SPECIAL EQUIPMENT IN THE COAL-HYDROGENATION DEMONSTRATION PLANT

BY J. A. MARKOVITS, K. C. BRAUN, J. T. DONOVAN, AND J. H. SANDAKER

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J. A. Markovits, K. C. Braun, J. T. Donovan, and J. H. Sandaker

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1/ A brief description of the equipment and materials used in the construction of the Hydrogenation Demonstration Plant at Louisiana, Mo., by the U. S. Bureau of Mines.

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ACKNOWLEDGMENTS

It took nearly 3 years to complete the design and construction of the Coal Hydrogenation Demonstration Plant. The lion’s share of the work was done by the Engineering Section of the Coal-to-Oil Demonstration Branch and the Bechtel Corporation’s engineering personnel. Vendors mentioned in the text participated in designing the equipment finally selected. The drafting force of the Engineering Section deserves special mention for the careful preparation of illustrations.

INTRODUCTION

Coal has been hydrogenated in Germany on a rather extensive scale for the past 20 years, but the process is new to the United States. Although pressures up to 15,000 pounds per square inch have been employed on a commercial scale in the synthesis of anhydrous ammonia, and there is evidence that valves, tubing, and equipment have been developed recently up to 30,000 p.s.i. working pressure at moderately elevated temperatures for limited industrial production of plastics, such pressure ranges are unusual in American industry, and equipment designed for them is not commonly available.

In the Coal Hydrogenation Demonstration Plant at Louisiana, Mo., 10,000 p.s.i. operating pressures are combined with temperatures up to 1,000° F., and the materials to be processed under these conditions range from gases to mixtures of viscous liquids containing high percentages of abrasive solid particles. These service conditions presented unusual engineering problems requiring thorough investigation. Limited financial resources and rigid time schedules inherent in the synthetic liquid fuels act often forced decisions before the best answer to a problem could be developed. Sometimes the ideal technical solution proved to be beyond the financial limitations of the demonstration-plant program.

Few items of equipment were commercially available for the process chosen. Consequently, special equipment had to be designed and built. Where there was a choice between importing equipment from Germany or building it here, the latter was done. It was the hard way, but it was considered desirable in order to acquire design craftsmanship and to give American manufacturers some conception of the basic requirements for designing future commercial-size plants. Within the limitations of the project, the equipment was chosen and sized, so that the results of operations would be representative of those expected from modern, full-scale hydrogenation plants.

High-pressure joints were discussed at length with the major manufacturers. Wherever possible, the self-sealing principle was adopted - internal pressure being used to help maintain the seal. Utilization of this principle produced large savings in space and material.
Tubing and flange standards of the American Standards Association stop at the 2,500 p.s.i. steam working pressure level. Contacts with leading flange manufacturers indicated that the size of the demonstration plant did not warrant the expenditure for the engineering work of setting up manufacturers' standards. Bureau of Mines investigations showed that extrapolation of the standard American method of flange design would not be practical, as neither the use of the conventional safety factors nor the assumptions used for yielding the gasket material are feasible in extremely high pressure work. After comparing the flanging methods used in 15,000 p.s.i. ammonia practice with the German 700 atmosphere coal-hydrogenation flanging method, the latter was selected, as it showed big material savings and promised greater flexibility.

The selection of materials for high pressure vessels and tubing was one of the most controversial problems. Not only was it necessary to provide steels to withstand high pressures and temperatures, but also to resist attacks by hydrogen, hydrogen sulfide, and other corrosive agents. Steels used for various services in German hydrogenation plants were first reviewed and their reproduction was discussed with American manufacturers. The resulting selections are given under the various items described.

This circular does not intend to give a full description of all the materials and equipment used in the construction of the plant. Rather, it aims to confine itself to the more or less special materials and equipment required for extremely severe services or to methods new in American industrial practice.

The demonstration plant was designed and built by the Bechtel Corp. of San Francisco, Calif., in cooperation with the Bureau of Mines.

SHORT DESCRIPTION OF THE PROCESS

The primary chemical difference between coal and crude petroleum is that there is approximately twice as much hydrogen in the latter. Thus, for the hydrogenation of coal to finished gasoline, hydrogen amounting to approximately 9 percent of the weight of the moisture- and ash-free coal has to be combined with the coal to form the products.

