelerant were found.

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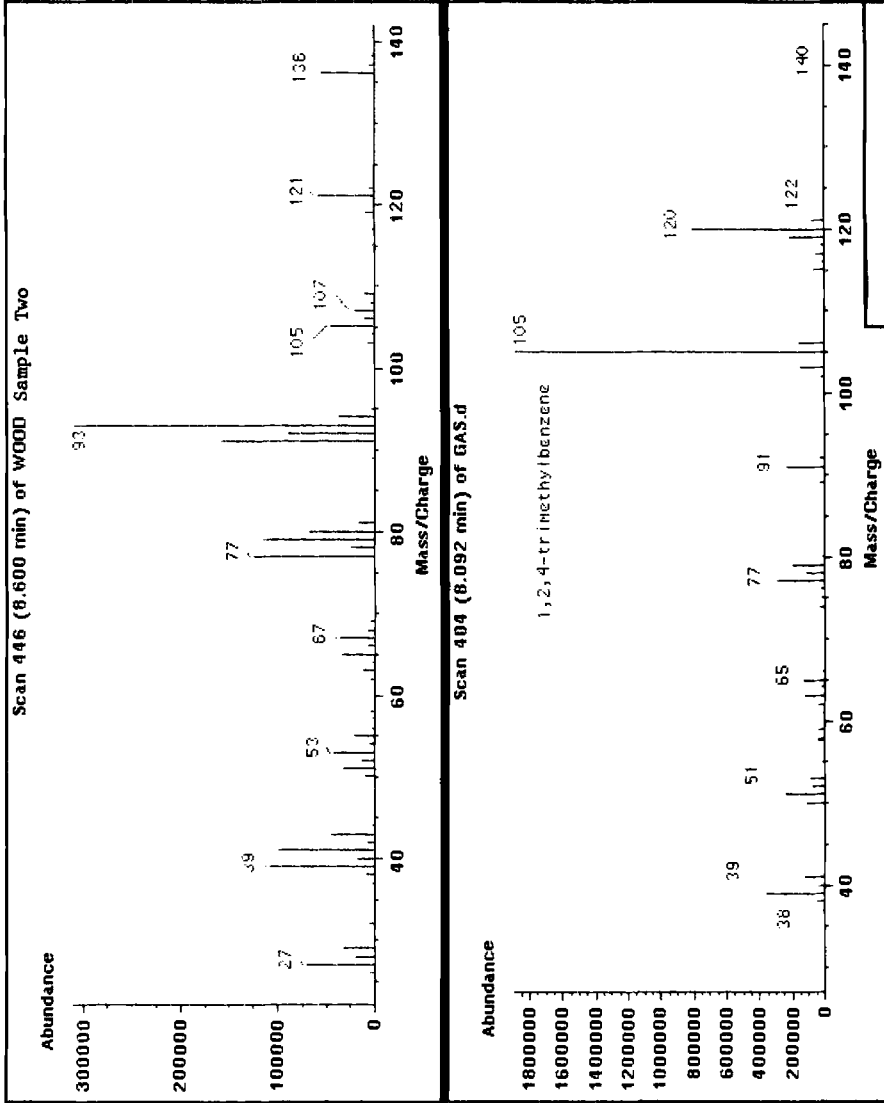


FIG. 6—Mass spectra of a terpene from Sample 2 (top) and 1,2,4-trimethylbenzene (bottom).

