APPENDIX A

TABLES OF OPERATING DATA

TABLE NO. 4

Analysis of German Coals Used For Hydrogenation:

Type of Coal	Brown Coal		Bituminous Coal	
Plant	Leuna	Wesseling	Gelsenberg	Pölitz
% Ash in dry coal	12.8	5.9	3.0	6.6
Element Analysis on dry, ash-free basis				
% C	71.9	68.7	83.8	81.9
% H	5.7	5.0	5.3	4.9
% O	17.9	24.9	8.0	11.5
% N	1.0	1.3	1.75	1.1
% S	5.8	0.40	1.0	0.8
% Cl	----	0.11	0.09	0.05
% available H	4.1	2.4	4.6	3.9
Volatile Matter	57.9	53.5	37.5	37.2

TABLE NO. 5

Summary of Operation Data for Sump Phase Hydrogenation

Material Treated	Brown Coal	Brown Coal Tar	Bituminous Coal	Pitch
Temperature, °C	470-480	450-460	485	485
Pressure, atm.	300&700	300	700	700
Catalyst	Iron Oxide	Iron sulfide	Sodium Sulfide Iron Oxide Iron Sulfide	Iron Sulfide
Total reaction volume per stall, in M^3	27	27	36	36

TABLE NO. 5 (cont'd.)

Material Treated	Brown Coal	Brown Coal Tar	Bituminous Coal	Pitch
Specific feed rate tons/m3 hr	1.1 - 1.3	0.75	0.9 - 1.0	0.7
Total feed rate, tons/hr	30 - 35	20	33 - 37	25
Concentration of material in feed, wt. %	36 - 40	75	40	60
Total gas flow, M/hr	35-45,000	27,000	50,000	40,000
Gas Circulated with paste, M^3/hr.	25-30,000	20,000	30,000	25,000
Gas used for cooling, M^3/hr	10-15,000	7000	20,000	15,000
Water added to product, M^3/hr.	1	1	1	0.7
% Conversion of Coal	97	-	95	----
Tons of middle oil/ton feed	0.48	0.38	0.62	0.29
Tons of C3 plus C4/ton feed	0.06	0.03	0.12	0.02
Hydrogen Consumed /ton middle oil, M^3	1500	250	1600	700-800

TABLE NO. 6

Analysis of Sump Phase Oils

Middle Oil From	Brown Coal	Bitminous Coal
Specific Gravity 20°C	0.962	0.974
Aniline point Phenol-free oil, °C.	-10	-20
% Phenolics	20	15
Element Analysis		
% C	86.05	87.28

TABLE NO. 6 (cont'd.)

Middle Oil From	Brown Coal	Bituminous Coal
Element Analysis		
% H	9.37	9.54
% O	3.90	3.08
% N	0.55	1.06
% S	0.13	0.04
gms H / 100 gms. C	10.90	10.93
Heavy Oil from		
Specific gravity, 50°C.	1.030	1.038
Element Analysis		
% C	88.62	89.27
% H	8.40	8.26
% O	2.62	1.64
% N	0.28	0.77
% S	0.08	0.06
gms H/100 gms C.	9.46	9.25

TABLE NO. 7

Summary of Operating Data for Gas Phase Hydrogenation

Stage	Prehydrogenation	Gasoline Production Coal & Tar	Pitch
Temperature, °C	390-410	400-420	500
Pressure, Atm.	300	300	700
Catalyst	Tungsten Disulfide	Tungsten Dissulfide & Act. Clay	Chromo-Zinc Malybdenum Act. Clay
Total Catalyst volume per stall, M³	28	28	32
Specific feed rate, tons/M³ hr.	0.6-0.8	1.2	0.45

TABLE NO. 7 (cont'd.)

Stage	Prehydrogenation	Gasoline Production Coal & Tar	Pitch
Total feed rate, tons/hr	20	35	14
Total gas flow M3/hr	55,000	27,000	38,000
Gas circulated with oil, M3/hr	35,000	20,000	30,000
Gas used for cooling M3/hr	20,000	7,000	8,000
Tons gasoline produced /ton feed/pass*	0.20	0.60	0.50
Tons C3 plus C4/ 1 ton feed/pass*	0.08	0.075	0.12
Hydrogen consumed/ton middle oil, M3	500	200	950

TABLE NO. 8

Comparison of Screw Type with Ball Type Sludge Coking Ovens

Type of Oven	Screw		Ball	
Plant	Wesseling	Leuna	Wesseling	Gelsenberg
Steam Consumption tons/ton feed	6.5	19.5	15.5	20.0
% solids in moisture free product	75.5	81.0	99.5	96.2
% ash in solids	86.2	83.0	64.2	41.1
% Benzol soluble oil in discharge product	24.5	19.0	0.5	3.8
Coke analysis of moisture free product				
% tar	6.1	8.7	0.5	1.3
% Coke	93.2	91.3	99.4	95.0
Ash content of the coke	70.8	75.2	64.4	42.0

*Single pass through the Converters Yields.

