

Before operations were resumed, it was necessary to dismantle all of the hot piping in the stall for refacing of tube ends and installation of new lens gaskets and new bolts.

Upon completion of this work the unit was pressure-tested, the fires lighted, and production of 77 to 78 octane-number (motor method), 8 to 10-pound vapor-pressure gasoline was resumed on October 24. The product-feed heat exchanger continued to give considerable trouble with uneven outlet temperatures on the feed side. This exchanger is composed of a number of double-pipe hairpin units hung in a vertical position. The product entering the hot side is partly condensed, and the resulting liquid tends to collect in the annular space between the pipes until the column of liquid is high enough to be forced out ahead of the vapor stream. When the liquid column is forming, the heat-transfer coefficient falls off, resulting in a drop in temperature of the feed leaving the exchanger. These changes in temperature were so large that they were not compensated by the preheater, which caused a 10° to 20° F. variation in the temperature of the feed entering the converter, thereby reducing somewhat the efficiency of the reaction. There also was a tendency for the preheater tubes to run hot (1,050°-1,075° F.) owing to the erratic and low efficiency of the exchanger and to the parallel flow arrangement of the preheater. Consideration is being given to designing a new tubular exchanger and to series flow in the preheater. The injection pumps equipped with improved blocks and better lubrication gave good service, and packing life increased to 15 days. Approximately 300,000 gallons of vapor-phase feed stock was converted to finished gasoline during the successful 30-day run. The principal flows, temperatures, and yields are shown in table 3. Figure 6 is a flow diagram of operating conditions, and table 4 contains analytical data illustrative of the quality of the streams.

Mechanical Features and Plant Improvements

Preparation of Coal and Paste

Coal grinding and drying usually is done in a mill swept with hot air or flue gas. However, it was found that a minimum of oxidation and low-drying temperatures as well as a minimum of either very coarse or very fine particles was desirable for the hydrogenation process. Accordingly, the design was changed to include recirculation of the gases and drying in a gas of less than 8 percent oxygen content at 300° to 400° F. Experience in grinding Rock Springs coal also disclosed the need for much closer control of temperatures, pressure drops, and flows than in the usual installation.

To reduce the carry-over of coarse particles in the finely ground coal, it was necessary to use a fixed, inverted V-type diffuser in a large double-cone classifier. Additional studies are under way to improve the quality of the ground coal and increase the capacity to design rates.

The use of a Waytrol to feed the coal has presented many problems. First, the coal entering the Waytrol from the storage bin is hot and contains fines (through 200-mesh) in the range of 50 to 70 percent; various devices have had to be used to prevent flooding of this coal onto the Waytrol belt. At present, a system of baffles in the chute from the Star feeder on the pulverized-coal bin, together with a canvas skirt, has proved the most successful. Recently, a variable-speed motor has been used on the Star feeder in conjunction with the Waytrol to prevent flooding.

TABLE 4. - Analysis of feed and product streams during
initial operation of vapor-phase run No. 2

	Feed	Products				
	To hydro	Raw gaso- line	Wash oil naphtha	Bottoms	Cold catchpot	Stripping still gasoline
Distillation, °F.:						
I.R.P.	149	86	184	406	142	75
5 percent.....	217	114	200	426	191	111
10 percent.....	275	135	311	433	215	157
20 percent.....	364	168	336	440	249	249
50 percent.....	459	228	371	468	358	360
90 percent.....	553	336	397	532	492	-
F. P.	609	378	425	608	576	461
Recovery, percent.....	98.7	94.2	99.1	99.1	98.5	80.5
Gravity.....	25.5	55.8	33.1	24.6	37.6	51.3
Tar acid.....	7.2	0	-	0.5	6.2	-
Color.....		+30				

Average tests on gasoline prepared for fleet road tests

Octane rating (ASTM D908-48 T C.F.R. Research Method).....	83
Octane rating (ASTM 357-48 C.F.R. Motor Method).....	78
Vapor pressure, p.s.i.	9.0
A.P.I. gravity at 60° F.	54.8
Sulfur content, percent.....	0.03
Doctor test (3SE20.31).....	Negative
Corrosion test (ASTM D130-30).....	Negative
Tar acids.....	Nil
Existent gum, mg./100 ml.	1.0
Induction period, min.	720 + min.

