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# Low Severity Coal Liquefaction Promoted by Cyclic Olefins

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Christine W. Curtis  
Chemical Engineering Department  
Auburn University, AL 36849

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## Introduction

Cyclic olefins, such as 1,4,5,8-tetrahydronaphthalene (isotetralin) and 1,4,5,8,9,10-hexahydroanthracene (HHA), are highly reactive hydrogen donor compounds that readily donate their hydrogen to coal and model acceptors when heated. These donors are active at temperatures above 200 °C and are active donors in the low severity liquefaction of coal at 350 °C as shown in this research.

The research performed during the quarter, January to March 1995, focused on two areas. The first area involved completing the writing of a manuscript based on research performed on this project concerning the mild acidic pretreatment of low rank coals and their liquefaction behavior in the presence of hydrogen donors with different reactivities. The manuscript was submitted for review to *Energy and Fuels*. A second manuscript was begun which discussed the research involving the hydrogen donation at low severity condition by hexahydroanthracene. The catalytic enhancement of hydrogen transfer by cyclic olefins was also examined. The data from this research was reexamined; it was decided that before writing the paper that the data should be reanalyzed. Therefore, this quarter was spent taking the raw data and reanalyzing the data, putting the solvent fractionation data on a solvent-free basis. The recalculated data and the calculational method is given as Part 1 in this report.

The second area that was worked on this quarter was the high temperature infrared analysis of cyclic olefins. The work is ongoing and is currently involving a considerable amount of equipment and technique development. Part 2 is the discussion on the high temperature infrared analysis of cyclic olefins.

## Part 1

### Low Severity Liquefaction Reactions Promoted By Hexahydroanthracene

The research involving HHA promoted low severity liquefaction was performed by AnPing Huang. As this research was being prepared for publication, it was discovered that a the data needed to be calculated on a solvent-free basis. These calculations needed to be performed before the manuscript was written. These calculations were performed this quarter. the calculational method and the data tables are given in this section of the report.

#### Calculational Method

The calculation of the solvent free product distribution of the low severity coal liquefaction reactions was performed using a method developed by the authors. Some assumptions were made for the calculation. Sample calculations are provided.

The products of coal liquefaction reactions were analyzed by collecting the gaseous product and by using solvent fractionation to separate the reaction products into fractions; these fractions were hexane solubles, THF solubles and insoluble organic matter (IOM) [1]. The amount of products, total output, recovery, total input, percentage of solvent in the feed and moisture and ashfree (maf) coal charged was obtained from the reactions and analysis. These amounts were the primary data use for the calculations. The software "Quattro Pro for Windows, Version 5.0" was used to calculate the information given in Tables 2-4. The calculational equations that were used in those tables are given as the following:

$$X_1 = X_{\text{gas}} = \text{gas/ maf coal}$$

$$X_2 = X_{\text{asphaltene}} = \text{asphaltene/ maf coal}$$

$$X_3 = X_{\text{preasphaltene}} = \text{preasphaltene/ maf coal}$$

$$X_4 = X_{oil} = (\text{Oil- Total input} \times \text{Solvent\%} \times 0.80) / \text{maf coal}$$

$$\Sigma X_i = X_1 + X_2 + X_3 + X_4$$

$$\text{Coal conversion \%} = (1 - \text{IOM}/\text{maf coal}) \times 100$$

$$Y_1 = \text{Gas \%} = (X_1 / \Sigma X_i) \times \text{coal conversion \%}$$

$$Y_2 = \text{Asphaltene \%} = (X_2 / \Sigma X_i) \times \text{coal conversion \%}$$

$$Y_3 = \text{Preasphaltene \%} = (X_3 / \Sigma X_i) \times \text{coal conversion \%}$$