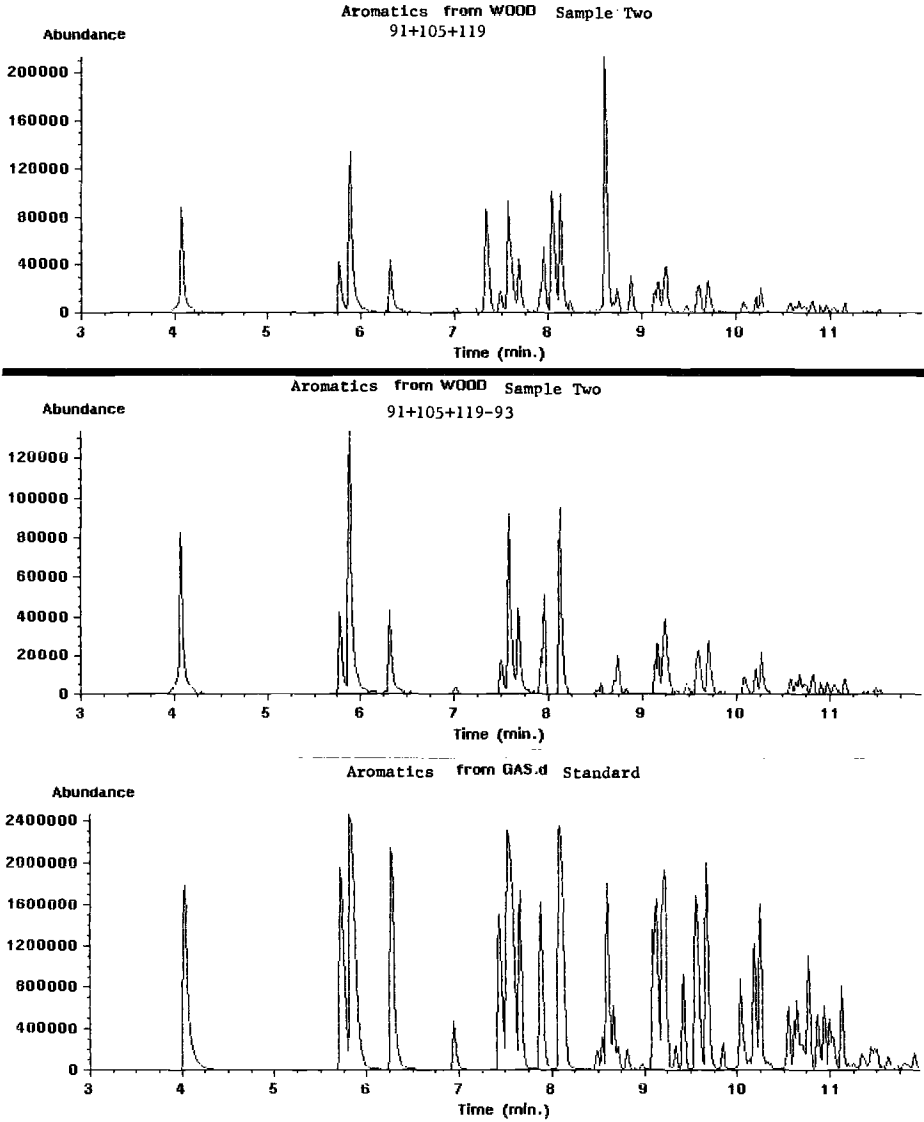


FIG. 7—Aromatic ion profiles of Sample 2 before (top) and after (middle) subtracting mass 93, and the gasoline standard (bottom).

