PART I

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

Coal-Hydrogenation Demonstration Plant

Two extended liquid-phase runs and one vapor-phase run were completed in 1951. A third liquid-phase operation is under way. On December 1 the combined operating time was 150 stream-days. Approximately 2,500 tons of Western Kentucky coal was converted into nearly 350,000 gallons of 77.5 motor method or 83.5 research octane gasoline, which was shipped out for military testing. An additional 2,500 tons of Illinois No. 6 coal (see fig. 2) was converted into vapor-phase charging stock, and the liquid-phase processing of 4,400 tons of subbituminous coal from Lake DeSmet, Wyo., was started in late October. Products from the latter two liquid-phase operations will next be converted to gasoline in a single vapor-phase run.

It should be noted that, throughout this section of the report, the data presented refer to coal actually processed. Coal was required for the production of steam, power, and filtered water but is not included. Furthermore, the hydrogen was made from natural gas.

Pertinent operating information is included, and processing data characterizing and finally comparing the hydrogenation of Rock Springs, Wyo., Western Kentucky, and Illinois No. 6 coals are summarized in tables 1 through 5, with appropriate discussions. A complete report is in preparation for each coal run and will be published in the form of a report of investigations. Significant and steady progress in the development of techniques, equipment, and technical data is producing the results required for the economic commercial production of fuels and chemicals from coal by the hydrogenation process, and these results are outlined in the following progress report.

Earlier operations included a short vapor-phase run made on North Dakota lignite-tar distillate oil in 1949, followed in 1950 by four comparatively short exploratory liquid-phase runs and one vapor-phase run, all on Rock Springs coal. These operations helped to train operators and work out the many changes required for improved plant performance.

TABLE 1. - Analyses of coals (moisture-free basis)

Proximate	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6
Volatile matter	45.1	39.4	42.9
Fixed carbon	49.5	53.4	48.8
Ash	5.4	7.2	8.3
Total	100.0	100.0	100.0
Ultimate			
Hydrogen	5.3	5.2	5.0
Carbon	72.3	74.2	72.3
Nitrogen	1.5	1.4	1.4
Oxygen	14.4	9.2	9.9
Sulfur	1.1	2.8	3.1
Ash	5.4	7.2	8.3
Total	100.0	100.0	100.0
Heating value	12,970 B.t.u.	13,390 B.t.u.	13,020 B.t.u.

Over-all material balance - Illinois No. 6 coal

Input:			
M.F. coal (includes catalyst)	114,250 lb.		
Pasting oil	168,210		
Make-up hydrogen gas	23,425		
Total input		305,885 lb.	100 percent
Recovery:			
Light oils	59,490		
Heavy oil (includes H.O.L.D.)	192,040		
Off gases	26,620		
Subtotal	278,150 lb.		
Water and soluble salts	15,765 lb.		
Total recovery		293,915 lb.	96.1 percent
Unaccounted for		11,970 lb.	3.9 percent

Hydro yields (recovery)

Hydrocarbon gases	3.84 M c.f./ton, m.a.f. coal
Light oils	3.52 bbl./ton, m.a.f. coal
Heavy oils	.39 bbl./ton, m.a.f. coal

TABLE 2. - Typical operating conditions and yields in liquid-phase hydrogenation

	Wyoming (Rock Springs)		Western Kentucky No. 11 bed		Illinois
	Period 1	Period 2	Period 2	Period 5	No. 6
Pressure .... p.s.i.g.	10,200	8,100	8,300	8,400	7,900
Temp. 1st converter .... °F.	870	866	865	864	866
2d converter .... do.	874	892	866	865	861
Paste injection g.p.t.1/	550	520	456	465	486
Coal, m.f. .... t.p.d.	54.7	53.4	54.9	65.4	57.1
H_2 gas to stalls .... M cu. ft./ton	194.5	182.0	171.5	138.0	138.1
Purge gas .... do.	28.2	30.7	10.9	5.9	5.6
Products from hydro:					
H.O.L.D. .... g.p.t.	223.6	214.3	175.5	166	214.5
C.C.P. product .... net g.p.t.	341.9	336.8	279.0	276	265.0
Gasoline .... g.p.t.	37.2	43.7	37.9	36.8	19.3
Naphtha .... do.	-	-	27.8	39.5	44.7
Middle oil .... do.	125.0	117.8	68.9	43.4	70.6
Flushing oil .... do.	12.4	-	-	-	4.0
L.O. bottoms .... do.	167.3	175.3	144.3	156.1	126.5
Total vapor-phase charging stock recovered .... bbl./ton m.a.f. coal	4.07	4.06	3.44	3.07	3.52
Net heavy oil produced .... bbl./ton	-0.20	-0.10	0.69	0.79	0.39
Total oil produced .... bbl./ton m.a.f. coal	3.87	3.96	4.13	3.86	3.91
Liquefaction .... percent of m.a.f. coal	96.1	96.0	96.2	96.7	94.2
Gasification (C_4 and lighter) .... M c.f./ton	7.18	6.98	4.01	3.90	3.5
Weight percent on m.a.f. coal	26.7	26.1	13.7	13.2	12.1
Hydrogen consumption:					
Total percent m.a.f. coal	15.1	14.5	10.8	10.0	9.7
Reaction percent m.a.f. coal	9.1	8.3	6.5	7.25	6.8

1/G.p.t. - gallon per ton of moisture-free coal.

TABLE 3. - Typical analytical data from liquid-phase hydrogenation

	PASTING OIL			COAL PASTE			H.O.L.D.		
	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6
Distillation.....°F.									
I.b.p.	475	475	477				591	360	485
5 percent	573	560	566				628	550	580
10 percent	595	604	596				646	606	609
20 percent	627	636	627				660	646	647
50 percent	706		674						702
70 percent			698						
90 percent									
E.P.		680					686	680	734
Recovery, percent.		49.0	70.0				80.0	81.0	60.0
Sp. gr. or °A.P.I...	1.18	1.117	1.107	1.30	1.20	1.222	1.33	1.25	1.238
C_6H_6 - P.E. insol...	25.3	8.4	7.03	55.6	41.3	46.5	46.5	25.2	24.4
C_6H_6 insol.	16.7	5.5	5.38	49.9	43.7	44.6	34.5	19.0	20.1
Tar acids									
Tar bases									
Aromatics									

	C.C.P. PRODUCT			GASOLINE			NAPHTHA		
	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6
Distillation.....°F.									
I.b.p.	190	190	184	96	79	83	306	210	219
5 percent	396	240	224	115	95	105	340	312	276
10 percent	434	297	288	134	118	118	354	360	349
20 percent	535	390	383	183	141	139	362	378	377
50 percent	618	593	576	262	214	186	382	402	404
70 percent	674	668	655	334	294	214	394	418	420
90 percent		731	740	400	399	315	420	446	455
E.P.	712	750		420	418	370	460	516	501
Recovery, percent.	84.0	93.5	90.0	92.6	89.9	93.3	99.0	99.0	98.3
Sp. gr. or °A.P.I...	10.2	11.2	11.5	50.7	58.5	63.3	18.9	17.1	17.9
C_6H_6 - P.E. insol...									
C_6H_6 insol.									
Tar acids	13.3		20.7	5.7	2.1	0.6	24.0	38.0	35.3
Tar bases	0.6		0.2	3.6	1.7	1.4	2.2	0.9	0.5
Aromatics					15.9	19.2		32.8	36.5

	MIDDLE OIL			LIGHT-OIL BOTTOMS		
	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	Illinois No. 6
Distillation.....°F.						
I.b.p.	412	242	390	547	592	590
5 percent	454	434	455	607	622	631
10 percent	484	464	483	625	638	639
20 percent	512	494	505	646	654	656
50 percent	550	534	535	695	686	696
70 percent	572	552	555	730	712	730
90 percent	603	578	586	795		
E.P.	638	610	625		760	756
Recovery, percent.	98.1	99.0	98.2	90.0	87.0	74.0
Sp. gr. or °A.P.I...	11.8	12.0	11.8	1.07	1.06	1.06
C_6H_6 - P.E. insol...						
C_6H_6 insol.						
Tar acids	15.8	22.0	23.6			
Tar bases	2.0	0.1	0.0			
Aromatics		45.4	54.7			

