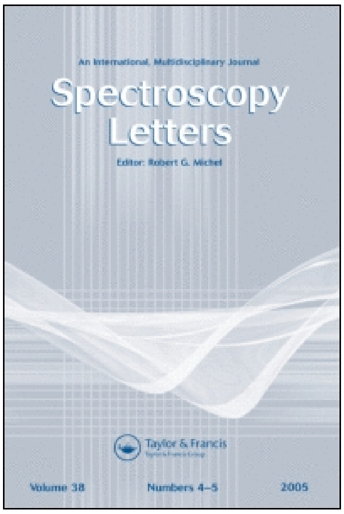

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Thermal Instability of Hydroarcmatics—Limitation on Quantitative Mass Spectrometric Analyses

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THERMAL INSTABILITY OF HYDROAROMATICS--LIMITATION ON QUANTITATIVE MASS SPECTROMETRIC ANALYSES

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Discrepancies in the determination of anthracene in dihydroanthracene were revealed when mass spectrometric analyses were compared with ultraviolet spectrophotometric data.1/ A limited investigation indicated that dehydrogenation of the hydroaromatic in the mass spectrometer inlet system can be minimized but not eliminated.

Analysis for aromatic hydrocarbons in the presence of hydroaromatics is essential to establish the purity of a hydroaromatic compound and to evaluate the products from dehydrogenation and reduction reactions. Investigations at the Pittsburgh Coal Research Center of the U.S. Bureau of Mines requiring such analyses include the electrochemical reduction of coal and the dehydrogenation of coal. The ultraviolet spectrophotometric analyses for the aromatic structures present in the reaction products from polycyclic compounds are reliable, but the hydroaromatic content cannot be determined by this method. The mass spectra of these reaction products indicate the molecular weights of the hydroaromatics present in the mixtures but an analytical method to obtain quantitative analyses for all products has not been achieved.

Low-ionizing voltage (7.5 indicated) mass spectrometric data were obtained using a Consolidated Electrodynamics Corp. model 21-103C mass spectrometer2/ equipped with an inlet system in which the temperature could

Table 1.- Mass spectrometric analyses of aromatics in dihydroaromatics

Inlet temperature, °C	Volume percent Anthracene[a] in dihydroanthracene	 Phenanthrene[b] in dihydrophenanthrene
150	4.8	-[c]
160	4.6	7.0
170	8.0	-
180	9.4	-
200	9.5	-
250	11.7	-
275	17.2	-
300	32.3	11.0
320	65.0	17.0

[a] Ultraviolet analysis - 3.4 percent.
[b] Ultraviolet analysis not available.
[c] Not determined.

be varied from ambient to 300° C. The ion source temperature was about 250° C.

The effect of temperature on determinations for anthracene in dihydroanthracene and phenanthrene in dihydrophenanthrene is shown in table 1. Erroneous results for anthracene and phenanthrene are obtained if the normal operating temperature of 300° C is used, although somewhat greater thermal stability is indicated for dihydrophenanthrene.

In analyzing such mixtures, the temperature selected must be high enough to ensure complete vaporization of the sample but low enough to avoid dehydrogenation. Tests thus far indicate a temperature of about 160° C best meets these requirements for the analysis of hydroanthracenes and hydrophenanthrenes. The ultraviolet analysis for the sample shown in table 1, first column, was 3.4 percent anthracene. Mass spectrometric results varied from 4 to 6 percent on 4 different days, suggesting that the condition of the ion source may also be involved. The dehydrogenation of dihydrophenanthrene is of smaller magnitude as the temperature is increased from 160° to 300° C, but data are still unreliable. Limitations on the accuracy of the higher molecular weight hydroaromatic determinations cannot be assessed until pure compounds are studied. A combination of both ultraviolet and mass spectrometric analyses will be necessary to determine the components present in samples containing hydroaromatic structures.

References

1. Friedman, Sidney, Research Chemist, Pittsburgh Coal Research Center, Bureau of Mines, U.S. Department of the Interior, Pittsburgh, Pa., private communication.

2. Reference to trade names is made for identification only and does not imply endorsement by the Bureau of Mines.

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