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Contamination of Brake Fluid by Power Steering Fluid

ABSTRACT: Analysis of a contaminated brake fluid revealed the contaminant to be power steering fluid. The sample received at the laboratory presented two immiscible phases, which were both organic. Both fluids were analyzed by GC-MS and compared with a library of known automotive fluids. The bottom layer (brake fluid) consisted of glycols while the top layer (power steering fluid) consisted of aliphatics ranging from C₁₆ to C₂₅₊. An interesting phenomenon of diffusion of some of the glycols from the brake fluid phase into the power steering fluid phase was observed and needs to be taken into consideration in the interpretation of the results.

KEYWORDS: forensic sciences, brake fluid, power steering fluid, gas chromatography, mass spectrometry, glycols, petroleum distillates

Recently, our laboratory received a sample of contaminated brake fluid extracted from the brake system of a 99 Isuzu Amigo. The vehicle presented a failure in the brake system shortly after being serviced at a brake shop. The sample was constituted of two phases, as shown in Fig. 1. Our laboratory was requested to identify the nature of the contaminating layer, assuming that one layer was brake fluid. The ultimate goal was to provide an explanation for the failure of the brake system.

Among the different contaminants of brake fluid, the most common is probably water. It reduces the boiling point of the fluid, creates vapor at the "hot spots" of the circuit, and prevents proper compression (1). Another frequent contaminant is petroleum distillates such as engine oil, automatic transmission fluid or power steering fluid (2,3). In this instance, the presence of two immiscible phases suggested that the contaminant was a petroleum distillate rather than water, since the latter is highly miscible in brake fluid.

We conducted the examination and chemical analysis of both phases, and identified the brake fluid as well as the contaminating fluid.

Material and Methods

First, color of the two layers were recorded and compared with known standards of different vehicle fluids such as coolant, brake fluid, power steering fluid, automatic transmission fluid, and engine oil. Then, 0.1% solutions of each phase were prepared in diethyl ether (spiked at 100 ppm with perchloroethylene (PCE) as an internal standard). Extracts were injected in a Hewlett-Packard gas chromatograph-mass spectrometer 5890 Series II-5971 equipped with a capillary column, HP 5 crosslinked (30 m × 0.25 mm × 1.0 μm). Helium was used as the carrier gas at a flow rate of 1 mL/min (set at 170°C). The oven temperature was programmed as follows: 60°C for 3.5 min, then 15°C/min to 285°C for 6.5 min. One μL of

solution was injected in the splitless mode. The quadrupole temperature was set at 170°C and the mass spectrometer was set to scan from 33 to 400 amu at 2.07 scans/second. Resulting chromatograms were compared to the chromatograms of more than 50 known standards of different vehicle fluids that are representative of products encountered on the market (4).

Results

Color

The top layer is transparent and does not contain any particles. The bottom layer is light brown and shows slight particle contamination in suspension, as shown in Fig. 1.

GC-MS analysis

Figure 2A shows the total ion chromatogram of the bottom layer of the sample, and Fig. 2B shows the total ion chromatogram of STP DOT 4 brake fluid. The presence of the glycol compounds in both chromatograms is typical of brake fluid.

In the first chromatogram, it is possible to observe the presence of diethylene glycol monomethyl ether [CAS 111-77-3] at 8.4 min, diethylene glycol [CAS 111-46-6] at 8.8 min, triethylene glycol monomethyl ether [CAS 112-35-6] at 12.3 min and triethylene glycol monoethyl ether [CAS 112-50-5] at 12.9 min. N-butyl-1-butanamine [CAS 111-92-2] at 8.8 min (coeluting with diethylene glycol) and n-cyclohexyl cyclohexanamine [CAS 101-83-7] at 14.6 min are very likely present as corrosion inhibitors.

The second chromatogram shows some common peaks as well as some extra peaks such as diethylene glycol monobutyl ether [CAS 112-34-5] at 11.9 min and 2,5,8,11,14-pentaohexadecan-16-ol [CAS 23778-52-1] at 15.1 min.

Figure 3A represents the total ion chromatogram of the top layer of the sample, Fig. 3B represents the total ion chromatogram of STP power steering fluid, and Fig. 3C shows the total ion chromatogram of an automatic transmission fluid Dexron III-Mercon.