TABLE 1. - Typical operating conditions and yields in vapor-phase unit

	Wyoming (Rock Springs)	Western Kentucky No. 11 bed	
		Period 1	Period 2
Pressure .... p.s.i.g.	10,200	10,250	10,250
Converter temperatures:			
Average middle of catalyst beds ... °F.	876	887	898
Average bottom of catalyst beds ... do.	890	905	916
Catalyst	Zn-Cr-Mo	Zn-Cr-Mo	Zn-Cr-Mo
Feed:			
Injection .... gal./day	16,680	20,320	22,180
Virgin .... do.	11,860	10,370	14,550
Recycle .... do.	4,820	9,950	7,630
Gas flows:			
Make-up hydrogen gas .... M c.f./day	1,008	1,037	1,114
Total gas to stall .... do.	9,200	8,670	9,360
Purge gas .... do.	745	58	29
Products from hydrogenation:			
Catchpot liquid, net .... gal./day	17,110	21,530	22,710
Gasoline .... do.	11,930	11,380	15,080
Wash oil .... do.	360	-	-
Bottoms .... do.	4,820	10,150	7,630
Stabilized gasoline, bbl./bbl. virgin feed	1.01	1.09	1.04
Gasification (C_4 and lighter)			
M c.f./day	145.	90.	118.
Weight percent on feed consumed	14.3	9.6	8.9
Space time rate			
Feed .... lb./cu. ft. catalyst/hr.	51.37	65.4	69.5
Gasoline produced .... do.	29.07	29.9	39.8
Reaction hydrogen .... weight percent on virgin feed	3.5	5.4	4.2

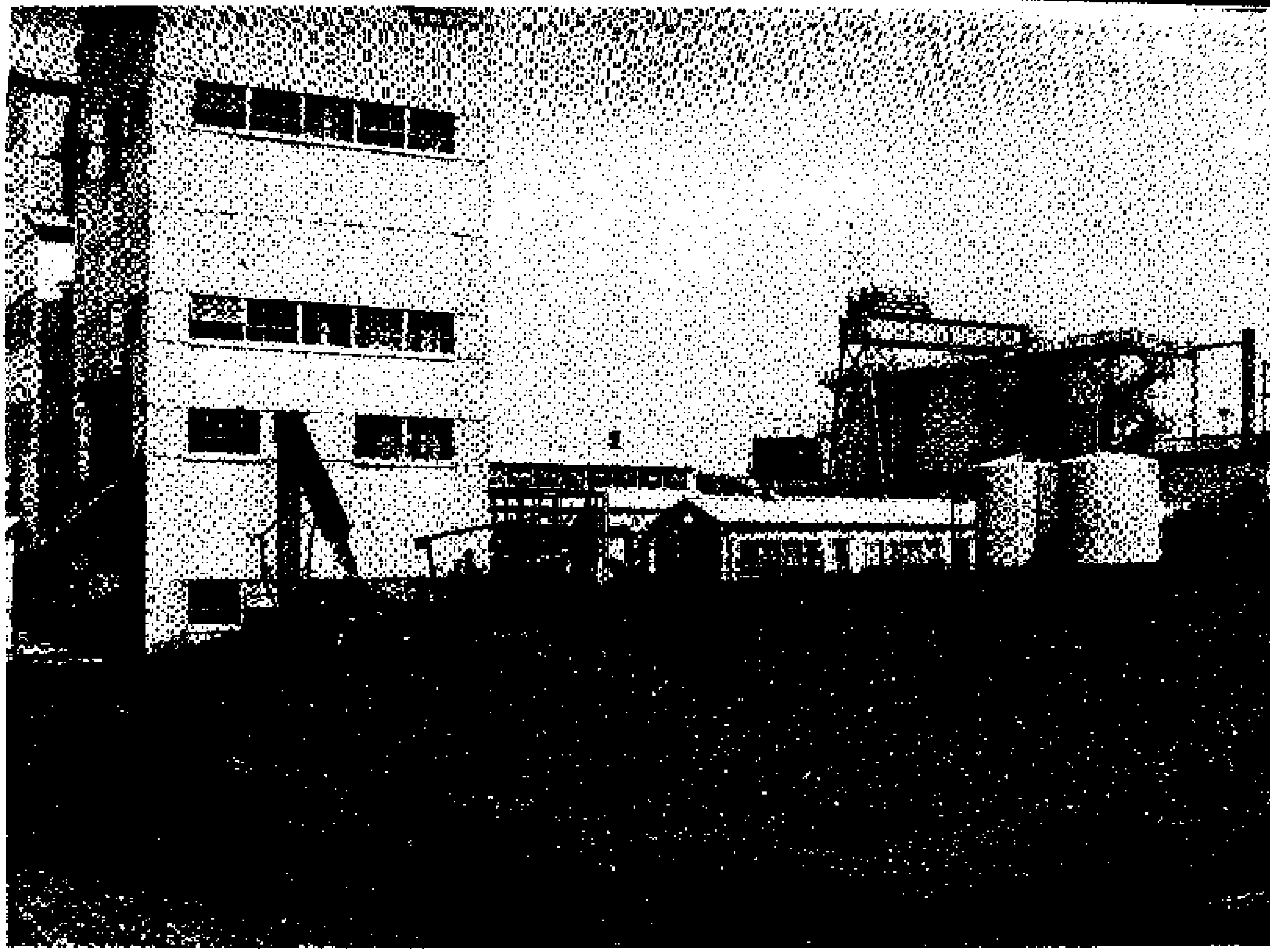
TABLE 5. - Typical analytical data from vapor-phase hydrogenation

	FEED: To hydrogenation			PRODUCTS: Cold catchpot			Raw gasoline			Wash-oil naphtha			Bottoms			S. S. gasoline		
	Wyoming coal (Rock Springs)	Western Kentucky No. 11 bed		Wyoming coal (Rock Springs)	Western Kentucky No. 11 bed		Wyoming coal (Rock Springs)	Western Kentucky No. 11 bed		Wyoming coal (Rock Springs)	Western Kentucky No. 11 bed		Wyoming coal (Rock Springs)	Western Kentucky No. 11 bed		Wyoming coal (Rock Springs)	Western Kentucky No. 11 bed	
		Period 1	Period 2		Period 1	Period 2		Period 1	Period 2		Period 1	Period 2		Period 1	Period 2		Period 1	Period 2
Distillation ... °F.																		
I.b.p.	144	196	149	131	147	135	94	87	88	184	Non Produced	Non Produced	400	369	368	75	87	79
5 percent	214	324	203	180	186	172	116	114	108	280			410	390	386	111	136	95
10 percent	262	376	270	199	216	195	140	134	133	311			422	401	399	157	211	133
20 percent	338	404	365	228	262	231	170	164	164	336			426	411	407	249	310	179
50 percent	436	458	443	317	377	342	228	228	222	371			444	439	432	360	378	382
90 percent	548	577	570	472	505	488	336	331	327	397			514	543	524		444	440
E.P.	614	625	615	562	588	568	376	364	366	425			604	610	591	464	462	472
Recovery, percent	96.7	97.8	96.9	98.2	98.0	98.5	98.0	94.4	97.0	99.1			99.0	99.0	99.3	80.5	92.5	93.0
Gravity, °A.P.I..	28.3	21.0	25.2	41.9	39.3	42.4	54.5	55.2	55.6	33.1			25.7	24.8	24.4	51.3	51.9	53.4
Tar acids	9.4	9.8	12.1	1.2	0.8	1.4	0.4	0.7	0.7	-			2.4	1.5	1.8	-	-	-
Tar bases	3.2	0.8	1.2				0.6	0.4	1.0				0.8	0.1	0.1			
Aromatics	46.8	52.2	45.6				25.2	27.1	25.6				61.4	58.5	60.6			

