

Table III. Slope of Molecular Weight vs. Concentration

Asphaltene	Slope
Starting	11.1
48% O silylated	4.9
Benzene eluted from silica gel	3.7
Et ₂ O eluted from silica gel	8.0

how association would be expected to vary among the different asphaltene products. Since the slopes and association are greater in benzene, we will consider only that series (Table III). We feel that these preliminary results tend to support the hydrogen bonding donor-acceptor (5, 6) model of coal-derived asphaltene association (2-4) more than the charge transfer complexation model previously suggested to explain association in petroleum asphaltene (10-12). Actually, the donor-acceptor concept is a much more universal and broad principle (9).

The reasoning for this is as follows: silylation eliminates phenolic OH functions and would be expected to reduce H-bonding; the benzene eluted fraction from silica gel is known to have a reduced basic nitrogen concentration (5, 6) which would also reduce the hydrogen-bonding possibilities; the Et₂O eluted fraction, which contains the major portion of both the basic nitrogen and oxygen, still contains some phenolic OH (5, 8), and therefore would be expected to show the smallest decrease in associative properties relative to starting asphaltene.

In conclusion, the extrapolated molecular weights, at zero concentration, for coal-derived asphaltenes and their derivatives are found to be almost identical in the solvents benzene and tetrahydrofuran. The average deviation found is 2.5% for eight such asphaltenes in benzene and tetrahydrofuran. This implies that coal-derived asphaltenes approach complete dissociation as their concentrations approach infinite dilution in both benzene and tetrahydrofuran in contrast to petroleum-derived asphaltenes which are apparently not dissociated in either benzene (13), or tetrahydrofuran (1) over the ranges 2-7 wt % and 1.2-2.4 wt %, respectively.

For the former case, with Athabasca asphaltenes, a solvent of higher polarity, such as pyridine, was required to cause significant variation in the observed molecular weights as a function of concentration.

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I. Schwager
W. C. Lee
T. F. Yen*

University of Southern California
Chemical Engineering Department
University Park
Los Angeles, California 90007

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Interference of 1-Chloro-2-propanol in the Determination of Bis(chloromethyl) Ether in Air by Gas Chromatography-Mass Spectrometry

Sir: Selected ion monitoring (SIM) is frequently used for the determination of bis(chloromethyl) ether (BCME), a highly toxic and carcinogenic compound (1-4). The retention time of BCME on a GC column and the strongest mass fragments at $m/e = 79$, 81, 49, and 51 produced under electron-impact (EI) conditions are used (5-7). In this correspondence it will be shown that under these circumstances and using a high resolution glass capillary column with the commonly applied GC phase SE-30, the presence of BCME is not unambiguously established.

Recently we investigated the headspace above soil contaminated with degradation products of burned polyurethane foam used for thermal isolation. The air was sampled with Tenax GC. The compounds trapped were desorbed by heat and subsequently separated on a high-resolution SE-30 glass capillary column. Figure 1 shows the resulting gas chro-

matogram. Identification of the compounds was carried out on a Varian MAT 112 GC/MS/DS combination using direct coupling of the capillary column and continuous repetitive measurement of mass spectra. Figure 2 shows the EI mass spectrum at the top of GC peak 1. Besides the base peak at $m/e = 45$ and lower mass fragments, the spectrum contained fragments at $m/e = 79/81$ and $49/51$ of which the intensity ratio corresponded with the ratio in a reference spectrum of BCME. Under the gas chromatographic conditions used, the retention time of GC peak 1 was identical with that of BCME, so it seemed likely that BCME was present in the headspace. However, in that case $m/e = 45$ and some lower mass fragments, which are absent in the reference spectrum of BCME, must be attributed to another compound eluting at the same time as BCME from the column.

