PART 1

PROCESSING, COAL-TO-OIL DEMONSTRATION PLANTS, LOUISIANA, MO.

Coal-Hydrogenation Demonstration Plant

In 1950, four liquid-phase and one vapor-phase hydrogenation runs were made in the Coal Hydrogenation Demonstration Plant near Louisiana, Mo., with a combined "on-stream" time of about 100 days. Approximately 3,000 tons of Rock Springs, Wyo., coal was converted into nearly 300,000 gallons of gasoline with a motor-method octane number of 78. The main operating conditions for the liquid-phase runs are summarized in table 1; analytical data of the streams are presented in table 2, and the flow sheet (see fig. 2) is illustrative of the operations.

Liquid-Phase Operations

Run No. 2 (Light Oil, Tar, and Coal)

From December 1949 through March 1950, the plant was down for extensive mechanical work involving cleaning, repairs, alterations, and testing. During this extended shutdown about 60 important process and equipment changes were made. Liquid-phase coal-hydrogenation run No. 2 was started on March 31 with a light start-up oil blend to test the controls and bring the unit up to operating conditions. On April 8, tar hydrogenation was started, and it continued until April 12. Throughout this period operations were very smooth, and it was possible to obtain useful information and data on tar hydrogenation. Operating conditions and yields are shown in the first column of table 1.

On April 12 Rock Springs coal was added to the system. Shortly afterward flow conditions, and hence operations, became very erratic. The instruments, which operated smoothly on tar, became undependable or failed to function, frequently upsetting the entire system. Some of this trouble was attributed to cold weather.

Owing to failures of plunger packing and leakage through or under the removable valve seats, the performance of the high-pressure injection pumps was very erratic, with the result that liquid flow conditions were unsteady. Cracks developed in the fluid end blocks of all but four of the injection pumps.

On April 15 and 16, several runaway reactions started in the converters but were brought under control. Erratic temperatures also indicated solids build-up in the converters. Simultaneously, a leak developed on the top head of the cold catchpot, which steadily became worse. A normal shutdown was in progress on April 16, when a violent runaway reaction occurred, causing leaks on lines, which forced an emergency shutdown.

Inspection revealed that the first converter was full of coke and the second converter contained 9 feet of coke in the bottom. The line between the converters was lined with coke. The preheater, hot catchpot, wash-oil scrubber, and cold catchpot were opened and found to be clean.

