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FOSSIL ENERGY

DEASPHALTING, DEASHING, AND UPGRADING OF COAL LIQUIDS

Quarterly Technical Progress Report for April—June 1979

By
Fredrick J. Riedl
Armand J. deRosset

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UOP Incorporated
Corporate Research Center
Des Plaines, Illinois

U. S. DEPARTMENT OF ENERGY

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UOP Inc.
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Ten UOP Plaza
Des Plaines, IL 60016

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ABSTRACT

Deashing of UOP coal liquefaction product, using the UOP Demex process, has been completed. Three rejection levels of 17, 30 and 26 wt-% were attained. Ash content of the composite product was < 20 ppm.

Fixed bed hydrotreating of the deashed oil was completed. A 0.75 wt-% increase in hydrogen content was obtained under relatively mild operating conditions.

A second coal liquefaction operation was completed, using the hydrotreated deashed oil as solvent A 90-93 wt-% (MAF basis) coal conversion was achieved.

Demex deashing of this second liquefied coal product is to be started in July.

Task 1.2
Work Accomplished

The Demex deashing of UOP coal liquefaction product has been completed. The deashing work was done on the UOP Demex deashing Plant 633, (Figure 1) and comprises 10 separate runs over a 4-month period of time. Two of the runs, 90 and 96, were of sufficient duration to provide dependable data.

In general, Demex deashing proceeds as follows: liquefied coal product and solvent are combined at operating pressure upstream of the deasher and then passed downflow through the in-line mixer into the deasher. Separation takes place in the deasher. The oil-rich upper phase flows to a stripper for removal of the solvent, while the solid-rich bottom phase containing the ash is removed through the lock-hopper system on the bottom of the deasher.

The liquefied coal product (3779-39) used for the deashing runs is described in Table 1. The analysis was calculated by averaging analytical data for stream samples taken at the beginning, in the middle and at the end of the liquefaction run. The liquefied coal product contained 85.4 wt-% toluene soluble oil. This oil contained 11 wt-% C_7 insoluble. The insoluble portion contained 56.7 wt-% ash.

Plant 633, Run 90

A summary of the deashing, Run 90, is presented in Table 2. Just after establishing a lined-out condition, a temperature upset occurred requiring an additional 30 hours of operation. The final 50 hours gave a resultant 17.3 wt-% rejection of solids and a 82.7 wt-% recovery of deashed oil which showed an ash content of less than 0.001 wt-% (10 wt-ppm).

Deasher solids from Run 90 representative of lined-out conditions were blended and analyzed. These data are shown in Table 3. The solids were also Soxhlet extracted with toluene in liquid phase, and both the toluene soluble oil and insoluble portions were submitted for analysis. Results are also shown on Table 3. The toluene soluble oil contained 6.79 wt-% hydrogen, compared to the original coal hydrogen content of 6.12 wt-% (Table 9). The insoluble portion would be a source of fuel for the coal plant.

Plant 693, Run 96

A second operation, Plant 633, Run 96, was begun using the same operating conditions as in Run 90. The liquefied coal product and solvent entered the deasher separately. The results of this run are presented in Table 4. A rejection of about 30 wt-% was obtained at base temperature and 26 wt-% at 4°C above base temperature.

A sample of the deasher solids (at the 30 wt-% rejection level) was analyzed. These data are presented on Table 5. These solids were Soxhlet extracted with toluene. The toluene soluble and insoluble portions were submitted for analysis. Results are also shown in Table 5. The amount of toluene soluble oil is slightly more than twice that observed in the previous operation at 17 wt-% rejection. The toluene soluble oil also has the expected higher hydrogen content. The insoluble portion is of poor quality, containing only 4.01 wt-% hydrogen. It could be used as a plant fuel.

From the results of these operations, it is concluded that it will be necessary to mix the solvent and liquefied coal product prior to entry to the deasher. It also appears that an in-line mixer is satisfactory.