The demonstration plant was designed to operate at the 700-atmosphere pressure level in two major steps. Liquid-phase hydrogenation accomplishes liquefaction of coal, and vapor-phase hydrogenation converts the liquefied coal to gasoline and byproducts. The output of the plant will be 200 to 300 barrels of gasoline per day, depending on the coal and catalyst used.

Figures 1 and 2 are flow sheets of the hydrogenation and product-recovery processes. Figure 3 is the plot plan, and figure 4 is a panoramic view of the plant, looking south. Marks or designations of vessels or other equipment mentioned in this report correspond to those shown on the flow sheets.

The raw coal is first crushed to minus 3/4-inch size, then pulverized to minus 60-mesh in a ball mill, and dried to 1 or 2 percent moisture content by a gas-fired recirculating dryer. The pulverized coal is mixed with a small amount of catalyst and with heavy oil previously obtained from the liquid-phase process into a paste containing approximately 47 percent solids. The
Figure 1. - Coal-hydrogenation process flow sheet.
Figure 3. - Coal-to-oil demonstration plant plot plan.
Figure 4. - Panorama looking south at open side of stall.
catalyst will consist either of 1 to 2 percent iron oxide or 0.05 percent tin oxalate as an alternate.

The viscous paste is injected into the paste preheater by steam-driven plunger pumps working at 10,000 p.s.i. The preheater is a modified radiant type in which the high-pressure tubing is protected by a superheated steam jacket. A small amount of hydrogen is injected into the paste ahead of the preheater to reduce the viscosity, and the paste is heated in the first section of the preheater to about 570° F. At this stage, additional hot hydrogen and recycle heavy oil are added to jump the temperature to the 640° F. level, circumventing coal-swelling difficulties that would occur at around 600° F. After passing through the remainder of the preheater, the mixture leaves at about 815° F. and passes into the first of two converters. For 95 percent conversion of the coal to liquid and gaseous products, the residence time is approximately 1 hour. The reaction is highly exothermic, and to maintain the reaction temperature at 930° F., cooling hydrogen is added to the converters in controlled amounts at different points.

After the second converter, the reacted products enter the "hot catchpot," where the hydrogen and light ends separate from the solid containing heavy oil fraction. "Letting down" the heavy oil to near atmospheric pressure is the next step. This is extremely difficult, because the hot liquid contains large quantities of absorbed gases and up to 30 percent of abrasive solids. The heavy oil is freed from ash, catalyst, and unreacted coal by centrifuging or by flash distillation with 1,100° F. superheated steam. The oil fraction constitutes the bulk of the pasting oil used in the process.

The gases and vapors leaving the top of the hot catchpot are cooled in a heat-exchanger system, and the condensed liquid and vapor are separated in the "cold catchpot." The gas from the cold catchpot, containing about 70 percent hydrogen, is washed with water at full operating pressure for the removal of NH₃, H₂S, and water-soluble salts, and with oil for removal of light hydrocarbons. After this purification, the hydrogen stream is recycled through the system with fresh make-up hydrogen. The liquid products from the cold catchpot and from the wash-oil scrubber go through let-down systems, where the pressure is reduced in two steps, first to 25 and then to 7 atmospheres, and are then charged to the distillation unit.

The bottoms from the liquid-phase distillation unit join the pasting-oil stream. Gasoline, naphtha, and middle-oil cuts are separated to establish weight relations and to prepare, by blending, a feed stock of uniform composition for the vapor-phase hydrogenation. This stock is combined with a nearly equal amount of vapor-phase recycle middle oil and saturated with H₂S or with sulfur for the vapor-phase hydrogenation step. The addition of some form of sulfur is necessary to preserve the activity of the vapor-phase catalyst.

The vapor-phase injection pumps, working at 10,000 p.s.i., push the charge, with a small amount of hydrogen, through a feed-product exchanger and a radiant-type vapor-phase preheater. The stream leaves the preheater completely vaporized at 920° F. This vapor enters a single converter containing six fixed catalyst beds. The catalyst is fuller's earth treated with 7.5 percent hydrofluoric acid and impregnated with compounds of zinc, molybdenum, chromium, and sulfur. The catalyst is very rugged and at 700 atmospheres
performs the triple duty of the former German saturation, splitting, and dehydrogenation operations. The reaction is quite sensitive to temperature variations, and recirculated cooling hydrogen is added at every tray to keep the temperature between the 912° and 950° F. operating limits. After the reaction is balanced, the feed-product exchanger provides most of the heat necessary, and the duty of the preheater becomes negligible.

