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UPGRADING OF COAL LIQUIDS

Monthly Technical
Progress Report
For December, 1977

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ABSTRACT

Hydrotreating process variable studies on raw EDS process gas oil were continued at 250 psig above base pressure. Two additional series of studies have been made. Tests were run at 11°C above base temperature and 5°C below base temperature, and three space velocities. In all cases heptane insolubles were completely converted and substantial amounts of nitrogen were removed. Nitrogen conversions as high as 99.9 wt-% were obtained.

The second preparative H-Coal gas oil hydrotreating run, aimed at producing a FCC feedstock with high hydrogen content, has been completed.

Mass spectroscopy (MS) analyses of the EDS naphtha before and after upgrading have been completed. The upgraded naphtha was reformed over a commercial Platforming® catalyst over a series of conditions. MS analyses, as well as other required analyses, of four selected reformates were completed.

Task 1

Work Accomplished

Work on hydrotreating raw EDS process gas oil over a commercial hydrotreating catalyst was continued. Inspections of the feedstock are given in Table 1.

Hydrotreating serves to convert practically all heptane insolubles to distillate and to lower substantially the heterocyclics content.

Hydrotreating process variable studies on EDS process gas oil were continued in Plant 505. The primary objective of these studies was to

investigate the effects of process variables and to arrive at optimum conditions for preparing suitable feedstocks for subsequent hydrocracking studies.

Two additional series of data have been obtained at 250 psig above base pressure. One series of studies was carried out at about 11°C above base temperature and three space velocities. Data are summarized in Table 2. Another series of studies was conducted at about 5°C below base temperature and otherwise identical space velocities. Results are tabulated in Table 3.

Task 1

Work Forecast

Hydrotreating process variable studies on raw EDS process gas oil should be completed by the end of January.

All hydrocracking runs on H-Coal gas oil have been completed (See Figure 1). Preparation of the interim report will be under way when all required analyses of the hydrocracked products have been completed and all product distribution data are obtained.

Task 2

Work Accomplished

Preparation of hydrotreated H-Coal gas oils for FCC studies was continued. Table 4 gives the inspections of the charge stock (3532-1).

The hydrotreated H-Coal gas oils from Plant 601, Run 760 (reported in FE-2566-09) were blended to provide twelve gallons of FCC feedstock. Inspections of this hydrotreated oil are given in Table 5. Table 6 is a

summary of an overall material balance made for the run for the purpose of obtaining product distribution data. Total over 100% represents hydrogen added to the feedstock.

The second preparative H-Coal gas oil hydrotreating run (Plant 638H, Run 17) was continued for 16 additional periods (192 hours) to reprocess products obtained earlier (see Table 7, FE-2566-09) which contained less than 12% hydrogen. Data obtained up to the termination of the run are summarized in Table 7, a continuation of Table 7, FE-2566-09. Table 8 shows an overall material balance for the run. All products were blended. Preliminary distillation data show that this hydrotreated oil contains 55 vol-% gasoline and has an end point of 592°F. Only the 385°F$^+$ fraction of this upgraded oil will be used as FCC feedstock.

Task 2

Work Forecast

FCC studies on two hydrotreated H-Coal liquids and the rerun H-Coal gas oil will start in the beginning of January. All FCC runs should be completed by the end of the month.

Task 3

Work Accomplished

Mass spectroscopy (MS) analyses of the EDS process naphtha before and after upgrading have been completed. Table 9 shows the inspections of the raw naphtha and the upgraded naphtha. Table 10 gives the results from MS analyses of the raw naphtha while Table 11 shows the product distribution

and MS analysis of the C_6+ fraction of the upgraded EDS naphtha.

The upgraded naphtha was reformed over a commercial Platforming® catalyst over a range of conditions to obtain data for constructing a yield octane curve. Complete data are summarized in Table 12. This table shows the reforming conditions, product yields, and inspections of the C_5+ products. The table also includes the Research Octane Number (RON) of the C_5+ products obtained at various conditions.

Mass spectroscopy analyses of the C_6+ fractions from four selected reformates have been completed. Results are summarized in Tables 13-16. These tables also give product distribution and RON of the C_5+ products.