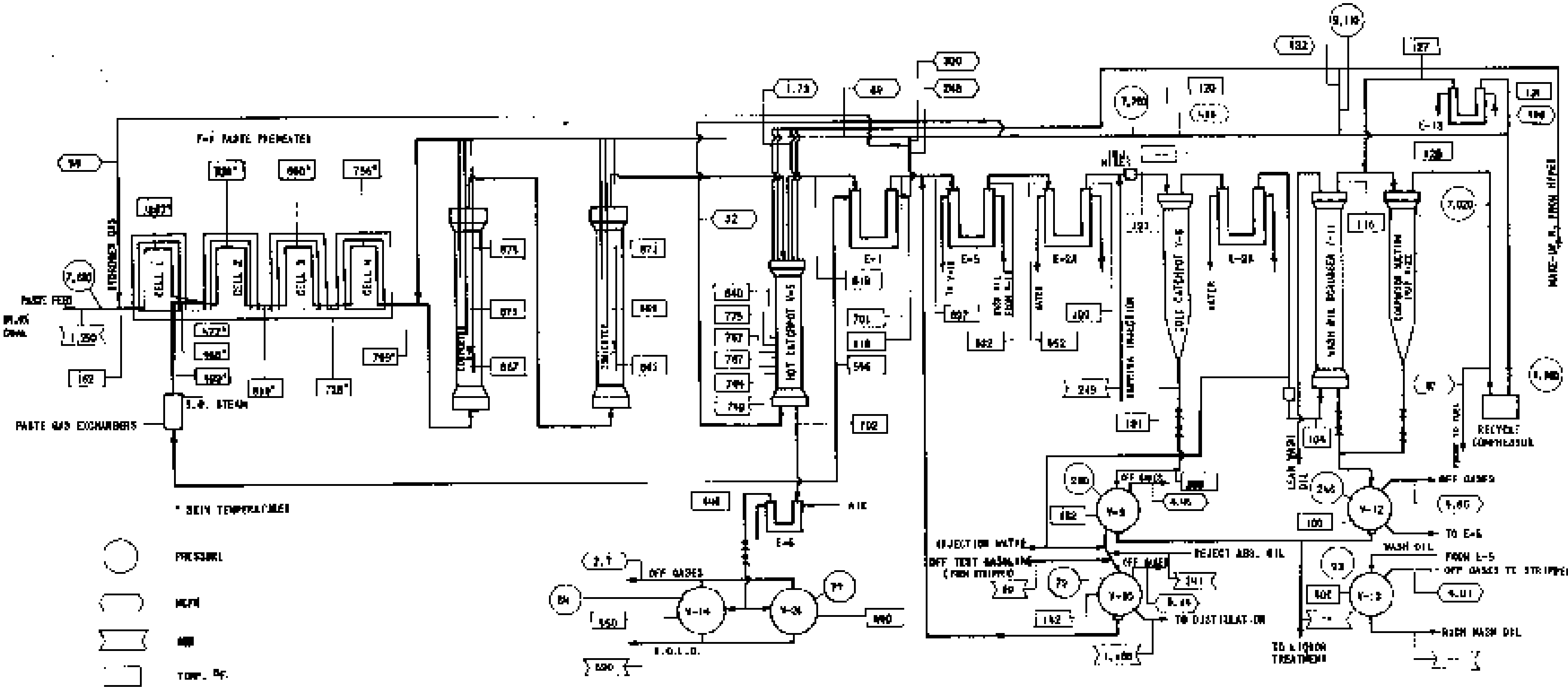

Figure 2. - Liquid-phase hydrogenation unit - typical operating conditions - Rock Springs coal hydrogenation.

TABLE 1. - Operational data, liquid-phase runs for 1950 selected periods representative of lined-out operations

	Run 2		Run 3	Run 4	Run 5	
	Tar oil	Coal	Coal	Coal	Coal	Coal
Pressure, p.s.i.g.	10,300	10,300	7,500	7,750	10,200	8,100
Recycle hydrogen, percent H_2	81	79	83	82	80	81
Paste injection, g.p.d.	-	27,900	32,000	33,300	30,800	29,100
Paste oil, g.p.d.	21,200	23,900	24,300	24,900	22,400	21,600
Coal, m.f., t.p.d.	0	22	45	52	56	56
Coal, m.f., wt. percent	0	13.5	27.3	29.3	33.5	34.2
Catalyst, type	2 percent MoO_3 on charcoal	Tin Oxalate	Tin Oxalate	Tin Oxalate	Tin Oxalate	Tin Oxalate
Weight percent on coal	0.5 (on fresh feed)	0.1	0.1	0.1	0.1	0.1
Naphtha injection, g.p.d.	5,100	4,200	4,250	5,990	7,300	8,700
Wash oil injection, g.p.d.	20,800	18,800	15,700	-	14,500	18,500
Gas flows, cu. ft./day:						
Make-up hydrogen gas	1,500,000	2,050,000	2,300,000	3,160,000	3,960,000	4,150,000
Total hydrogen to stalls	7,800,000	8,200,000	9,900,000	10,200,000	10,900,000	10,200,000
Compressor recycle	12,900,000	13,000,000	11,600,000	11,900,000	12,300,000	13,100,000
Total paste gas	5,470,000	5,200,000	7,200,000	7,250,000	7,200,000	7,030,000
Cold paste gas	1,250,000	1,060,000	1,500,000	1,300,000	1,200,000	1,230,000
Hot paste gas	1,220,000	4,100,000	5,700,000	5,950,000	6,000,000	5,800,000
Total cooling gas	1,500,000	2,000,000	2,000,000	2,150,000	2,630,000	2,470,000
Hot catchpot agitation	820,000	940,000	700,000	750,000	890,000	700,000
Purge gas	-	300,000	1,200,000	2,100,000	1,580,000	1,720,000
Temperatures, °F.:						
Paste preheater inlet	154	163	-	162	164	159
1st section outlet	433	406	456	466	464	462
Preheater outlet	834	854	849	847	852	853

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TABLE 1. - Operational data, liquid-phase runs for 1950 selected periods representative of lined-out operations (Cont'd.)