The stripper bottoms, deashed oil (3776-8), is a blend of all stripper bottom products. This product was analyzed and the inspections are shown on Table 6. The ash content amounted to 0.002 wt-%, which is satisfactory for processing over a fixed-bed catalyst.

Task 1.3
Work Accomplished

The feedstock for hydrotreating is the deashed oil 3776-8, Table 6. The hydrotreating of this material was done in UOP Research Pilot Plant 638H, Run 36. A flow diagram is shown in Figure 2. The deashed oil along with fresh make up and recycle hydrogen are pumped downflow over a 300 cc fixed-bed black oil conversion catalyst. Water is injected into the reactor effluent line to pick up ammonia. The water is removed in the 3-phase separator and the hydrotreated deashed oil is sent to a debutanizer column for removal of light gases.

Plant 638H, Run 36

The deashed oil was processed at 40°C below base temperature, 240 psig below base pressure, and 2.0 LHSV relative to base, for a period of 8.8 days. A summary of the operation is given in Table 7. Periodically, samples from the plant were sent for hydrogen and carbon analyses. As noted, the hydrogen content lined out at about 10.3 wt-% with no indication of catalyst deactivation during the run. Normally a product distribution, hydrogen consumption and hydrogen distribution would be presented for the operation. However, analytical data on the oxygen content of the hydrotreated deashed oil is delayed and the above data will be reported in the next quarterly report.

Task 1.4

Work Accomplished

The inspections of the hydrotreated deashed oil 3776-9, Illinois No. 6, coal, and the blend charged for coal liquefaction to UOP Research Pilot Plant 666, Run 44, are shown on Tables 8, 9, 10. The inspection of the deashed oil blend was determined from a sample separated in the laboratory.

Coal oil slurry was pumped concurrently upflow under hydrogen pressure through an empty stainless steel tube at elevated temperature. Gas was separated for recycle or venting. The product, comprising liquefied coal, unconverted coal, water, and ash was collected and the water removed.

Plant 666, Run 44

Product distributions will be calculated and presented in the next quarterly report, for two periods of operation, midway and near the end of the run. Operating conditions during these periods along with the net hydrogen consumptions are all shown on Table 11. The net hydrogen consumption amounted to 960-1010 SCF/bbl. This represents formation of water, ammonia, hydrogen sulfide, solubility losses as well as uptake by coal during liquefaction. The hydrogen consumption is based on the difference of measured hydrogen input and output to and from the plant.

At the operating conditions employed, conversion of coal (MAF basis) was 90-93 wt-%. Samples to determine these conversions were selected during periods at the beginning and middle of the liquefaction run. These stream samples were drawn over short 20 to 30 minute intervals during the test period and separated in the laboratory into a liquid product, solids, and water. The results are shown on Table 12 along with the calculated conversions.

Task 2.0
Work Accomplished

To prevent excessive feed usage and/or loss, Synthoil centrifugate is being used as an off test feedstock to establish operating conditions and to work out mechanical problems on the Demex-deashing unit.

The work plan and progress, Figure 3, has been modified in regard to the DOE liquids. The new work plan and progress is shown in Figure 4.

ERRATA

Report FE-2645-02, Table 4, page 7. Read for line 2 under "Operating Conditions":

T - T (base) °C	5	5

Table 1

Inspections of Liquefied Coal Product 3777-39

Total Product Analysis	Wt-%
Oil	85.42
Insoluble in Toluene	4.33
Water	10.25
Total	100.00

Oil Portion	
C_7 Insoluble	11.08

Insoluble Portion	
Ash	56.74
% H	2.16
% C	33.34

Table 2

Deashing Liquefied Coal Product 3777-39

Plant 633, Run 90

P-P (base) = 480 psi, Solvent/Oil ratio = base

Hrs. on Stream	T-T (base), °C	% Rejection (Deasher Botts), wt-%	Deashed Oil, wt-%	Ash in Deashed, Oil, wt-%
0-30	4	12.3	87.7	<0.001
30-45	33	23.0	77.0	
45-60	6	10.8	89.2	<0.001
60-85	3	17.2	82.8	<0.001
85-110	3	17.3	82.7	<0.001