Task 3

Work Forecast

Elemental analyses of the C_5+ reformates should be completed in the beginning of January. Preparation of the interim report on reforming of H-Coal derived naphthas will start in January, as soon as elemental analyses of the C_5+ reformates are completed.

Table 1

Inspections of Raw EDS Liquid Product

Drum No.	2
UOP Sample No.	3532-1
°API @ 60°F	7.6
Sp. Gr. @ 60°F	1.0173
Distillation, ASTM D-1160	
IBP, °F	278
5%	428
10%	435
20%	452
30%	469
40%	511
50%	561
60%	619
70%	720
80%	825
90%	908
95%	953
EP	978
% Over	96.0
% Bottoms	4.0
Hydrogen, Wt-%	8.73
Carbon, Wt-%	86.00
Sulfur, Wt-ppm	6500
Nitrogen, Wt-ppm	4580
Oxygen, Wt-ppm	9100
Con. Carbon, Wt-%	4.02
Heptane Insolubles, Wt-%	8.11
882°F- Liquid	
Vol-%	86.7
Heptane Insolubles, Wt-%	1.75
Benzene Insolubles, Wt-%	0.01

Table 2

Hydrotreating Raw EDS Process Liquid Product(3532-1)
Plant 505, Run 861

Period No.	Hours on Stream	P-P(base), psig	LHSV / LHSV(base)	T-T(base), °C	Prod. N Content, ppm	N Conv., Wt-%
Feed					4580	
17	197-207	250	2.00	12	81.6	98.22
18	207-217	250	2.00	11	77.3	98.31
19	217-227	250	2.00	12	82.7	98.19
20	235-241	250	3.00	11	234.0	94.89
21	241-247	250	3.00	11	246.0	94.63
22	247-256	250	3.20	11	225.5	95.08
23	263-273	250	1.00	11	7.8	99.83
24	273-283	250	0.80	11	4.6	99.91
25	283-293	250	0.80	10	4.3	99.91
26	301-309	250	2.00	12	77.6	98.31
27	309-317	250	2.06	9	118.0	97.45
28	317-325	250	1.98	9	114.0	97.51

Table 3

Hydrotreating Raw EDS Process Liquid Product(3532-1)
Plant 505, Run 862

Period No.	Hours on Stream	P-P(base), psig	LHSV / LHSV(base)	T-T(base), °C	Prod. N Content,ppm	N Conv., Wt-%
Feed					4580	
1	22-30	250	2.20	-5	204.0	95.55
2	30-38	250	2.20	-5	200.0	95.63
3	38-47	250	2.00	-5	159.5	96.52
4	47-55	250	2.00	-5	215.8	95.29
5	55-63	250	2.00	-4	223.2	95.13
6	63-71	250	2.00	-5	228.3	95.02
7	77-83	250	3.14	-4	460.6	89.94
8	83-89	250	3.06	-4	568·0	87.60
9	89-97	250	3.08	-4	565.0	87.66
10	107-117	250	0.90	-4	10 .1	99.78
11	117-127	250	0.74	-4	12.8	99.72
12	127-137	250	0.84	-4	20.6	99.55
13	145-153	250	2.24	-6	244.0	94.67
14	153-161	250	1.98	-5	222.0	95.15
15	161-169	250	1.96	-4	183.0	96.00

Table 4

Inspections of H-Coal Atmospheric Still Bottoms

	As Received		After Flash Distillation
HRI Sample No.	LO-585	LO-586	
UOP Sample No.	37-1118	37-1117	3581-11
°API @ 60°F	7.4	12.1	8.9
Sp. Gr @ 60°F	1.0187	0.9854	1.0078
Distillation, ASTM D-1160			
IBP, °F	412	410	415
5%	463	467	462
10%	485	474	490
20%	509	495	518
30%	530	519	538
40%	558	545	553
50%	580	566	571
60%	603	591	590
70%	631	621	615
80%	661	666	645
90%	715	710	689
95%	768	772	715
EP	857	855	769
% Over	99.0	99.0	99.0
% Bottoms	1.0	1.0	1.0
Hydrogen, Wt-%			9.14
Carbon, Wt-%			88.98
Sulfur, Wt-ppm	951	690	600
Nitrogen, Wt-ppm	3534	2066	4100
Oxygen, Wt-ppm	5006	5480	
Con. Carbon, Wt-%	0.41	0.49	<0.01
Heptane Insolubles, Wt-%	0.37	0.53	0.05
FIA, Vol-%			
A	90.9	83.7	90.2
O	0	0	-
P&N	9.1	16.3	9.8
Stm.Jet Gum, mg/100 ml	620	933	37