The products then pass through a cooler and a cold catchpot, where the condensed oil and hydrogen are separated. The hydrogen is returned to the circulating compressor, and the liquid passes through a double let-down system to the vapor-phase distillation unit. The bottoms are recirculated to the vapor-phase hydrogenation system; the overhead is stabilized to a 10-pound R.V.P. gasoline, washed with caustic and water, and sent to final storage. Tail gases pass through an absorber before being used for fuel. Ammonia, CO₂, and H₂S liquors are treated with sulfuric acid before going to the sewer, and the recovered H₂S is utilized to saturate the vapor-phase charge.

Make-up hydrogen of about 11 percent by weight of the moisture- and ash-free coal is furnished from the former Missouri Ordnance Works natural-gas reformer, scrubber, and compressor system. Here, natural gas is crunched with steam, and the resulting H₂ and CO are shifted, with more steam, to H₂ and CO₂. The mixture is compressed through three stages of a 7-stage compressor to 450 p.s.i., and CO₂ is scrubbed out with water at this pressure. After the 7th stage (around 13,000 p.s.i.), the remaining CO is removed by catalytically reacting into methane and water. The resulting 97 percent pure hydrogen is ready for injection into the gas-circulating system.

HIGH-PRESSURE VESSELS

Converters

The largest forged vessels in the plant are the converters. In these vessels the hydrogenation reaction takes place at around 10,000 p.s.i. and above 900° F. In order to utilize a low-alloy steel shell, a 3-inch lining of asbestos cement was placed between the inner liner, or reaction basket, and the shell proper. This layer of insulating material holds the shell temperature below 500° F., thereby permitting full utilization of the low-temperature properties of steel, reducing temperature stresses, and minimizing hydrogen attack. The primary consideration in the construction of these heavy-wall vessels was to keep the over-all weight as low as possible within safe and practical design limits.

All of the high-pressure vessels, including the converters, hot and cold catchpots, compressor suction traps, and wash-oil scrubber, were built by the Midvale Co., Philadelphia, Pa.

a. Liquid-Phase Converters V-1 and V-4

These vessels, shown in figure 5, are alloy-steel forgings, 32 inches inside diameter by 49-1/2 inches outside diameter by 39 feet 1-1/2 inches long, face to face of end flanges, with the ends flanged out to 61 inches outside diameter. The heads are flat steel forgings 21-1/2 inches thick by 57
Figure 5. - Liquid-phase converter assembly.
inches in diameter and provided with the necessary openings for the entry of the coal paste and hydrogen and the exit of the products of reaction. There are no openings whatever in the converter shell. The shell steel is 3 percent chrome with 0.65 percent nickel, 0.30 percent molybdenum, and 0.25 percent carbon, with an ultimate tensile strength of 100,000 p.s.i. and an elastic limit of 55,000 p.s.i.

Past experience has shown that a minimum of 3 percent chromium is desirable to resist the penetration of hydrogen and the corrosive effect of H₂S at 500°F and 10,000 p.s.i. The other constituents are necessary for sufficient strength in an 8-3/4-inch thick converter wall where the thickness is based on a working unit stress not greater than one-half the elastic limit at the shell temperature.

The A.P.I.-A.S.M.E. Code stipulates that when pressure vessels have a wall thickness greater than 10 percent of the inside diameter, the Lamé formula, based upon maximum principal stress theory, shall be used in determining the required wall dimensions. Therefore, the wall thickness is:

\[
t = \frac{D_1}{2} \sqrt{\frac{s}{p} - 1} \cdot f \cdot C
\]

Where: \( t \) = wall thickness, in inches
\( p = 10,300 \) p.s.i. working pressure
\( D_1 = 32 \)-inch inside diameter
\( s = 25,000 \) p.s.i. maximum allowable stress up to 650°F
\( C = 0 \), corrosion allowance