AVERAGE TESTS ON FINISHED GASOLINE PREPARED FOR FLEET ROAD TESTS

	Wyoming	Western Kentucky Period 1	Western Kentucky Period 2
Octane rating (A.S.T.M. D908-48 T.C.F.R. Research Method)	83	-	-
Octane rating (A.S.T.M. 357-48 C.F.R. Motor Method)	78	76.6	76.7
Vapor pressure .... Reid p.s.i.g.	9.0	10.0	9.0
Gravity .... °A.P.I. at 60° F.	54.8	53.3	54.8
Sulfur content .... percent	.3	-	-
Doctor test (FST 520.31)	Neg.	Neg.	Neg.
Corrosion test (A.S.T.M. D-130-30)	Neg.	Neg.	Neg.
Existent gum .... mg./100 ml.	1.0	1.2	4.0
Induction period .... min.	720	-	-
Tar acids	Nil	0.7	0.9

	Wyoming	Western Kentucky Period 1	Western Kentucky Period 2
Distillation, °F.			
I.b.p.	92	97	98
10 percent	136	130	124
50 percent	225	225	212
90 percent	318	328	328
95 percent	336	354	351
E.P.	366	381	372
Recovery, percent	99.0	97.6	98.0
Residue, percent	1.0	1.2	1.0



212686 O 52 3

Figure 2 - 2,500-ton pile of Illinois coal ready for processing.

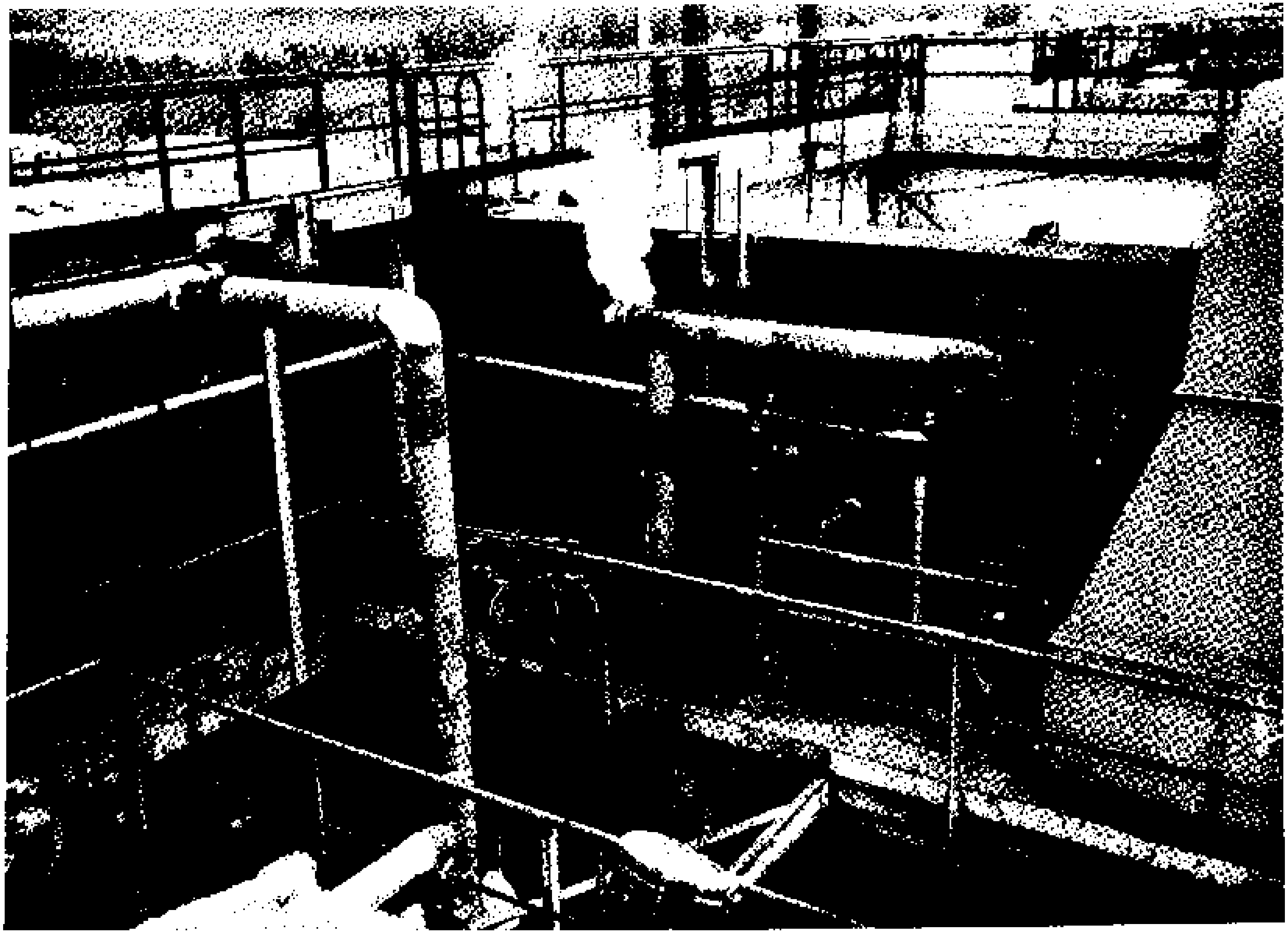


Figure 3. - Top of liquid-phase hot stall, showing new welded piping construction with clean-outs on lines to hot catchpot.

Figure 3. - Top of liquid-phase hot stall, showing ...