Table 3

Inspection of Deasher Solids 3810-37

Plant 633, Run 90

	wt-%
Hydrogen	5.06
Carbon	71.55
C_7 Insoluble	92.2
Coal Ash	15.28

Soxhlet Extraction with Toluene (Liquid Phase)

	wt-%
Oil (Toluene soluble)	
Yield	35.67
Hydrogen	6.79
Carbon	87.50
Insoluble portion	
Yield	64.33
Hydrogen	4.52
Carbon	65.05
Ash	23.49

Table 4

Deashing Liquefied Coal Product 3777-39

Plant 633, Run 96

P-P base = 480 psi, Solvent/Oil ratio = base

Hrs. on Stream	T-T (base), °C	Rejection (Deasher Botts), wt-%	Deashed Oil, wt-%	Ash in Oil, wt-%
0-9	0	28.4	71.6	
9-21	-3	30.5	69.5	
21-37	0	30.8	69.2	
37-71	4	26.0	74.0	<0.001

Table 5

Inspections of Deasher Solids 3810-39

Plant 633, Run 96

	wt-%
Hydrogen	7.52
Carbon	82.10
C_7 Insoluble	53.2
Coal Ash	6.85

Soxhlet Extraction with Toluene

	wt-%
Oil Toluene Soluble	
Yield	74.00
Hydrogen	8.17
Carbon	88.67
Insoluble portion	
Yield	24.35
Hydrogen	4.01
Carbon	60.44
Ash	26.50

Table 6

Inspections of Deashed Oil 3776-8

°API @ 60°F	8.2
Sp. Gr. @ 60°F	1.0129
Distillation, ASTM D 86	
IBP, °F	384
5%	468
10%	495
20%	545
30%	583
40%	629
50%	680
60%	740
70%	810
80%	932
90%	
95%	
EP	
% Over	80.0
% Bottoms	20.0
Hydrogen, wt-%	9.52
Carbon, wt-%	88.62
Sulfur, wt-ppm	1500
Nitrogen, wt-ppm	4010
Oxygen, wt-ppm	7550
Ash, wt-%	0.002
Toluene Insoluble, wt-%	0.16
C_7 Insoluble, wt-%	3.21
Viscosity: cSt @ 210°F	4.563
SUS @ 210°F	41.3
Molecular weight	257

Table 7

Product Hydrogen Content

Hydrotreating Deashed Oil 3776-8

Plant 638H, Run 36

LHSV/LHSV base = 2.0, P-P (base) = -240 psi

Period No	Hrs on Stream	T-T (base), °C	Product Analysis, wt-% H	C
1	22-32	-39	10.14	88.55
5	62-72	-40	10.08	89.06
8	92-102	-41	10.12	88.90
11	122-132	-40	10.36	89.52
12	132-142	-40	10.38	89.48
13	142-152	-38	10.38	88.94
15	162-172	-39	10.22	89.11
17	182-192	-38	10.31	89.20
19	202-212	-39	10.24	88.79

Table 8

Inspections of Hydrotreated Deashed Oil 3776-9

°API @ 60°F	11.8
Sp. Gr. @ 60°F	0.9874
Distillation, ASTM D 86	
IBP, °F	340
5%	438
10%	489
20%	538
30%	570
40%	610
50%	651
60%	701
70%	769
80%	859
86%	930
95%	
EP	
% Over	86.0
% Bottoms	14.0
Hydrogen, wt-%	10.27
Carbon, wt-%	87.92
Sulfur, wt-ppm	177
Nitrogen, wt-ppm	2155
Oxygen, wt-ppm	
C_7 Insoluble, wt-%	0.26
Viscosity: cSt @ 210°F	3.534
SUS @ 210°F	37.9