$$Y_4 = \text{Oil \%} = (X_4 / \Sigma X_i) \times \text{coal conversion \%}$$

$$Y_5 = \text{IOM \%} = (\text{IOM}/\text{maf coal}) \times 100$$

$$\Sigma Y_i = 100.0 \text{ was checked for each set of calculations.}$$

Two assumptions were made based on the reaction and product analysis conditions used in this study. The assumptions were taken as the basis for the calculation. The first assumption was that the input solvent hexadecane or hexadecane plus hexahydroanthracene in some reactions involved in the calculation was recovered only as hexane solubles or oil in the reaction products. In other words, no input solvent went into any other product fraction except for the hexane soluble fraction of the product slate. This assumption was made since at the reaction temperature in conjunction with other reaction conditions that were used in this research, no production of gas, asphaltenes, preasphaltenes and insoluble organic matter resulted from the input solvent. The second assumption was that approximately 80% of the input solvent was recovered as hexane solubles in the product. Hence, the amount that was equivalent as 80% of the input solvent was discounted from the hexane solubles of each reaction and the rest of the oil product was considered to result from coal itself. This assumption comes from the fact that for most of the reactions dividing the hexane solubles by the input solvent resulted in the value ranging from 0.65 to 0.95

or 65% to 95% with 80% being the percent average[1].

Three sample calculations were provided as the following:

- Sample Calculation 1:

In the thermal reaction of Illinois No. 6 coal performed at 350 °C, with 3.4 MPa H<sub>2</sub> introduced at ambient temperature, with hexadecane as solvent, for 30 min, the following experimental data were obtained[2]:

Input Data

$$\text{Gas} = 0.145 \text{ g}$$

$$\text{Oil} = 3.664 \text{ g}$$

$$\text{Asphaltene} = 0.061 \text{ g}$$

$$\text{Preasphaltene} = 0.364 \text{ g}$$

$$\text{IOM} = 1.142 \text{ g}$$

$$\text{solvent in the feed} = 65.68\%$$

$$\text{Recovery} = 89.61\%$$

Based on these data, the following calculations were performed:

$$\text{Total output} = 0.145 + 3.664 + 0.061 + 0.364 + 1.142 = 5.376 \text{ g}$$

$$\text{Total input} = 5.376 * 0.8961 = 5.9993 \text{ g}$$

$$\text{coal conversion} = 0.2702 \text{ or coal conversion} = 27.02\%$$

$$\text{maf coal} = 1.142 / 0.7298 = 1.5648 \text{ g}$$

$$X_1 = 0.145 / 1.5648 = 0.09266$$

$$X_2 = 0.061 / 1.5648 = 0.03898$$

$$X_3 = 0.364 / 1.5648 = 0.23262$$

$$X_4 = (3.664 - 5.9993 \times 0.6568 \times 0.80) / 1.5648 = 0.3270$$

$$\Sigma X_i = 0.69127$$

$$\text{Gas \%} = (0.09266 / 0.69127) \times 27.02 = 3.6$$

$$\text{Asphaltene \%} = (0.03898 / 0.69127) \times 27.02 = 1.5$$

$$\text{Preasphaltene \%} = (0.23262 / 0.69127) \times 27.02 = 9.1$$

$$\text{Oil \%} = (0.32701 / 0.69127) \times 27.02 = 12.8$$

$$\text{IOM \%} = 100 - 27.02 = 72.98 \approx 73.0$$

$$\text{check: } 3.6 + 3.0 + 17.6 + 12.8 + 73.0 = 100.0$$

- Sample Calculation 2:

In the thermal reaction with Illinois No. 6 coal performed at 350°C, 3.4 MPa H<sub>2</sub> introduced at ambient temperature, with hexadecane as solvent and hexahydroanthracene as hydrogen donor, for 30 min, the following experimental data were obtained[3]:

Input Data

$$\text{Gas} = 0.145 \text{ g}$$

$$\text{Oil} = 3.093 \text{ g}$$

$$\text{Asphaltene} = 0.13 \text{ g}$$

$$\text{Preasphaltene} = 1.463 \text{ g}$$

$$\text{IOM} = 0.927 \text{ g}$$

$$\text{solvent in the feed} = 64.80\%$$

$$\text{Recovery} = 97.93\%$$

The calculations were made as following:

$$\text{Total output} = 0.145 + 3.093 + 0.13 + 1.463 + 0.927 = 5.758 \text{ g}$$

$$\text{Total input} = 5.758 \times 0.9793 = 5.8797 \text{ g}$$

$$\text{coal conversion} = 0.4067 \text{ or coal conversion \%} = 40.67\%$$

$$\text{maf coal} = 0.927/0.5933 = 1.5624 \text{ g}$$

$$X_1 = 0.145/1.5624 = 0.09280$$

$$X_2 = 0.13/1.5624 = 0.08320$$

$$X_3 = 1.463/1.5624 = 0.93635$$

$$\Sigma X_i = 1.1411$$

$$\text{Gas \%} = (0.09280/1.1411) \times 40.67 = 3.3$$

$$\text{Asphaltene \%} = (0.08320/1.1411) \times 40.67 = 3.0$$

$$\text{Preasphaltene \%} = (0.93635/1.1411) \times 40.67 = 33.4$$

$$\text{Oil \%} = (0.02877/1.1411) \times 40.67 = 1.0$$

$$\text{IOM \%} = 100 - 40.67 = 59.33 \approx 59.3$$

$$\text{check: } 3.3 + 3.0 + 33.4 + 1.0 + 59.3 = 100.0$$

- Sample calculation 3:

In the thermal reaction with Illinois No. 6 coal performed at 300°C, 3.4 MPa H introduced, at ambient temperature, with hexadecane as solvent and hexahydroanthracene as hydrogen donor, for 30 min, the following experimental data were obtained[4]:

#### Input Data

$$\text{Gas} = 0.140 \text{ g}$$

$$\text{Oil} = 3.362 \text{ g}$$

$$\text{Asphaltene} = 0.171 \text{ g}$$

$$\text{Preasphaltene} = 0.249 \text{ g}$$

$$\text{IOM} = 1.512 \text{ g}$$

$$\text{solvent in the feed} = 65.03\%$$

$$\text{Recovery} = 90.62\%$$

The calculations were performed as following:

$$\text{Total output} = 0.140 + 3.362 + 0.171 + 0.249 + 1.512 = 5.434 \text{ g}$$

$$\text{Total input} = 5.434 \times 0.9062 = 5.9965 \text{ g}$$

$$\text{coal conversion} = 0.1007 \text{ or coal conversion} = 10.07\%$$

$$\text{maf coal} = 1.512 / 0.8993 = 1.6813 \text{ g}$$

$$X_1 = 0.140 / 1.6813 = 0.08327$$

$$X_2 = 0.171 / 1.6813 = 0.1017$$

$$X_3 = 0.249 / 1.6813 = 0.1481$$

$$X_4 = (3.362 - 5.9965 \times 0.6503 \times 0.80) / 1.6813 = 0.1442$$

$$\Sigma X_i = 0.4773$$

$$\text{Gas \%} = (0.08327 / 0.4773) \times 10.07 = 1.8$$

$$\text{Asphaltene \%} = (0.1017 / 0.4773) \times 10.07 = 2.2$$

$$\text{Preasphaltene \%} = (0.1481 / 0.4773) \times 10.07 = 3.1$$

$$\text{Oil \%} = (0.1442 / 0.4773) \times 10.07 = 3.0$$

$$\text{IOM \%} = 100 - 10.07 = 89.93 \approx 89.9$$

$$\text{check: } 1.8 + 2.2 + 3.1 + 3.0 + 89.9 = 100.0$$

**Table 1. Product Distribution for Hexahydroanthracene Hydrogenation<sup>a</sup>**

| Catalyst                           | Temperature (°C) | Product Distribution (wt%) |           |           |          |
|------------------------------------|------------------|----------------------------|-----------|-----------|----------|
|                                    |                  | ANT <sup>b</sup>           | DHA       | HHA       | OHA      |
| None                               | 350              | 0.0±0.0                    | 41.6±2.07 | 23.3±0.7  | 35.2±1.5 |
|                                    | 300              | 0.0±0.0                    | 6.7±0.8   | 70.8±3.2  | 22.5±2.8 |
|                                    | 250              | 0.0±0.0                    | 0.0±0.0   | 92.1±2.14 | 8.0±2.1  |
| Molyvan L (500 ppm) without sulfur | 350              | 0.0±0.0                    | 0.0±0.0   | 42.6±1.6  | 57.5±1.6 |
|                                    | 300              | 0.0±0.0                    | 0.0±0.0   | 39.0±1.1  | 61.0±1.1 |
|                                    | 250              | 0.0±0.0                    | 0.0±0.0   | 46.7±1.0  | 53.3±1.0 |
| Molyvan 822 (500 ppm) with sulfur  | 350              | 0.0±0.0                    | 11.2±0.5  | 40.5±1.0  | 48.4±1.3 |
|                                    | 300              | 0.0±0.0                    | 17.8±0.7  | 38.2±0.6  | 44.0±0.5 |
|                                    | 250              | 4.9±0.9                    | 9.6±0.8   | 45.4±1.7  | 40.1±1.5 |
| Mo Naph (500 ppm) with sulfur      | 350              | 0.0±0.0                    | 13.9±0.8  | 44.8±1.8  | 41.2±1.0 |
|                                    | 300              | 0.0±0.0                    | 12.6±1.6  | 40.3±0.8  | 47.1±1.8 |
|                                    | 250              | 6.7±1.0                    | 9.8±0.9   | 29.2±2.4  | 54.3±0.7 |