	Run 2		Run 3	Run 4	Run 5	
	Tar oil	Coal	Coal	Coal	Coal	Coal
First converter - °F.:						
Reaction, top zone.............	840	756	860	874	874	878
middle zone..........	839	792	854	873	865	854
bottom zone..........	840	728	801	867	-	839
Second converter - °F.:						
Reaction, top zone.............	832	836	860	871	874	893
middle zone..........	828	839	860	869	874	892
bottom zone..........	821	840	853	862	866	877
Temperatures, °F.:						
Hot catchpot -						
Vapors.....................	792	793	826	840	851	798
Liquid (average)..........	654	686	746	770	688	690
Cold catchpot, liquid..........	117	126	189	193	187	190
Products from hydro, g.p.d.						
Heavy oil let-down.............	11,900	12,100	18,000	19,300	12,000	11,100
Cold catchpot product - net....	9,220	12,800	12,960	15,840	16,810	16,900
Gasoline......................	275	550	805	1,540	2,300	2,200
Naphtha.......................	885	1,170	2,380	1,350	-	-
Middle oil....................	2,370	4,050	2,260	3,590	5,530	5,500
Flushing oil..................	-	-	-	-	510	-
Light oil bottoms..............	5,690	6,750	7,510	9,360	8,470	9,200
Total vapor-phase charging stock, bbl./ton of coal.................	2.7	2.6	2.9	3.0	3.3	3.3
Gasification (C_4 and lighter):						
Cu. ft./day....................	128,000	264,000	290,000	340,000	402,000	390,000
Weight percent on paste oil and/or coal consumed.........	1/ 8.4	2/ 20.3	23.9	25.2	26.7	26.4

1/ Paste oil consumed.
2/ Paste oil plus coal consumed.

TABLE 2. - Typical analytical data. Liquid-phase coal-hydrogenation runs

	Coal paste	Pasting oil, °F.	H.O.L.D., °F.	Gasoline, °F.	Naphtha, °F.	Middle oil, °F.	Light oil btms., °F.	Cold catchpot products
Distillation, °F.:								
I.B.P.		475	591	92	306	412	547	190
5 percent		573	620	116	340	454	607	396
10 percent		595	646	145	354	484	625	434
20 percent		627	660	180	362	512	646	535
50 percent		706	30% - 686	251	382	550	695	618
70 percent				296	394	572	730	674
90 percent				355	420	603	795	94% - 712
E.P. percent				398	460	638		
Recovery, percent				97.0	99.0	98.1		
Gravity	1.30	1.18	1.33	51.9	18.9	11.8	1.07	10.2
C_6H_6 + P.F.	55.6	25.3	46.5					
C_6H_6	49.9	16.7	34.5					
Tar acid				1.3	24.0	15.8		13.3
Tar base				2.1	2.2	2.0		0.6

Coal analysis:

Proximate

	Weight percent
Moisture	3.0
Vol. matter	43.8
Fixed carbon	48.0
Ash	5.2

Ultimate

	Weight percent
Ash	5.4
Sulfur	1.1
Hydrogen	5.3
Carbon	72.3
Nitrogen	1.5
Oxygen	14.1

Screen

	Weight percent
On 35	0.1
Through 35, on 60	1.2
Through 60, on 100	4.5
Through 100, on 200	22.3
Through 200, on 230	27.3
Through 230	44.6