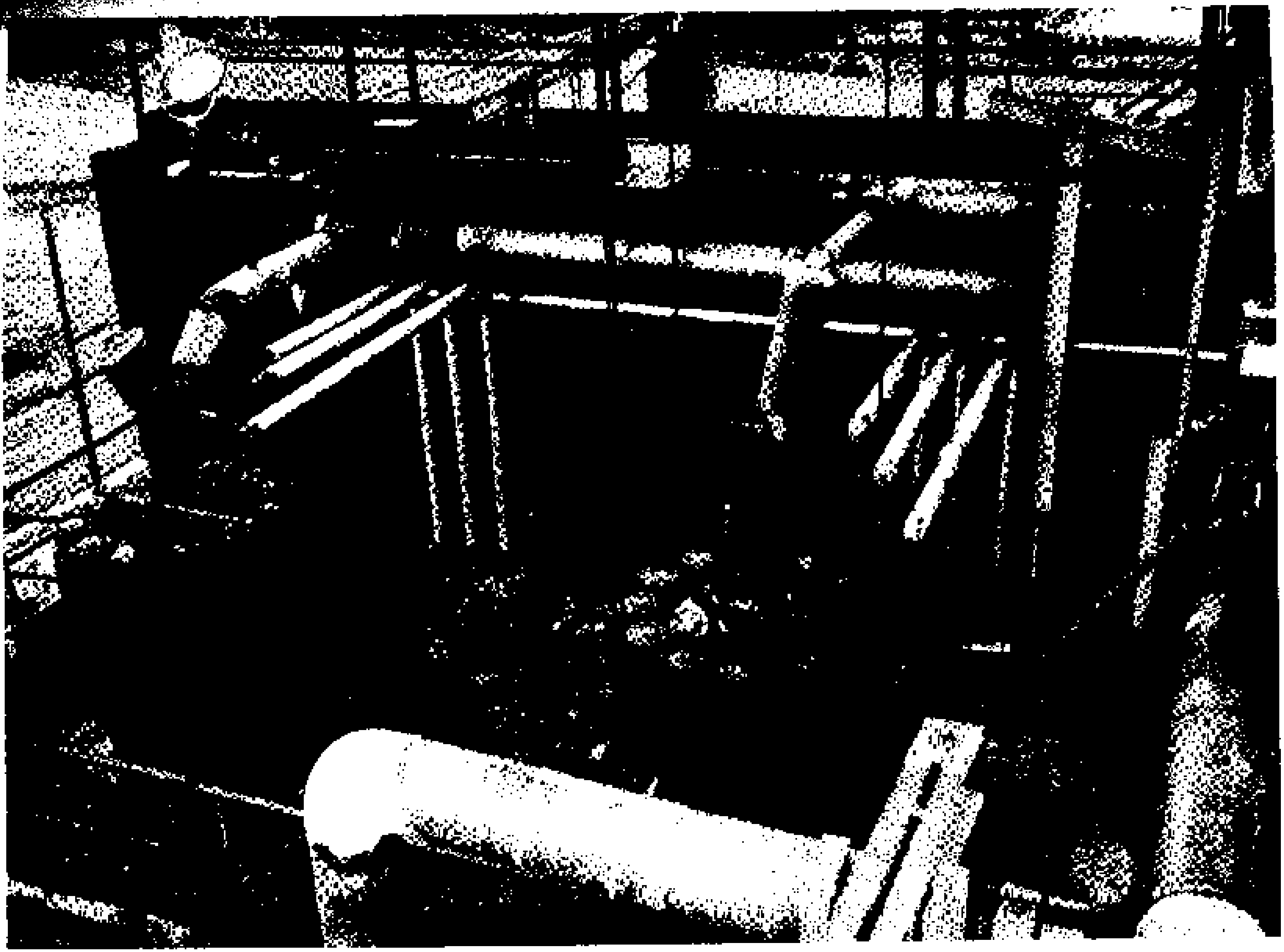


Figure 4. - Top of liquid-phase hot stall during turn-around, showing new welded lines from converters and flexible supports.

Operations

The exploratory runs on Rock Springs coal supplied valuable products and the information required to solve many difficulties which had interfered with safe, continuous operation. After major changes in equipment and techniques, it was possible to process 2,500 tons of coal in a single continuous liquid-phase run. Experience and observations determined that:

1. The chevron-type packing was satisfactory for use in high-pressure injection pumps, provided it is properly supported and is lubricated enough at the low-pressure end. Months of test experience with a dozen or more packing arrangements, using both metallic and nonmetallic materials, resulted in replacing expensive machined-metal packing with low-cost, easily replaceable, neoprene-impregnated duck. The packing was developed in cooperation with a leading manufacturer, who now supplies similar material to several commercial users.

2. All flanged joints in the hot piping systems, if not required for assembly and clean-out, must be eliminated, and the long piping runs must be properly anchored and flexibly supported to avoid high stresses and resultant leaks in the remaining joints. The original hot piping installation, using flanges and flanged fittings indiscriminately, was patterned generally after German practice, and within a few months contributed directly to three emergency shut-downs and one fire. Substitution of welded connections, wherever possible, made it relatively easy to secure and maintain a tight system (see figs. 3 and 4).

3. The improved, 3-percent-chrome-steel, injection pump blocks with exterior ball valves performed satisfactorily at moderate speeds. Although the workability of this unconventional design, adopted as a stopgap measure, was unknown, pumps did not vapor-lock or become clogged with solids.

4. The wash-oil hydrogen-purification system required revisions to prevent hold-up and carry-over in the high-pressure scrubbing tower, troubles that had interfered with compressor and instrument performance and prevented the efficient removal of hydrocarbons and nitrogen in maintaining hydrogen purity with minimum fresh hydrogen usage. Spasmodic operation of the scrubber over a period of several months proved that the equipment needed modification to provide better cooling of the incoming gas, better distribution of the scrubbing oil, adequate liquid-disengagement space above and below the packing, and maintenance of a constant liquid level by an instrument arranged with both the top and bottom take-off points from the free space below the packing.

5. It was found that the flash-distillation solids-removal unit must be separated from the high-pressure unit because the direct withdrawal of hot heavy-oil let-down (H.O.L.D.) seriously interfered with hot catchpot level control and did not provide a uniform flow at a satisfactory temperature for flash distillation. Provisions were made to allow feeding from a tank through a separate heater under close automatic control. The residue-removal system requires further improvement, because the original pumping installation is inoperable, and a stopgap method using an open trough is inefficient and somewhat hazardous.

6. Difficulties with instrument control, particularly of levels, were attributable to variations in system pressure. These variations occurred in attempting to maintain pressure by varying the make-up hydrogen input while maintaining a uniform rate of purge. It was proved that fixing the rate of hydrogen input and varying the purge for pressure control maintained uniform system pressure and gas quality required for smooth operation.

7. The continuous horizontal Bird centrifuge removed benzene-insoluble material from the H.O.L.D., but equipment of this type is not satisfactory for adjusting the asphalt level of the pasting oil.

8. The liquid-phase converter volume had to be reduced to achieve satisfactory control of reactions and to lessen the tendency for solids to deposit in the converters. The converters originally were lined to leave 130 cubic feet free space in each. However, the plant was not generally sized for converters of such generous proportions, and early results proved they must be reduced in volume to 90 cubic feet each by use of smaller-diameter liners.

9. Extensive use must be made of properly sized, strategically located, restricting orifices and target assemblies downstream of control valves, particularly when solids were handled, in order to achieve satisfactory valve life. This is true especially of let-down valves when operating at system pressures above 8,000 p.s.i.

10. It proved necessary to discontinue using rotary pumps in paste-circulating service because these pumps would not maintain enough head pressure and capacity for acceptable periods. Neither the original valveless screw pumps nor the replacement gear pumps proved satisfactory, and it was necessary to replace them with steam-driven duplex pumps equipped with hardened rods, Madsen valves, and Darcova cup plungers. The application of specially equipped reciprocating pumps to this difficult pumping duty is a marked advance over previously known methods of handling extremely viscous high-solid-content liquids.

Liquid-Phase Run 6 (Western Kentucky Coal)

During an extended winter shut-down, the liquid-phase system was completely cleaned and inspected, and mechanical work required in adopting the above changes was completed. After the usual pressure testing and activation of instruments and controls, coal was charged to the unit from March 30 to May 17. During the run, 2,160 tons of moisture- and ash-free (m.a.f.) coal was processed, and the inventory of light oils was increased by 291,500 gallons and the heavy oils by 43,500 gallons. This dried, washed coal, containing 7.2 percent ash, hydrogenated well to light oil (vapor-phase feed) at a conversion pressure of 7,700 p.s.i. and temperature of 875° F., using a 0.5-percent iron catalyst. Coal was hydrogenated at rates of 50 to 75 tons per day, with excellent conversion and control results.

All equipment in the stall and high-pressure area remained tight throughout the entire run. Despite high paste rates and considerable injection-pump valve trouble, which developed as the run progressed, flow, pressure, and temperature conditions remained relatively smooth and controllable throughout the run. The rather frequent difficulties with ball valves in the paste pumps were traced to partly plugged suction lines. Accordingly, these lines were redesigned to alleviate plugging and to facilitate cleaning before inadequate suction conditions developed (see fig. 5). It is hoped that spring loading of suction valves also will improve packing and plunger life, as well as the over-all injection-pump performance (fig. 6). While the service life of most high-pressure control valves was excellent, the performance of heavy-oil letdown valves continued somewhat erratic (3 to 7 days) despite the installation of 3/32-inch restricting orifices immediately downstream from these valves. Hot-catchpot level-control difficulties were accentuated by a tendency for reaction to start in the vessel, causing deposits of coke. To alleviate these conditions, more agitation gas is now being used and arrangements have been made for adding hydrogen or flushing oil as quench to the inlet-product tube. The wash-oil scrubber with the revised level-control arrangements worked very well. During the run, arrangements were completed for a desander line to facilitate removal of fine, sandy solids from the converters during operation and to assist in emptying these vessels on shut-downs.

The flash-distillation system was operated about half of the time, usually along with the Bird centrifuge, partly to reduce further the solids in the pasting oil but primarily to control the asphalt level of the oil. The fume problem and the manpower requirements of the process remained serious obstacles to continuous use of the unit. An experimental run, dropping part of the residue on a small, water-cooled metal conveyor, gave promising results. Arrangements for obtaining and installing this type of solids-removal equipment are under way.