Distillation

	°F.
I.R.P., percent.....	92
10.....	136
50.....	225
90.....	318
95.....	336
F.P.	356
Recovery.....	99.3%
Residue.....	1.0%

Pumps

In the first run, performance of the low-pressure, valveless, modified screw-type, paste-circulating pumps was very poor, resulting in low delivery rates and discharge pressures after short operation. The excessive wear of the stators and rotors is believed to be due in part to the service and the partial plugging of the suction line. New stators and rotors were installed, the suction lines to the individual pumps were simplified, and the performance of the pumps has been fairly



Figure 7. - High-pressure injection pumps, Coal-Hydrogenation Demonstration Plant. Original with valves in blocks at left; improved model with external valves at right.

satisfactory since these changes. To test various types of pumps in this service, a standard duplex reciprocating pump has been installed and is giving excellent initial service.

High-pressure injection pumps (see fig. 7) have caused considerable difficulty from two sources: packing for the fluid-end plungers and fatigue cracks in the fluid-end blocks under variable stresses. The results of a number of tests with various types of packings are summarized in table 5.

TABLE 5. - Packing used on high-pressure injection pumps

<u>Type</u>	<u>Remarks</u>
1. Neoprene-impregnated duck chevron.	This was the original packing that worked satisfactorily to 7,500 lb.
2. O&C type ring, soft, nonmetallic (modified chevron).	Rods ran very hot and tight above 7,500 lb. Pump never reached operating condition.
3. Supported ring, soft, metallic (chevron).	This packing was not successful owing largely to failure of the bakelite rings. The rubber seal rings were destroyed and the buna-rubber chevron rings badly worn on the inner surface. Scored rods.
4. Phosphor-bronze metal packing, modified German design.	Brinell hardness, 71-76. Did not prove successful because the basic pump design was not rigid enough.
5. Soft metallic.	Unable to make packing tight on inboard end. When packing was tight enough to hold on outboard end, pump would barely run.
6. Braided copper rings.	Did not hold; too much leakage around rods and stuffing box.
7. Alternate soft metallic and Thiekol-treated duck chevron.	Not successful; similar to No. 5. Also scored rods.
8. Same as No. 1, with modification.	Spacer rings now steel with bronze lining, plus braided copper ring next to lantern ring, with much better pump alignment.
9. Bakelite rings with bronze spacers.	Not successful; too much leakage. Poor wear characteristics.
10. Rubber duck chevron with copper faced steel support rings.	In use at present. Quite promising.

The failure of the fluid-end blocks to stand up under high pressures has been a continual problem. New blocks with a minimum of openings were installed and have given acceptable service to date.

A program for developing new high-pressure pumps for coal-hydrogenation service is now under way, and one prototype pump incorporating the following features is under actual construction:

1. Rigid construction will assure maintenance of alignment.
2. The power cylinders are centrally located, with the high-pressure pumping cylinders on each end.
3. High-tensile-strength materials will be used throughout.
4. Suitable provision has been made for accurately guiding the floating plungers through the packing.
5. The high-pressure liquid cylinders are of the shrunk-on, double-barrel design.
6. By careful design and workmanship, all sharp corners, side openings, and other stress raisers have been eliminated.

Instruments and Controls

Many changes have been made to modify and improve automatic temperature-control and flow measurements. Temperature measurements, except in the converters, and skin temperatures on high-pressure lines have been satisfactory.

The initial pressure recorders were designed for a maximum of 1-1/2 times the operating pressure, or 15,000 p.s.i. The 22- to 24-percent chromium-steel Bourdon tubes gave very short service and frequently ruptured at 10,000 p.s.i., necessitating the adoption of tubes made of A.I.S.I. type-316 stainless steel designed to withstand 30,000 p.s.i. Control of flow throughout the process has been improved greatly by regulating the pressure difference across the recycle compressors indirectly from the flow measurement to the stall, as it is of prime importance to maintain steady flow of gas through the system regardless of fluctuations in the actual differential. Likewise, greatly improved pressure control and over-all operation has been achieved by maintaining a controlled flow of make-up hydrogen to the unit at all times while automatically regulating purging to maintain pressure.

Differential pressure across an orifice plate originally was measured by either a strain gage or special high-pressure mercury-filled manometer with an inductive-type transmitter. The strain gages, measuring a maximum differential of 7.2 p.s.i., have proved unsuccessful for this service and have been replaced by mercury manometers.

The helical rotor flow meters suspended directly in hydrogen lines to measure small flows have proved unsatisfactory and have been taken out of service.

The Gagatron has proved successful in liquid-level indication and control of the hot catchpot level. The pneumaticator system used alternately for this service is also quite satisfactory.