TABLE NO. 9

Element Balance for Sump Phase Operation with Brown Coal

	Total Tons	Carbon Tons	Hydrogen Tons	Oxygen Tons	Nitrogen Tons	Sulfur Tons	Residue Tons
Dry Brown Coal	1000.00	619.12	48.06	181.93	5.81	53.72	91.36
Catalyst	89.48			61.53			27.95
Fresh Gas H_2	52.95		52.95				
N_2	13.94				13.94		
CO	3.03	1.30		1.73			
CO_2	2.48	0.68		1.80			
CH_4	5.76	4.31	1.45				
Total Fresh Gas	78.16	6.29	54.40	3.53	13.94	-	-
Total Inlet	1167.64	625.41	102.46	246.99	19.75	53.72	119.31
Middle Oil	417.17	353.98	44.24	14.84	1.68	2.43	
High Volatile Gasoline	19.73	16.42	3.31				
Total Liquid Product	436.90	370.40	47.55	14.84	1.68	2.43	
Outlet Gas H_2	7.29		7.29				
N_2	13.94				13.94		
CO	13.27	5.69		7.58			
CO_2	91.26	24.90		66.36			
CH_4	37.68	28.20	9.48				
C_2	31.80	25.40	6.40				
C_3	36.41	29.75	6.66				
C_4	19.39	16.02	3.37				
C_5	2.90	2.41	0.49				
H_2S	34.66		2.05			32.61	
Total outlet Gas	288.60	132.37	35.74	73.94	13.94	32.61	-
Miscellaneous Solids in Sludge	214.13	18.01	0.72	38.99	0.67	21.00	134.74

TABLE NO. 9 (cont'd.)

	Total Tons	Carbon Tons	Hydrogen Tons	Oxygen Tons	Nitrogen Tons	Sulfur Tons	Residue Tons
Water of Reaction	148.25		16.59	131.66			
Phenols	2.18	1.67	0.14	0.37			
H_2S in Disch. H_2O	1.21		0.07			1.14	
NH_3 in Disch. H_2O	3.21		0.57		2.64		
CO_2 in Disch. H_2O	4.49	1.22		3.27			
Oil Lost in Coking	75.05	70.50	4.18	0.27	0.05	0.05	
Total Miscellaneous	448.52	91.40	22.27	174.56	3.36	22.19	134.74
Total Outlet	1174.02	594.17	105.56	263.34	18.98	57.23	134.74
Balance Difference	÷ 6.38	-31.24	÷3.10	÷16.35	-0.77	÷3.51	÷15.93
Balance Difference in % of Inlet	÷ 0.54	-5.00	÷3.02	÷ 6.62	-3.90	÷6.53	÷12.93

TABLE NO. 10

Element Balance for Sump Phase Operation with Bituminous Coal

	Total Tons	Carbon Tons	Hydrogen Tons	Nitrogen Tons	Sulfur Tons	Oxygen Tons	Residue Tons
Dry Coal	1000	769	45.0	11.3	5.2	107.7	61.8
Catalyst	44.8		1.4		2.7	20.8	19.9
Pasting Oil	1363	1239.3	99.5	10.6		13.6	
Solids in Pasting Oil	117.4	72.5	2.6	0.8	2.9	0.7	37.9
Heavy Oil Added	63.9	57.7	5.2	0.4		0.6	
Flushing Oil	52.0	46.8	4.3	0.4		0.5	
Fresh Gas(1,150,000M^3)	140.0	14.6	95.7	29.3		0.4	
Total Inlet	2781.1	2199.9	253.7	52.8	10.8	144.3	119.6

TABLE NO. 10 (cont'd.)