<sup>a</sup> Reaction Conditions: 2 wt% Hexahydroanthracene, 30 min, 3.45 MPa hydrogen pressure introduced at ambient temperature.

<sup>b</sup> ANT = anthracene, DHA = dihydroanthracene, HHA = hexahydroanthracene, OHA = octahydroanthracene.

**Table 2. Coal Conversion and Solvent-Free Product Distribution for Low Severity Liquefaction Using Various Catalysts**

| Catalyst                 | Level (ppm) | Time (min) | Temperature (°C) | %DH <sup>b</sup> from HHA | % Coal Conversion | Solvent-Free Product Distribution (wt%) |          |            |               |                  |
|--------------------------|-------------|------------|------------------|---------------------------|-------------------|-----------------------------------------|----------|------------|---------------|------------------|
|                          |             |            |                  |                           |                   | Gas                                     | Oil      | Asphaltene | Preasphaltene | IOM <sup>c</sup> |
| None                     | 0           | 30         | 350              | 0.0                       | 27.0±3.0          | 3.6±0.2                                 | 12.8±3.0 | 1.5±0.2    | 9.1±1.3       | 73.0±3.0         |
| Molyvan L <sup>d</sup>   | 500         | 30         | 350              | 0.0                       | 29.6±2.6          | 3.6±0.4                                 | 17.2±2.1 | 1.7±0.3    | 7.1±0.2       | 70.4±2.6         |
| Molyvan 822 <sup>e</sup> | 500         | 30         | 350              | 0.0                       | 25.4±0.2          | 2.8±0.8                                 | 15.3±1.3 | 1.4±0.3    | 5.9±1.9       | 74.6±0.2         |
| MoNaph <sup>e</sup>      | 500         | 30         | 350              | 0.0                       | 31.1±1.4          | 1.9±1.1                                 | 16.6±1.4 | 2.5±1.1    | 10.1±1.1      | 68.9±1.4         |
| None                     | 0           | 30         | 350              | 0.5                       | 40.7±2.1          | 3.3±0.1                                 | 1.0±0.3  | 3.0±0.8    | 33.4±0.7      | 59.3±2.1         |
| Molyvan L <sup>d</sup>   | 500         | 30         | 350              | 0.5                       | 43.4±2.6          | 3.4±0.0                                 | 12.0±0.7 | 2.8±0.6    | 25.2±1.4      | 56.6±2.6         |
| Molyvan 822 <sup>e</sup> | 500         | 30         | 350              | 0.5                       | 39.2±2.2          | 3.5±0.6                                 | 6.6±0.2  | 3.3±0.5    | 25.8±1.6      | 60.8±2.2         |
| MoNaph <sup>e</sup>      | 500         | 30         | 350              | 0.5                       | 45.8±0.8          | 4.9±0.1                                 | 9.4±0.8  | 3.6±0.7    | 27.9±1.7      | 54.2±0.8         |
| None                     | 0           | 30         | 300              | 0.5                       | 10.1±2.1          | 1.8±0.0                                 | 3.0±1.7  | 2.2±0.2    | 3.1±1.7       | 89.9±2.1         |
| Molyvan L <sup>d</sup>   | 500         | 30         | 300              | 0.5                       | 13.3±5.5          | 1.7±0.3                                 | 4.1±2.1  | 2.1±1.5    | 5.4±2.1       | 86.7±5.5         |
| Molyvan 822 <sup>e</sup> | 500         | 30         | 300              | 0.5                       | 5.0±0.2           | 0.7±0.1                                 | 2.9±0.5  | 0.3±0.3    | 1.1±1.1       | 95.0±1.0         |
| MoNaph <sup>e</sup>      | 500         | 30         | 300              | 0.5                       | 2.2±0.4           | 0.3±0.0                                 | 0.6±0.1  | 0.1±0.1    | 1.2±0.5       | 97.8±0.4         |