Figure 8. - Stellite-faced targets following restricting orifices in heavy-oil let-down lines. Hole was cut through wall of target at right at point indicated by arrow. Unused target in center.

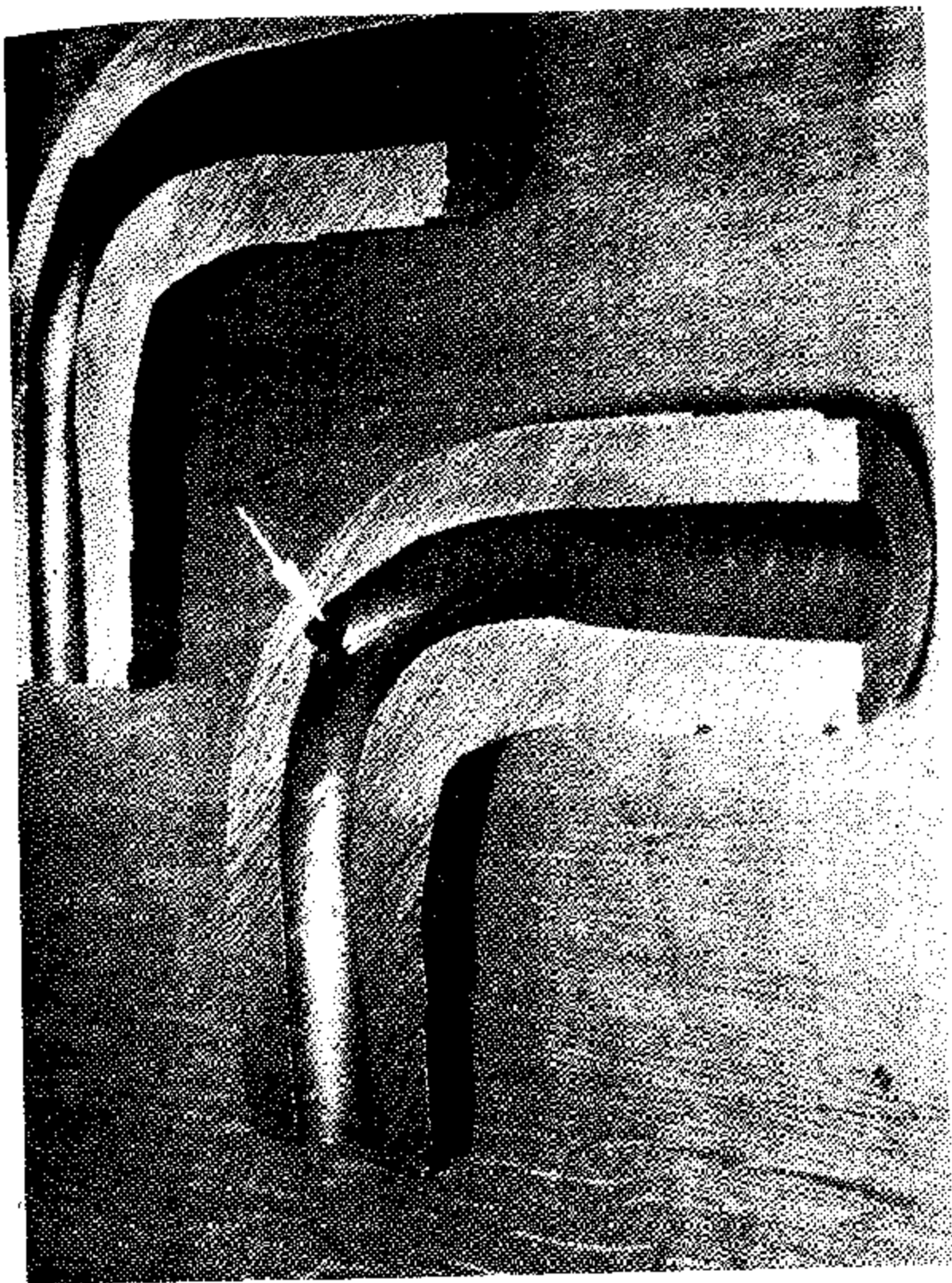


Figure 9. - Two halves of elbow following ore of targets shown in figure 8. Wall was cut completely through at point indicated by arrow.

