

FE-2645-06

Distribution Categories UC-90 and UC-90d

DEASPHALTING, DEASHING AND UPGRADEING OF COAL LIQUIDS

Quarterly Technical Progress Report  
for  
The Period January-March, 1980

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Date Published - June, 1980

PREPARED FOR THE UNITED STATES  
DEPARTMENT OF ENERGY

Under Contract No. ET-78-C-01-2645

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## ABSTRACT

Continuing work on deashing SRC-I filter feed has yielded deashed oils containing 0.004 wt-% ash or less. The lowest reject bottoms yield was 18.8 wt-%. This fails to meet the target of 15% maximum rejection.

A summary of the deashing work on SRC-I filter feed has also shown that rejection decreased with use of higher boiling point solvents, with addition of up to 10% light aromatic component to the solvent, and with increased operating temperature.

The hydrotreatment of the deashed oil derived from SRC-I filter feed yielded oils containing 9.99 wt-% and 10.17 wt-% hydrogen. Recoveries of oil ranged from 99.20 to 99.65 wt-%. Hydrogen consumption was 1080-1090 SCFB hydrogen.

Two deashed oils derived from UOP coal liquefaction product have been catalytically cracked. Conversions ranged from 44 to 60 vol-%.

## Work Accomplished

### Task 2.0

The deashing of two drums of SRC-I filter feed, 3777-3 and 3777-4, (Tables 1 and 2) was continued in UOP Research Plant 633.

UOP deashing proceeds as follows: preheated SRC-I filter feed is combined with preheated solvent upstream of the deasher. The combined feed is passed downflow through the in-line mixer into the deasher (Figure 1). Phase separation takes place in the deasher. The oil-rich upper phase flows to the stripper for removal of solvent. The solid rich bottom phase, containing the ash, is removed through a lock hopper system, which is controlled by a capacitance sensing interface detector. Both deashed oil and deasher bottoms are weighed and analyzed. The percent deashed oil and percent reject bottoms are calculated on the basis of the total solvent-free effluent.

Solvents used in this study included a light paraffin, designated "Paraffin A"; a higher boiling paraffin, designated "Paraffin B"; and a light aromatic. The aromatic was used both neat and at 10% concentration in the paraffin.

Four runs are reported covering effects of solvent type and residence time on deashing the SRC-I filter feed. This solvent study was done to determine the best solvent to achieve the objective of a maximum 15 wt-% bottoms rejection.

### Plant 633, Run 97

A summary of deashing Run 97 is presented in Table 3. This was the initial work on SRC-I filter feed. The in-line mixer was not used for this run. No change in rejection was noted when the deashing temperature was increased. When the residence time was increased by 100%, only a slight decrease in bottoms rejection, from 48.3% to 40.9%, was observed.

For comparison, data are presented from a subsequent operation, Run 116, reported in FE-2645-05. The in-line mixer had been reinserted prior to Run 116 after mechanical problems were resolved. The rejection dropped from 48.3% to 24.5%. The major part of this reduction is attributed to the insertion of the mixer, rather than the addition of a light aromatic component to the solvent.

Inspections of the deashed oil from Run 97 show a low C<sub>7</sub> insoluble content. This would be expected in view of the high rejection. The ash content of the deashed oil was less than 10 ppm. Inspection of the reject bottoms shows the effect of the dilution with SRC solvent. Both the ash and toluene insoluble contents, 4.96 wt-% and 30.0 wt-% respectively, are low when compared to the reject bottoms from Run 116 which are 19.8 wt-% and 94.3 wt-% respectively.

#### Plant 633, Run 120

A summary of deashing Run 120 is given in Table 4. The solvent used in this operation was a light aromatic. The high ash content of the deashed oil and the low rejection show the operation to be essentially one of settling. A doubling of the residence time showed a minor decrease in rejection, from 7.8 down to 6.2 wt-%. A decrease in temperature did not result in an increased rejection as expected, but decreased rejection from 6.2 to 0.5 wt-%. The ash content of the deashed oil did not change with the drop in temperature. These results are consistent with deashing by settling, rather than phase separation.

#### Plant 633, Run 122

A summary of deashing Run 122 is given in Table 5. The solvent was changed to a higher boiling point paraffin B. The operating temperature was also increased slightly to 55°C above base temperature. Up to 26 hours on stream, feedstock was 3777-3 (Table 1). Thereafter, feedstock 3777-4 (Table 2) was used. The rejection was 22.5 wt-% as compared to 24.5 wt-% using paraffin A in Run 116 (Table 3). This deashed oil contained 30 ppm ash and had a low toluene insoluble content of 0.17 wt-%.

The recovery in Run 122 was 80.4 wt-%. All losses were assigned to reject bottoms, which remained in the deashing reactor when both the level control and lock hopper systems malfunctioned. The 22.5% rejection reported is the difference between the recovered overhead and 100 wt-%.

#### Plant 633, Run 123

A summary of deashing Run 123 is given in Table 5. The solvent was modified by addition of 10 vol-% light aromatic to the higher boiling paraffin B solvent. The operation was started at 44°C above base temperature to obtain an initial rejection value of 21.4 wt-%. The deashing temperature was raised to 81°C above base and the plant was run out to 108 hours. This 37°C increase in temperature resulted in a modest decrease in rejection from 21.4 wt-% down to 18.8 wt-%. The 15 wt-% rejection level was not reached.

A summary of the solvent study at a 1.0 residence time is given below.

Conditions

|                 | 97 <sup>a</sup>                                       | 116   | 122   | 123   | 123   | 120 <sup>b</sup>  |
|-----------------|---|---|---|---|---|-------------------|
| Run             | 97 <sup>a</sup>                                       | 116   | 122   | 123   | 123   | 120 <sup>b</sup>  |
| Test, hrs.      | 64-88   | 12-29   | 12-25   | 12-28   | 28-108  | 4-20              |
| T-T(base), °C   | 49  | 48  | 55  | 44  | 81  | 45                |
| Solvent Type    | Paraffin A<br>90% Paraffin A<br>10% Light<br>Aromatic | Paraffin B<br>90% Paraffin B<br>10% Light<br>Aromatic | Light<br>Aromatic |
| Rejection, Wt-% | 48.3  | 24.5  | 22.5  | 21.4  | 18.8  | 7.8               |
| Reject Bottoms  |   |   |   |   |   |                   |
| Ash, Wt-%       | 4.96  | 19.80   |   | 21.0  | 22.3  | ~100              |
| Deashed Oil     |   |   |   |   |   |                   |
| Ash, Wt-%       | ~0.001  | 0.001   | 0.003   | 0.002   | 0.004   | 1.18              |

<sup>a</sup>No in-line mixer used

<sup>b</sup>Settling occurred in this run

Comparison of Runs 116 and 123 at corresponding temperatures indicates that the desired lowering of rejection was obtained by replacing paraffin A with the heavier paraffin B in the solvent blend. Comparison of Runs 122 and 123 indicates that rejection is also lowered by addition of 10% aromatic. The temperature effect shown in Run 123 is opposite that displayed in petroleum deasphalting, where rejection increases with increase in temperature.