Then:

\[
t = \frac{32}{2} \cdot \left( \sqrt{\frac{25,000}{25,000 - 10,300}} - 1 \right) \cdot f \cdot 0
\]

\[
t = 16 \left( \sqrt{2.4} - 1 \right)\]

\[
t = 8.75 \text{ inches}
\]

The converter heads are subjected to more severe temperature; consequently, it was decided to use a slightly higher content of the same alloying elements with heat treatment to provide approximately the same physical properties. The head steel is 4 to 6 percent chrome with 1 to 1.25 percent nickel and 0.40 to 0.80 percent molybdenum. The head studs, of which there are twelve in each head on a 45-1/2-inch diameter bolt circle, are 5-3/4 inches diameter of S.A.E.-4340, with an ultimate tensile strength of 120,000 p.s.i. and an elastic limit of 90,000 p.s.i. The stud nuts are of S.A.E.-4140, with an ultimate tensile strength of 100,000 p.s.i. and an elastic limit of 70,000 p.s.i. The shell proper weighs about 80 tons, each head about 7-1/2 tons, and the assembled converter, with internals, about 105 tons.
In determining the required head thickness, the problem was approached by assuming a head thickness of 18 inches and substituting this value in Roark's' formulas for determining tension stresses in flat, bolted cover plates:

\[ S_t = \frac{3}{2} \frac{w}{m} \left( \frac{m}{(m - 1)} \ln \frac{a}{r_o} - (m - 1) \frac{r_o^2}{2a} \right) \]

Where: \( a = \frac{22.75 \text{ inches}}{2} \) = Bolt circle diameter

\( r_o = \frac{16.625 \text{ inches}}{2} \) = Gasket contact diameter

\( w = 8,940,000 \text{ lbs.} \) - Total force on head from internal pressure

\( t = 18 \text{ inches} \)

\( m = 3, \) reciprocal of Poisson's ratio

Then:

\[ S_t = \frac{3 \times 8,940,000}{2 \times 3 \times 324} \left[ \frac{3}{4} \left( \ln \frac{22.75}{16.625} - (3 - 1) \left( \frac{16.625}{4 \times 22.75} \right)^2 \right) \right] \]

\[ S_t = 17,500 \text{ p.s.i.} \] (without holes)

This stress and thickness was then checked by using the A.P.I.-A.S.M.E. formula,

\[ t = d \sqrt{\frac{C}{P}} \]

Where \( t = \text{Head thickness in inches} \)

\( d = \text{Diameter of shortest head span = 33.25 inches} \)

\( p = 10,300 \text{ p.s.i. working pressure} \)

\( s = 25,000 \text{ p.s.i. maximum allowable working stress} \)

\( C = 0.30 \ \frac{1.4 \text{ wh}}{H} = 0.558 \)

\( h_G = \text{Radial distance from the bolt circle to the diameter d, in inches} \)

\( H = \text{Total hydrostatic end force on area bounded by the O.D. of the gasket in pounds} \)
Figure 6. - Delta gasket detail.
Then: \[ t = 33.25 \sqrt{\frac{0.558 (10,300)}{25,000}} = 15.9 \text{ inches} \]

The working stress in an 18-inch thick head by this same formula

\[ = \frac{(15.9)^2}{18} \times 25,000 \]

\[ = 19,500 \text{ p.s.i. (without holes)} \]

Using basic calculation per A.S.T.M. code W-316 and reinforcing for metal removed, the head thickness equals 18 inches at 25,000 p.s.i. stress. It was decided to increase head thickness to 21-1/2 inches owing to the possible higher temperatures that might occur in the top and bottom heads.

The bolts were sized in the conventional manner, based upon a working load of 8,940,000 pounds, which resulted in a working stress for the twelve 5-3/4-inch diameter studs of 30,400 p.s.i., or about one-third of the elastic limit.

The asbestos-cement lining for the converter shell is mixed in the proportion of 1:1:1 by volume of granulated asbestos, quick-setting Portland cement, and water. The mixture is poured and tempered in sections in the annular space between the shell and the basket. The basket is stainless steel, A.S.T.M.-A240-347, and has a reaction space of about 110 cubic feet. This steel was chosen for its good hydrogen and corrosive-gas resistance at temperatures up to 1,000° F. The basket is 25-1/2 inches inside diameter, with 1/4-inch thick wall perforated with 5/16-inch-diameter holes on 3-inch centers for pressure equalization.

