APPENDIX B

ANALYTICAL TECHNIQUES

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During the past year, the following were the main projects in the analytical area:

1. The Carle Model 520 gas chromatograph, for online analysis of the gas phase protion of LHF products, was delivered in late May 1982. Several major modifications to the original plumbing scheme were required during test-out to obtain the desired chromatographic separations. These changes were made and the instrument has been routinely used since mid-June.

2. The Envirochem/Unacon Model 810B sampling concentrating purge and trap G.C., which was ordered to do capillary column analysis of the C_6 to C_{22} carbon range "heart cut" of the LHF product samples, arrived in early July. The instrument was set up and a method was developed to fractionate and analyze the heart cut.

During the course of the year, a number of problems developed with this instrument ranging from electrical component failures to valve leaks. Maintenance and down time have been extensive.

3. Efforts continued to find a routine reliable analytical method for determining hydrocarbon group-types in LHF samples. Result discrepancies of duplicate samples submitted for FIA analysis, have made that method very suspect. As a cross check, six pairs of duplicate samples were sent to an independent petroleum testing laboratory for FIA analysis. The reproducibility and "probable" accuracy of their results were just as suspect as those obtained by our analytical laboratory. Although reasons for these discrepancies cannot fully be explained, we have found that most of our LHF samples have a natural fluorescence under U.V. light. This may be contributing to, or masking the color bands of the FIA test.

Liquid chromatography as an alternate to FIA analysis has not materialized. Attempts by several LC manufacturers to separate our LHF samples into three distinct fractions (saturates, olefins,

aromatics) were unsuccessful. Present column technology is not suitable to the wide boiling range of our samples. The best that has been done is separation of two fractions--a combination saturates plus olefins fraction and an aromatic fraction.

Samples from one remaining LC manufacturer still are to be evaluated. This work has been slowed by maintenance downtime of the Envirochem 810B chromatograph. The 810B has been essential in analyzing the LC fractions submitted by the various manufacturers.