While ash content of the DAO was consistently low under deashing conditions, the target rejection of 15 wt-% maximum could not be met.

Task 3.0

Hydrotreating the deashed oil obtained from deashing SRC-I filter feed was completed in UOP Plant 638 (Figure 2).

The deashed oil was processed with hydrogen, downflow, over a fixed bed of 630 cc of UOP black oil conversion catalyst. Water was injected into the reactor effluent line to pick up ammonia. The water was then removed in a 3-phase separator. The hydrotreated deashed oil was sent to a stripper and debutanizer column for removal of light gases and hydrogen sulfide.

Two feed blends of deashed oil were made, based on the extent of deashing rejection. The first blend, 3777-47 (Table 6), consisted of oil from deashing operations where rejections ranged from 19 to 25 wt-%. The second feed blend, 3777-48 (Table 7), consisted of oil from deashing operations having rejections ranging from 41 to 48 wt-%. No oil from operations in which settling occurred were hydrotreated.

Plant 638, Run 48

The deashed oils were processed over the commercial black oil conversion catalyst at 20°C below base temperature, 300 psi below base pressure and at 2.0 LHSV relative to base over a period of 244 hours. Deashed oil 3777-47 was processed for the first 184 hours, deashed oil 3777-48 was processed from 184-244 hours. A summary of the operation is shown in Table 8. Product samples taken from the plant during the run were sent for C<sub>7</sub> insoluble, and carbon and hydrogen analyses. The operation ran well with only a slight catalyst deactivation, as measured by the product hydrogen content, over the first 184 hours of operation.

Liquid products from the line-out through period 16, ending at 184 hours, were blended and designated hydrotreated deashed oil 3777-50. Inspections of this product are shown in Table 9. Liquid products from periods 17 through 22 (184-244 hrs.) were also blended and designated hydrotreated deashed oil 3777-51. Inspections of this product are shown in Table 10.

Product distributions, hydrogen consumption and distribution of hydrogen consumptions were calculated for each of the feedstocks. The data are presented in Tables 11 and 12 for feedstock 3777-47 and in Tables 13 and 14 for feedstock 3777-48. High yields, 99.2 and 99.65 wt-% of hydrotreated oil were achieved.

#### Task 5.0

Two additional FCC runs were made on deashed oils in UOP Research Plant 593. Feedstock for Run 303 (Table 15) was deashed oil 3777-42, described in Table 10, Report FE-2645-04. Feedstock for Run 304 (Table 16) was hydrotreated deashed oil 3777-46, described in Table 18, Report FE-2645-04. Both these stocks, as well as that fed in the previously reported FCC Run 302 (Table 19, FE-2645-05) were derived from liquefaction of Illinois No. 6 coal, using deashed oil as solvent. Cracking conditions were as follows:

$$\begin{aligned} P-P(\text{base}) &= -10 \text{ psi} \\ T-T(\text{base}) &= 3 \text{ and } 31^\circ\text{C} \\ \frac{\text{Cat/Oil}}{\text{Cat/Oil(base)}} &= 1 \text{ and } 1.4 \end{aligned}$$

Due to a feedstock shortage, only two tests (at the lower temperature condition) could be made on deashed oil 3777-42. Four tests were made at each temperature on the hydrotreated deashed oil 3777-46. Only the acceptable tests are listed in the tables.

The cut point of the plant fractionator was set too low during Run 304. The yield data and gravities for the gasolines and cycle oils have been adjusted, based on a 380°F temperature at the 90 vol-% point of the Engler distillation.

The volume percent conversion of these feedstocks is abstracted from the tables and shown below. Conversion data from the first hydrotreated deashed oil 3777-43 presented in Report FE-2645-05 are also presented for comparison:

| Feed No.                   | 3777-42 | 3777-43 | 3777-46 |
|----------------------------|---------|---------|---------|
| Hydrogen, Wt-%             | 9.77    | 10.19   | 10.57   |
| <u>Volume % Conversion</u> |         |         |         |
| T-T(base) °C               |         |         |         |
| 3                          | 44.2    | 49.4    | 57.5    |
| 31                         |         | 55.7    | 60.2    |

The data show the expected increasing conversion with increasing hydrogen content of the feed. Overall conversions appear to be low, and reflect the low hydrogen contents of the feeds.

This completes Task 5 for the UOP coal liquefaction product.

## Work Forecast

### Task 4.0

Task 4.0 (Hydrocracking) cannot reasonably be performed for SRC-I filter feed, because of lack of success in obtaining deashed oil from this stock at a reasonably low rejection.

Since the filter feed comprises about two parts recycle solvent and one part SRC, rejections above 15% represent less than 50% recovery of SRC.

In the case of UOP coal liquefaction product, supply of feedstock was exhausted in FCC experiments. Preparation of additional feedstock, using hydrotreated deashed oils 3777-50 and 3777-51 as solvent, will be necessary.

### Task 5.0

Task 5.0 (FCC) cannot reasonably be performed for SRC-I filter feed for reason noted above.