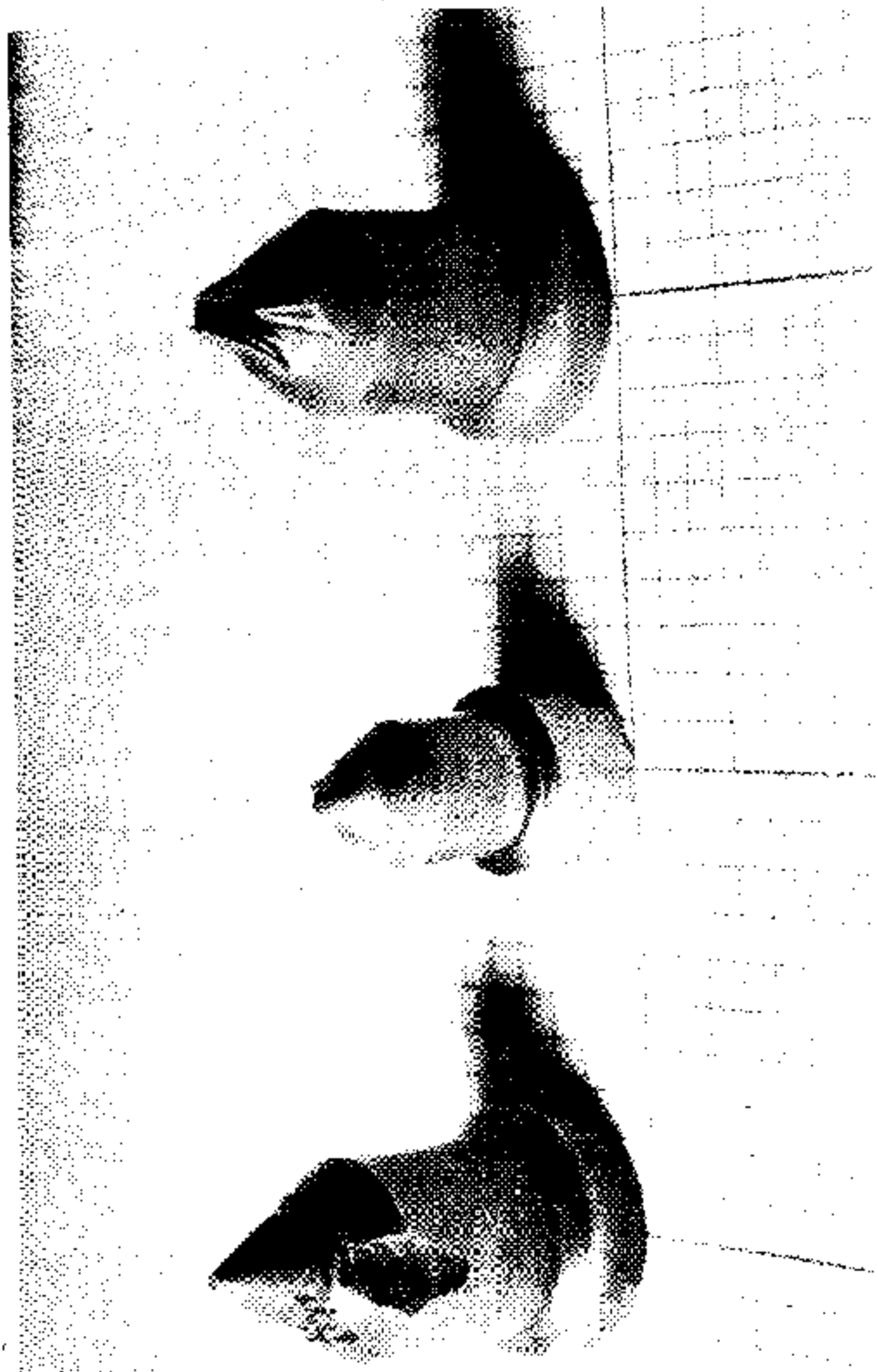


Figure 10. - Cemented tungsten carbide valve tips showing wear during use. Left: Tip from mild throttling service, showing erosion at shut-off position. A chip also spalled off owing to mechanical shock. Center: Eroded tip from severe throttling control valve on heavy-oil let-down containing coal ash after 24 hours. Right: Eroded tip from hand-operated severe throttling valve on heavy-oil let-down to flash distillation after 16 hours.