These three chromatograms (Figs. 3A, 3B, and 3C) present a similar pattern of aliphatic compounds ranging from approximately

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Received 5 Oct. 2002; and in revised form 22 Dec. 2002 and 25 Jan. 2003; accepted 25 Jan. 2003; published 1 May 2003.

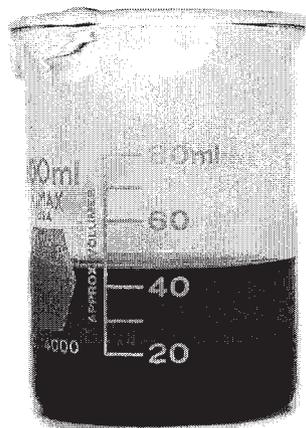


FIG. 1—Photograph of the contaminated fluid, showing the two immiscible layers.

C_{16} to C_{25+} , with the automatic transmission fluid having a more distinctive n-alkane pattern. Extraneous compounds are observed in the top layer of the suspected liquid, as shown in Fig. 3A. These are the two principal glycols found in the bottom layer at 12.3 and 12.9 min as well as the amine at 14.6 min.

Figures 4A, 4B and 4C are the extracted ion profiles for ions 55, 57 and 83 between 14 and 25 min for the three chromatograms 3A, 3B and 3C. The top layer does not present any obvious n-alkane patterns. However, when comparing the intensities of the three ions, it can be assumed that the bulk of the aliphatics is constituted of isoparaffinic products. On the other hand, the presence of cycloalkanes is observed.

The power steering fluid presents a slightly more distinctive pattern of alkanes, but they are not as evident as in the transmission fluid. Even pristane and phytane (next to C_{17} and C_{18} respectively) are easily distinguishable in the latter. Cycloalkanes are observed in both fluids.

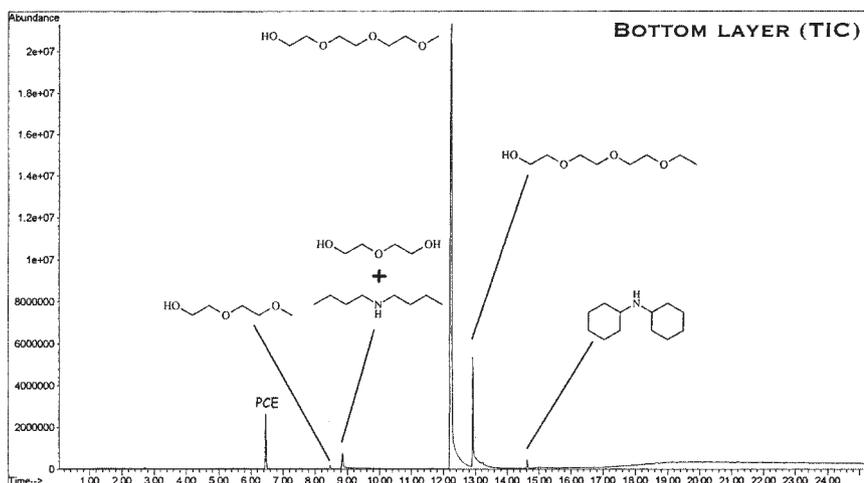


FIG. 2A—Total ion chromatogram of the bottom layer (PCE represents the internal standard).