Table 1

Inspections of SRC-I Filter Feed 3777-3

|                                |        |
|--------------------------------|--------|
| Drum No.                       | 1      |
| °API @ 60°F                    | -4.4   |
| Sp. Gr. @ 60°F                 | 1.1133 |
| Distillation ASTM D-86         |        |
| IBP°F                          | 445    |
| 5%                             | 490    |
| 10%                            | 515    |
| 20%                            | 551    |
| 30%                            | 586    |
| 40%                            | 622    |
| 50%                            | 675    |
| 60%                            | 741    |
| 70%                            | 830    |
| 80%                            |        |
| 90%                            |        |
| 95%                            |        |
| EP                             |        |
| % Over                         | 70.0   |
| % Bottoms                      | 30.0   |
| Hydrogen, Wt-%                 | 7.55   |
| Carbon, Wt-%                   | 83.85  |
| Sulfur, Wt-ppm                 | 8300   |
| Nitrogen, Wt-ppm               | 12670  |
| Oxygen, Wt-ppm                 | 22700  |
| Ash ASTM, Wt-%                 | 3.42   |
| C <sub>7</sub> Insoluble, Wt-% | 25.4   |
| Toluene Insoluble, Wt-%        | 14.9   |
| Pour Point, °F                 | +50    |
| Viscosity SUS @ 210°F sec      | 125.1  |
| Furol @ 122°F sec              | 230.0  |

Table 2

Inspections of SRC-I Filter Feed 3777-4

|                                |        |
|--------------------------------|--------|
| Drum No.                       | 2      |
| °API @ 60°F                    | -5.9   |
| Sp. Gr. @ 60°F                 | 1.1266 |
| Distillation ASTM D-86         |        |
| IBP°F                          | 439    |
| 5%                             | 491    |
| 10%                            | 521    |
| 20%                            | 565    |
| 30%                            | 599    |
| 40%                            | 635    |
| 50%                            | 680    |
| 60%                            | 739    |
| 70%                            | 850    |
| 80%                            |        |
| 90%                            |        |
| 95%                            |        |
| EP                             |        |
| % Over                         | 70.0   |
| % Bottoms                      | 30.0   |
| Hydrogen, Wt-%                 | 7.37   |
| Carbon, Wt-%                   | 83.39  |
| Sulfur, Wt-ppm                 | 8650   |
| Nitrogen, Wt-ppm               | 11800  |
| Oxygen, Wt-ppm                 | 21400  |
| Ash ASTM, Wt-%                 | 4.19   |
| C <sub>7</sub> Insoluble, Wt-% | 24.75  |
| Toluene Insoluble, Wt-%        | 14.78  |
| Pour Point, °F                 | +45    |
| Viscosity SUS @ 210°F sec      | 126.6  |
| cSt @ 210°F sec                | 26.44  |

Table 3

Solvent Deashing of SRC-I Filter Feed 3777-3

Plant 633

| Solvent                     | 100% Light Paraffin A |        |        | 10% Light Aromatic<br>90% Light Paraffin A |
|-----------------------------|-----------------------|--------|--------|--|
| Run                         | ←                     | 97     | →      | 116  |
| Hours on Stream             | 8-64                  | 64-88  | 96-139 | 12-29                                      |
| <u>Conditions</u>           |                       |        |        |  |
| P-P(base), psi              | 430                   | 480    | 465    | 480  |
| T-T(base), °C               | 31                    | 49     | 43     | 48   |
| Solvent/Feed Ratio          | base                  | base   | base   | base                                       |
| <u>Residence Time, hr.</u>  | 1.0                   | 1.0    | 2.0    | 1.0  |
| Residence Time(base), hr.   |                       |        |        |  |
| Feed Total, g               | 27732                 | 11467  | 11775  | 7806                                       |
| <u>Product Recovered</u>    |                       |        |        |  |
| Deashed Oil, g              | 14479                 | 5947   | 6826   | 5741                                       |
| Reject Bottoms, g           | 13423                 | 5563   | 4718   | 1864                                       |
| Total, g                    | 27902                 | 11510  | 11544  | 7605                                       |
| Recovery, Wt-%              | 100.6                 | 100.4  | 98.0   | 97.4                                       |
| <u>Deashed Oil</u>          |                       |        |        |  |
| Recovered, Wt-%             | 51.9                  | 51.7   | 59.1   | 75.5                                       |
| C <sub>7</sub> Insol., Wt-% | 2.49                  | 2.87   | 2.19   | 10.1                                       |
| Ash, Wt-%                   | <0.001                | <0.001 | <0.001 | 0.001                                      |
| Toluene Insol., Wt-%        |                       |        | 0.02   | 0.04                                       |
| <u>Reject Bottoms</u>       |                       |        |        |  |
| Recovered, Wt-%             | 48.1                  | 48.3   | 40.9   | 24.5                                       |
| Ash, Wt-%                   |                       | 4.96   | 12.61  | 19.80                                      |
| Toluene Insol., Wt-%        |                       | 30.0   | 52.5   | 94.3                                       |
| C <sub>7</sub> Insol., Wt-% |                       |        | 68.3   | 99.5                                       |

Table 4

Solvent Deashing of SRC-I Filter Feed 3777-3

Plant 633

| Solvent                     | 100% Light Aromatic |       |       |
|-----------------------------|---------------------|-------|-------|
| Run                         | 120                 |       |       |
| Hours on Stream             | 4-20                | 20-24 | 32-40 |
| <u>Conditions</u>           |                     |       |       |
| P-P(base), psi              | 555                 | 595   | 425   |
| T-T(base), °C               | 45                  | 45    | 10    |
| Solvent/Feed Ratio          | base                | base  | base  |
| Residence Time, hr.         | 1.0                 | 2.0   | 2.0   |
| Residence Time(base), hr.   |                     |       |       |
| Feed Total, g               | 7775                | 1054  | 1919  |
| <u>Product Recovered</u>    |                     |       |       |
| Deashed Oil, g              | 7040                | 1057  | 1882  |
| Reject Bottoms, g           | 593                 | 70    | 10    |
| Total, g                    | 7633                | 1127  | 1892  |
| Recovery, Wt-%              | 98.2                | 106.9 | 98.1  |
| <u>Deashed Oil</u>          |                     |       |       |
| Recovered, Wt-%             | 92.2                | 93.8  | 99.5  |
| C <sub>7</sub> Insol., Wt-% | 15.30               | 15.1  | 18.2  |
| Ash, Wt-%                   | 1.18                | 0.79  | 0.78  |
| <u>Reject Bottoms</u>       |                     |       |       |
| Recovered, Wt-%             | 7.8                 | 6.2   | 0.5   |
| Ash, Wt-%                   |                     |       |       |