The heads of the converter are insulated with 12 inches of asbestos cement and are sealed to the flanged ends by annealed S.A.E.-1020 steel gaskets. The self-sealing gasket shown in figure 6 is of triangular cross section. The 33.250-inch inside diameter gasket fits into 0.35-inch-deep grooves in head and shell flange. About 0.002-inch copper plating was added to the polished gasket faces, which improved the seal considerably by eliminating the galling of contact surfaces during fluctuation in pressure. The initial seal is created on opposite tips of the gasket by the bolting force. The gasket becomes deformed after internal pressure is applied, as shown by dotted lines on figure 6. Sealing actually takes place along faces marked "a" and "b" in the cross section. The soft iron gasket is of small cross section, and the internal pressure forces the entire gasket to expand in diameter when the pressure forces the bolted head away from the shell. At this writing, it is still controversial how often a gasket can be reused.

Temperature measurement within the converter is taken through a centrally located, 40-foot long, 2-inch outside-diameter, 7/8-inch inside-diameter, seamless, steel pyrometer tube. This tube, held in place by several steel spiders, has a closed bottom and is designed to withstand external pressure of 10,300 p.s.i. at 1,000° F. Inside of the tube is a 1/8-inch schedule-40 pipe of A.S.T.M.-A276-304 stainless steel, to which are
attached 6 thermocouple elements on 6 foot centers. These elements transmit the temperatures at the various levels to a recording instrument in the instrument house.

b. Vapor-Phase Converter V-511

The vapor-phase converter shown in figure 7 is identical with the liquid-phase converter in dimensions and composition of shell and heads. The inner insulation also is the same, except that the liner is 1/16-inch, perforated, stainless steel. The insulation is recessed for the 3/8-inch cooling hydrogen lines, so that the space inside the insulation is unobstructed for the insertion and removal of the catalyst basket.

The removable catalyst basket, shown in detail in figure 8, is 24 inches inside diameter, 36 feet 4-3/4 inches long, and is made of 3/8-inch A.S.T.M.-A240-347 stainless-steel plate. Both top and bottom ends are bell-shaped and bolted to the body of the basket. Pressure between basket and shell is equalized by holes in the top bell. The catalyst space of about 100 cubic feet in the basket is subdivided into six compartments by perforated grids covered by a wire screen to support the 10 mm. diameter by 10 mm. long pellet catalyst. The grids are so constructed that they serve also as a gas-distribution chamber for uniform distribution of the hydrogen gas supplied to the bottom of each grid by separate 3/8-inch supply lines through the top head.

The pyrometer tube is similar to that in the liquid-phase converter, except that inside of the 2-inch outside diameter pyrometer tube there is a 3/8-inch diameter solid rod that carries 12 thermo-elements for measuring the temperature at two points in each of the 6 catalyst chambers. The pyrometer tube is enclosed in a shielding pipe, permitting removal of the tube without disturbing the catalyst. The shield is closed at the bottom and is made of A.S.T.M.-A240-347, schedule-160 pipe.

c. Hot Catchpot V-5

As previously stated, this vessel receives the products of reaction from the liquid-phase converters. The heavy oil is drawn off at the bottom, whereas gases and vapors leave through the top of the hot catchpot. The general construction and dimensions shown in figure 9, as well as the materials of construction - the shell, heads, gaskets, insulation, and perforated liner - are the same as for the liquid-phase converter, except that the shell is shorter - 25 feet 5-1/2 inches face to face of end flanges. The liquid inlet pipe through the top head extends 13 feet 6 inches below the head. The normal level of the liquid in the pot is about 5 feet above the bottom, and the top of the liquid outlet pipe, projecting through the center of the bottom head, is 4 feet above the bottom. To prevent settling of solids and coking at the bottom of the pot, a 1/2-inch, perforated, schedule-40 pipe bent to a 23-1/2-inch diameter ring is provided a few inches above the bottom for agitating and cooling the liquid with hydrogen.