Table 9

Inspections of Illinois No. 6 Coal

Sample No.	3776-7
Analyses, wt-%	
Moisture	14.90
Ash (ASTM D 3174)	6.54
Sulfur	2.46
Nitrogen	1.01
Carbon	59.72
Hydrogen	6.12
Oxygen	9.60

Table 10

Blends and Inspection of
Illinois No. 6 Coal and Coal Liquefaction Solvent 3776-9

Blend Data

Blend Identification No.	3776-10
Blend Composition	
Solvent, gm	61517
Coal, gm (moisture free)	36144

Inspections

Coal in Blend, moisture free, wt-%	36.83
Solvent in Blend, wt-%	55.09
Water in Blend, wt-%	8.08

Table 11

Coal Liquefaction, Plant 666, Run 44

Test No.	44-8	44-14
Operating Conditions		
P-P (base), psig	740	840
T-T (base), °C	5	5
LHSV/LHSV (base)	1.26	1.23
Net Hydrogen Consumption, SCF/Bbl	958	1011

Table 12

Coal Liquefaction, Plant 666, Run 44

Operating Conditions, Product Inspections, Coal Conversions

Hours on Stream	12-24	96-108
Operating Conditions		
P-P (base), psi	785	800
T-T (base), °C	4	5
LHSV/LHSV, (base)	1.12	1.25
Product Analyses, wt-%		
Sample No.	3810-45	3810-47
Liquid Product (coal oil)	86.46	88.27
Solids	5.09	7.13
Water	7.04	4.46
Loss	1.41	0.14
Total	100.00	100.00
C_7 Insoluble in Coal Oil	8.95	8.32
Ash in Solids	52.98	55.20
Coal Conversion MAF (basis)	93.0	90.3