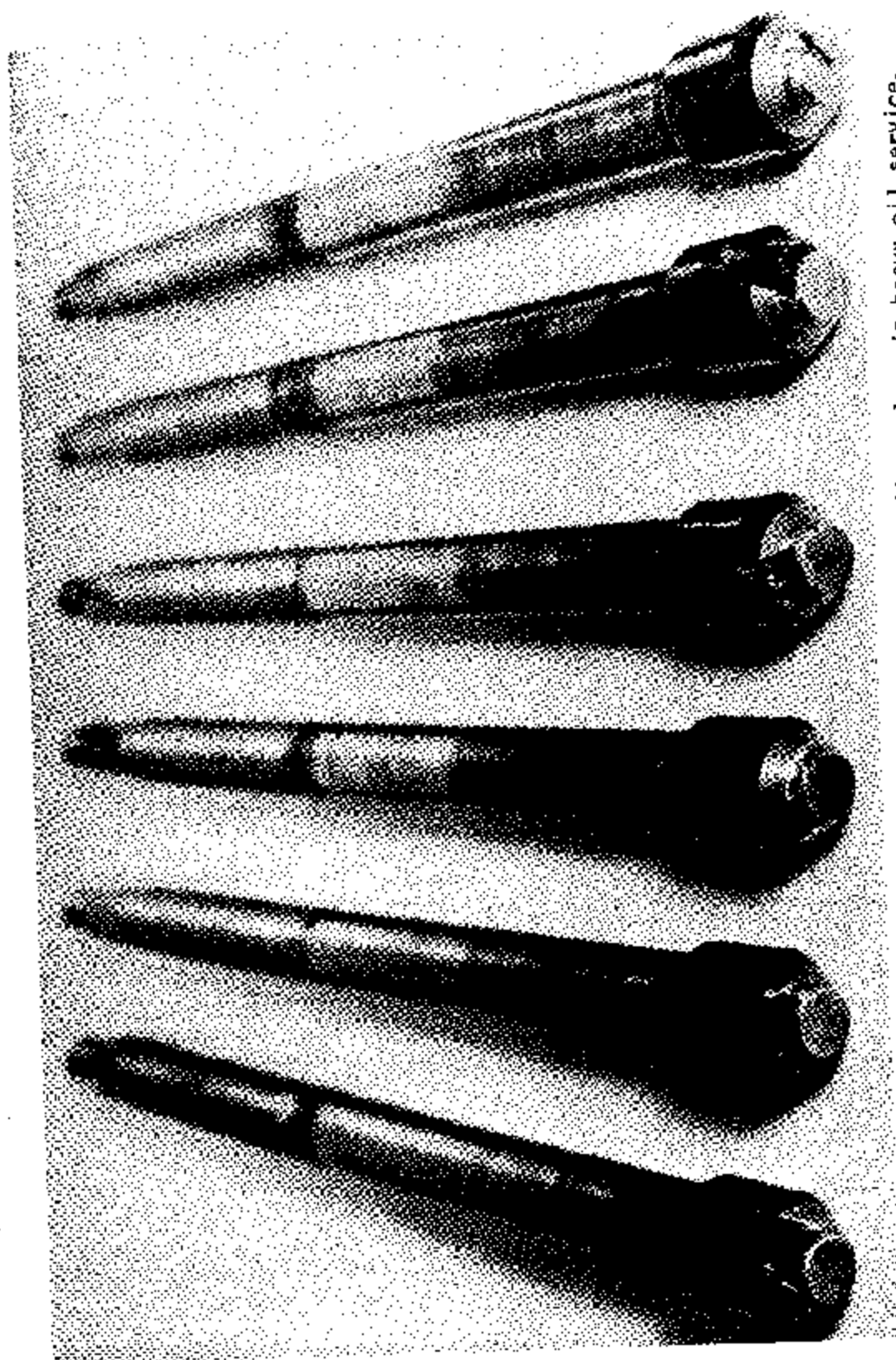


Figure 11. - Valve disks from block valves ahead of throttling valves in heavy-oil service.