Table 5

Solvent Deashing of SRC-I Filter Feed 3777-4

Plant 633

| Solvent                     | 100% Light Paraffin B | 90% Light Paraffin B<br>10% Light Aromatic |        |
|-----------------------------|-----------------------|--|--------|
| Run                         | 122                   | 123  | 123    |
| Hours on Stream             | 12-44                 | 12-28                                      | 28-108 |
| <u>Conditions</u>           |                       |  |        |
| P-P(base), psi              | 555                   | 545  | 545    |
| T-T(base), °C               | 55                    | 44   | 81     |
| Solvent/Feed Ratio          | base                  | base                                       | base   |
| Residence Time, hr.         | 1.0                   | 1.0  | 1.0    |
| Residence time(base), hr.   |                       |  |        |
| Feed Total, g               | 15255                 | 7626                                       | 38036  |
| <u>Product Recovered</u>    |                       |  |        |
| Deashed Oil, g              | 11828                 | 5869                                       | 30600  |
| Reject Bottoms, g           | 430                   | 1604                                       | 7102   |
| Total, g                    | 12258                 | 7473                                       | 37702  |
| Recovery, Wt-%              | 80.4                  | 98.0                                       | 99.1   |
| <u>Deashed Oil</u>          |                       |  |        |
| Recovered, Wt-%             | 77.5                  | 78.6                                       | 81.2   |
| C <sub>7</sub> Insol., Wt-% | 8.65                  | 9.84                                       | 10.75  |
| Ash, Wt-%                   | 0.003                 | 0.002                                      | 0.004  |
| Toluene Insol., Wt-%        | 0.17                  | 0.41                                       | 0.30   |
| <u>Reject Bottoms</u>       |                       |  |        |
| Recovered, Wt-%             | 22.5                  | 21.4                                       | 18.8   |
| Ash, Wt-%                   |                       | 21.0                                       | 22.3   |
| Softening Point, °F         |                       | 420  | 479    |

Table 6

Inspections of Deashed Oil 3777-47

|                                |        |
|--------------------------------|--------|
| °API @ 60°F                    | 2.2    |
| Sp. Gr. @ 60°F                 | 1.0583 |
| Distillation ASTM D-86         |        |
| IBP°F                          | 461    |
| 5%                             | 502    |
| 10%                            | 528    |
| 20%                            | 558    |
| 30%                            | 590    |
| 40%                            | 622    |
| 50%                            | 662    |
| 60%                            | 709    |
| 70%                            | 761    |
| 80%                            | 867    |
| 84%                            | 950    |
| EP                             |        |
| % Over                         | 84.0   |
| % Bottoms                      | 16.0   |
| Hydrogen, Wt-%                 | 8.25   |
| Carbon, Wt-%                   | 88.18  |
| Sulfur, Wt-ppm                 | 4700   |
| Nitrogen, Wt-ppm               | 9900   |
| Oxygen, Wt-ppm                 | 21900  |
| Ash ASTM, Wt-%                 | 0.009  |
| C <sub>7</sub> Insoluble, Wt-% | 9.68   |
| Toluene Insoluble, Wt-%        | 0.12   |
| Viscosity SUS @ 210°F sec      | 48.9   |
| cSt @ 210°F sec                | 6.93   |
| Conradson Carbon, Wt-%         | 9.16   |
| Molecular Weight, kg/kmol      | 245    |

Table 7

Inspections of Deashed Oil 3777-48

|                                |        |
|--------------------------------|--------|
| °API @ 60°F                    | 4.3    |
| Sp. Gr. @ 60°F                 | 1.0420 |
| <b>Distillation ASTM D-86</b>  |        |
| IBP°F                          | 442    |
| 5%                             | 489    |
| 10%                            | 513    |
| 20%                            | 542    |
| 30%                            | 569    |
| 40%                            | 600    |
| 50%                            | 635    |
| 60%                            | 678    |
| 70%                            | 729    |
| 80%                            | 799    |
| 87%                            | 901    |
| 95%                            |        |
| EP                             |        |
| % Over                         | 87.0   |
| % Bottoms                      | 13.0   |
| Hydrogen, Wt-%                 | 8.74   |
| Carbon, Wt-%                   | 88.05  |
| Sulfur, Wt-ppm                 | 4600   |
| Nitrogen, Wt-ppm               | 8400   |
| Oxygen, Wt-ppm                 | 16300  |
| Ash ASTM, Wt-%                 | 0.002  |
| C <sub>7</sub> Insoluble, Wt-% | 2.80   |
| Toluene Insoluble, Wt-%        | 0.02   |
| Pour Point, °F                 | +35    |
| Viscosity SUS @ 210°F sec      | 41.5   |
| cSt @ 210°F sec                | 4.63   |
| Conradson Carbon, Wt-%         | 6.01   |
| Molecular Weight, kg/kmol      | 240    |

Table 8

Hydrotreating Deashed Oils 3777-47 and 48

Plant 638, Run 48

Operational Summary

P-P(base) psi = -300, LHSV/LHSV(base) = 2.0

| Period No. | Hrs. on Stream | Feed-stock | T-T(base), °C | Product Analysis, Wt-% |       |                           |
|------------|----------------|------------|---------------|------------------------|-------|---------------------------|
|            |                |            |               | H                      | C     | C <sub>7</sub> Insolubles |
| Line Out   | 12-24          | 3777-47    |               |                        |       |                           |
| 1          | 24-34          |            | -20           | 10.08                  | 88.09 | 2.17                      |
| 2          | 34-44          |            | -19           | 9.79                   | 88.27 |                           |
| 3          | 44-54          |            | -19           | 9.74                   | 88.18 |                           |
| 4          | 54-64          |            | -22           |                        |       | 1.22                      |
| 5          | 64-74          |            | -18           |                        |       |                           |
| 6          | 74-84          |            | -22           | 9.75                   | 87.96 |                           |
| 7          | 84-94          |            | -18           |                        |       |                           |
| 8          | 94-104         |            | -21           |                        |       | 1.47                      |
| 9          | 104-114        |            | -20           |                        |       |                           |
| 10         | 114-124        |            | -20           |                        |       |                           |
| 11         | 124-134        |            | -20           | 9.72                   | 88.10 | 1.20                      |
| 12         | 134-144        |            | -16           |                        |       |                           |
| 13         | 144-154        |            | -18           |                        |       |                           |
| 14         | 154-164        |            | -24           | 9.77                   | 87.99 |                           |
| 15         | 164-174        |            | -31           | 9.90                   | 87.96 |                           |
| 16         | 174-184        |            | -20           |                        |       |                           |
| 17         | 184-194        | 3777-48    | -20           |                        |       |                           |
| 18         | 194-204        |            | -27           | 10.06                  | 88.16 | 0.33                      |
| 19         | 204-214        |            | -24           |                        |       |                           |
| 20         | 214-224        |            | -26           |                        |       |                           |
| 21         | 224-234        |            | -23           | 10.12                  | 88.39 | 0.22                      |
| 22         | 234-244        |            | -22           |                        |       |                           |