Table 5

Inspections of Hydrotreated H-Coal Gas Oil 3531-25

Sample No.	3531-25
°API @ 60°F	16.8
Sp. Gr. @ 60°F	0.9541
Distillation, ASTM-D86	
IBP°, F	373
5%	434
10%	455
20%	480
30%	492
40%	513
50%	528
60%	548
70%	570
80%	597
90%	640
95%	685
% Over	96.5
% Bottoms	3.5
Hydrogen, Wt-%	10.68
Carbon, Wt-%	88.77
Sulfur, Wt-%	6.7
Nitrogen, Wt-ppm	8563
Con. Carbon, Wt-%	<0.01
Heptane Insolubles, Wt-%	<0.01
Molecular Weight, Average	201
FIA, Vol-%	
A	73.0
O	0.0
P&N	27.0

Table 6

Hydrotreating Rerun H-Coal Atmospheric Still Bottoms (3531-11)

Product Distribution

Plant 601, Run 760

Product Distribution, Wt-% of Feed

Liquid Product[(a)]	100.1
C_5 and C_6 in Plant Gas	0.1
Gas (C_1-C_4)	0.1
H_2O	0.6
H_2S	0.8
NH_3	0.5
Total	102.2

H_2 Consumption, Wt-% of Feed	2.2
H_2 Consumption, SCF/bbl	1380

[(a)] Designated as hydrotreated H-Coal gas oil 3531-25.

Table 7

Hydrotreating Rerun H-Coal Atmospheric Still Bottoms (3531-11)

Plant 638H, Run 17

Preparative Run No. 2

P-P (base), psig: 500

Period No.	Hours on Stream	LHSV / LHSV(base)	T-T (base), °C	Product Analysis, Wt-% H	C
Feed				9.14	88.98
27	339-351	0.29	21	13.71	86.42
28	351-363	0.29	21	13.64	86.17
29	363-375	0.29	20	13.85	85.91
30	375-387	0.30	21	13.91	89.59
31	387-399	0.30	21	14.33	85.99
32	399-411	0.29	22	14.36	86.09
33	411-423	0.30	21	14.16	85.04
34	423-435	0.30	21	14.35	85.22
35	435-447	0.30	22	-	-
36	447-459	0.30	21	14.58	85.62
37	459-471	0.29	22	-	-
38	471-483	0.30	22	14.29	84.96
39	483-495	0.30	22	14.66	85.41
40	495-507	0.29	20	14.80	85.23
41	507-519	0.30	25	-	-
42	519-531	0.30	27	-	-

Table 8

Hydrotreating Rerun H-Coal Atmospheric Still Bottoms (3531-11)

Product Distribution

Plant 638H, Run 17

Product Distribution, Wt-% of Feed	
Liquid Product[(a)]	96.5
C_5 and C_6 in Plant Gas	2.2
Gas (C_1 - C_4)	4.3
H_2O	0.6
H_2S	0.3
NH_3	0.5
Total	104.2
H_2 Consumption, Wt-% of Feed	4.2
H_2 Consumption, SCF/bbl	2640

[(a)] Designated as hydrotreated H-Coal gas oil.

Table 9

Inspection Data of EDS Process Naphthas

	As Received	Upgraded
Sample No.	3531-7	3531-12
°API @ 60°F	38.4	44.1
Sp. Gr. @ 60°F	0.8328	0.8058
Distillation, ASTM D-86		
IBP, °F	142	202
5%	178	215
10%	208	226
20%	244	237
30%	268	250
40%	286	264
50%	302	287
60%	316	305
70%	319	322
80%	339	336
90%	348	351
95%	-	360
EP	380	374
% Over	-	98.6
Hydrogen, Wt-%	-	13.34
Carbon, Wt-%	-	86.18
Sulfur, Wt-ppm	9,978	0.1
Nitrogen, Wt-ppm	2,097	0.2
Oxygen, Wt-ppm	13,700	98.0
Chloride, Wt-ppm	18	1.0
FIA, Vol-%		
A	-	20.1
P&N	-	79.9
MS Hydrocarbon Types, Vol-%		
A	25.3	21.62
N	42.9(b)	65.48
P	13.2	12 90
Bromine Index	39(a)	124.0
RON, Clear	83.2	64.5
N Jet Gum, mgl/100 ml	44.0	-

(a) Bromine number.

