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

Instruments and Controls

Instrumentation of the plant is believed to be more complete than in any other domestic or foreign high-pressure plant. The instruments include liquid level, flow, pressure, and temperature controls, and most of these function dependably. Except in the converters, temperature measurement has been fairly satisfactory. The response of skin thermocouples used on lines carrying suspended solids is slow and the indicated temperatures are 20° to 70° below the actual temperatures, even when well-insulated and with no appreciable coke deposits on the pipe walls. Improved paste-preheater temperature control has been achieved by controlling from the skin temperature of the steam-jacket tubes rather than from the heavy-wall-product tubes.

Temperature measurements in pyrometer tubes have been made more accurate and reliable by carefully assembling the small, fiber-glass-covered thermocouple wires inside a small inner tube rather than around a tube or rod. The couples emerge through small slits into which the spring-clip contact points can recess, and the wiring and contact points are protected in this way. Reliability of the temperature measurement has been improved by careful cleaning, polishing, and drying of the heavy-walled pyrometer tube. Means are sought to measure converter temperatures, using short couples through the walls, particularly in the vapor phase, as the present outer catalyst support tube surrounding the heavy-walled pyrometer tube seriously interferes with thermocouple-temperature response. The measurement of paste and other solids-containing oils requires improvement. An area flowmeter and the displacement meters proved unsatisfactory, although venturi-type flowmeters appear to have some promise for this service. Meanwhile, the paste rate is approximated from tank gages, injection-pump strokes, and preheater-temperature indications. The measurement of small flows, particularly in the refinery, has been improved by use of 50- and 100-inch meters in place of 20-inch meters.

Although the initial operation was promising, the gagetron has not yet proved serviceable for control of hot catchpot level. However, entirely satisfactory service has been achieved from the pneumercator system since using filtered fresh hydrogen and somewhat shorter bottom tubes, which are not adversely affected by the increased flow of bottom agitation gas required to keep the vessel clean. Temperature measurements of the liquid and vapor in the vessel and of the streams flowing from the vessel have proved very useful in checking the level and the quantity of flow from the vessel.

A major improvement in diaphragm-control-valve operation can be largely attributed to the modified, well-lubricated, soft-metal base packing and to use of better valves, plugs, and seats.

Stellite No. 1 plugs and seats are adequate in clean liquid or cooling gas services. In gas and solids bearing oil service, such as H.O.L.D., Kennametal (tungsten carbide) has given the best results. Norbide (boron carbide) was tried but has not given good performance, owing largely to imperfections in manufacturing the plugs and seats.

Further tests are in progress to develop restricting orifices and target devices that will allow the valves to function without excessive wear, mainly in starting and stopping the flow.

that worked quite successfully and, for the coals processed to date, has given satisfactory hydrogenation results.

To lower the moisture content of the hot recirculating flue gas used to dry coal and to improve sizing of the ground coal, arrangements are under way to install a cooler in the recirculating flue gas line and to replace the ineffective pulverized-coal classifier with a vibrating screen.

The paste-making equipment operated more satisfactorily and dependably than in previous years. This was attributed to added provisions for external lubrication of bearings submerged in the paste mixer and to added adjustable support arrangements to prevent the lower blades from rubbing the tank bottom.

Although continuous operation is still difficult, the mechanical functioning of the coal Waytrol was improved. To obtain more accurate weighing, especially at high throughput rates, it is planned to increase the speed of the Star feeder and the Waytrol belt. Plans also are under consideration for batch-weighing the coal from the Redler conveyor into the powdered-coal bin.

Pumps

Two standard duplex reciprocating pumps, equipped with crown valves, special liners, and Darcova cup pistons, are now used for paste circulation with excellent results.

Considerable trouble is still experienced with the oversized centrifugal paste-oil-circulation pumps, which leak excessively around the packing. By installing new bearings and sleeves, and providing for adequate clearance between the housing and impeller to eliminate excessive pressure against the packing, the situation has been improved considerably and the packing life increased from, in some cases, a few hours operation to 15 days or more. A smaller, steam-driven, heavy duty centrifugal pump of open end impeller-type construction is on hand for trial runs before testing pumps, similar to those used for circulating paste.