A more satisfactory explanation can be found if it is as-

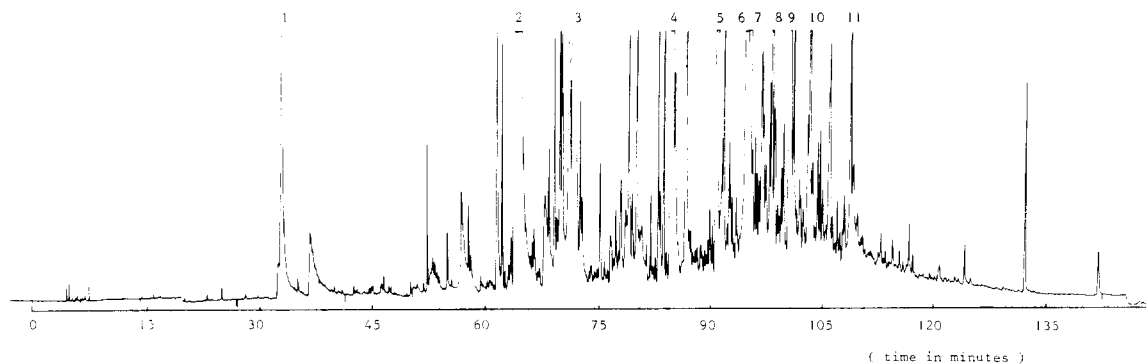


Figure 1. Gas chromatogram of the air sample. Column, 60 m \times 0.25 mm i.d. glass capillary column coated with SE-30; temp. program, 0–200 °C with 2 °C/min; carrier gas, helium, 0.8 kg/cm². Identified compounds: (1) 1-chloro-2-propanol; (2) aniline; (3) *O*-toluidine; (4) quinoline; (5,6,7) methylquinolines; (8) ethylquinoline; (9) dimethylquinoline; (10) anthracene; (11) phenanthrene

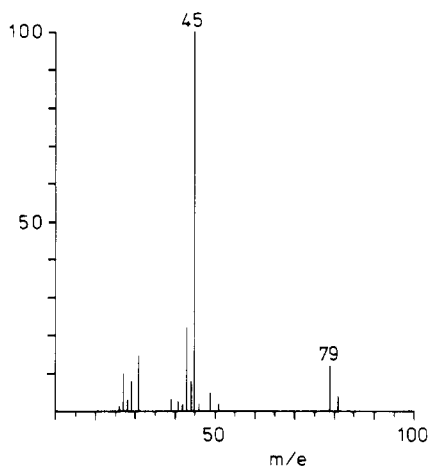


Figure 2. Electron impact mass spectrum of compound 1

sumed that the compound under investigation is 1-chloro-2-propanol. Indeed, the spectrum of GC peak 1 corresponded roughly with a reference spectrum of 1-chloro-2-propanol.

Figure 3 shows the isobutane chemical-ionization mass spectrum of GC peak 1. The spectrum contained fragments at $m/e = 95/97$ corresponding with the $(M + H)^+$ ion of 1-chloro-2-propanol and at $m/e = 77/79$ corresponding with the loss of water, a characteristic fragment of isobutane CI mass spectra of secondary alcohols (8). Based on these CI results, GC peak 1 was definitely identified as 1-chloro-2-propanol. This compound gives the same retention time on a SE-30 column and produces mass fragments at $m/e = 79/81$ and $49/51$ in the same intensity ratio as BCME. Therefore, 1-chloro-2-propanol could be mistaken as BCME by using SIM on a SE-30 column and again it is demonstrated that, even using sophisticated methods for the determination of a specific compound, this still may yield false results.

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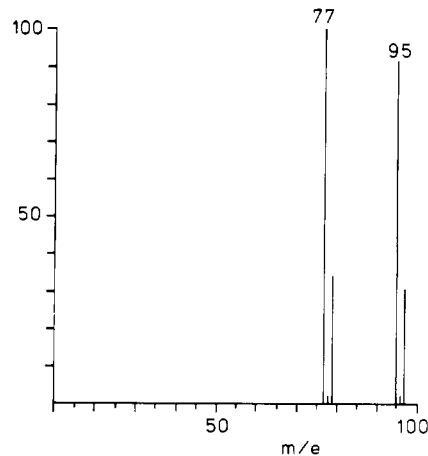


Figure 3. Isobutane chemical ionization mass spectrum of compound 1

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Frans L. Schulting*
Eric R. J. Wils

Chemical Laboratory TNO
Rijswijk (ZH)
The Netherlands

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