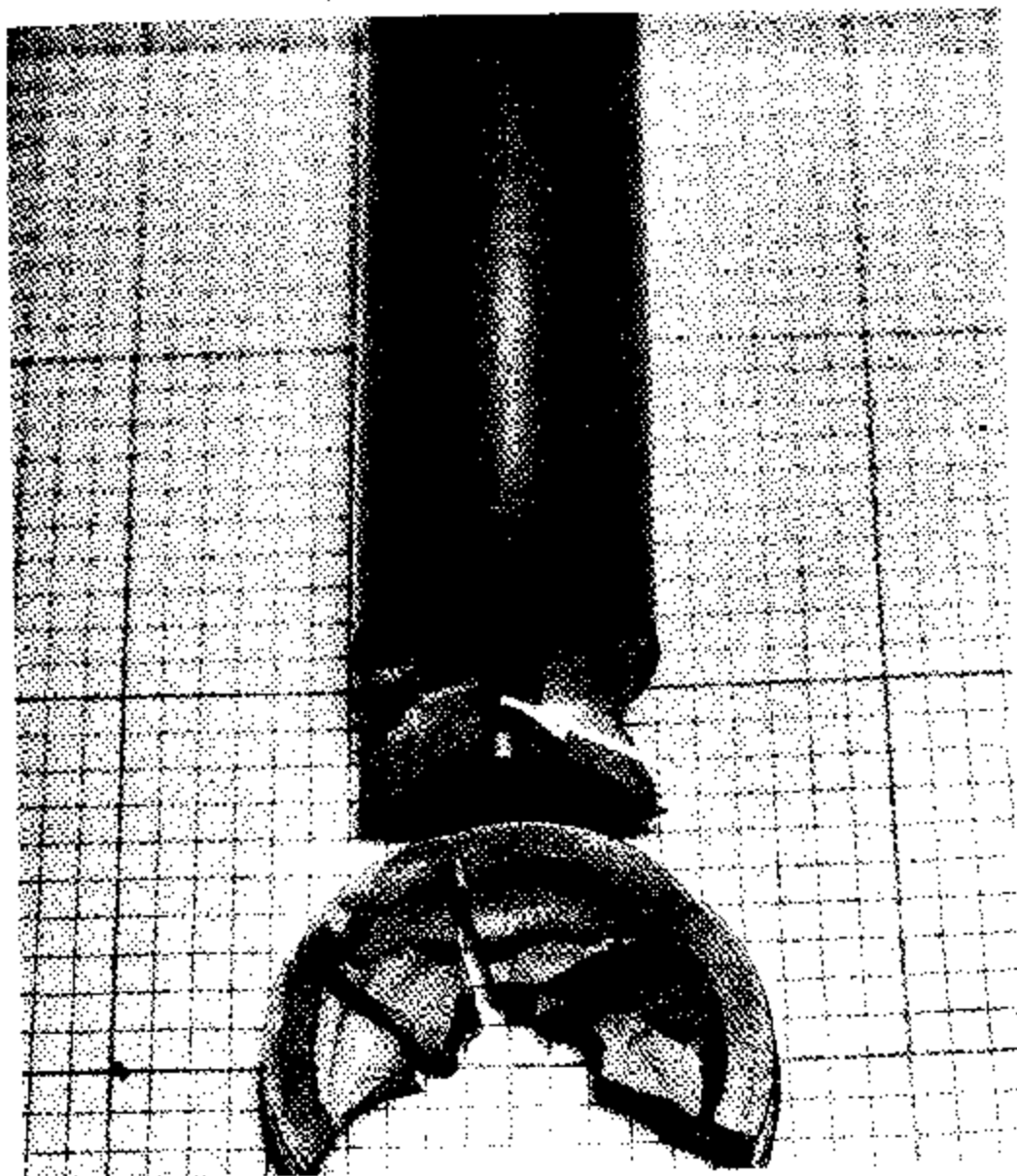


Figure 12. - Cemented tungsten carbide seal insert from severe throttling after 24 hours service. Erosion back of seat can be seen at arrow point.

Heaters and Exchangers

The four-cell, steam-jacketed, radiant-type paste heater with vertical hairpins has shown little tendency to form coke at localized hot spots, and the wear on return bands has been negligible. Heat-transfer rates are satisfactory for the service. Temperature control has been improved by regulation of the fuel gas from the skin temperature on the steam tubes rather than from the heavy-wall paste tubes.

The duty or load of the majority of the heat exchangers is low when compared to the usual commercial units. The exchangers are of two classes: (a) those with high pressures on one side only, such as water-cooled condensers, and (b) those having high pressures on both sides, such as product-gas exchangers. The former have given satisfactory service. Screwed and seal-welded joints in the headers of the exchangers with high pressure on both sides of the transfer walls could not be made tight. The seal welds had to be cut out and replaced with standard high-pressure welds capable of holding the full pressure.

The heat recovery from the vapor-phase feed-product exchanger has been very low because of radiation from the large metal mass and irregular flow resulting from the series-parallel design. This has resulted in a heavy load on the preheater, even though flow rates equal to only half of the design flows have been attained. A study is under way to provide a satisfactory tube and shell-type product-feed exchanger.