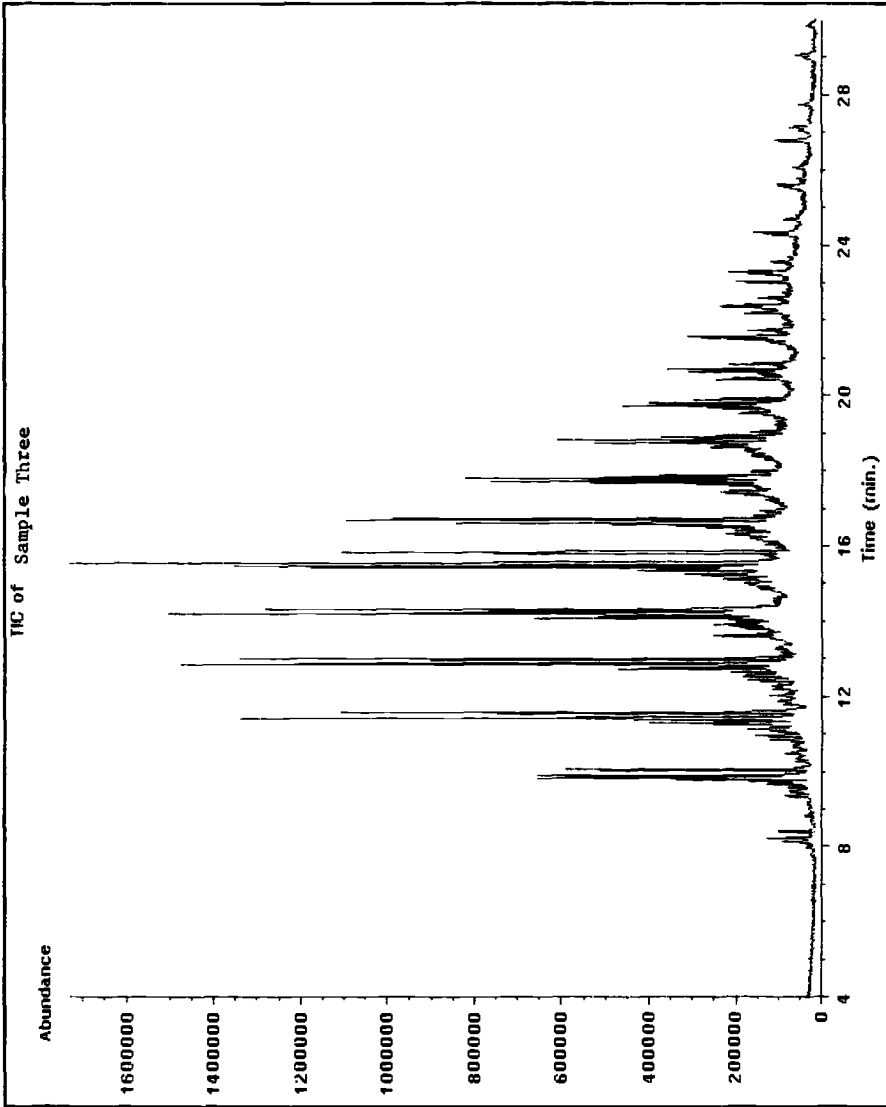


FIG. 8—Total ion chromatogram of Sample 3.