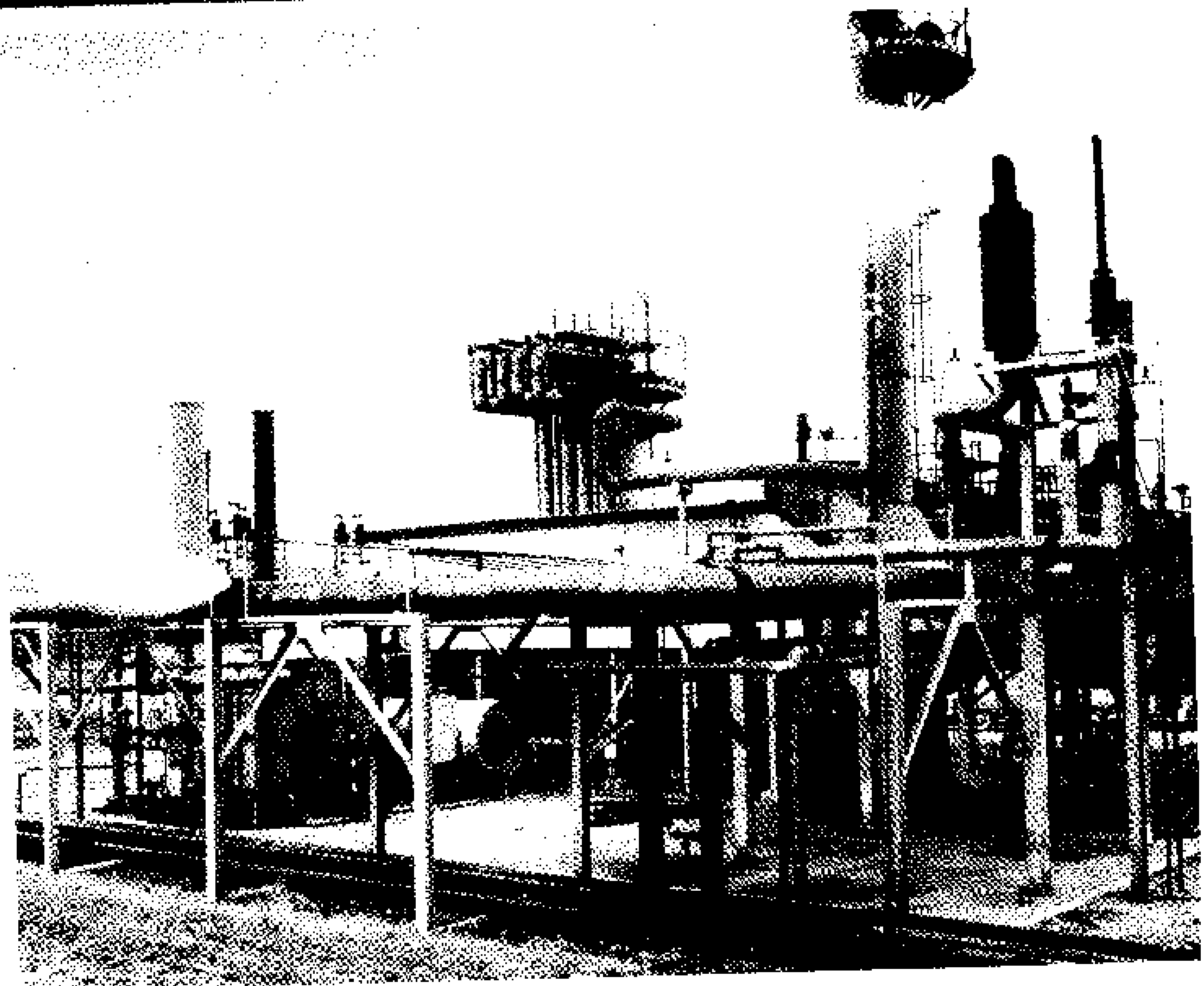

Figure 3. - Flash distillation unit for separating lower boiling oils from the heavy-oil let-down - Coal-Hydrogenation Demonstration Plant.

Run No. 3 (Rock Springs, Wyo., Coal)

The unit was shut down from April 16 to May 10 for cleaning, tightening, repair of control valves and thermocouples, and pressure testing.

To achieve smooth and steady operation before coal hydrogenation actually began, light-oil bottoms were charged to the unit and circulated for several days at 7,500 p.s.i. pressure. Coal paste was charged to the unit on May 16, and hydrogenation continued until an electric-power failure on May 21 forced a partial shutdown and change-over to pasting oil. Coal-paste injection was resumed but was again interrupted when a plugged paste mixer forced a change-over to pasting oil. Although paste injection was not stopped, operations were rather erratic, especially in the first converter. Owing to the uncertain condition of the unit, a normal shutdown occurred on May 23. Upon inspection it was found that the vessels were clean. The unstable condition now can be attributed largely to faulty temperature measurement.

Solids removal during the run was not satisfactory owing to the low capacity of the horizontal centrifuge and the inoperability of the vertical centrifuge in this service. The flash-distillation unit (see fig. 3) was put in operation for the first time. This method of solids removal from heavy-oil let-down showed promise, but many changes will be made before the adequacy of this method can be determined. The changes now being made include installation of a feed heater and condenser to obtain a better controlled operation.