Five periods were selected during the run for yield and process analyses. Period 1 lasted from April 7 to 14 and represented a low-throughput run with 0.1 percent stannous oxalate catalyst. Period 2 was from April 18 to 25 and represented a low throughput on mixed catalyst (0.05 percent stannous oxalate and 0.8 percent copperas). Period 3 covered April 29 to May 4 and represented gradually increasing throughput with 2.5-percent copperas catalyst addition and lower reaction temperatures. Periods 4 and 5 were from May 4 to 8, and from May 8 to 13, respectively, with a high throughput and 2.4-percent copperas catalyst addition. A division into periods 4 and 5 was made because preliminary calculations indicated an increased liquefaction beginning May 8.

Data from periods 2 and 5 are presented in summary form, in figure 7 and tables 1, 2, and 3. From the tabulated data it is possible to draw some comparison between the hydrogenating properties of the coals and the effect of mechanical and process improvements. It should be noted that this was the first run during which the solids-removal system functioned adequately at all times to maintain an average pasting oil of 5.5 percent benzene insolubles and 8.4 percent petroleum-ether insolubles, as opposed to the previous 16.7 percent benzene insolubles and 25.3 percent petroleum-ether insolubles. This improved pasting oil allowed the average percentage of coal in the paste to be raised from 35 to 43; converter-coal throughput increased from 18 to 30 pounds per cubic foot of reactor volume per hour. The higher-unit coal throughput and the improved converter temperature, flow, and pressure conditions resulted in a lowered asphalt production and a decrease in apparent gasification from 26.5 to 13.5 percent. As a result of this and the lower oxygen content of the coal (that is, 9.2 percent as opposed to 14.4 percent in Rock Springs coal), the reaction hydrogen was only 7.0 percent for Western

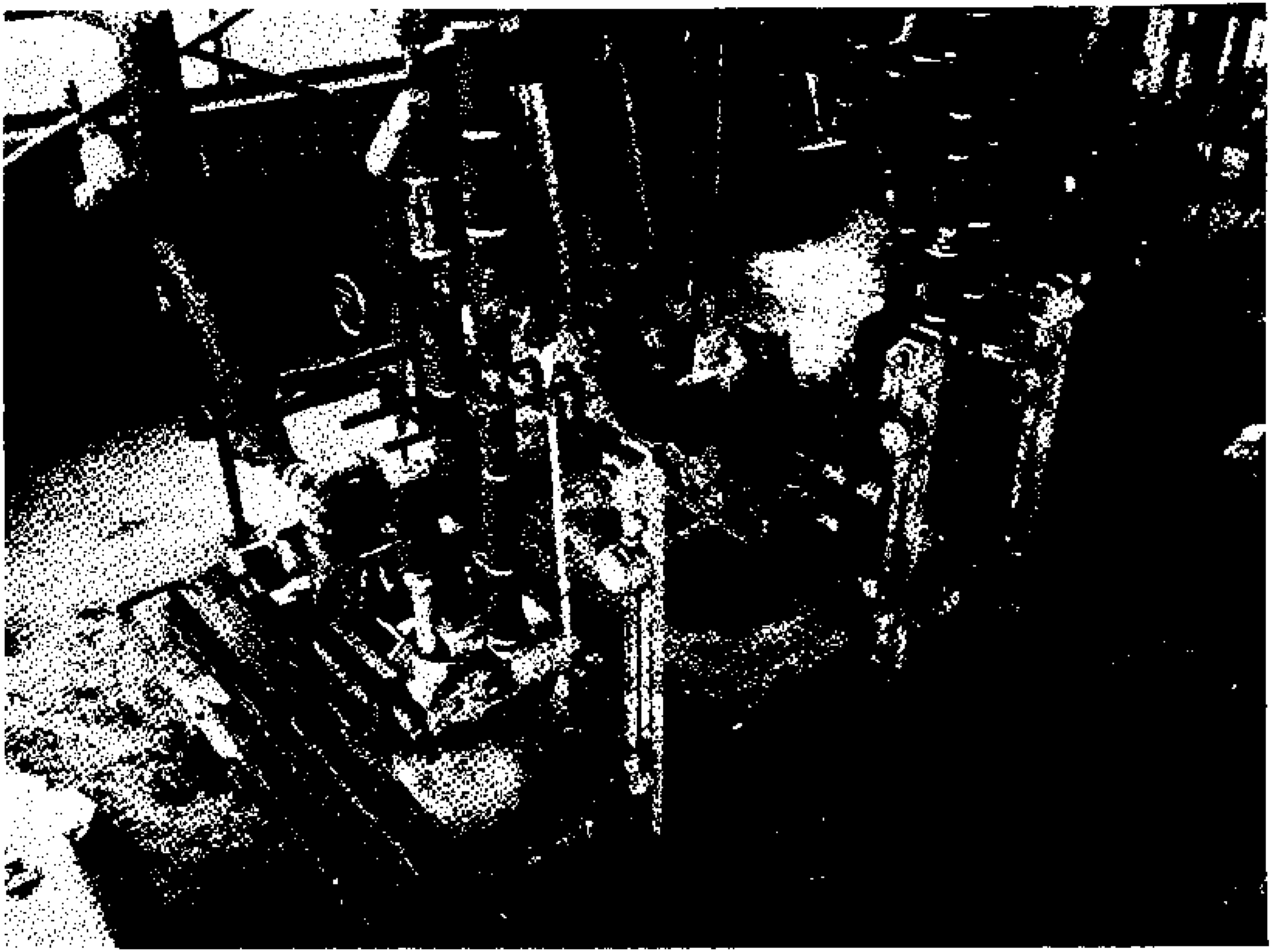


Figure 5. - New piping arrangement in paste-transfer pumps P-6A and B.