FIGURE 1

UOP DEASHING UNIT

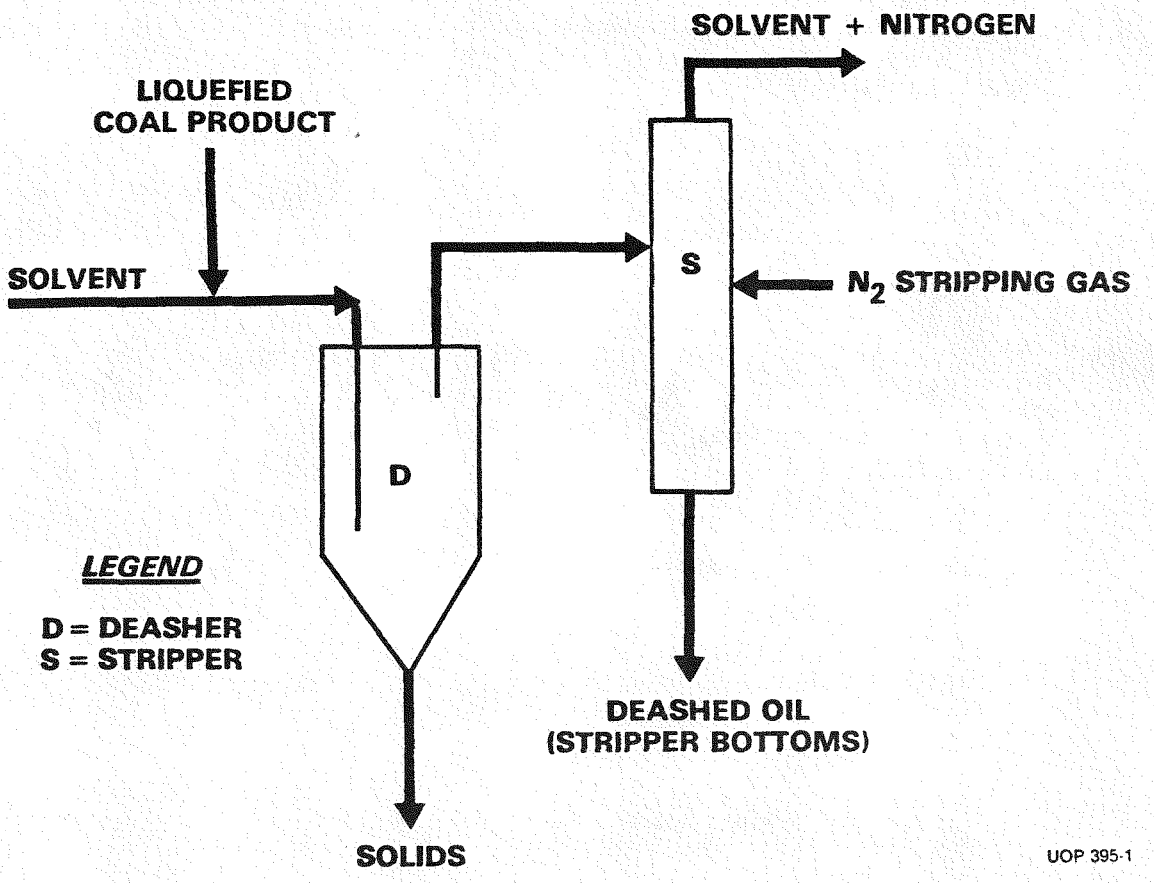

FIGURE 2

GAS OIL HYDROTREATING PLANT

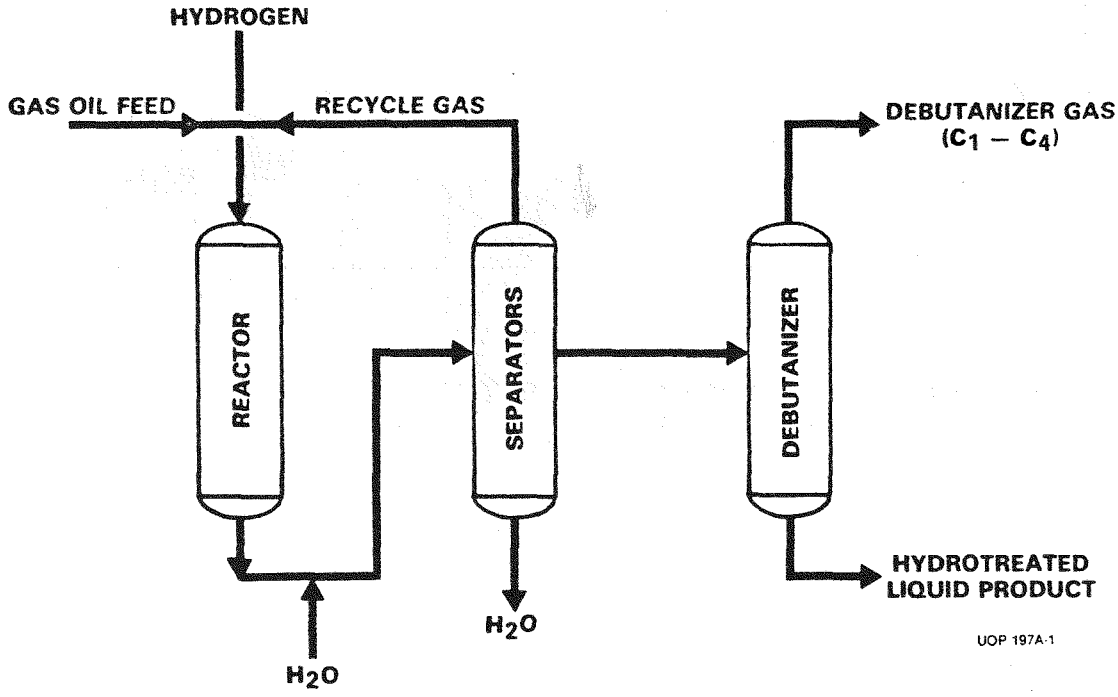