Distillation and gas manufacturing were on a nearly routine basis. Much better fractionation was achieved on the liquid-phase still by introducing intermediate reflux in the fractionating column.

Except for the problem of solids removal, the over-all operation was excellent until the power failure and subsequent difficulties with temperature measurement in the converters. It is considered that the improved operation resulted primarily from higher and steadier oil and gas flows, improved coal-feeder operation and paste circulation, improved performance of the newly stellited control valve tips, and lower operating pressure.

This was the first run during which all equipment remained free of coke and during which instrument operation was relatively reliable.

Run No. 4 (Rock Springs Coal)

The unit was pressure-tested with inert gas to 10,000 p.s.i. During the test no leaks occurred on vessel heads, but it was necessary to retighten all flanges in hot lines, especially between the two converters. Hydrogen was introduced on June 3, and the system was pressured to 7,500 p.s.i., which was considered a safe maximum operating pressure for the injection pumps. On June 7 pasting oil was charged to the system, furnace temperatures were raised to about 650° F., and all instruments were put into service. On June 8 paste containing 15 percent coal was charged, and this concentration was increased to 30 percent coal within a few hours. Operations continued smooth and steady after that, and, except for constant maintenance on the injection pumps, the run approached routine basis. The failure on June 16 of the spare injection pump before the other could be repacked necessitated rapid change over to flushing oil and reduction of converter temperatures. This interruption was brief, and the unit was lined-out at normal flows and temperatures within 4 hours.

It was necessary to purge large amounts of gas from the system to maintain adequate hydrogen purity in the circulating gas. This was due to the inability of the wash-oil scrubber to remove the impurities because the pumps were unable to inject enough wash oil. Light naphtha was introduced into the cold catchpot to reduce foaming and to help purify the circulating gas. This, and a higher cold catchpot temperature (180° to 210° F.), seemed to eliminate foaming in the system.

Conditions for the run were similar to those in run 3. The flow of gas through the preheater was maintained at 300,000 c.f.h. and the paste injection rate at 23 g.p.m. Converter temperatures were kept at 870° to 875° F., with a preheater outlet of 850° F., and there has been no evidence of "coking" or settling of solids in the converters.

During the 20-day run, approximately 1,000 tons of Rock Springs coal was charged to the unit with a production of 130,000 gallons of oil, or a make of three barrels of oil per ton of coal.

It proved to be impossible to increase the coal in the paste above 30 percent and yet maintain a 44 to 46 percent solids limit on the paste because of the insufficient solids removal. Even though the continuous horizontal centrifuge produced a concentrate containing 60 percent solids, the filtrate still contained 15 to 18 percent. The vertical centrifuge proved unsuccessful during the last run and was replaced with a German-made vertical centrifuge especially designed for this service. The operation of the German machine was not entirely satisfactory, the longest run being a matter of hours rather than days, followed by 6 to 8-hour shut-downs for cleaning. It was found that the centrifuge was badly worn when received, and several parts had to be remade. Additional mechanical changes and experimental runs, together with the replacement of gears, shear pins, etc., will be necessary to make this unit operable and to clean up the pasting oil to a point where more coal can be used in the paste and an improved asphalt conversion can be achieved with higher reaction temperatures. It is expected that these changes also will improve the centrifugability of the oils.

The coal-preparation equipment operated satisfactorily.

The two high-pressure paste-injection pumps gave good service, but it was necessary to repack the rods continuously, 16 to 18 hours being the longest run on any one pump. This decreasing packing life probably can be attributed to wear of the rods and packing followers and to excessive ash in the paste. The duplex, reciprocating, steam-driven paste-circulation pump fitted with "Dercova" valves gave excellent service. The wash-oil pumps had to be operated at a reduced rate owing to the fatigue cracks in the blocks. The regular naphtha pump worked only a few days at 7,500 pounds pressure before failure of the block in the side weld, and naphtha injection was transferred to one of the water-injection pumps. Water was injected into the same line as the naphtha just before the cold catchpot. Flushing-oil injection was continuous and without difficulty.

Measurement of temperature in the converters was greatly improved after cleaning and drying the pyrometer tubes. This was the first run during which all temperature instruments remained entirely dependable.

By around-the-clock maintenance, the instruments were kept in good working order and performed very well. Several sticky potentiometer slide wires on controlling instruments caused minor upsets during the run, but nothing important.