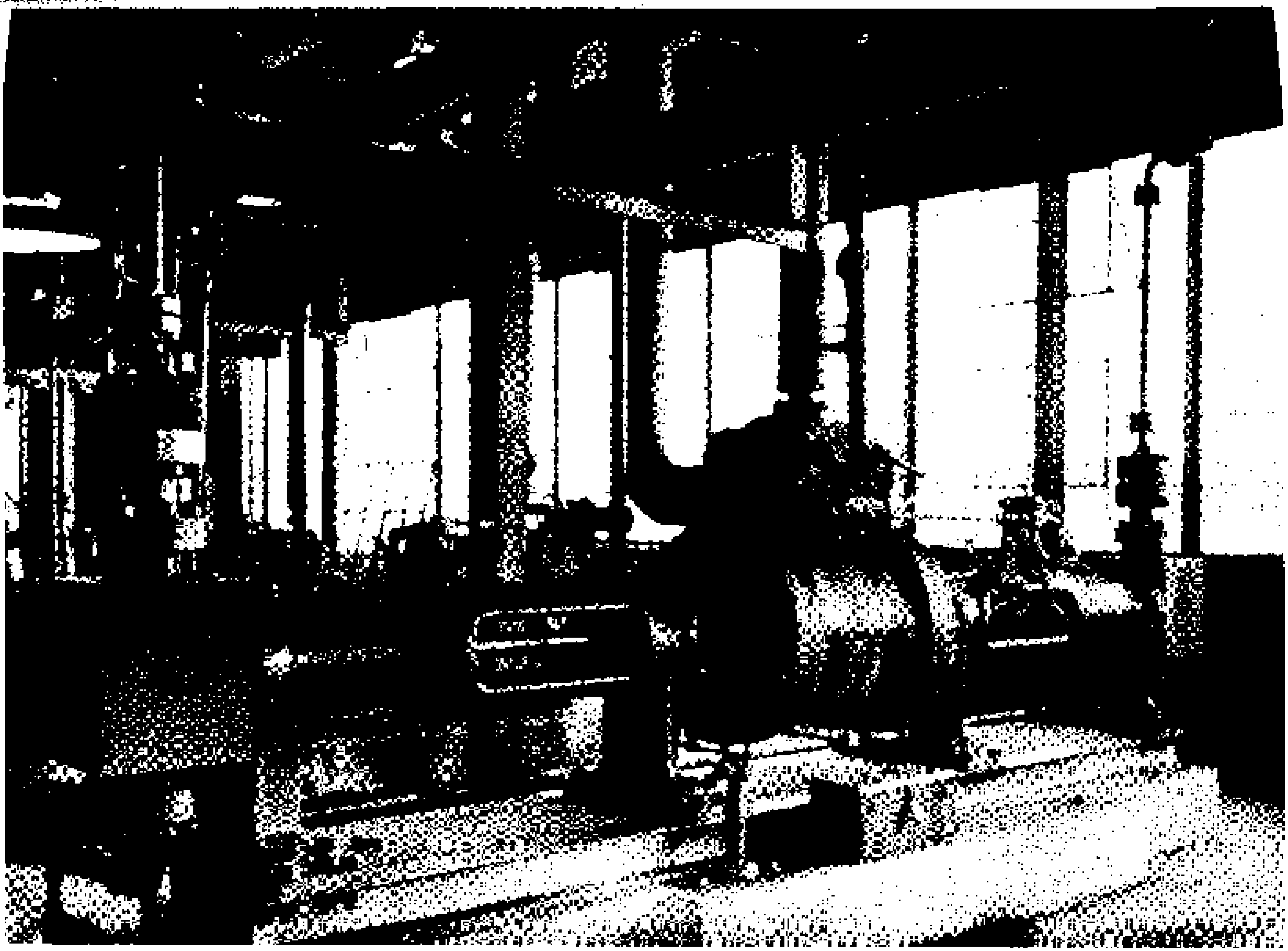


Figure 6. - High-pressure injection pump.

Kentucky coal compared to 8.7 percent for the Rock Springs coal. The greatly improved operation of the hydrogen wash-oil system resulted in a loss of hydrogen to purge of only 5.9 M cubic feet per ton of coal against 30.7, which was necessary on previous runs when the purity was maintained mainly by purge. The over-all yields, particularly of tar acids, were appreciably higher for the Western Kentucky coal.

Vapor-Phase Run 3 (Western Kentucky Coal)

During the extended winter shut-down, the vapor-phase system also was completely cleaned and inspected. The preheater was changed to series flow, welded construction was adopted in the hot stall, changes were made to obtain improved temperature control of the converter feed, and arrangements were made to secure and maintain more accurately blended feed-charge stock.

After the unit was pressure-tested, hydrogen was introduced to the vapor-phase system for a 3-day catalyst-activation period before liquid-phase feed was started for a 30-day run. The fresh vapor-phase charge from Western Kentucky coal was fed to the unit at an average rate of 12,740 gallons per day, and the total charge was converted to an average of 12,713 gallons per day of gasoline. Approximately 60,000 gallons of distillate produced from slop and creosote oil later was fed to the unit at a slightly reduced rate for comparable yields of gasoline.

The operations were performed smoothly at 10,000 pounds per square inch (p.s.i.), with the inlet to the converter at 845° F. and with average bed temperature of 900° F. The over-all mechanical performance of the high-pressure plant equipment was adequate throughout the run. Likewise, all equipment in the distillation and gas-manufacturing plants remained satisfactory at all times.

At the end of the run the following finished products from the refinery (see fig. 8) were available for plant consumption and testing: 225,000 gallons of regular gasoline, 7,000 gallons of aviation-base stock, 400 gallons of jet fuel, all from Western Kentucky coal, and 60,000 gallons of regular gasoline produced from creosote-slop distillate. Samples of the first three items and gasoline from Rock Springs coal were subjected to complete tests in a commercial laboratory, preliminary to arranging for military performance tests under more severe operating conditions than is normally required of post transport equipment. A summary of these tests is presented in a later section of this report.

A number of interesting process and mechanical improvements contributed materially in making this run more successful than previous operations. The smooth, dependable operation of injection pumps and recycle compressors allowed smooth flows to the unit at all times. Inasmuch as the preheater was repiped to operate all tubes in series and the exchanger was provided with better insulation, it was possible to hold the outlet temperature from the heater within a relatively narrow operating range. The new, close-range (800° to 900° F.) recording-temperature controller regulating cooling gas to the preheater outlet line kept the converter-entrance temperature within the ± 2° F. required for smooth control of bed temperatures.

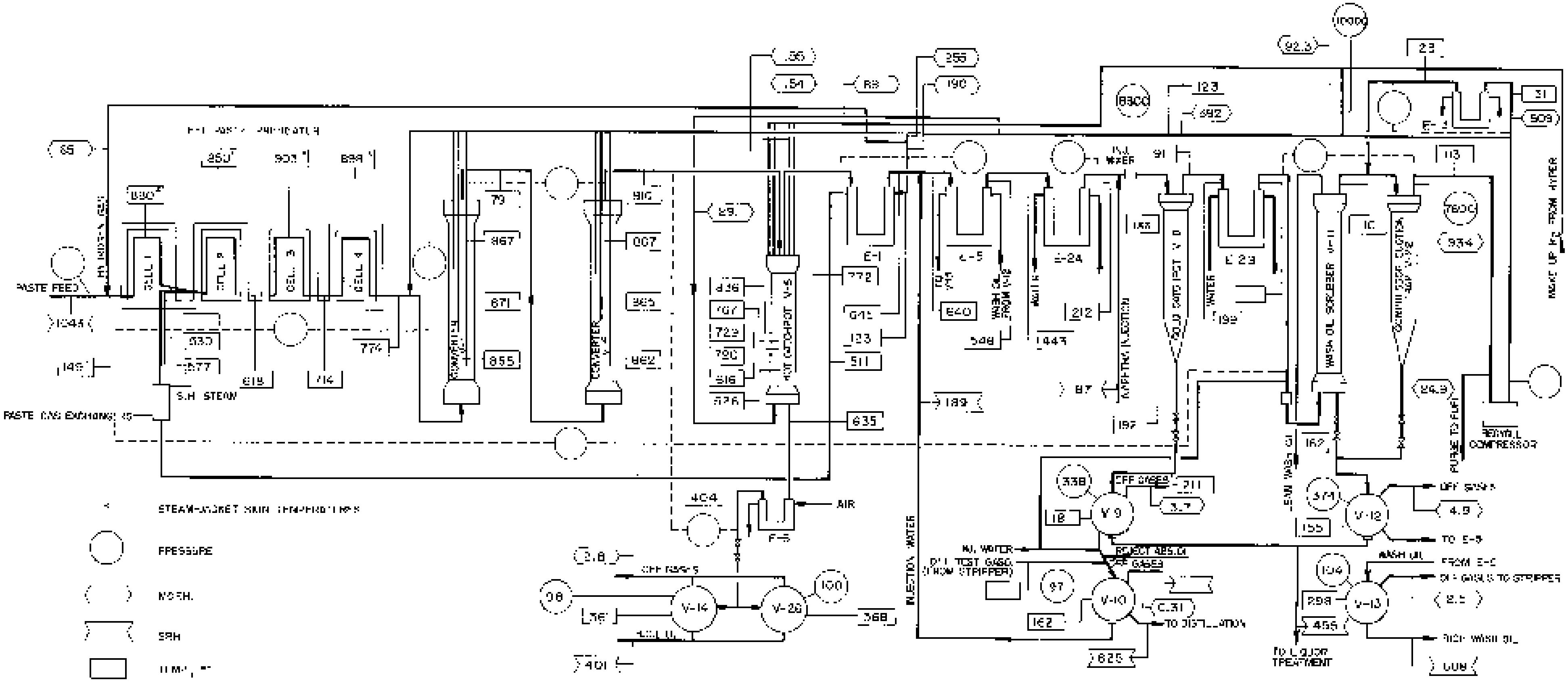


Figure 7. - Liquid-phase hydrogenation unit, typical operating conditions for series 2, Western Kentucky coal hydrogenation.