Measuring and regulating the liquid level in the hot catchpot is a very important but troublesome operating function, and in an attempt to make it fool-proof various methods were provided and are expected to work simultaneously. The primary method consists in introducing hydrogen into the liquid.
Figure 8. - Vapor-phase converter basket detail.
Figure 9. - Hot catchpot assembly.
Figure 10. - Hot catchpot liquid-level controller installation.
Figure II. - Cold catchpot and compressor suction trap assembly.
and the vapor space of the hot catchpot and measuring the difference in pressure between the two lines as an indication of the height of the liquid. It should be noted that the total pressure is around 10,000 p.s.i., whereas the head variation is but a few feet. Two pairs of 3/8-inch inside diameter hydrogen lines enter the hot catchpot through the top head. One of each pair supplies hydrogen to the vapor space just below the head, the other to the liquid 1 foot 9 inches above the bottom. The flow of hydrogen through the tubes must be sufficient to prevent asphalt from creeping back into the lines.

One pair of these 3/8-inch diameter lines is provided with orifice plates and recording flow indicators. Equal flow is maintained in both lines by hand-controlled valves. This will insure equal pressure drops through the lines, and the pressure difference due only to the liquid head will actuate the transmitter of the liquid-level controller, as indicated in figure 10.

The other pair of lines is provided with capillary throttling tube coils, which maintain uniform hydrogen flow because the choke effect of the capillary coil will not be affected by the small variations in pressure caused by the 3/8-inch feed-line resistance. The Brown recording level controller is electrically connected to the differential pressure transmitters, which in turn are connected by 3/16-inch instrument lines to the respective pairs of 3/8-inch hydrogen feed lines.

Operation of the above-mentioned liquid-level control device is checked by another instrument. This check operates on the principle that liquids or vapors of different specific gravities that come between a radium source and a Geiger-Mueller detector have a different degree of gamma-ray absorption not influenced by temperature, pressure, or chemical composition of the liquid in the vessel. The changing rate of gamma rays striking the detector causes variations in the electrical impulses. These impulses are amplified and transmitted electrically to a Brown electronic recording controller. The detector is manufactured by the Engineering Laboratories, Inc., of Tulsa, Okla., and utilizes the penetrating power of gamma rays from an iridium-platinum needle containing a small amount of radium-salt mounted on the pyrometer tube and a gamma-ray detector on the outside of the vessel.

The temperature of the hot catchpot contents is observed by pyrometer readings. The pyrometer tube in this case extends from the bottom up about 2 feet above the top of the liquid. The thermo-elements are spaced 12 inches on centers, so that there are at least two elements in the vapor space.

d. Cold Catchpots V-8 and V-508

Figure 11 shows an assembly of these vessels. They are 24 inches inside diameter, 37-3/4 inches outside diameter, and 16 feet 11-3/8 inches inside length. The pots are flanged at the top to 47 inches outside diameter and tapered at the bottom to a 2-inch diameter liquid outlet. The top head is 45 inches outside diameter by 15-7/8 inches thick and is provided with product inlet and gas outlet openings. The 2-1/8-inch-diameter product inlet pipe extends 2 feet below the head, where it is divided into two 1-1/2-inch diameter pipes, terminating in 3/8-inch by 1-7/8-inch rectangular outlets discharging downward at 1/2° F. and tangentially along the inner surface of the
shell. This arrangement will provide efficient separation of gases from liquids without foaming. At the elevation of the impinging stream the vessel is lined with an interchangeable carbon steel wear cylinder 2 feet long by 3/8-inch wall thickness. The shell steel analysis is 2.50 to 3.25 percent nickel, 0.75 percent chromium, and 0.30 to 0.40 percent molybdenum, the heads are carbon steel, and the head studs, nuts, and gaskets are the same as for the converters and the hot catchpot.

As the normal operating temperatures are low, 150° F. for the liquid phase and 80° F. for the vapor phase, the cold catchpots are neither insulated nor lined on the inside. For the same reason, the shell steel contains only enough alloy metals to meet the required physical properties.