Table 9

Inspections of Hydrotreated Deashed Oil 3777-50

|                                |         |
|--------------------------------|---------|
| Sample No.                     | 3777-50 |
| °API @ 60°F                    | 10.4    |
| Sp. Gr. @ 60°F                 | 0.9972  |
| Distillation ASTM D-86         |         |
| IBP°F                          | 270     |
| 5%                             | 418     |
| 10%                            | 477     |
| 20%                            | 519     |
| 30%                            | 550     |
| 40%                            | 580     |
| 50%                            | 611     |
| 60%                            | 651     |
| 70%                            | 701     |
| 80%                            | 785     |
| 89%                            | 952     |
| 95%                            |         |
| EP                             |         |
| % Over                         | 89.0    |
| % Bottoms                      | 11.0    |
| Hydrogen, Wt-%                 | 9.99    |
| Carbon, Wt-%                   | 88.45   |
| Sulfur, Wt-ppm                 | 598     |
| Nitrogen, Wt-ppm               | 5800    |
| Oxygen, Wt-ppm                 | 7900    |
| C <sub>7</sub> Insoluble, Wt-% | 1.91    |
| Toluene Insoluble, Wt-%        | 0.03    |
| Viscosity SUS @ 210°F sec      | 35.8    |
| cSt @ 210°F sec                | 2.867   |
| Conradson Carbon, Wt-%         | 3.95    |
| Molecular Weight, kg/kmol      | 243     |

Table 10

Inspections of Hydrotreated Deashed Oil 3777- 51

|                                |         |
|--------------------------------|---------|
| Sample No.                     | 3777-51 |
| °API @ 60°F                    | 12.4    |
| Sp. Gr. @ 60°F                 | 0.9833  |
| Distillation ASTM D-86         |         |
| IBP°F                          | 250     |
| 5%                             | 412     |
| 10%                            | 468     |
| 20%                            | 514     |
| 30%                            | 550     |
| 40%                            | 577     |
| 50%                            | 608     |
| 60%                            | 641     |
| 70%                            | 691     |
| 80%                            | 760     |
| 90%                            | 920     |
| 92%                            | 970     |
| EP                             |         |
| % Over                         | 92.0    |
| % Bottoms                      | 8.0     |
| Hydrogen, Wt-%                 | 10.17   |
| Carbon, Wt-%                   | 87.93   |
| Sulfur, Wt-ppm                 | 436     |
| Nitrogen, Wt-ppm               | 4500    |
| Oxygen, Wt-ppm                 | 6610    |
| C <sub>7</sub> Insoluble, Wt-% | 0.42    |
| Toluene Insoluble, Wt-%        | <0.01   |
| Viscosity SUS @ 210°F sec      | 34.3    |
| cSt @ 210°F                    | 2.436   |
| Conradson Carbon, Wt-%         | 2.33    |
| Molecular Weight , kg/kmol     | 237     |

Table 11

Hydrotreating Deashed Oil 3777-47

Overall Product Distribution

Plant 638, Run 48, Periods L.O.-16

| Product Distribution                           | Wt-%        |
|--|-------------|
| C <sub>1</sub> -C <sub>4</sub>                 | 0.46        |
| C <sub>5</sub> -C <sub>6</sub> (in Debut. Gas) | 0.34        |
| Combined Product <sup>a</sup>                  | 99.65       |
| H <sub>2</sub> O                               | 0.50        |
| NH <sub>3</sub>                                | 0.44        |
| H <sub>2</sub> S                               | <u>0.33</u> |
| Total  | 101.72      |
| Total Hydrogen Consumption SCFB                | 1082        |

<sup>a</sup>Designated as Hydrotreated Deashed Oil 3777-50

Table 12

Hydrotreating Deashed Oil 3777-47

Distribution of Hydrogen Consumption

Plant 638, Run 48, Periods L.O.-16

| Hydrogen Distribution                          | <u>Wt-%</u> |
|--|-------------|
| C <sub>1</sub> -C <sub>4</sub>                 | 3.28        |
| C <sub>5</sub> -C <sub>6</sub> (in Debut. Gas) | 1.56        |
| Combined Product <sup>a</sup>                  | 86.94       |
| H <sub>2</sub> O                               | 3.01        |
| NH <sub>3</sub>                                | 4.19        |
| H <sub>2</sub> S                               | <u>1.02</u> |
| Total  | 100.00      |
| Total Hydrogen Consumption SCFB                | 1082        |

<sup>a</sup>Designated as Hydrogenated Deashed Oil 3777-50

Table 13

Hydrotreating Deashed Oil 3777-48

Overall Product Distribution

Plant 638, Run 48, Periods 17-22

| Product Distribution                           | <u>Wt-%</u> |
|--|-------------|
| C <sub>1</sub> -C <sub>4</sub>                 | 0.44        |
| C <sub>5</sub> -C <sub>6</sub> (in Debut. Gas) | 0.43        |
| Combined Product <sup>a</sup>                  | 99.20       |
| H <sub>2</sub> O                               | 0.75        |
| NH <sub>3</sub>                                | 0.44        |
| H <sub>2</sub> S                               | <u>0.47</u> |
| Total  | 101.73      |
| Total Hydrogen Consumption SCFB                | 1088        |

<sup>a</sup>Designated as Hydrotreated Deashed Oil 3777-51

Table 14

Hydrotreating Deashed Oil 3777-48

Distribution of Hydrogen Consumption

Plant 638, Run 48, Periods 17-22

| Hydrogen Distribution                          | <u>Wt-%</u> |
|--|-------------|
| C <sub>1</sub> -C <sub>4</sub>                 | 3.45        |
| C <sub>5</sub> -C <sub>6</sub> (in Debut. Gas) | 2.11        |
| Combined Product <sup>a</sup>                  | 82.38       |
| H <sub>2</sub> O                               | 5.30        |
| NH <sub>3</sub>                                | 4.98        |
| H <sub>2</sub> S                               | <u>1.78</u> |
| Total  | 100.00      |
| Total Hydrogen Consumption SCFB                | 1088        |