(b) Vol-% of Polars and olefins are 8.7 and 9.9, respectively (See Table 10).

Table 10

MS Analysis of EDS Naphtha 3531-7

Series	Hydrocarbon Types	Wt-%	Vol-%
C_nH_{2n+2}	Paraffins	11.3	13.2
	Naphthenes		
C_nH_{2n}	Monocycloparaffins		
	Cyclopentanes	9.8	10.4
	Cyclohexanes	14.1	15.0
C_nH_{2n-2}	Bi, Dicycloparaffins	10.9	10.5
C_nH_{2n-4}	Tricycloparaffins	7.4	7.0
	Aromatics		
C_nH_{2n-6}	Alkylbenzenes	17.5	17.0
C_nH_{2n-8}	Indanes/Tetralins	8.7	7.9
C_nH_{2n-10}	Dinaphthenebenzenes	0.5	0.4
C_nH_{2n-12}	Naphthalenes	Trace	Trace
	Polars		
$C_nH_{2n-4}O$	Furans	Trace	Trace
$C_nH_{2n-6}O$	Phenols	9.1	7.4
$C_nH_{2n-7}N$	Naphthenopyridines	0.1	0.1
$C_nH_{2n-4}S$	Thiophenes	1.5	1.2
	Olefins*		
C_nH_{2n}	Monoolefins	2.3	2.6
C_nH_{2n-2}	Diolefins and/or Monocycloolefins	5.4	5.8
C_nH_{2n-4}	Triolefins and/or Dicycloolefins	1.4	1.5
	Total	100.0	100.0

Carbon Number Distribution, Vol-%

Carbon No.	Aromatics J = 6	J = 8	J = 10	J = 12	Polars J = 6^O
6	0.3				1.8
7	3.7				5.4
8	3.9	0.1			0.1
9	4.8	3.6	0.3		0.1
10	4.0	4.2	0.1	Trace	
11	0.3			Trace	
Total	17.0	7.9	0.4	Trace	7.4

*The total olefin number was obtained by SiO_2 separation, but the mono-, di-,tri-olefin split is estimated since no calibration coefficients are available.

Table 11

Distribution of Upgraded EDS Naphtha 3531-12

Product Distribution	Wt-%	Vol-%
n-Pentane	0.4	0.5
Isopentane	0.1	0.1
C_6 Plus	99.5	99.4
Total	100.0	100.0

MS Analysis of C_6 Plus Fraction

I. Hydrocarbon Types	Vol-%
Paraffins	12.90
Naphthenes	
Monocycloparaffins	50.15
Bi, Dicycloparaffins	15.33
Tricycloparaffins	0.0
Aromatics	
Alkylbenzenes	17.82
Indans, Tetralins	3.80
Naphthalenes	0.0
Total	100.00

II. Carbon Number Distribution, Vol-%

Carbon No.	Paraffins	Monocyclo-Paraffins	Aromatics J = 6	Aromatics J = 8	Aromatics J = 12
6	0.05	6.90	0.65		
7	5.59	15.17	4.13		
8	4.52	11.18	4.73	0.0	
9	2.41	8.94	4.16	1.72	
10	0.0	5.45	3.91	2.08	0.0
11	0.33	2.51	0.24	0.0	0.0
12	0.0	0.0	0.0	0.0	0.0
13	0.0	0.0	0.0	0.0	0.0
Total	12.90	50.15	17.82	3.80	0.0