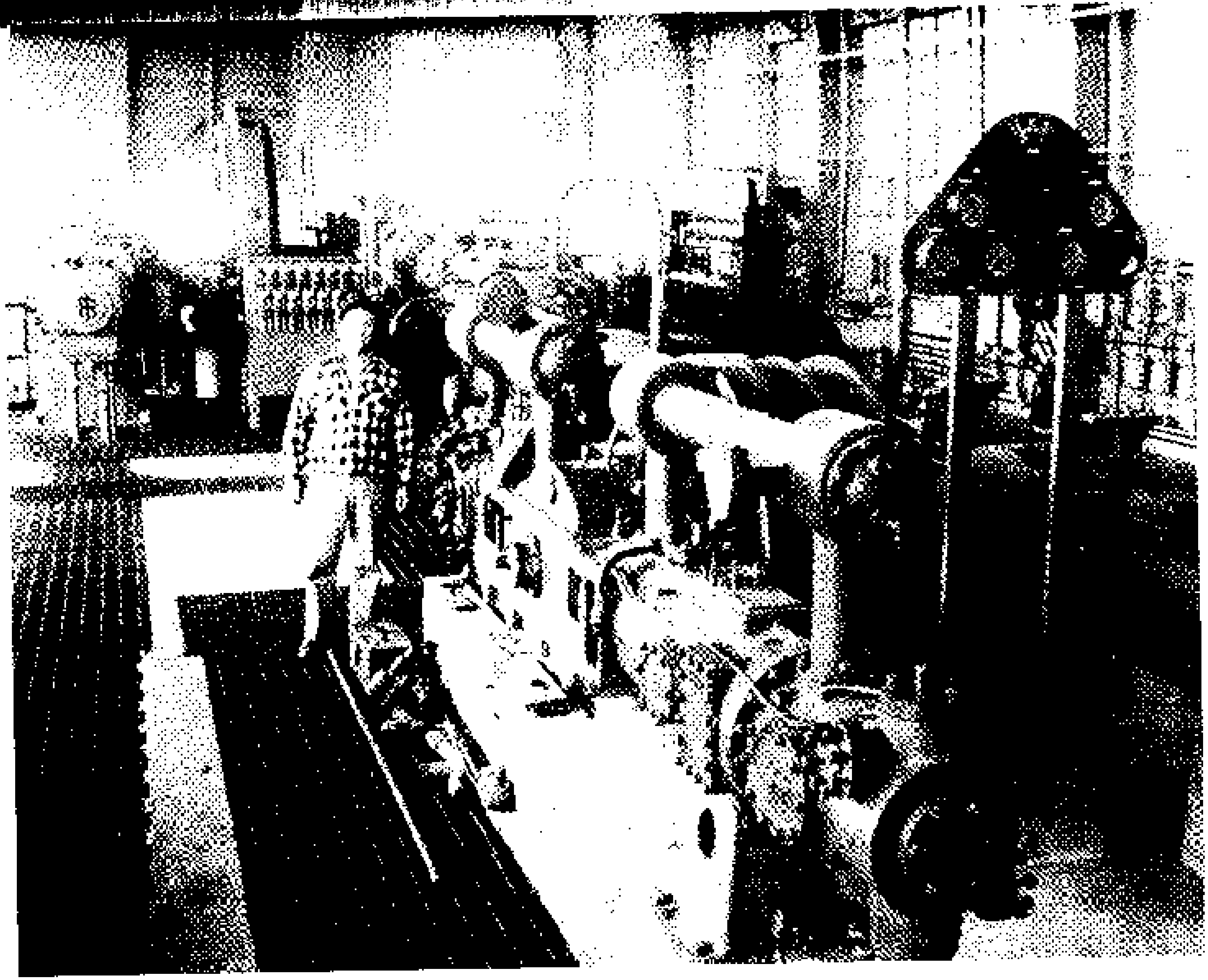

Figure 4. - Inert gas compressor. Make-up hydrogen compressor in background. Both discharge at pressures up to 15,000 p.s.i. - Coal-Hydrogenation Demonstration Plant.

Figure 5. - German high-pressure injection pump as installed in Coal-Hydrogenation Demonstration Plant. This pump is hydraulically operated. Fluid surge drum and steam turbine-driven circulating pump in left background.