High-Pressure Vessels, Piping, and Fittings

With few exceptions (see figs. 8-12), the high-pressure vessels, piping, and fittings have given good service. The delta-type gasket-closure seals on the vessels have not leaked during any of the runs, although it is sometimes difficult to seal new gaskets during pressure tests. The solid lens rings in cold-piping joints have been entirely satisfactory. Leaks have resulted during testing and operation on the bellows lens rings used in hot piping. It usually has been necessary to retighten, reface, and regasket all of the hot piping before attempting a pressure test. As a result of this experience, arrangements are under way to eliminate flanged joints by use of welded piping wherever possible.

Valves in severe throttling duty have given good service, where followed by restricting orifices. However, when these orifices were not used, the life of the valve plug tips has been very short - 15 to 20 hours - because of rapid destruction of the tip and seat.

Except for a few installations, the high-pressure shut-off valves have given good service. In several installations, however, the plugs and seats of these valves have been badly eroded. This was attributed to one of two causes. First, inspection of several shut-off valves has shown the presence of fine hairline cracks across the stellite plug. It is believed that erosion started in these cracks. Second, there is the evidence that solid particles have been wedged between the seat and plug, preventing a positive shut-off and causing eventual erosion of the plug and seat.

The service of the mild-throttling valves, satisfactory in most installations, has not been as good as the shut-off valves. In the inspection after the first run, many Kennametal seat rings were found to be cracked, especially on the smaller valves, some of which had been used to obtain a positive shut-off. Since that time, however, special care has been taken in setting the stop nuts on these valves to prevent overclosing and damage to the seats.

In relatively severe service, such as manual let-down from the cold catchpot and break-in line from the injection pumps, the mild throttling valves have been badly eroded, especially in the throat after the seat. It was felt that these installations on relatively clean oil did not warrant installation of severe throttling valves. This problem is now being discussed with the manufacturer to determine whether erosion can be reduced by a change in design of the throat.

Gas-Synthesis Demonstration Plant

Oxygen Plant

The oxygen plant continued in routine operation during all periods when oxygen supply was necessary for use in the gasification work. The longest period of continuous operation, which began August 8, 1949, and continued until December 22, was not completely reported last year. Operating statistics for this run may be summarized briefly as follows: More than 67 million std.c.f. of oxygen was produced at an average rate of 21,300 std.c.f. per operating hour. The average power used was 19.4 kw.-hr. per M c.f. of oxygen produced. The plant was in production for 3,150.5 of the 3,328 hours, or 94.7 percent of the elapsed time.

Gasification

Gasification was centered in two projects. The first was the continuation of operation of the Koppers powdered-coal-oxygen gasifier, and the second was the operation of a Kerpely-type fixed-bed gas producer on oxygen of varying purities, steam, and three varieties of cokes.

Koppers Gasifier

The Koppers powdered-coal gasifier equipment and program were described in the 1949 Annual Report. The gasifier is a horizontal, refractory-lined, cylindrical unit measuring 6-1/2 feet inside diameter by 9 feet long, fed at both ends with a mixture of powdered coal and oxygen, and with steam entering as an envelope around this mixture.

Between the initial operations in May 1949 and the shutdown at the end of April 1950, 46 test periods of operation were completed. Of these, approximately half were made during the calendar year 1950. For data of representative runs see table 6.