<sup>a</sup>Designated as Hydrotreated Deashed Oil 3777-51

Table 15

Fluid Catalytic Cracking of Deashed Oil 3777-42

Plant 593, Run 303

| Test No.                                   | 1         | 2      |
|--|-----------|--------|
| Operating Conditions                       |           |        |
| P-P(base), psig                            | -10.0     | -9.0   |
| T-T(base), °C                              | -4        | 6      |
| [Cat./Oil]                                 | 1.02      | 0.99   |
| [Cat./Oil(base)]                           |           |        |
| Conversion, Vol-%                          | 41.5      | 46.8   |
| Product Distribution, Wt-%                 |           |        |
| C <sub>3</sub> -                           | 3.9       | 5.3    |
| C <sub>4</sub>                             | 1.8       | 1.9    |
| C <sub>5</sub>                             | 1.5       | 1.5    |
| C <sub>6</sub> -EP Gasoline                | 17.3      | 19.8   |
| Cycle Oil                                  | 60.1      | 55.3   |
| Carbon                                     | 9.8       | 11.6   |
| Wt. Recovery                               | 94.4      | 95.4   |
| Products, Vol-%                            |           |        |
| C <sub>5</sub> -EP Gasoline                | 23.1      | 26.2   |
| Cycle Oil                                  | 58.5      | 53.2   |
| Inspections of C <sub>5</sub> -EP Gasoline |           |        |
| °API @ 60°F                                | 39.8      | 40.7   |
| Sp. Gr. @ 60°F                             | 0.8260    | 0.8217 |
| Distillation, ASTM D-86                    |           |        |
| IBP, °F                                    | 113       | 122    |
| 5%   | 145       | 153    |
| 10%  | 167       | 167    |
| 50%  | 271       | 367    |
| 90%  | 390       | 380    |
| 95%  | 404       | 403    |
| EP   | 445       | 436    |
| RON, Clear                                 | ← 97.3 →  |        |
| RON, 3 ml TEL/Gallon                       | ← 101.3 → |        |
| FIA, Vol-%                                 |           |        |
| A  | 47.2      | 50.0   |
| O  | 26.2      | 20.9   |
| P&N  | 26.6      | 29.1   |

Table 15 (Cont'd.)

| Test No.                 | 1      | 2      |
|--------------------------|--------|--------|
| Properties of Cycle Oil  |        |        |
| °API @ 60°F              | 5.0    | 3.4    |
| Sp. Gr. @ 60°F           | 1.0366 | 1.0489 |
| Distillation, UOP No. 1  |        |        |
| IBP, °F                  | 471    | 458    |
| 5%                       | 492    | 477    |
| 10%                      | 504    | 494    |
| 15%                      | 512    | 504    |
| % Over at 650°F          | 61.0   | 63.0   |
| °API of 650°F- @ 60°F    | 12.7   | 11.0   |
| Sp. Gr. of 650°F- @ 60°F | 0.9813 | 0.9930 |
| °API of 650°F+ @ 60°F    | -5.2   | -7.3   |
| Sp. Gr. of 650°F+ @ 60°F | 1.1203 | 1.1393 |
| $C_3^-$ , Mole %         |        |        |
| $H_2$                    | 52.2   | 43.0   |
| $C_1$                    | 17.2   | 21.4   |
| $C_2$ (Total)            | 14.3   | 16.8   |
| $C_3$ Olefins            | 13.0   | 13.8   |
| $C_3$                    | 3.3    | 5.0    |
| Total                    | 100.0  | 100.0  |
| $C_4$ , Vol-%            |        |        |
| $C_4$ Olefins            | 67.7   | 61.6   |
| $i-C_4$                  | 18.9   | 26.0   |
| $n-C_4$                  | 13.4   | 12.4   |
| Total                    | 100.0  | 100.0  |
| $C_5$ , Vol-%            |        |        |
| $C_5$ Olefins            | 72.4   | 59.5   |
| $i-C_5$                  | 23.8   | 34.8   |
| $n-C_5$                  | 3.8    | 5.7    |
| Total                    | 100.0  | 100.0  |

Table 16

Fluid Catalytic Cracking of Hydrotreated Deashed Oil 3777-46

Plant 593, Run 304

| Test No.  | 1      | 2      | 3      |
|---|--------|--------|--------|
| Operating Conditions                                  |        |        |        |
| P-P(base), psig                                       | -10.0  | -10.0  | -10.0  |
| T-T(base), °C   | 3      | 6      | 4      |
| $\left[ \frac{\text{Cat.}}{\text{Oil}} \right]$       | 1.05   | 1.09   | 1.10   |
| $\left[ \frac{\text{Cat.}}{\text{Oil(base)}} \right]$ |        |        |        |
| Conversion, Vol-%                                     | 58.2   | 57.0   | 57.2   |
| Product Distribution, Wt-%                            |        |        |        |
| C <sub>3</sub> -                                      | 6.3    | 6.8    | 6.2    |
| C <sub>4</sub>  | 4.9    | 5.3    | 5.3    |
| C <sub>5</sub>  | 3.8    | 4.0    | 4.1    |
| C <sub>6</sub> -EP Gasoline                           | 29.9   | 27.3   | 30.1   |
| Cycle Oil   | 45.0   | 46.1   | 45.9   |
| Carbon  | 6.3    | 6.3    | 6.6    |
| Wt. Recovery  | 96.2   | 95.8   | 98.2   |
| Products, Vol-%                                       |        |        |        |
| C <sub>5</sub> -EP Gasoline                           | 41.4   | 38.4   | 42.3   |
| Cycle Oil   | 41.8   | 43.0   | 42.8   |
| Inspections of C <sub>5</sub> -EP Gasoline            |        |        |        |
| °API @ 60°F   | 47.2   | 41.5   | 47.2   |
| Sp. Gr. @ 60°F  | 0.7918 | 0.8179 | 0.7918 |
| Distillation, ASTM D-86                               |        |        |        |
| IBP, °F   | 106    | 136    | 105    |
| 5%  | 129    | 168    | 129    |
| 10%   | 145    | 184    | 149    |
| 50%   | 241    | 262    | 236    |
| 90%   | 368    | 360    | 357    |
| 95%   | 387    | 375    | 372    |
| EP  | 425    | 428    | 372    |
| RON, Clear  | 94.8   | 94.8   | 94.8   |
| RON, 3 ml TEL/Gallon                                  | 101.1  | 101.1  | 101.1  |
| MON, Clear  | 81.5   | 81.5   | 81.5   |
| MON, 3 ml TEL/Gallon                                  | 89.2   | 89.2   | 89.2   |
| FIA, Vol-%  |        |        |        |
| A   | 41.4   | 50.3   | 40.7   |
| O   | 11.0   | 9.3    | 10.4   |
| P&N   | 47.6   | 40.4   | 48.9   |

Table 16 (Cont'd.)