A variable-speed motor was installed on the pulverized-coal Star feeder to the Waytrol. The Waytrol performed very well during this run, and this was attributed to the reduction of fines in the coal and a change in chute arrangement to handle and control flooding of the weighing mechanism.

All valves, including diaphragm-control valves, gave good service, and no replacements were required.

The liquid-phase still and absorber-stripper system were in continual service after June 7. This unit operated on a routine basis.

It was necessary to flare the 25-atm. off gases, as their heating value was too low for use with the furnace burner equipment. The 7-atm. off gases of high heating value were used as fuel.

Hydrogen was produced by No. 5 hydrogen cracking furnace at rates up to nearly 100 percent of design. This large production was necessary to maintain the hydrogen purity in the system by venting rather than washing the circulating hydrogen to maintain hydrogen purity above 78 percent.

No. 4 hyper compressor (see fig. 4) operated continuously at full capacity during most of the run, and no serious trouble was experienced with gas manufacturing.

Run No. 5 (Rock Springs Coal)

The unit was down from June 27 through August 20 for cleaning, mechanical repairs, and preparations to install six improved injection-pump blocks. Following usual inert-gas tests, hydrogen circulation was established for instrument testing and reactivation, and a number of trial pumping runs were made at various stall pressures, using the recently installed German hydraulic-driven paste pump (see fig. 5). These operations were finally terminated when the plunger metallizing started to disintegrate and crack off with possible damage to the packing. Arrangements have been made to obtain two new case-hardened plungers for this large pump.

Three of the original high-pressure injection pumps were equipped with improved fluid-end blocks with the valves in separate housings bolted to the inlet and outlet openings. They operated fairly successfully at low to moderate pumping speeds.

Normal hydrogenation was resumed on August 21 for a combination 8,000 and 10,000 p.s.i. run, which continued satisfactorily until September 10, when a leaky flange near the inlet to the first converter and subsequent fire necessitated emergency shutdown. With the supply of Rock Springs coal nearing depletion, a shutdown had been planned for September 12. During the 20-day run approximately 1,130 tons of coal was converted to 143,000 gallons of oil - a yield of approximately 3 barrels per ton. With the exception of a 4-day period, the operation was carried out at 8,000 p.s.i. and 880° to 890° F. converter temperature. Other conditions were as already outlined, except that, owing to carry-over, the wash-oil scrubber could not be operated effectively. From August 27 through September 2, the plant was operated at 10,000 p.s.i. with fair success. However, it again was considered advisable to reduce pressure to improve injection-pump operation and to conserve the rapidly depleting stock of let-down valves. A second reason for the lower pressure was to reduce the reaction rates and assist in maintaining smooth operations in the second converter, which had exhibited a decided tendency to lose reaction. Because of this experience, consideration is being given to decreasing the reaction volume by installing a liner in each converter.

During the last part of the run, some experimental work was done to develop a suitable process and equipment for using "red mud" Bayer mace catalyst. Several tons of the "red mud" was made into a slurry with about 20 percent water in portable ribbon mixer. Trial batches of the slurry were pumped into the ball-mill drier with the coal, and other batches were sprayed directly onto the coal pile. Both of these methods, although workable, were exceedingly laborious with the available equipment. Except during inclement weather, it seems quite feasible to add the catalyst to the coal, using a crane bucket, which also could be utilized to mix the semi-dry mud lumps into the coal. No experience with the actual catalyst can be reported, as about 150 tons of coal treated with 5 percent of red-mud slurry did not reach the plant for hydrogenation.

In addition to the production of oils required for the vapor-phase run, valuable experience and information were obtained during the run. It was found that:

1. The improved pump blocks with exterior ball valves, located midway along the plunger travel, will pump any of our oils or water satisfactorily at speeds up to 12 r.p.m. Some improvement in valve life and pumping capacity may be achieved with balls of better materials and reduction of the ball lift. It may also be advisable to spring-load suction valves.

2. The life of chevron packing used in injection pumps was improved markedly by the addition of adequate forced lubrication to the lantern ring and by use of exterior drip lubrication to the plungers. Further development is planned to improve the packing life by forced lubrication into the outer end of the stuffing box, so that all chevron rings and the plungers may be adequately sealed and lubricated by a positive oil film.

3. A number of the bronze-faced, steel support rings for the chevron packing developed center cracks during the 10,000 p.s.i. operation and will require redesign, use of heavier parts, and stronger material.