Table 12

Platforming® Upgraded EDS Naphtha

Plant 508, Run 1456

Period No.	Feed		1		2		3		4		5	
Reforming Conditions												
P-P (base), psig			0		0		0		0		0	
T-T(base), °C			-53		-73		-86		-97		-114	
LHSV/LHSV(base)			1.55		1.52		1.53		1.52		1.52	
Product Yields	Wt-%	Vol-%	Wt-%	Vol-%	Wt-%	Vol-%	Wt-%	Vol-%	Wt-%	Vol-%	Wt-%	Vol-%
H_2			3.2		3.0		2.8		2.7		2.3	
C_1-C_3			0.5		0.3		0.1		0.1		-	
nC_4	-	-	0.1	0.2	-	-	-	-	0.1	0.1	-	-
iC_4	-	-	-	-	-	-	-	-	-	-	-	-
C_5 Plus	100.0	100.0	96.2	90.3	96.7	91.4	97.1	92.1	97.1	92.5	97.6	93.7
Total	100.0	100.0	100.0	90.5	100.0	91.4	100.0	92.1	100.0	92.6	100.0	93.7
Product (C_5+)Inspection												
°API @ 60°F	44.1		33.5		34.9		35.3		36.1		37.1	
Sp.Gr. @ 60°F	0.8058		0.8576		0.8504		0.8483		0.8443		0.8393	
Distillation, ASTM D-86												
IBP, °F	202		200		190		197		190		198	
5%	215		215		212		216		212		216	
10%	226		227		226		228		223		227	
30%	250		261		260		260		255		260	
50%	287		300		296		298		290		298	
70%	322		339		334		338		332		337	
90%	351		379		372		375		368		378	
95%	360		402		387		390		383		396	
EP	374		441		423		420		410		436	
RON, Clear	64.5		99.7		97.9		95.9		95.1		92.1	
Elemental Analysis												
Hydrogen, Wt-%	13.34		10.40		10.74		11.23		11.45		11.43	
Carbon, Wt-%	86.18		89.30		89.48		88.51		88.18		88.89	
Sulfur, Wt-ppm	0.1		<0.1		0.1		<0.1		0.1		< 0.1	
Nitrogen, Wt-ppm	0.2		0.1		0.1		0.1		0.1		0.1	

Table 12, Cont'd.

Period No.	6		7		8		9		10	
Reforming Conditions										
P-P(base), psig	0		0		0		0		0	
T-T(base), °C	-119		-120		-69		-39		-6	
LHSV/LHSV(base)	1.42		1.52		1.51		1.53		1.51	
Product Yields	Wt-%	Vol-%	Wt-%	Vol-%	Wt-%	Vol-%	Wt-%	Vol-%	Wt-%	Vol-%
H_2	2.2		2.1		3.1		3.4		3.6	
C_1-C_3	-		-		0.1		0.5		2.5	
nC_4	-	-	-	-	-	-	0.1	0.1	0.8	1.1
iC_4	-	-	-	-	-	-	-	-	0.5	0.8
C_5 Plus	97.8	94.1	97.9	94.1	96.8	91.3	96.0	89.6	92.5	85.7
Total	100.0	94.1	100.0	94.1	100.0	91.3	100.0	89.6	100.0	87.6
Product (C_5+) Inspection										
°API @ 60°F	57.8		37.7		34.4		32.6		30.5	
Sp. Gr. @ 60°F	0.7475		0.8363		0.8529		0.8623		0.8735	
Distillation, ASTM D-86										
IBP, °F	190		202		194		198		194	
5%	209		219		215		219		214	
10%	220		229		226		231		226	
30%	251		261		257		264		258	
50%	288		298		293		303		294	
70%	326		338		332		343		336	
90%	364		375		370		388		389	
95%	380		393		385		409		406	
EP	407		452		419		460		460	
RON, Clear	90.6		90.4		98.2		101.5		104.5	
Elemental Analysis										
Hydrogen, Wt-%	11.59		11.60		10.51		10.18		9.94	
Carbon, Wt-%	88.06		88.35		88.96		90.35		89.73	
Sulfur, Wt-ppm	0.1		0.1		< 0.1		<0.1		<0.1	
Nitrogen, Wt-ppm	0.1		0.1		0.1		0.1		0.1	

Table 13

Platforming® Upgraded EDS Naphtha
Product Distribution and MS Analysis

Plant 508, Run 1456, Period 2

Product Distribution	Wt-%	Vol-%
H_2	3.0	
C_1-C_3	0.3	
nC_4	-	-
iC_4	-	-
nC_5	0.7	0.9
iC_5	-	-
C_6 Plus	96.0	90.5
Total	100.0	91.4