| Test. No.                      | 1      | 2      | 3      |
|--------------------------------|--------|--------|--------|
| <b>Properties of Cycle Oil</b> |        |        |        |
| °API @ 60°F                    | 3.3    | 3.6    | 3.7    |
| Sp. Gr. @ 60°F                 | 1.0497 | 1.0474 | 1.0466 |
| <b>Distillation, UOP No. 1</b> |        |        |        |
| IBP, °F                        | 443    | 441    | 444    |
| 5%                             | 459    | 460    | 460    |
| 10%                            | 472    | 478    | 471    |
| 15%                            | 481    | 487    | 480    |
| % Over at 650°F                | 69.0   | 68.0   | 70.0   |
| °API of 650°F- @ 60°F          | 9.8    | 10.2   | 9.8    |
| Sp. Gr. of 650°F- @ 60°F       | 1.0014 | 0.9986 | 1.0014 |
| °API of 650°F+ @ 60°F          | -8.9   | -8.5   | -8.4   |
| Sp. Gr. of 650°F+ @ 60°F       | 1.1542 | 1.1504 | 1.1495 |
| <b>C<sub>3</sub>-, Mole %</b>  |        |        |        |
| H <sub>2</sub>                 | 50.5   | 52.0   | 51.9   |
| C <sub>1</sub>                 | 12.4   | 12.8   | 12.2   |
| C <sub>2</sub> (Total)         | 11.3   | 10.9   | 10.9   |
| C <sub>3</sub> Olefins         | 19.0   | 18.2   | 18.6   |
| C <sub>3</sub> Total           | 6.8    | 6.1    | 6.4    |
|                                | 100.0  | 100.0  | 100.0  |
| <b>C<sub>4</sub>, Vol-%</b>    |        |        |        |
| C <sub>4</sub> Olefins         | 44.5   | 48.0   | 45.2   |
| i-C <sub>4</sub>               | 39.5   | 36.6   | 38.9   |
| n-C <sub>4</sub>               | 16.0   | 15.4   | 15.9   |
| Total                          | 100.0  | 100.0  | 100.0  |
| <b>C<sub>5</sub>, Vol-%</b>    |        |        |        |
| C <sub>5</sub> Olefins         | 36.3   | 51.2   | 38.0   |
| i-C <sub>5</sub>               | 57.4   | 44.0   | 56.1   |
| n-C <sub>5</sub>               | 6.3    | 4.8    | 5.9    |
| Total                          | 100.0  | 100.0  | 100.0  |

Table 17

Fluid Catalytic Cracking of Hydrotreated Deashed Oil 3777-46

Plant 593, Run 304

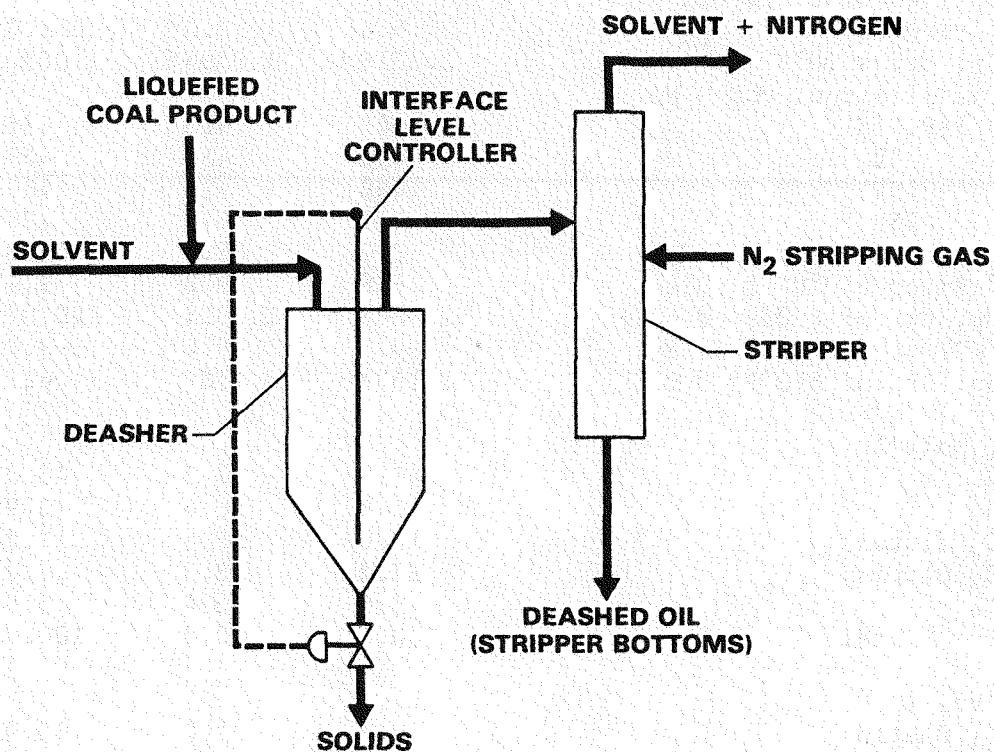
| Test No.   | 5                   | 6                   | 7                   |
|--|---------------------|---------------------|---------------------|
| Operating Conditions   |                     |                     |                     |
| P-P(base), psig  | -10                 | -9                  | -10                 |
| T-T(base), °C  | 32                  | 26                  | 31                  |
| $\left[ \frac{\text{Cat.}/\text{Oil}}{\text{Cat.}/\text{Oil(base)}} \right]$ | 1.43                | 1.5                 | 1.34                |
| Conversion, Vol-%  | 59.8                | 58.4                | 62.4                |
| Product Distribution, Wt-%   |                     |                     |                     |
| C <sub>3</sub> -   | 9.0                 | 9.0                 | 9.5                 |
| C <sub>4</sub>   | 5.9                 | 6.1                 | 6.1                 |
| C <sub>5</sub>   | 35.9 <sup>a</sup>   | 34.7 <sup>a</sup>   | 35.5 <sup>a</sup>   |
| C <sub>6</sub> -EP Gasoline }  | 41.4 <sup>a</sup>   | 43.0 <sup>a</sup>   | 38.2 <sup>a</sup>   |
| Cycle Oil  | 8.1                 | 7.7                 | 9.0                 |
| Carbon   |                     |                     |                     |
| Wt. Recovery   | 100.3               | 100.5               | 98.3                |
| Products, Vol-%  |                     |                     |                     |
| C <sub>5</sub> -EP Gasoline  | 43.6 <sup>a</sup>   | 42.2 <sup>a</sup>   | 42.7 <sup>a</sup>   |
| Cycle Oil  | 37.7 <sup>a</sup>   | 39.6 <sup>a</sup>   | 34.9 <sup>a</sup>   |
| Inspections of C <sub>5</sub> -EP Gasoline                                   |                     |                     |                     |
| °API @ 60°F  | 44.4 <sup>a</sup>   | 45.0 <sup>a</sup>   | 43.3 <sup>a</sup>   |
| Sp. Gr. @ 60°F   | 0.8044 <sup>a</sup> | 0.8017 <sup>a</sup> | 0.8095 <sup>a</sup> |
| Distillation, ASTM D-86  |                     |                     |                     |
| IBP, °F  | 96                  | 102                 | 104                 |
| 5%   | 119                 | 128                 | 129                 |
| 10%  | 136                 | 144                 | 143                 |
| 50%  | 236                 | 240                 | 235                 |
| 90%  | 353                 | 358                 | 350                 |
| 95%  | 373                 | 394                 | 373                 |
| EP   | 411                 | 436                 | 415                 |
| RON, Clear   | ↔ 97.1 ↔            |                     |                     |
| RON, 3 ml TEL/Gallon   | ↔ 101.8 ↔           |                     |                     |
| MON, Clear   | ↔ 83.7 ↔            |                     |                     |
| MON, 3 ml TEL/Gallon   | ↔ 88.5 ↔            |                     |                     |
| FIA, Vol-%   |                     |                     |                     |
| A  | 45.9                | 44.2                | 49.0                |
| O  | 13.9                | 15.5                | 11.1                |
| P&N  | 40.2                | 40.3                | 39.9                |