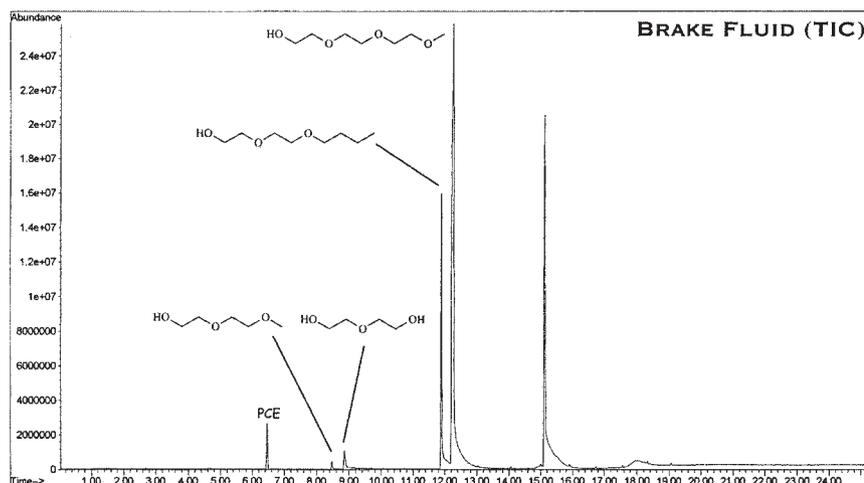


FIG. 2B—Total ion chromatogram of STP DOT 4 brake fluid (PCE represents the internal standard).

Discussion

Brake fluid is highly miscible in water and therefore, the presence of two layers in the contaminated liquid led to the hypothesis that both components are organic. Gas chromatography has already been shown as a valuable tool in analyzing high molecular weight petroleum products (5).

The glycol content of the bottom layer is a sufficient characteristic to conclude that it is brake fluid (4). The material safety data sheet of the STP brake fluid analyzed as a reference sample describes the content of the fluid as a "mixture of glycol ethers, polyglycols, oxidation inhibitors and corrosive inhibitors" (6). This was confirmed by the analysis shown in Fig. 2B, except that the corrosion inhibitors were not detected.

The chromatographic characteristics of the top layer are somewhat similar to those of power steering fluid and automatic transmission fluid. Miscibility testing showed that brake fluid was more dense than power steering fluid or automatic transmission fluid,

which corroborates the position of the layers in the sample. These two last liquids have been shown to be difficult to distinguish based only on chromatographic patterns (4). Brake fluids and power steering fluids present different colors, ranging from transparent to brown. However, the authors have never encountered an automatic transmission fluid that was not red. The top layer was almost transparent and this allowed us to conclude that the liquid was almost certainly not automatic transmission fluid.

Another interesting phenomenon is the partial dissolution of some of the two principal glycols from the brake fluid (bottom layer) in the aliphatic mixture (top layer). This added a few more peaks to the chromatogram of the power steering fluid. This phenomenon has been previously observed and tested in our laboratory. A solution of 50% power steering fluid and 50% brake fluid was prepared, mixed, and allowed to rest. Then, both layers were analyzed and compared with the original fluids. A slight transfer of glycols in the aliphatic layer was observed, but to a much lower extent than in the present case. However, this laboratory experiment did not include the envi-

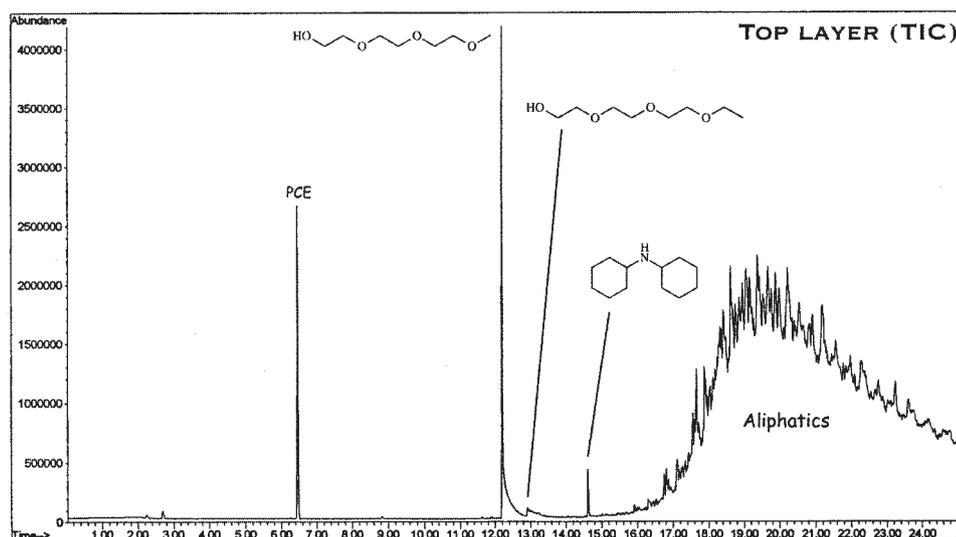


FIG. 3A—Total ion chromatogram of the top layer (PCE represents the internal standard).