Many improvements have been made in the design and operation of the high-pressure injection pumps. These included harder, more accurately machined plungers, spring-loaded packing, and improved lubrication to the outer end of the packing. The suction and discharge ball valves, located in flanged fittings midway of the plunger travel, continue to give 7 to 24 days service. In the case of paste pumps, valve life has been materially lengthened by improving the suction conditions, that is, constant paste-circulating pressure, and revised suction manifolds to facilitate periodic cleaning before serious plugging occurs. However, an entirely satisfactory valve has not been developed for paste service. The injection pumps in clean light-oil service have shown increased capacity and dependability after introduction of spring-loaded suction valves. As the service life of the new blocks also is limited, a new injection pump has been designed, using compound cylinder construction, centrally located steam drive, and lubricated spring-loaded flexible packing. The first simplex pump has been delivered, installed, and is in operation; the others are in production.

Heaters and Exchangers

The performance of the steam-jacketed paste preheaters and the direct-fired vapor-phase heater has been proved. Direct-fired paste-heater designs have been proposed for commercial plants and tests on unjacketed tubes will be tried, using the demonstration-plant heater and starting with a few hairpin tubes in the first heater section. The steam jacket already has been removed from the hydrogen heating-tube hairpin, with good results. The vapor-phase heater has been repiped for series flow, the temperature control has been improved, and tendencies toward hot tubes and subsequent coke formation have been alleviated.

Double-tube heat exchangers gave satisfactory service, except for the seven-parallel-pass, vapor-phase, feed-product exchanger, which has been removed for alterations. Several small shell and tube heat exchangers are on order, and tests will be scheduled to obtain data for commercial design.

High-Pressure Vessels, Piping, and Fittings

The original high-pressure piping materials have proved quite satisfactory. However, it seems that a considerable increase in allowable stresses may be permitted by the use of other materials that would decrease the cost of commercial plants. Cooperative work is under way to test the suitability of centrifugally cast steel tubing for high-pressure work.

The demonstration plant incorporates much more welded construction than did European hydrogenation plants. Our operating experience to date indicates this to be justified, and additional welding is being introduced into hot, high-pressure alloy lines. An extended period of operation will be required to confirm the practicability of this step. If and when confirmed, commercial plants may use welded construction with a minimum number of flanged joints for assembly, inspection, and clean-out purposes.

In adopting this construction, the hot stall piping was removed and refabricated to eliminate as many flanged joints as possible. For clean-out connections, a welding tee was used, with a blind lens ring and a blind flange to close the opening. A system of counterbalanced weights supported the hot piping. After the initial pressure test was made, hot stall piping was heated to an average temperature of 800° F. to permit observation of line growth and performance of the counterbalancing. Only minor corrections were necessary to allow for free expansion of the lines in the proper direction. With the new piping construction and through close attention in assembling flanged fittings and vessel heads, leakage problems in the stall area have become negligible.

Equipment for Removing Solids

Both the Bird centrifuge and flash-distillation unit have been used during the year for removing solids. Because the Bird centrifuge does not have the capacity to remove all the solids and has little effect on the asphalt level, it was necessary to operate flash distillation at maximum rates most of the time. By this means, it was possible for the first time to maintain the benzene-insoluble solids in the pasting oil between 4 to 7 percent and the asphalt, particularly sticky asphalt, at a low level, where its presence seldom interfered with pump packing, strainers, or other equipment and instruments.

The speed of the Bird centrifuge was increased from 2,000 to 2,340 revolutions per minute (r.p.m.) in an attempt to improve separation of solids. At this higher speed, it was impossible to operate the centrifuge at a rate much over 800 gallons per minute (g.p.m.) without overloading and breaking shear pins. Many pins were sheared during runs; and at one time, when straight H.O.L.D. was being processed, it was necessary to remove the rotor and conveyor for cleaning, owing to the formation of a solid cake that cemented the rotor and conveyor together.

The flash-distillation unit was revised extensively, and it was operated early in the year to process the heavy, solid-bearing oils from liquid-phase run 5. H.O.L.D. is supplied from tankage and heated to 650° to 700° F. in a radiant-type furnace. A spray condenser system was installed to condense most of the exhaust steam from the quench tower and recover oil entrained in the steam. The most troublesome problem encountered was handling of pitch from the bottom of the flash drum. The original Magma plunger pumps proved inadequate, mainly because it was impossible to prevent plug-up from solids and keep the suction line, pumps, and discharge line hot enough to assure flow at all times. After many attempts, the pumps were abandoned in favor of a chute leading directly from the bottom of the flash drum into a portable receiving tank, which is hauled away and emptied. A test was made on a water-cooled conveyor to handle the pitch. The test looked promising, and a full-scale conveyor is on order.