	Total Tons	Carbon Tons	Hydrogen Tons	Nitrogen Tons	Sulfur Tons	Oxygen Tons	Residue Tons
Middle Oil	526						
Cold Separator Oil		965.3	91.4	8.0	0.3	24.0	
Heavy Oil	563						
Phenol Oil	3.7	2.8	0.3			0.6	
Oil in Slurry	922	844	62.5	8.1		7.4	
Solids in Slurry	294	138.3	5.9	1.2	7.6	1.7	139.3
Slurry Gas	30.1	14.6	8.0	5.5		2.0	
Lean Gas	146	79.7	34.4	21.8	0.5	9.6	
Rich Gas	166.7	123.5	31.4	4.8	0.8	6.2	
Reaction Water	96		10.7			85.3	
NH_3 in Water	6.8		1.2	5.6			
H_2S in Water	0.2		0.0		0.2		
CO_2 in Water	8.9	2.4				6.5	
TOTAL OUTLET	2763.4	2170.6	245.8	55.0	9.4	143.3	139.3
Balance Difference	-17.7	-29.3	-7.9	+2.2	-1.4	-1.0	+19.7
Balance Difference in % of inlet	-0.64	-1.34	-3.12	+4.16	-13.0	-0.70	+16.5

TABLE NO. 11

Element Balance for 5058 Gas Phase Operation with Brown Coal Middle Oil

	Total Tons	Carbon Tons	Hydrogen Tons	Oxygen Tons	Nitrogen Tons	Sulfur Tons
Middle Oil	1102.98	935.8	116.92	39.26	4.52	6.40
High Volatile Gasoline	27.04	22.51	4.53			
H_2S	3.36		0.20			3.16

TABLE NO. 11 (cont'd.)

		Total Tons	Carbon Tons	Hydrogen Tons	Oxygen Tons	Nitrogen Tons	Sulfur Tons
Fresh Gas	H_2	52.34		52.34			
	N_2	13.70				13.70	
	CO	3.14	1.35		1.79		
	CO_2	2.61	0.71		1.90		
	CH_4	5.62	4.21	1.41			
Total Fresh Gas		77.41	6.27	53.75	3.69	13.70	
Total Inlet		1210.79	964.66	175.40	42.95	18.22	9.56
Gasoline		1000.00	858.32	139.46	0.94	1.14	0.14
Outlet Gas	H_2	3.25		3.25			
	N_2	11.84				11.84	
	CO	0.22	0.09		0.13		
	CO_2	0.92	0.25		0.67		
	CH_4	9.21	6.89	2.32			
	C_2	2.92	2.33	0.59			
	C_3	27.38	22.37	5.01			
	C_4	81.82	67.62	14.20			
	C_5	0.89	0.74	0.15			
	H_2S	2.44		0.14			2.30
Total Outlet Gas		140.89	100.29	25.66	0.80	11.84	2.30
Miscellaneous							
Water of Reaction		45.13		5.05	40.08		
Phenol		2.13	1.63	0.14	0.36		
NH_3 in Disch. H_2O		5.78		1.03		4.75	
H_2S in Disch. H_2O		6.45		0.38			6.07
CO_2 in Disch. H_2O		1.06	0.29		0.77		
Total Miscellaneous		60.55	1.92	6.60	41.21	4.75	6.07
Total Outlet		1201.44	960.53	171.72	42.95	17.73	8.51
Balance Difference		-9.35	-4.13	-3.68	--	-0.49	-1.05
Balance Difference in % of Inlet		-0.76	-0.43	-2.10	--	-2.69	-10.98

TABLE NO. 12

Element Balance for 5058 Gas Phase Operation with Bituminous Coal Middle Oil

	Total Tons	Carbon Tons	Hydrogen Tons	Nitrogen Tons	Sulfur Tons	Oxygen Tons
Middle Oil	1000	863.8	108	4.9	4.3	19.0
Fresh Gas 489,000 M^3	59.4	6.2	40.5	12.5		0.2
Total Inlet	1059.4	870.0	148.5	17.4	4.3	19.2
Cold Separator Oil Gasoline	360	852	131.9	0.1		
Cold Separator Oil Middle Oil	624					
Phenol Oil	0.4	0.3				0.1
Lean Gas	31.4	8.5	12.6	9.8		0.5
Rich Gas	7.8	5.6	1.5	0.5	0.1	0.1
Reaction Water	18.1		2.1			16.0
NH_3 in Disch Water	7.7		1.4	6.3		
H_2S in Disch Water	6.2		0.4		5.8	
CO_2 in Disch Water	0.4	0.1				0.3
Total Outlet	1056.0	866.5	149.9	16.7	5.9	17.0
Balance Difference	-3.4	-3.5	+1.4	-0.7	+1.6	-2.2
Balance Difference in % of Inlet	-0.32	-0.40	+0.95	-4.0	+37.3	-11.5