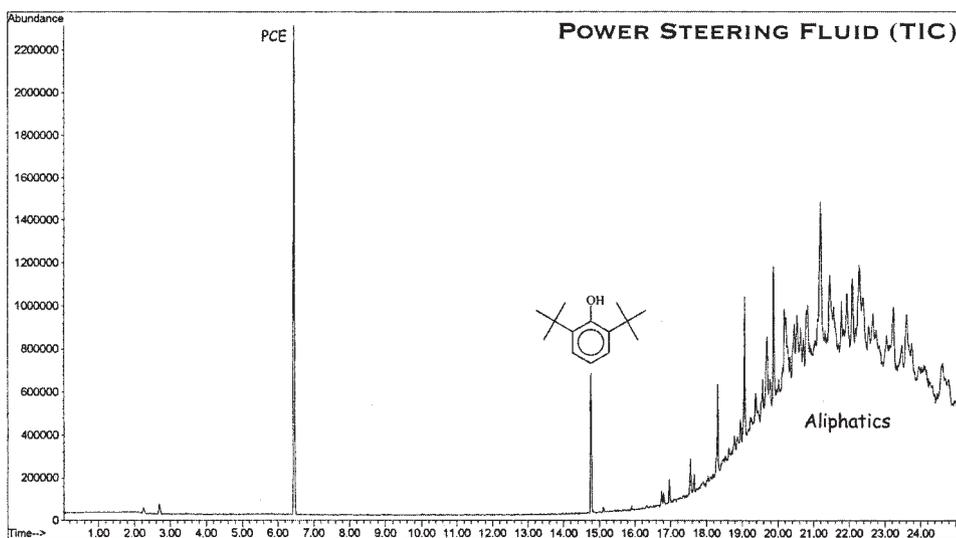


FIG. 3B—Total ion chromatogram of STP power steering fluid (PCE represents the internal standard).

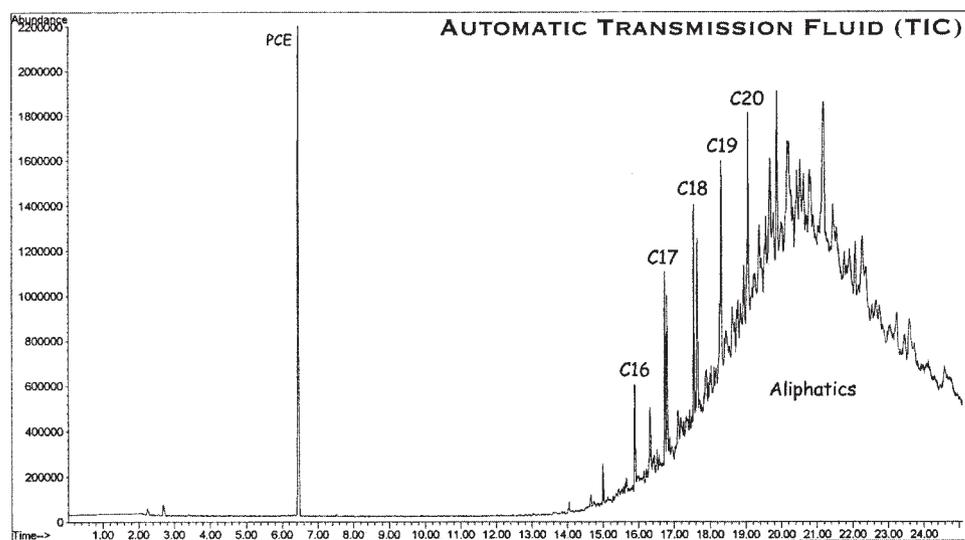


FIG. 3C—Total ion chromatogram of Dexron III-Mercon automatic transmission fluid (PCE represents the internal standard).