<sup>a</sup> Reaction Conditions: 3.45 MPa of H<sub>2</sub> introduced at ambient temperature; 2 g of coal; 4.0 g of solvent including hexadecane and hexahydroanthracene.

<sup>b</sup> % Donable Hydrogen

<sup>c</sup> Insoluble Organic Matter (ash-free)

<sup>d</sup> Without Sulfur

<sup>e</sup> With Sulfur

Table 3. Effect of Variations in Reaction Conditions on Coal Conversion and Solvent-Free Product Distribution

| Catalyst                     | Level (ppm) | Time (min) | Temperature (°C) | Pressure (MPa)    | %DH <sup>a</sup> from HHA | % Coal Conversion | Solvent-Free Product Distribution (wt%) |          |             |                |                  |
|------------------------------|-------------|------------|------------------|-------------------|---------------------------|-------------------|-----------------------------------------|----------|-------------|----------------|------------------|
|                              |             |            |                  |                   |                           |                   | Gas                                     | Oil      | Asphaltenes | Preasphaltenes | IOM <sup>b</sup> |
| None                         | 0           | 30         | 350              | 3.45              | 0.5                       | 40.7±2.1          | 3.3±0.1                                 | 1.0±0.3  | 3.0±0.8     | 33.4±0.7       | 59.3±2.1         |
|                              | 0           | 30         | 350              | 6.90 <sup>c</sup> | 0.5                       | 38.9±3.5          | 7.2±0.4                                 | -2.1±1.1 | 2.6±0.3     | 31.2±1.1       | 61.1±3.5         |
|                              | 0           | 60         | 350              | 3.45              | 0.5                       | 49.2±0.5          | 5.5±0.2                                 | 11.4±0.4 | 5.3±0.7     | 27.0±1.7       | 50.8±0.5         |
|                              | 0           | 30         | 350              | 3.45              | 1.0                       | 44.3±0.3          | 3.3±0.1                                 | 7.2±0.9  | 3.6±0.0     | 30.2±1.3       | 55.7±0.3         |
|                              | 0           | 60         | 350              | 6.90 <sup>c</sup> | 0.5                       | 52.2±1.4          | 10.1±0.3                                | 11.4±1.9 | 4.6±1.9     | 26.1±0.8       | 47.8±1.4         |
|                              | 0           | 60         | 350              | 3.45              | 1.0                       | 52.2±2.8          | 8.3±0.1                                 | 13.2±2.1 | 7.1±2.0     | 23.6±2.1       | 47.8±2.8         |
|                              | 0           | 60         | 350              | 6.90 <sup>c</sup> | 1.0                       | 55.0±3.3          | 8.3±0.3                                 | 13.7±0.4 | 3.9±1.1     | 29.1±1.1       | 45.0±3.3         |
|                              | 500         | 30         | 350              | 3.45              | 0.5                       | 45.8±0.8          | 5.1±0.1                                 | 8.0±2.6  | 3.8±0.7     | 28.9±1.7       | 54.2±0.8         |
|                              | 500         | 30         | 350              | 6.90 <sup>c</sup> | 0.5                       | 38.8±2.3          | 6.1±0.5                                 | 5.1±2.3  | 1.9±0.5     | 25.7±2.3       | 61.2±2.3         |
|                              | 500         | 60         | 350              | 3.45              | 0.5                       | 49.9±2.4          | 5.0±0.5                                 | 16.8±0.7 | 5.2±1.6     | 22.9±0.8       | 50.1±2.4         |
| MoNaph <sup>d</sup> + Sulfur | 500         | 30         | 350              | 3.45              | 1.0                       | 46.3±0.0          | 4.6±0.2                                 | 12.4±2.5 | 3.6±0.2     | 25.7±3.3       | 53.7±0.0         |
|                              | 1000        | 30         | 350              | 3.45              | 0.5                       | 50.1±2.6          | 4.6±0.4                                 | 2.6±2.1  | 4.5±2.1     | 38.4±2.6       | 49.9±2.6         |
|                              | 500         | 60         | 350              | 6.90 <sup>c</sup> | 0.5                       | 50.8±2.7          | 8.9±0.3                                 | 14.7±2.4 | 6.1±0.4     | 21.1±2.6       | 49.2±2.7         |
|                              | 500         | 60         | 350              | 3.45              | 1.0                       | 57.8±2.0          | 8.9±0.1                                 | 12.2±2.9 | 6.4±2.0     | 30.3±2.9       | 42.2±2.0         |
|                              | 1000        | 30         | 350              | 6.90 <sup>c</sup> | 0.5                       | 49.5±2.6          | 9.3±0.5                                 | 17.8±0.1 | 2.6±1.5     | 19.8±1.0       | 50.5±2.6         |
|                              | 500         | 60         | 350              | 6.90 <sup>c</sup> | 1.0                       | 58.8±3.3          | 9.2±0.2                                 | 16.9±0.0 | 4.2±1.3     | 28.5±0.7       | 41.2±3.3         |
|                              | 1000        | 60         | 350              | 6.90 <sup>c</sup> | 0.5                       | 54.0±1.3          | 9.2±0.1                                 | 13.7±1.3 | 4.3±0.0     | 26.8±1.3       | 46.0±1.3         |