TABLE NO. 13

Element Balance for 6434 Gas Phase Operation with Bituminous Coal Middle Oil

	Total Tons	Carbon Tons	Hydrogen Tons	Nitrogen Tons	Sulfur Tons	Oxygen Tons
Middle Oil	1000	865.5	134		0.5	
Fresh Gas 192,000 M^3	23.3	2.4	15.9	4.9		0.1
Total Feed	1023.3	867.9	149.9	4.9	0.5	0.1
Gasoline	565					
Cold Separator Oil		801	136			
Middle Oil	372					
Lean Gas	19.0	7.2	4.9	6.2	0.5	0.2
Rich Gas	63.0	49.4	10,8	1.0	1.6	0.2
NH_3 in Disch H_2O	0.04		0.1	0.03		
H_2S in Disch H_2O	< 0.01					
CO_2 in Disch H_2O	0.02	< 0.01				0.02
Total Outlet	1019.0	857.6	151.7	7.2	2.1	0.4
Balance Difference	-4.3	-10.3	±1.8	±2.3	±1.6	±0.3
Balance Difference in % of Feed	-0.42	-1.12	-1.06	±47	±320	±300

APPENDIX B

CATALYST PREPARATION

Preparation of 5058 Catalyst.

Old catalyst was roasted in air at 800°C. to convert the tungsten to the oxide.

$$WS_2 + 3\tfrac{1}{2}O_2 \rightarrow WO_3 + 2SO_2$$

The tungsten oxide produced by roasting was either treated alone or combined with an impure tungsten oxide called "gelberde" which contained 92 percent WO_3. These oxides were then treated with filtrate from previous catalyst batches. The filtrate was an aqueous solution of ammonium polysulfide and ammonium thiotungstate, and to this added 10 percent make-up ammonia and hydrogen sulfide. After heating to 50°C. and stirring for about 3 hours most of the oxide was converted into ammonium thiotungstate.

$$WO_3 + (NH_4)_2S_x \rightarrow (NH_4)_2WS_4$$

The solution was filtered to remove undissolved material, and the filtrate was heated up to 70°C. Then the ammonium thiotungstate was allowed to slowly crystallize out with continual stirring for 8 hours, during which time the temperature was lowered to 20°C. The slurry was filtered with nitrogen pressure, and the filtrate was recycled to dissolve more tungsten oxide.

The crystals of $(NH_4)_2\ WS_4$ were not washed, but dried in a nitrogen atmosphere at 100°C for about 3 hours. Great care was taken to prevent access of air to the salt on account of oxidation to the oxysulfide which would result in loss of activity.

$$(NH_4)_2\ WS_4 + 3O_2 \longrightarrow (NH_4)_2\ WO_2S_2 + 2SO_2$$

The dry salt was next heated to 400°C in a stream of hydrogen to convert it into tungsten disulfide.

Preparation of 5058 Catalyst (cont'd.)

$$(NH_4)_2\,WS_4 + H_2 \longrightarrow WS_2 + 2NH_3 + 2H_2S$$

This operation was conducted in a four pass heated kiln with internal screws. The top two passes were heated and had hydrogen introduced to decompose the salt, while the lower two were unheated and used nitrogen for cooling of the tungsten disulfide.

The WS_2 was dry ground so that the average particle size was about 0.5-1 millimeter. Too fine a powder reduced the mechanical strength of the finished pellets. The powder was then pelleted in a regular pellet press to make pills 10 millimeters in diameter by 10 millimeters high. During the operation it was necessary to maintain an inert atmosphere with nitrogen or carbon dioxide around the dies to prevent oxidation. The pellets were tumbled around in a mill to remove sharp edges and produce smooth uniform cylinders. The finished catalyst was stored under a nitrogen atmosphere until ready for use.

Some of the important data about 5058 catalyst are given in the following table:

Diameter	10 mm
Height	10 mm
Volume	0.785 cm^3
Weight	3.15 grams
Outer surface/pellet	4.7 cm^2
Weight of 1 liter pellets	2600 grams
Pellet surface/ 1 liter of pellets	0.39 M^2
Compression strength	300 kg/cm^2
Compression strength after 1½ years service	270 kg/cm^2

Preparation of 6434 Catalyst.