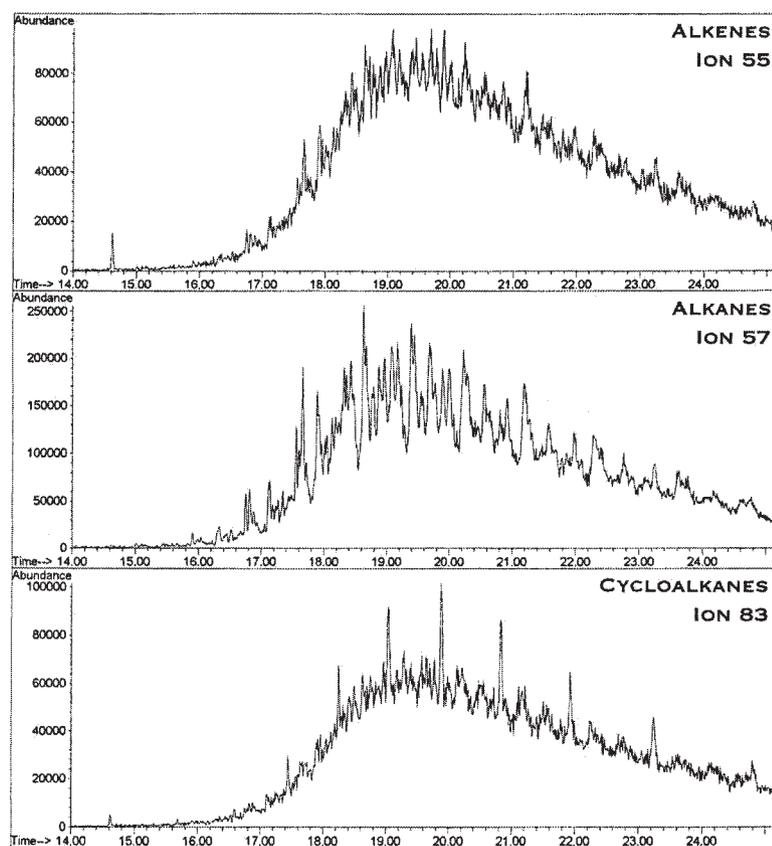


FIG. 4A—Extracted ion profile of ions 55, 57, and 83 of the top layer between 14 and 25 min.

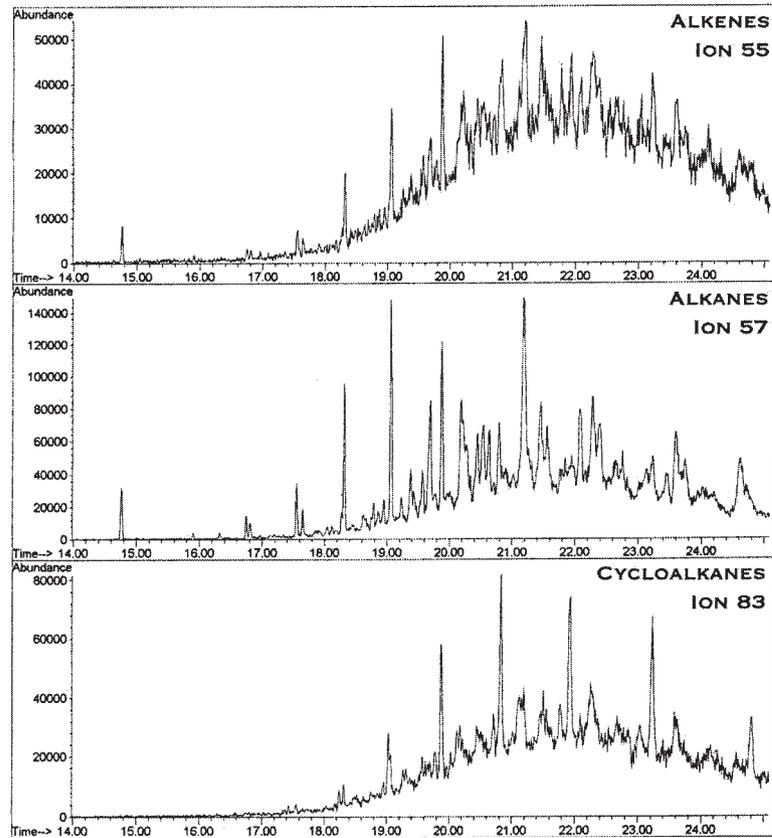


FIG. 4B—Extracted ion profile of ions 55, 57, and 83 of STP power steering fluid between 14 and 25 min.