Corrosion and Erosion Experience

Corrosion in low-pressure carbon-steel equipment is similar to that found in oil refineries. It is most severe on the water side of heat exchangers. In one exchanger installed in 1949 and operated less than 80 days, 18 tubes out of 84 corroded through from the water side. Heat exchangers and vessels handling gases and liquids containing hydrogen sulfide and other sulfur compounds have had relatively high corrosion rates. The liquid-phase overhead exchanger has corroded at the rate of 0.02 inch per year. Based on actual operating time, this rate would be 0.08 inch per year. The liquid-phase reflux drum corroded at the rate of 0.01 inch per year, or 0.04 inch per year based on actual operating time. Other exchangers and vessels have corroded at similar rates, and in some cases there has been extensive pitting. In the high-pressure equipment corrosion was not severe, although equipment using water, either as a coolant or as process liquid, was affected. The water-injection pumps and the suction manifolds have pitted, and some scale has formed. Tubes in box-type coolers have scaled on the outside and pitted near the water line. There has been no significant corrosion on the inside of tubing or equipment handling the hydrocarbon-process liquids.

Erosion has been more serious than corrosion. The heavy-oil-let-down, containing a high percentage of ash, unconverted solids, and absorbed gases, has caused deep erosion where velocities approach the speed of sound. Throttling-control valves made of the most abrasion-resistant metals, in some instances, have been eroded beyond use in less than 24 hours. The normal life of these parts when less than 500 gallons per hour (g.p.h.) is handled has been under 48 hours. Modifications of design and changes in materials are being made to attempt to increase their life. Two 1-inch high-pressure elbows, which followed a restricting orifice in the lines handling this material, were cut completely in less than 3 weeks' operation. These 1-inch elbows were replaced with 2-1/2-inch high-pressure tees having solid-shaped plug target which deflected the material in the same manner as an elbow. Three weeks' operation of these units resulted in fairly severe erosion of the plugs and tees. The wear plate in the vessel was also deeply eroded. A replaceable plug having a spherical cup, hard-faced with abrasion-resistant metal, then was tried. This change virtually eliminated the wear on the tees and has reduced erosion of the wear plate in the vessel considerably.

Evaluation of Hydrogenation Fuel Products

Motor Gasoline

Extensive fleet tests in Army ordnance transport and engineer equipment have shown that the synthetic gasoline (see fig. 11) will give fully satisfactory performance under sustained 100-percent-overload conditions. Use of the gasoline in all Bureau of Mines equipment at the Louisiana, Mo., Station also has been successful. To evaluate the products further and extend the military test program, samples of finished vapor-phase gasoline made from Rock Springs and Western Kentucky No. 11-bed coals, an aviation base stock, and a blended jet fuel from Western Kentucky No. 11-bed coal were submitted to a commercial testing laboratory for evaluation. The tests are summarized in the following tables:

Finished gasoline	Western Kentucky No. 11 bed	Rock Springs, Wyo. 1/
Gravity, °A.P.I.	54.1	54.3
10 percent evaporation ... °F.	125	135
50 percent evaporation ... do.	220	224
90 percent evaporation ... do.	327	320
Residue ... percent	1.0	1.0
Vapor pressure ... p.s.i.	9.9	8.4
Octane number:		
Motor method	77.3	75.3
Research method	83.7	81.5
Existent gum content ... mg./100 ml.	1.6	4.0
Sulfur content ... percent	0.023	0.016
Corrosion copper strip	Negative	Positive
Oxygen stability ... minutes	480+	480+
Composition after removal of phenols and bases, percent		
Aromatics	29.5	25.2
Olefins	2.5	1.8
Naphthenes	26.2	28.1
Paraffin content	41.8	44.9
Aniline point ... °C.	26.4	28.0

1/ The gasoline from Rock Springs coal had been stored in a 55-gallon drum for 1 year.