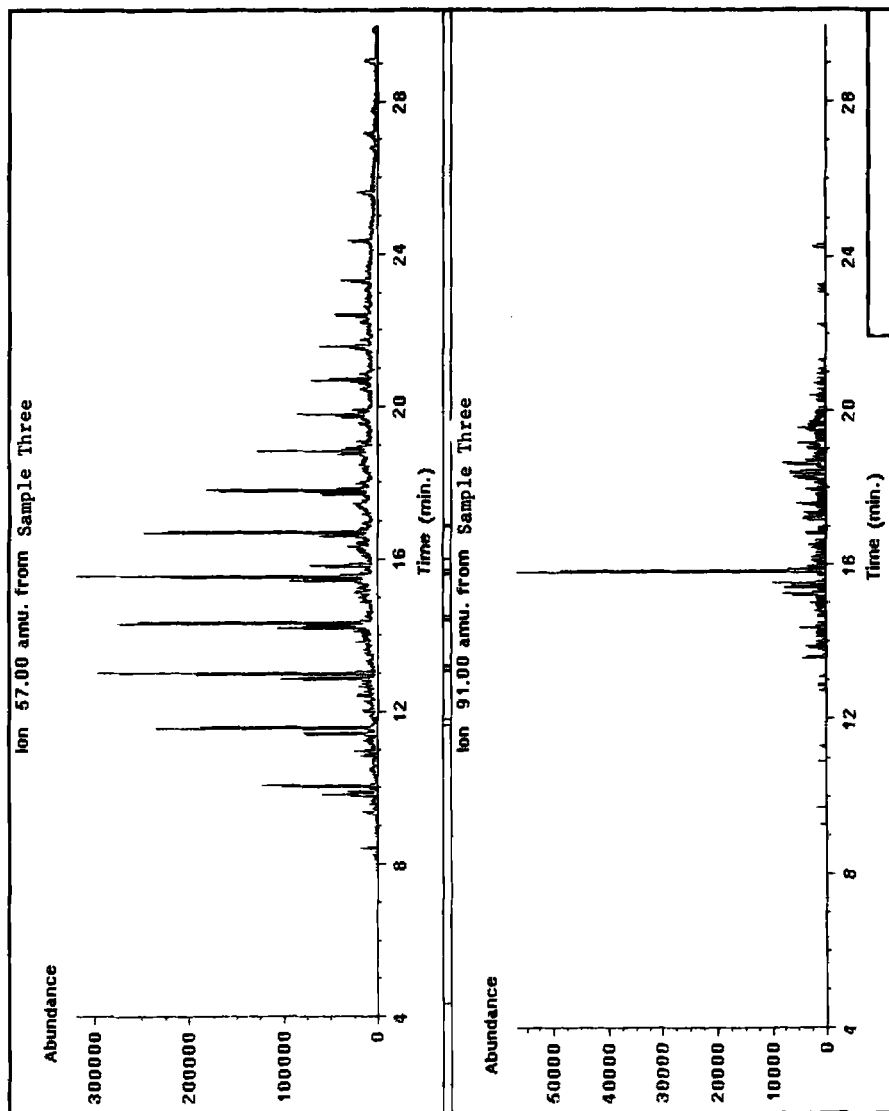


FIG. 9—Alkane ion profile (top) and aromatic ion profile (bottom) of Sample 3.