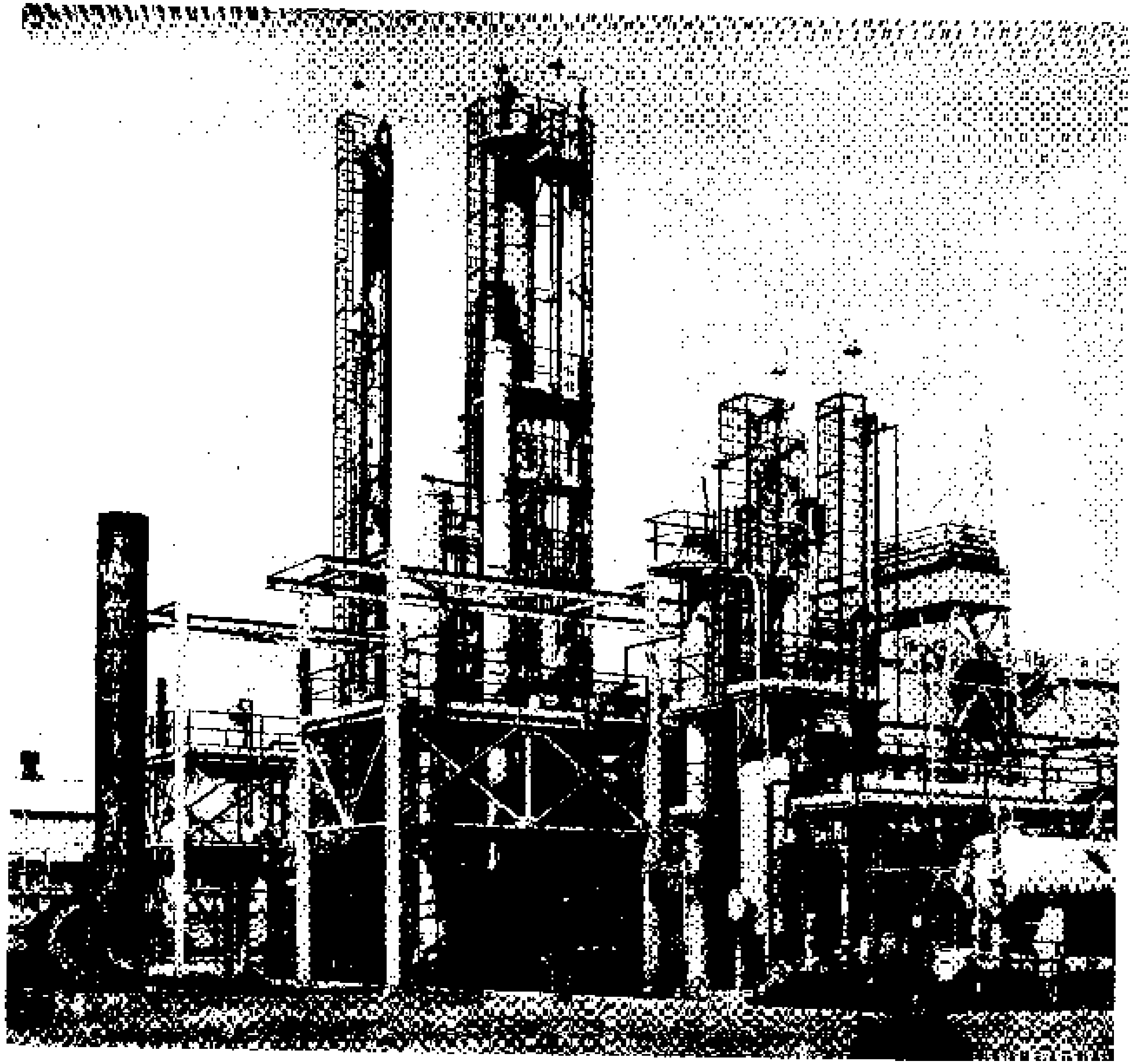


Figure 8. - Refinery undergoing exchanger and insulation repairs.

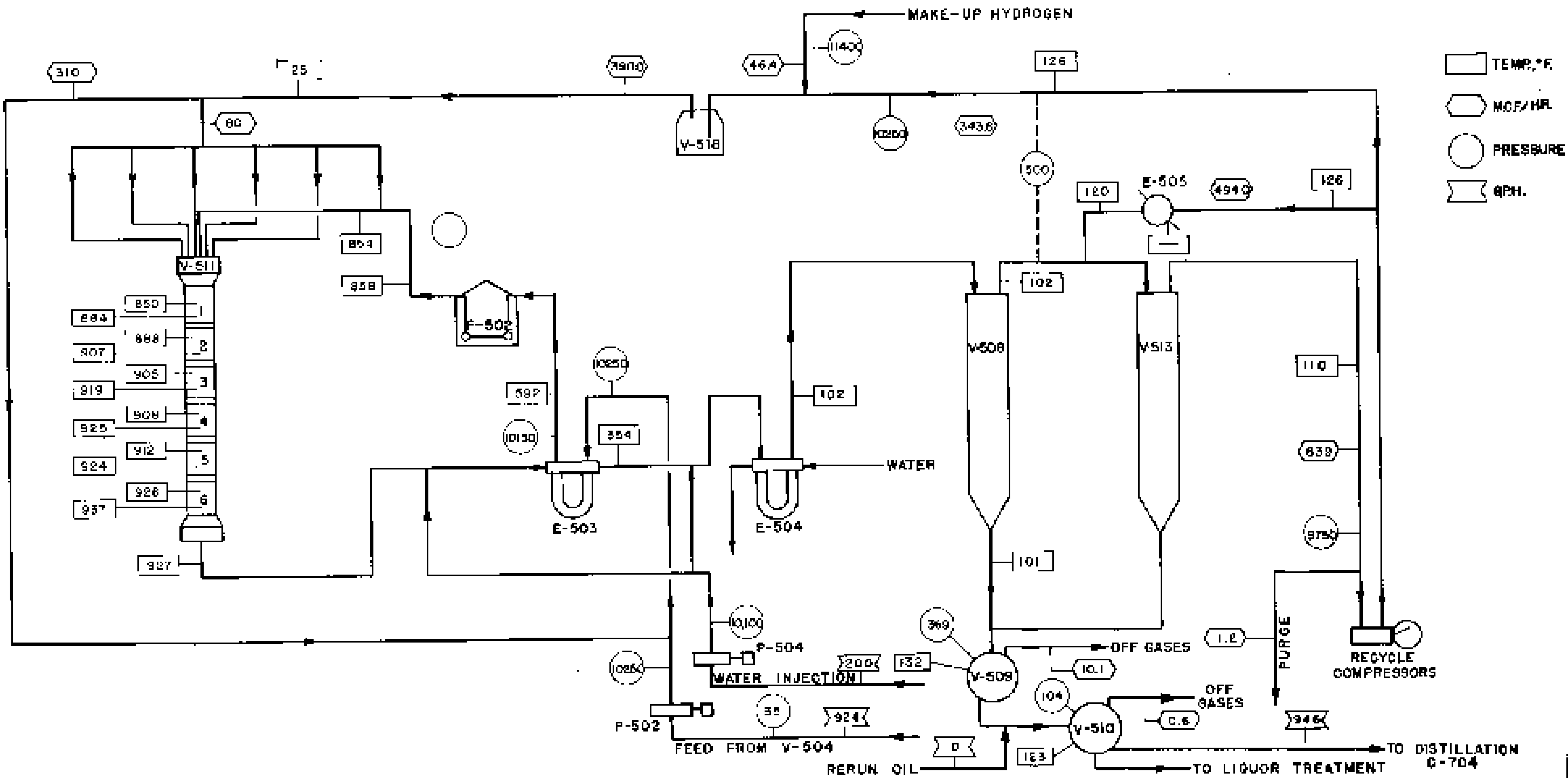


Figure 9. - Vapor-phase hydrogenation unit; typical operating conditions for period 2, Western Kentucky coal hydrogenation.