Reformate (C_5 Plus)	
Yield, Vol-%	91.4
RON, Clear	97.9

MS Analysis of C_6 Plus Fraction, Vol-%

I. Hydrocarbon Types	
Paraffins	15.37
Naphthenes	
Monocycloparaffins	7.82
Bi, Dicycloparaffins	1.18
Tricycloparaffins	0.0
Aromatics	
Alkylbenzenes	64.20
Indans, Tetralins	9.53
Naphthalenes	1.90
Total	100.00

II. Carbon Number Distribution, Vol-%

Carbon No.	Paraffins	Monocyclo-paraffins	Aromatics J = 6	Aromatics J = 8	Aromatics J = 12
6	0.95	2.30	8.18		
7	2.97	1.39	19.57		
8	3.74	1.93	15.44	0.0	
9	3.24	1.37	11.25	3.51	
10	2.44	0.61	7.11	4.93	1.57
11	2.03	0.22	2.55	1.09	0.31
12	0.0	0.0	0.10	0.0	0.02
13	0.0	0.0	0.0	0.0	0.0
Total	15.37	7.82	64.20	9.53	1.90

Table 14

Platforming® Upgraded EDS Naphtha
Product Distribution and MS Analysis

Plant 508, Run 1456, Period 4

Product Distribution	Wt-%	Vol-%
H_2	2.7	
C1-C3	0.1	
nC_4	0.1	0.1
iC_4	-	-
nC_5	0.5	0.7
iC_5	0.1	0.2
C_6 Plus	96.5	91.6
Total	100.0	92.6

Reformate (C_5 Plus)	
Yield, Vol-%	92.5
RON, Clear	95.1

MS Analysis of C_6 Plus Fraction, Vol-%

I. Hydrocarbon Types	
Paraffins	16.55
Naphthenes	
Monocycloparaffins	15.56
Bi, Dicycloparaffins	1.02
Tricycloparaffins	0.0
Aromatics	
Alkylbenzenes	56.24
Indans, Tetralins	8.88
Naphthalenes	1.75
Total	100.00

II. Carbon Number Distribution, Vol-%

Carbon No.	Paraffins	Monocyclo-Paraffins	Aromatics J = 6	Aromatics J = 8	Aromatics J = 12
6	0.65	3.30	4.35		
7	2.10	2.91	16.48		
8	2.99	3.57	14.08	0.0	
9	5.07	2.90	10.92	3.02	
10	3.03	1.62	7.47	4.77	1.59
11	2.70	1.01	2.81	1.09	0.16
12	0.01	0.25	0.13	0.0	0.0
13	0.0	0.0	0.0	0.0	0.0
Total	16.55	15.56	56.24	8.88	1.75

Table 15

Platforming® Upgraded EDS Naphtha
Product Distribution and MS Analysis

Plant 508, Run 1456, Period 6

Product Distribution	Wt-%	Vol-%
H_2	2.2	
C_1-C_3	-	
nC_4	-	-
iC_4	-	-
nC_5	0.6	0.7
iC_5	0.1	0.2
C_6 Plus	97.1	93.2
Total	100.0	94.1

Reformate (C_5 Plus)	
Yield, Vol-%	94.1
RON, Clear	90.6

MS Analysis of C_6 Plus Fraction, Vol-%

I. Hydrocarbon Types

Paraffins	15.38
Naphthenes	
Monocycloparaffins	24.25
Bi, Dicycloparaffins	0.99
Tricycloparaffins	0.0
Aromatics	
Alkylbenzenes	48.39
Indans, Tetralins	10.60
Naphthalenes	0.39
Total	100.00

II. Carbon Number Distribution, Vol-%

Carbon No.	Paraffins	Monocyclo-paraffins	Aromatics J = 6	Aromatics J = 8	Aromatics J = 12
6	0.32	5.90	3.60		
7	2.03	5.83	13.45		
8	3.04	5.21	11.86	0.0	
9	4.20	3.83	9.78	3.39	
10	2.99	2.28	7.10	5.78	0.36
11	2.79	1.05	2.60	1.43	0.03
12	0.01	0.15	0.0	0.0	0.0
13	0.0	0.0	0.0	0.0	0.0
Total	15.38	24.25	48.39	10.60	0.39

R----Rerunning
U----Unifining

Figure 1
Work Plan and Progress

Legend
H-Coal Liquids
EDS Liquids
Scheduled
Completed