Table 17 (Cont'd.)

| Test No.                         | 5                   | 6                   | 7                   |
|----------------------------------|---------------------|---------------------|---------------------|
| <b>Properties of Cycle Oil</b>   |                     |                     |                     |
| °API @ 60°F                      | 0.6 <sup>a</sup>    | 2.0 <sup>a</sup>    | 1.0 <sup>a</sup>    |
| Sp. Gr. @ 60°F                   | 1.0716 <sup>a</sup> | 1.0596 <sup>a</sup> | 1.0682 <sup>a</sup> |
| <b>Distillation, UOP - No. 1</b> |                     |                     |                     |
| IBP, °F                          | 444                 | 423                 | 438                 |
| 5%                               | 456                 | 450                 | 459                 |
| 10%                              | 468                 | 465                 | 469                 |
| 15%                              | 477                 | 473                 | 478                 |
| % Over at 650°F                  | 68.0                | 67.0                | 67.0                |
| °API of 650°F- @ 60°F            | 8.8                 | 9.8                 | 9.9                 |
| Sp. Gr. of 650°F- @ 60°F         | 1.0086              | 1.0014              | 1.0107              |
| °API of 650°F+ @ 60°F            | -9.8                | -9.4                | -9.7                |
| Sp. Gr. of 650°F+ @ 60°F         | 1.1627              | 1.1589              | 1.1617              |
| <b>C<sub>3</sub>-, Mole %</b>    |                     |                     |                     |
| H <sub>2</sub>                   | 48.9                | 47.3                | 48.0                |
| C <sub>1</sub>                   | 15.8                | 16.6                | 16.9                |
| C <sub>2</sub> (Total)           | 12.0                | 12.4                | 12.5                |
| C <sub>3</sub> Olefins           | 17.3                | 18.3                | 16.0                |
| C <sub>3</sub>                   | 6.0                 | 5.4                 | 6.6                 |
| Total                            | 100.0               | 100.0               | 100.0               |
| <b>C<sub>4</sub>, Vol-%</b>      |                     |                     |                     |
| C <sub>4</sub> Olefins           | 49.7                | 52.0                | 45.9                |
| i-C <sub>4</sub>                 | 36.7                | 35.4                | 38.6                |
| n-C <sub>4</sub>                 | 13.6                | 12.6                | 15.5                |
| Total                            | 100.0               | 100.0               | 100.0               |
| <b>C<sub>5</sub>, Vol-%</b>      |                     |                     |                     |
| C <sub>5</sub> Olefins           | 39.8                | 42.1                | 33.9                |
| i-C <sub>5</sub>                 | 54.8                | 52.7                | 59.3                |
| n-C <sub>5</sub>                 | 5.4                 | 5.2                 | 6.8                 |
| Total                            | 100.0               | 100.0               | 100.0               |

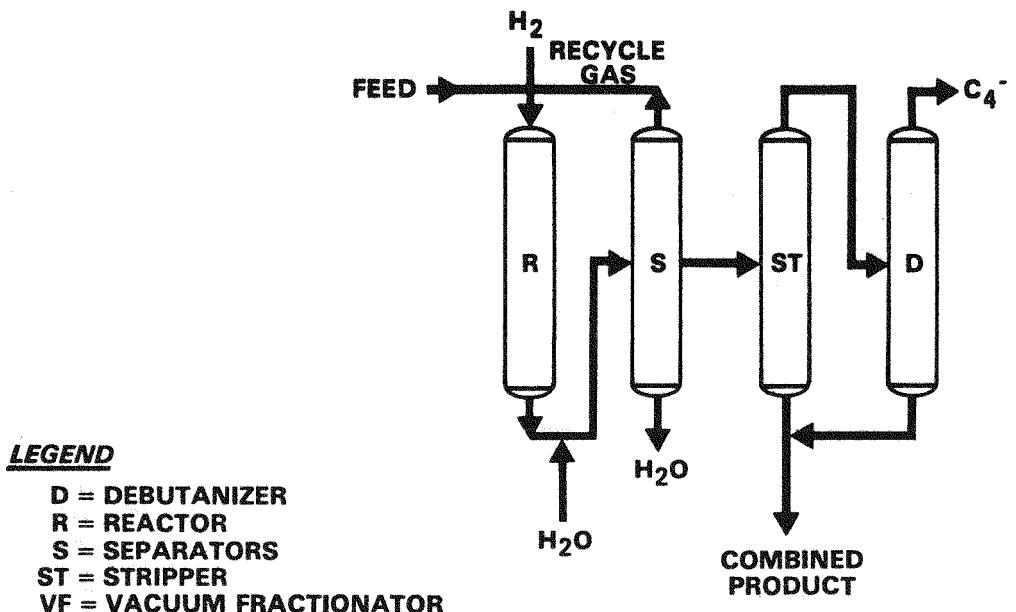
<sup>a</sup>Adjusted

**FIGURE 1**  
**UOP DEASHING UNIT**



UOP 437-1

**FIGURE 2**  
**BENCH-SCALE UNIT FOR HYDROTREATING**



UOP 197D-24