No serious difficulties were experienced with inaccurate pyrometer temperature readings and very few control-valve tips became worn or broken during the run to interfere with smooth let-down or other flow conditions. As many of the flanged joints were replaced by welds and the piping was properly supported and anchored, there was no leakage, even during periods when the operating temperatures changed rather abruptly. All of the cooling gas lines were recently steam-traced, and no difficulties were encountered in maintaining a free flow of cooling gas to the catalyst-bed levels.

Improved operations in the distillation area resulted in a more perfectly blended feed stock.

Despite improved converter inlet-temperature conditions, the feed-product heat exchanger is being changed to minimize uneven flow of liquid and gas, subsequent overloading of the fired heater, and uneven temperatures to the converter.

Data from these operations are presented in summary form in figure 9 and in tables 4 and 5. It will be noted that the Western Kentucky vapor-phase charge was lower in American Petroleum Institute (A.P.I.) gravity and higher in tar-acid content than the Rock Springs charging stock. However, due largely to improved converter control, it was possible to process the Western Kentucky material at a 20 percent higher rate and yet produce gasoline equivalent to the Rock Springs gasoline as regards octane and aromatics content. The gasification was somewhat higher for the Rock Springs feed, and the hydrogen usage was higher for the lower-gravity, higher-tar-acid content Western Kentucky stock. The lower gasification of the Western Kentucky stock, 9 percent versus 14 percent for Rock Springs, was reflected in a 5 percent higher gasoline yield.

Liquid-Phase Run 7 (Illinois No. 6 Coal)

During the vapor-phase run, the liquid-phase system was prepared for run 7. In addition to more or less routine cleaning, inspection, and maintenance, a new top pyrometer tube was installed in the hot catchpot, and provisions were made for tripling the hot catchpot agitation gas.

After pressure testing and activation of instruments, 15 percent coal paste was charged to the unit on August 12, 1951. The coal concentration was gradually increased until, within 2 days, the fresh-coal content of the paste was 40 percent. The run progressed smoothly at a rate of about 52 tons per day of moisture-free coal, until 6:45 p.m. on August 15, when all pyrometer indications in the second converter suddenly read off scale. Although there was no appreciable change in hydrogen purity or consumption, hot or cold catchpot products, or in other temperatures in the system, to be on the safe side it was assumed that there was a hot spot in the second converter. The feed was changed to 50-50 paste and pasting oil, and the converter was cooled with a full flow of cooling gas for several hours. When all converter temperature indications again became normal, full paste operation was resumed at moderate temperatures.

After closely observing the unit on August 16, it was concluded that any hot spot in the second converter had been of minor nature. Further investigation proved that the thermocouple wiring was charred and shorted above the

36-foot level. A new thermocouple assembly was installed the next morning, and normal operation at 870° to 880° F. was resumed on August 17.

During the following days, the coal rate was gradually raised to 65 tons per day under conditions simulating the 30,000-barrel-per-day commercial-plant estimate. Operations were smooth until 6 p.m. August 19, when a second minor hot spot developed in the second converter, and a third occurred the following evening. These disturbances apparently did not seriously interfere with satisfactory throughput for the 65 ton-per-day operation from August 18 to 21. On the morning of August 22, while attempting to raise the second converter temperature from 860° to 870° F. to decrease the hot catchpot product and increase the vapor-phase feed make, temperatures up to 1,200° F. were attained quite suddenly elsewhere. In view of the doubtful condition of temperature-measuring equipment, cooling gas tubes, and solids-formation conditions in the second converter, on August 22 it was decided to circulate the unit down for inspection, cleaning, and repairs to the converter as required.

Upon inspection it was found that the second converter contained a solids formation extending from a point 10 feet above the bottom to within a few feet of the top. The first converter and the hot catchpot were checked by a displacement method and found to be free of solids formation.

The following measures were taken to correct the hot-spot conditions:

(1) A new control valve was installed to provide automatic control of the bottom temperatures of the second converter.

(2) All cooling gas lines were steam-traced and insulated to prevent plugging.

(3) The catalyst addition, calculated as Fe, was to be increased from 0.5 to 1.0 percent.

(4) Paste-gas flows were to be increased by 10 percent.

(5) Second converter temperatures would be held a few degrees higher than the first converter temperatures.

(6) A new arrangement was made to install the pyrometer-tube thermocouple wiring to give less chance for thermocouple failures.

Paste injection for run 7B was begun September 2. Operation proceeded smoothly as temperatures, flows, and paste concentrations were raised to obtain a rate of 65 tons of moisture-free coal per day. On September 9 the unit was switched to pasting oil for 5 hours, owing to stoppage of the coal flow by a piece of metal jamming the Star feeder on the Waytrol unit.

High throughput production was started when enough data were obtained for the operation as outlined. The coal throughput was gradually raised to reach a 77- to 80-ton-per-day rate. To obtain the desired light-oil yield, the converter temperatures were slowly increased to a maximum of 885° F. After these conditions were attained and held for 5 days, trouble developed September 20 because of a local hot spot in the bottom of the second converter. Temperatures recorded by the two lower temperature controllers quickly went off scale,

Figure 10. - Illinois No. 6 coal entering plant and gasoline awaiting shipment to Army.

but a controller for the upper section of the converter recorded a top of only 950° F. The outlet temperatures dropped below normal as soon as cooling was begun. Conditions did not indicate a runaway reaction, but rather a local hot spot that could spread to a serious runaway if not quickly controlled. The unit was switched to pasting oil, and the converter temperatures were reduced to less than 800° F. The second converter top and midpoint temperatures dropped rapidly, but the bottom temperature was much slower to respond, indicating some deposition of solids.

It is of interest that some European experience indicates that the throughput can be increased above a given rate, but the maximum temperature that can be held in the converters reaches an upper limit, dependent upon the stall pressure. Above this temperature, local hot spots and a tendency toward ragged operation, with possibilities for runaway reactions, will develop. The converter temperatures were below the maximum point indicated by experience on German coals.

After the converter temperatures dropped below 800° F., injection of coal paste was resumed, using a 50-50 mixture of pasting oil and paste. The unit was returned to normal operation with 46-47 percent coal paste, and it was decided not to continue the high throughput operation, as some deposition of solids was suspected in the second converter. Therefore, the unit was operated at a 60- to 65-ton-per-day rate for several days, until it was deemed advisable to adjust conditions to obtain data for producing a 50-50 yield of heavy oil to light oil. On October 1, with the supply of coal exhausted, the unit was circulated down for a normal shut-down. Cleaning of the last row of preheater tubes and removal of some relatively soft solids from the second converter, together with inspection and mechanical work, were accomplished by October 29, before starting run 8 to process 4,400 tons of subbituminous coal from Lake DeSmet, Wyo.

Data from the processing of Illinois No. 6 coal (see fig. 10) are presented in summary form in tables 1, 2, and 3.

The comments after liquid-phase run 6, comparing the results from processing Rock Springs, Wyo., and Western Kentucky coals, apply in general equally well to the processing of Illinois No. 6 coal. Due in part to the relatively low oxygen-content of Illinois No. 6 coal, the gasification and process hydrogen usage were about 10 percent lower than for Western Kentucky coal. The oil and tar-acid yields were essentially the same and equally good for Illinois and Western Kentucky coals.

Mechanical Features and Plant Improvements

Preparation of Coal and Paste

By performing the coal-preparation unit operations on a 2-shift-per-day basis, it was possible to dry and pulverize 60 to 75 tons per day of moisture-free coal. During the later runs ferrous sulfate catalyst was used in place of tin oxalate. In autoclave bench-scale investigations, it had been found that excellent results could be obtained from this catalyst by spraying a water solution on the raw coal before drying and pulverizing. This method was tried but was discontinued, because the coal, already saturated with water, did not readily absorb the solution. The addition of wet catalyst was discontinued in favor of adding dry iron sulfate to the coal in the primary crusher, a method