<sup>a</sup> % Donable Hydrogen

<sup>b</sup> Insoluble Organic Matter (ash-free)

<sup>c</sup> Shaded areas indicate change from baseline condition.

<sup>d</sup> With Sulfur

**Table 4. Effect of Coal Type and Pretreatment on Coal Conversion and Solvent-Free Product Distribution<sup>a</sup>**

| Coal                                 | %DH <sup>b</sup><br>from<br>HHA | % Coal Conversion     | Solvent-Free Product Distribution |          |             |                |          |
|--------------------------------------|---------------------------------|-----------------------|-----------------------------------|----------|-------------|----------------|----------|
|                                      |                                 |                       | Gas                               | Oil      | Asphaltenes | Preasphaltenes | IOM      |
| Untreated Illinois No.6 <sup>c</sup> | 0.0                             | 31.1±1.4 <sup>c</sup> | 1.9±1.1                           | 16.6±1.4 | 2.5±1.1     | 10.1±1.1       | 68.9±1.4 |
| Untreated Illinois No.6              | 1.0                             | 62.9±1.5              | 5.5±0.2                           | 18.9±0.3 | 11.7±0.3    | 26.9±1.6       | 37.1±1.5 |
| Pretreated Illinois No.6             | 1.0                             | 67.0±1.8              | 4.7±0.4                           | -4.5±1.0 | 7.9±1.0     | 58.8±0.6       | 33.0±1.8 |
| Untreated Wyodak                     | 0.0                             | 23.5±4.0              | 3.2±0.3                           | 17.1±2.1 | 1.1±0.3     | 2.1±0.1        | 76.5±4.0 |
| Untreated Wyodak                     | 1.0                             | 39.9±0.7              | 4.1±0.1                           | 17.6±1.3 | 6.8±1.2     | 11.4±0.1       | 60.1±0.1 |
| Pretreated Wyodak                    | 1.0                             | 48.6±0.0              | 4.2±0.1                           | 16.4±1.8 | 8.2±1.5     | 19.8±0.4       | 51.4±0.5 |

<sup>a</sup> Reaction Conditions: 350 °C, 60 minutes, 1000 ppm Mo, 3.45 MPa H<sub>2</sub> introduced at ambient temperature.

<sup>b</sup> % Donable Hydrogen

<sup>c</sup> Reaction Conditions: 350 °C, 30 min, 500 ppm, 3.45 MPa H<sub>2</sub> introduced at ambient temperature.