4. A valve utilizing a power piston with a built-in positioner for direct movement of the stem in either direction was tried in differential pressure-control service. Although the motor seems to operate smoothly, it has been found necessary to redesign the tip to allow control over a relatively long stem movement.

5. The original control valves used in high-pressure gas and clean-oil let-down service had stellite seats and disks. To eliminate excessive breakage of tips in both the cooling-gas and mild-service let-down valves, the tips were changed to stainless steel. However, wear characteristics for this soft material were unsatisfactory. During run No. 5, the stellite tips used on all mild control service showed uneven wear, which resulted in leakage and necessitated frequent replacement of valves on both cooling-gas and liquid-level control service. On the basis of this experience, it is planned to obtain samples of heat-treated, "Co-Mo-Van", chromovan-type material for trial runs on control valves in various services. As the best operating conditions are definitely established and smoothed out, it is hoped to utilize more restricted orifices down stream from the valves to take up most of the pressure drop and allow the valves to control the flow evenly with only a few hundred pounds pressure drop across the actual control valve.

6. The duplex general service pump, equipped with hardened rods, Madsen valves, and Darcova cup plungers, continued to give excellent service in paste circulation at 250° F. However, similar pumping arrangements in centrifuge feed service gave very

poor results, apparently because the present Darcova cups quickly disintegrate in the 350° F. oil. The original single-ring, expander-type pistons give poor service, both pistons and liners wearing out quickly. An effort will be made to find or develop Darcova cups of suitable material.

7. The horizontal continuous centrifuge continued to operate satisfactorily throughout the run at rates of 5 to 20 g.p.m. Quality of filtrate improves slightly at extremely low rates. However, in the range cited, the solids in the filtrate remained substantially uniform at 15 percent when feeding a material containing 17 to 18 percent solids at 350° F.

8. Several difficulties were experienced with the wash-oil scrubber system. As there was considerable carry-over of heavy material from the cold catchpot, need for further adjustment to the tangential flow mist-vapor separator used in the cold catchpot is indicated. It will be necessary to increase the vapor space below the packing rings, so that both top and bottom connections of the level controller can be made from this free space below the packing, as the packing imposes a variable resistance that interferes with level indication.

9. The plant was under pressure and operated satisfactorily for 34 days before a serious lens-ring leak developed in the hot stall. As a result of this experience and a rather similar incident during run No. 3, the general piping layout was redesigned to eliminate about 75 percent of the flanged joints; new pipe supports were added to permit greater movement of the piping under thermal stresses, and the method of tightening the flanged joints was revised.

After September 11 the liquid-phase unit was down for cleaning, inspection, and repair of fire damage to piping, electrical wiring, and structural steel. During this time, operators and maintenance personnel readied the vapor-phase unit for operation early in October, and all distillate oils were rerun through the liquid-phase still to vapor-phase feed specifications.

Vapor-Phase Run No. 2

Preparation consisted primarily of revision to the instrumentation similar to that that worked best in the liquid phase, extensive repairs to the double-tube feed-product heat exchanger, and installation of improved pump blocks for the vapor-phase feed service. After the usual inert gas-pressure test, hydrogen was introduced, and the unit was heated from October 1 through October 4 to reactivate the catalyst at 925° F. before starting up at a 2 g.p.m. feed rate at 815° F. converter temperature on October 5. The feed rate and converter temperature were slowly raised to 15 g.p.m. and 910° F. maximum during the next 7 days. During this time the feed, containing 8 to 9 percent tar acids, resulted in a product containing 1 to 2 percent, rather than 0.4 percent, tar acids, despite the increased feed rate and temperatures. The distilled gasoline contained 0.4 to 1 percent tar acids and was slightly sour to the usual doctor test. A sample of the hydrogenated material taken from the product line between the converter and exchanger on October 12 showed excellent catalyst activity, was light in color and sweet, indicating that 30 to 40 percent of the feed was bypassed in the exchanger direct to the cold catchpot product. Despite the reduced and somewhat unsteady flow through the preheater and converter, reaction conditions proceeded quite smoothly.

After the development of a small leak on the converter inlet piping, the unit was shut down on October 13 for inspection and repair of the heat exchanger. The 1-1/4-inch tubes were rerolled and withstood a 300-pound differential nitrogen cold test.