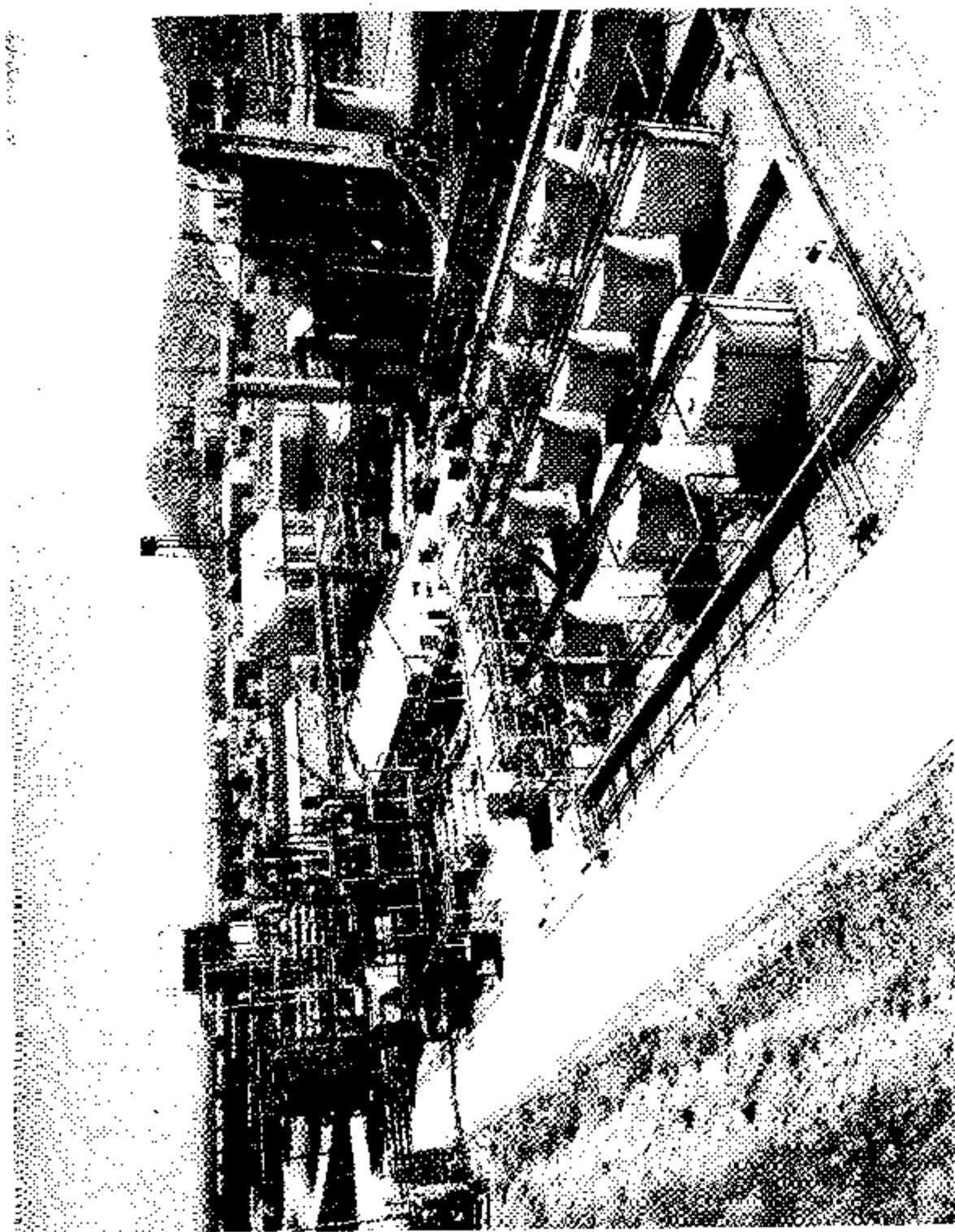


Figure 13. - General view of Gas-Synthesis Demonstration Plant.

TABLE 6. - Representative runs, Koppers powdered-coal gasifier

Run No.	32 3 hr.	35 5 hr.	36 6 hr.	43 11 days	Remarks
Duration.....	2,062	2,297	2,151	2,695	Burner nozzles projecting 16 inches into the gasifier were used in run No. 32.
Coal feed, lb./hr.	72	72	93	79	It was the special objective of this run to observe operation at a high oxygen- to-coal ratio.
Percent coal through 325-mesh....	10.6	9.0	9.3	8.0	
Std.c.f. oxygen/lb. coal.....	0.78	0.80	0.81	0.75	Run No. 35 was a standard run made to observe operation with "flush" burner nozzles.
Lb. steam/lb. coal.....	1,680	1,700	1,705	1,640	
Temp. steam, °F.					
Gasification temp., °F., by thermal balance.....	2,600	2,480	2,535	2,260	Run No. 36 was made with 1-inch project- ing burner nozzles and with "extra fire" grind coal. Better over-all results obtained in production of gas than in any other comparable run.
Carbon conversion, percent.....	89.9	83.4	89.0	73.8	
Lbs. steam decomposed/lb. coal..	0.08	0.13	0.15	0.12	
Materials required/M c.f. of CC + H ₂ , lbs. coal.....	37.5	38.8	36.7	41.0	Run No. 43 was an extended run made with 1-inch projecting burner nozzles. Data here given were taken during 7-1/2- hour period on 10th day.
Std.c.f. C ₂	399	349	342	326	
Lb. steam.....	29.3	30.4	29.6	30.8	