Figure 3

Work Plan and Progress

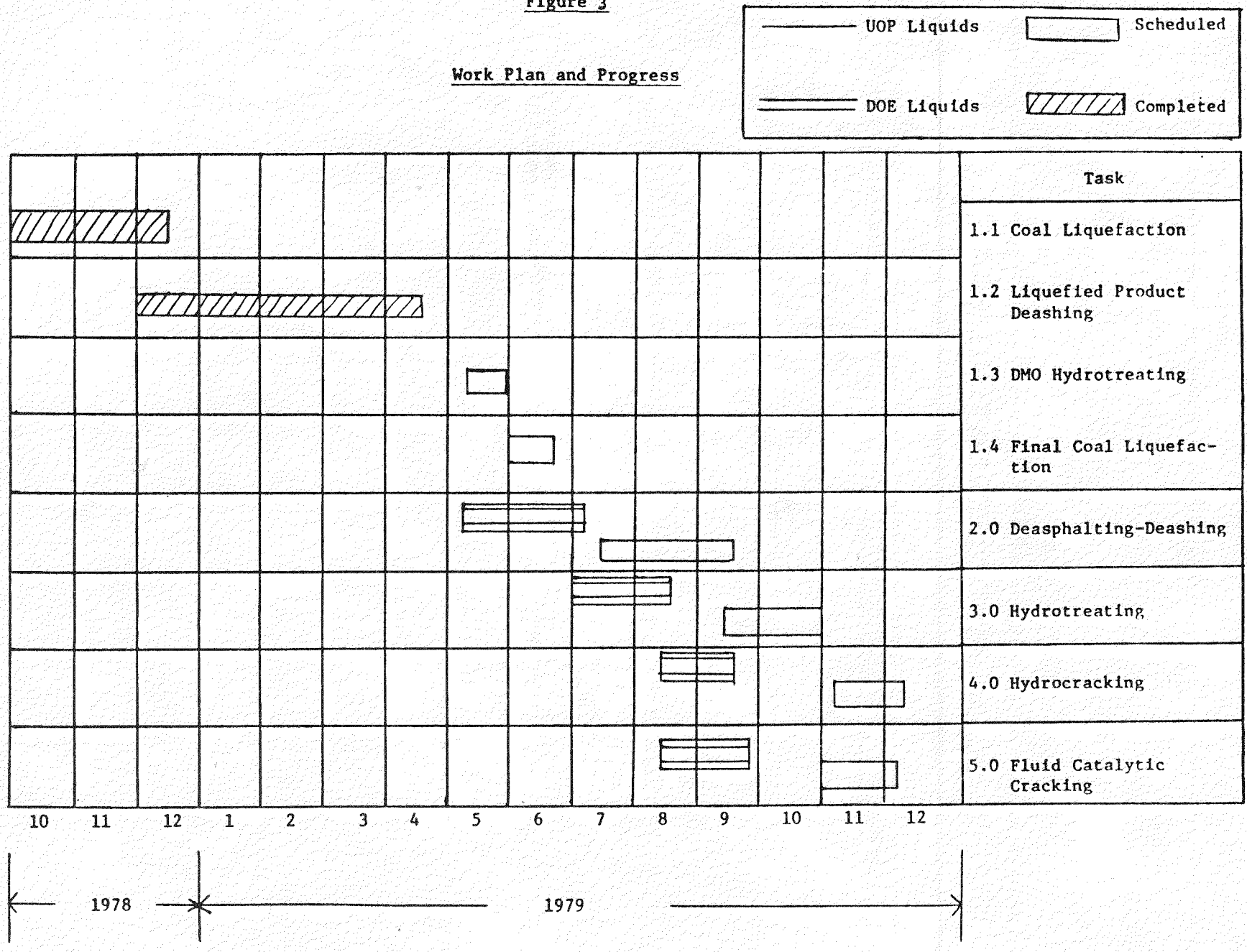

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Figure 4

Work Plan and Progress