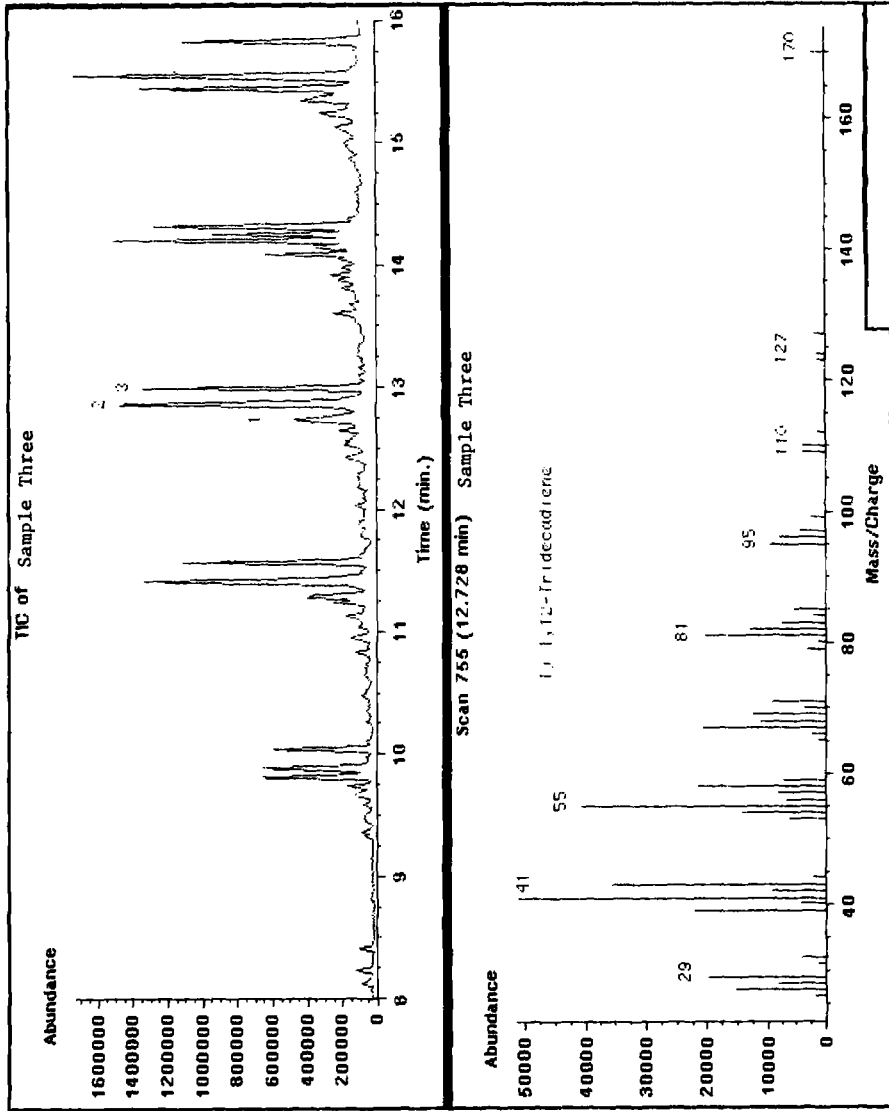


FIG. 10—Portion of the total ion chromatogram (top) and mass spectrum of the peak at 12.73 min (bottom) of Sample 3.

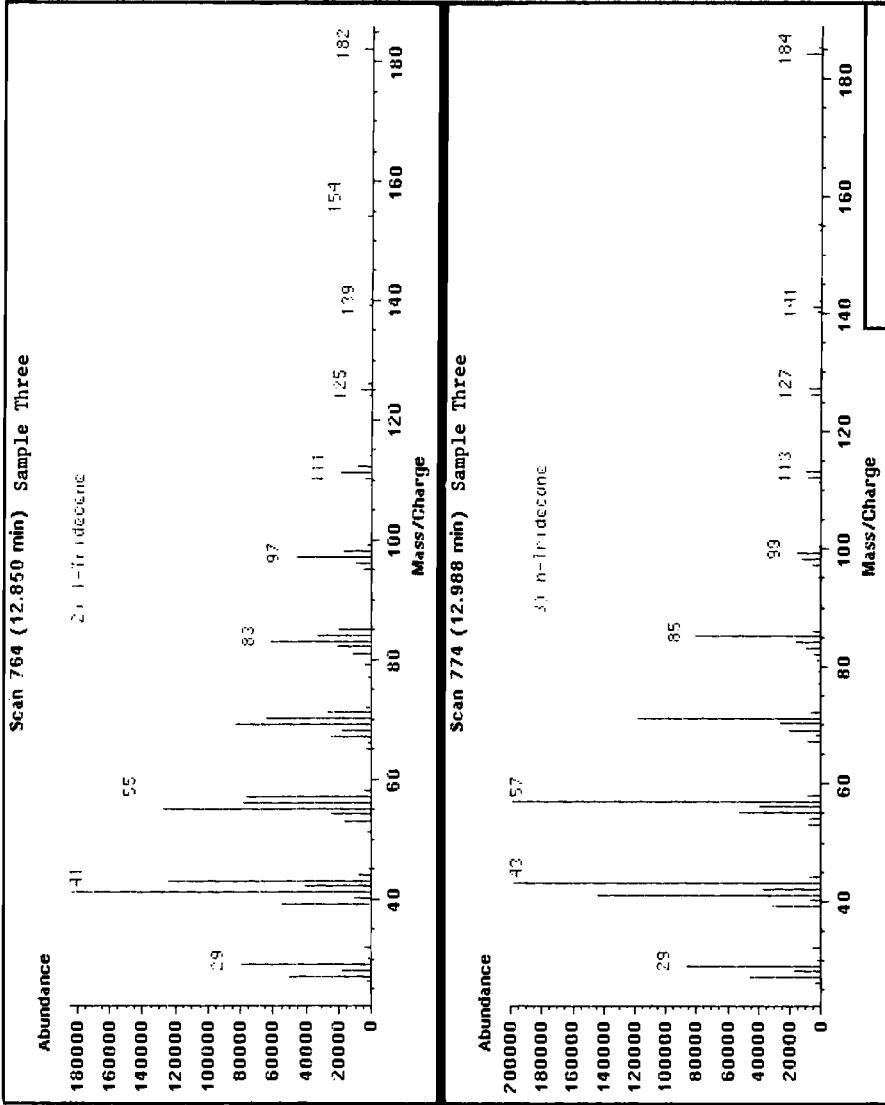


FIG. 11—Mass spectra of the peaks at 12.85 min (top) and 12.99 min (bottom) of Sample 3.

**References**

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