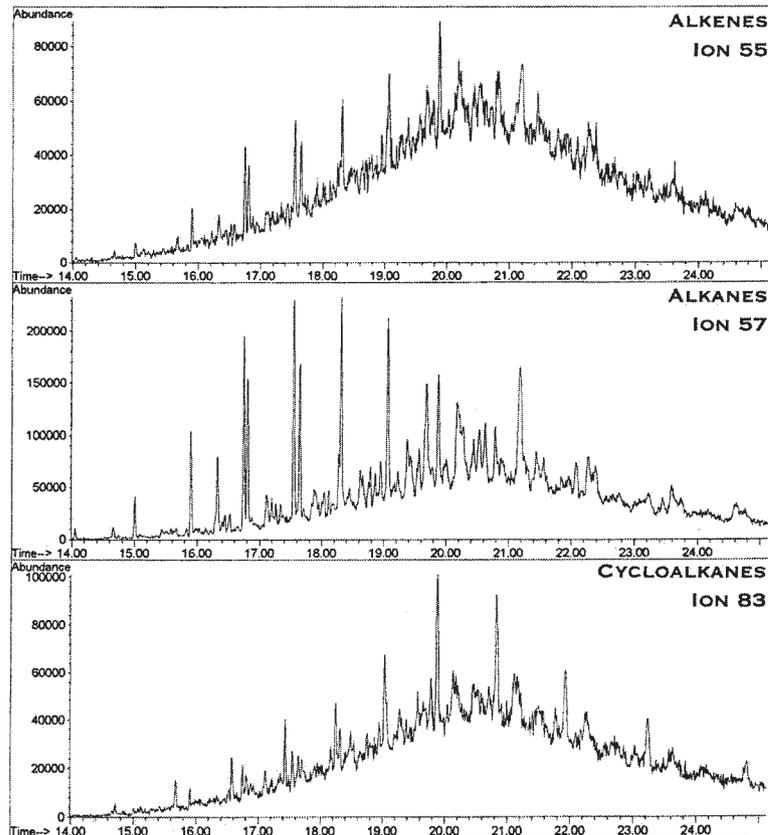


FIG. 4C—Extracted ion profile of ions 55, 57, and 83 of automatic transmission fluid between 14 and 25 min.

ronmental conditions such as pressure and temperature that the fluid undergoes during the operation in a vehicle. This might significantly contribute to the dissolution of glycols in the aliphatic mixture.

Contamination of brake fluid with power steering fluid can lead to catastrophic consequences (1). Petroleum distillates deteriorate rubber parts of the brake system and yield rapidly to a complete failure of the brake system. The most likely scenario to explain the presence of power steering fluid is that the mechanic might have confused the bottle of brake fluid with the bottle of power steering fluid when filling the brake system after servicing the vehicle.

Conclusion

It was possible to successfully identify the source of a second phase in a contaminated brake fluid as power steering fluid. The presence of a hydrocarbon mixture in the brake system leads to disastrous consequences. GC-MS was shown to be a valuable tool to identify different vehicle fluids. However, color examination was necessary to distinguish between automatic transmission fluid and power steering fluid and to allow the authors to reach a definitive conclusion.

References

1. Herguth Laboratories, Inc. Brake fluid contamination analysis complete and accurate. <http://www.herguth.com> 2002.
2. Herguth Laboratories, Inc. Typical brake fluid contamination laboratory analysis report. <http://www.herguth.com> 2002.
3. Lloyd JBF. A note on the detection and determination of mineral oil contamination in brake fluids. *J Forensic Sci Soc* 1982;22:289-90.
4. Stauffer E, Lentini JJ. Forensic identification of vehicle fluids. Abstract Book of the 16th Meeting of the International Association of Forensic Sciences. *J Forensic Med* 2002;45(4-5):58.
5. Lloyd JBF. Capillary column gas chromatography in the examination of high relative molecular mass petroleum products. *J Forensic Sci Soc* 1982;22:283-7.
6. First Brands Corporation. STP[®] heavy duty brake fluid material safety data sheet. 1998 MSDS0093.

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