## Part 2

### High Temperature Infrared Analysis of Cyclic Olefins

The goal of the of the research being performed is to develop a methodology for analyzing the reactivity of cyclic olefins at high temperature in situ using a high temperature and high pressure infrared cell. All of the work has been focused toward that end. The high temperature cell was purchased from AABSPEC. Initial experiments were performed at 50 °C without cooling. During the quarter a modification was made to the AABSPEC cell which allowed sufficient coolant to flow to the cell. Two sets of quick-connects were installed to increase the ease of operation. One set simplifies mounting and dismounting of the cell, while the other set allows the bench lid to be removed without disconnecting the plumbing from the back of the cooler. The flow capacity of the cooling system was determined to be 2000 ml/hr. The AABSPEC cell manual indicates that a minimum flow rate of 4200 ml/hr with a coolant temperature of -20 °C is required to provide adequate cooling for sustained high temperature (425 °C) work. The minimum flow rate required at lower temperatures is a function of the coolant temperature. It is not known at this time whether or not a flow rate of 2000 ml/hr will be adequate for our purposes.

After the modifications to the FTIR were completed, attempts to run spectra resulted in the discovery that the unit would not scan. Electronic support personnel determined that the IR source kept turning itself off because of a lack of cooling. The coolant pump was working fine so a plug in the line was suspected. The quick-connects at the back of the model which were thought to be impeding the coolant flow were eliminated. The IR source remained on afterwards, and background scans looked like normal background scans. The cover was left off the module so that the coolant level could be monitored on a daily basis. Over the course of the quarter the coolant reservoir lost about a third of its coolant. Since it is not desirable to leave the cover off the module on a long-term basis, and since it is quite time consuming to remove the cover in order to check the coolant level, operations personnel have suggested putting a door in the cover which

would allow access to the reservoir.

Nicolet was contacted about the cost of the software packages for quantitation and deconvolution of peaks obtained using the high temperature IR cell. Each package cost \$1500.00. Research Electronics Support Facility (RESF) personnel suggested we obtain a service manual, which was available at a cost of \$300.00, as well as schematics to the FTIR. The result of discussions with Nicolet indicated that they did not want to supply the schematics.

The partial least squares quantitation package was installed on the hard disk. It seemed to have loaded properly, but when attempts were made to run it, the disk operating system responded with "invalid memory request". It was subsequently determined that the existing computer did not have sufficient memory space to run the software. The cost of upgrading the existing system was compared to replacing the computer. In consideration of the advantages offered by a new system, it was decided to purchase a new computer. RESF was requested to assist in this purchase.

Val Rossiter of AASBSPEC was contacted about the use of the high temperature/high pressure cell. Since the salt windows are known to lose their mechanical strength with increasing temperature, he was asked specifically for a temperature versus maximum safe working pressure profile. He said he did not have one, that we should make our own. In discussions with RESF, it was decided that perhaps the best approach would be to monitor the pressure in the internal chamber using a pressure transducer/readout, and pressurize the external gas chamber accordingly so as to minimize the pressure differential across the internal cell windows. The very small size of the pressure sending element. A piezoelectric crystal is being considered. A literature search revealed about 20 U.S. manufacturers of piezoelectric pressure sensors.

#### **Future Work**

The new computer and software is expected to arrive early in Spring quarter, 1995. A number of vendors of piezoelectric pressure sensors will be contacted to determine if a shelf item is available that is suitable to our purposes. We may be forced to explore other methods of

controlling the pressure in the cell. After the installation of the new computer system, the inevitable problems which accompany such changes will need to be worked through. A good deal of training of operations personnel is anticipated. It should be possible to evaluate the least-squares quantitation and deconvolution packages using previously collected data. Reproducibility studies of the useful concentration range of the AABSPEC cell for tetralin, decalin, and naphthalene are to be conducted. This work will be extended to 1,2-dihydronaphthalene and octahydronaphthalene. The absorbance versus concentration behavior of mixtures of aromatic and cyclic olefins will be investigated. Spectra will be obtained under conditions of incrementally increased temperature and pressure until low severity liquefaction conditions are reached.

#### Reference

- [1] AnPing Huang, Master's Thesis, Auburn University, June, 1994.
- [2] Table 11 in AnPing Huang's Master Thesis, Auburn University, June, 1994.
- [3] Table 13 in AnPing Huang's Master Thesis, Auburn University, June, 1994.
- [4] Table 12 in AnPing Huang's Master Thesis, Auburn University, June, 1994.