Fullers earth was activated by treatment with 8-10 percent of 10 percent HF solution by adding the acid slowly and stirring for about 20 minutes. Then sufficient ammonium thiotungstate was dissolved in the mother liquor from which

Preparation of 6434 Catalyst (cont'd.)

it crystallized to give a 10 percent WS_2 content in the finished catalyst. The ammonium thiotungstate was prepared in exactly the same manner as was done for the 5058 catalyst. The mixture was then heated to about 120° C maximum and stirred for about 8 hours until approximately dry.

The mass was then cooled in nitrogen and ground to about 1 to 3 millimeter size before passing through a kiln at 400°C. similar to that used for the pure tungsten sulfide. A 1 to 1 mixture of hydrogen to hydrogen sulfide was used in the heating process instead of pure hydrogen sulfide and nitrogen. After cooling, about 28-30 percent water was added while stirring to the material to give it good cohesive properties in pelleting. The addition of this water did not make the catalyst mass appear wet, since the fullers earth absorbed it. The lumpy mass was screened to remove pieces over 3 millimeters, the oversize being reground, and then the power was pelleted in the same manner as was done with 5058.

After pelleting the pills were allowed to dry 3 to 4 hours to improve their hardness, and then tumbelled slightly in the mill. After final drying at 100-120°C, the catalyst was stored in a nitrogen atmosphere until used. Before use an initial heat treatment of 8 - 10 hours in hydrogen at 400-450°C improved the strength.

Some of the important facts about 6434 catalyst are given in the following table:

Diameter	10 mm
Height	10 mm
Volume	0.785 cm^3
Weight	1.24 grams
Outer surface/pellet	4.7 cm^2
Weight 1 liter pellets	800 grams
Pellet surface/liter	0.39 m^2
Compression strength	about 200 kg/cm^2

Preparation of Welheim Gas Phase Catalyst No. K536.

Approximately 90 kilos of crude kieselguhr paste having a water content of 60 percent were mixed carefully for 30 minutes with 4 kilos of 70-72 percent hydrofluoric acid solution. Then 10 kilos of Fullers earth were added and mixed thoroughly. Next 3.7 kilos of zinc oxide plus 3 kilos of flowers of sulfur were added together with 4.6 kilos 50 percent aqueous solution of chromic acid. After 10 kilos more of Fullers earth had been added and the whole charge thoroughly mixed, it was neutralized with about 9 kilos of 0.916 density ammonia solution. Then 1.4 kilos of ammonium thiomolybdate dissolved in 7 kilos of ammonia solution were added together with 4 kilos of Fullers earth and the whole mass thoroughly mixed.

The catalyst was dried to a water content of 30-33 percent and then pelleted into cylinders 10 millimeters in diameter by 10 millimeters high. These pills were dried 3 to 4 days at 75°C before storing. In order to activate the catalyst, it was necessary to heat it for 12 hours in hydrogen at 350°C. After activation the catalyst contained 0.7 percent Mo, 2.0 percent Cr, and 4.0 percent Zn as the active metallic consituents.

APPENDIX C

DRAWINGS

No. C-1	Heat Exchanger, 500 mm. diameter, Assembly
C-2	Gas-Fired Preheater, Section
C-3	Gas-Fired Preheater, Thermocouple Location.
C-4	Gas Phase Converter, 1000 mm diameter, Assembly
C-5	Hot Separator, 800 mm. diameter, Top & Bottom Cooling, Assembly
C-6	Hot Separator, 800 mm. diameter, Gas Injection, Assembly
C-7	Cold Separator, 1000 mm. diameter, Assembly
C-8	Expansion Valve, Assembly and Parts.
C-9	Electric Preheater, Assembly
C-10	Gas Phase Converter, 800 mm. diameter, Assembly.
C-11	Gas Phase Converter, 1000 mm. diameter, Assembly.
C-12	Coking Oven, Screw type, Section and Flowsheet.
C-13	Coking Oven, Ball Type, Flowsheet.
C-14	T.T.H. Converter, 1000 mm. diameter, Assembly.

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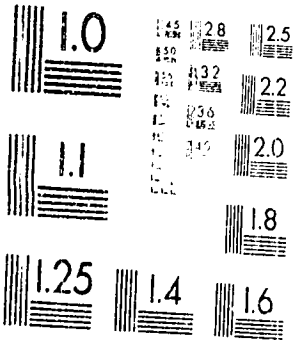
END OF REEL

JOB NO. E-108 AR-311-27



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