The normal liquid level in the cold catchpot is 7 feet 8-3/8 inches above the bottom outlet flange. A Penberthy special, high-pressure, transparent gage, 15,000 p.s.i. test, is connected between the bottom liquid outlet and the top gas outlet to indicate the liquid level. The level is controlled by a Brown recording level controller with high and low level alarms.

e. Compressor Suction Traps V-22 and V-513

These vessels, also shown in figure 11, are installed in the recycle-gas compressor suction lines from the cold catchpots to trap the moisture from the gas before it reaches the compressors. They are identical with the cold catchpots.

f. Wash Oil Scrubber V-11

The wash-oil scrubber is installed in the liquid-phase recycle-gas suction line between the cold catchpot and the compressor suction trap. The purpose of the scrubber is to wash out the hydrocarbons and impurities from the gaseous products of hydrogenation in order to maintain the partial pressure of hydrogen in the recycle gas at a minimum of 80 percent. The washing medium is naphtha from vapor-phase still C-704 and is injected into the scrubber through the top head, whereas the gas enters at the bottom. The scrubber is packed with 1-1/2- by 1-1/2-inch steel Raschig rings.

Design conditions are similar to those for the cold catchpot - 10,300 p.s.i. at 120° F. In details of the shell, heads and closures, materials of construction, and general dimensions, the scrubber is identical with the cold catchpot, except that it is much longer, being 41 feet 7-1/4 inches over-all. Like the cold catchpot, it is tapered at the bottom from 24 inches inside diameter in the body to a 2-inch-diameter bottom outlet. The wash-oil inlet pipe through the top head extends down 6 feet to a perforated-steel distribution plate, below which is a packing space 28 feet 8 inches deep. The packing is supported by a steel grate 3 feet 2-3/8 inches above the bottom head flange. The gas inlet pipe extends upward from the bottom to a point just below the packing grate and is surmounted by a serrated bubble cap. The normal liquid level in the scrubber is 4 feet above the packing grate. The liquid level is indicated and controlled in the same manner as in the cold catchpot.
Figure 12. - View looking west at stall showing Gantry crane.
Figure 13. - Details of Wickelofen wrapping.
Figure 14. - Wickelofen wrapping.
Figure 15. Wickelofen wrapping lathe.
Stall Structure

All high-pressure vessels are within a heavy-walled, reinforced-concrete structure 193 feet long by 28 feet wide by 58 feet high, open on the top and rear. To minimize the danger of possible fires or explosions, the structure is divided into 6 stalls by 14-inch concrete walls. A 130-ton Gantry crane with a 70-foot lift on a 365-foot craneway straddles the structure and facilitates handling of heavy equipment. Figure 12 shows the Gantry crane carrying a converter to the stalls visible in the right background.

The two end compartments of the stalls house the preheaters. Next to the preheater are the hot stalls of each phase, containing the converters, high-temperature exchangers, and hot catchpot. In the center are the two cold stalls with the cold catchpots, coolers, and compressor suction traps. In a full-scale plant it is not considered necessary to enclose the cold vessels and preheaters. The experimental nature of the operation was the reason for this extra precaution in the demonstration plant.

New Developments in High-Pressure Vessel Design

The demonstration plant high-pressure vessels described above were built in accordance with the established practice of forged construction. However, during the initial stages of plant design, consideration was given to layer-vessel design and to the spirally wound vessels (Wickelofen) still in the industrial development stage in Germany. The carbon-steel plate-layer vessels proved to be too heavy and expensive, whereas the spirally wound vessels were not available from American manufacturers.

Latest investigations indicate that the spirally wound vessels have excellent possibilities in future American full-scale operations. Therefore, a detailed description of this method seems to be pertinent to this report. The Germans developed this method of pressure-vessel construction shortly before the second world war as a modification of the layer vessel designed and constructed by the A. O. Smith Co. of Milwaukee, Wis. Figures 13, 14, and 15 help to visualize the spirally wound method of construction. The principle of design utilized by this method is the shrinkage of each successive layer of the wound strip upon the layer or core beneath. The shrinkage of the layers is achieved by continuously heating each strip electrically to 800° to 900° C. just before winding, followed by cooling to atmospheric temperature immediately after it is pressed onto the underlying layer. The resultant predetermined contraction of the metal strip will set up circumferential prestressing necessary for minimum wall thickness. This fact is borne out by analysis of the maximum principal stress theory on which the heavy-wall vessels for this plant were designed.

Assuming a forged vessel of 32-inch inside diameter and 49-1/2-inch outside diameter (the dimensions of the demonstration-plant converter):

\[
\text{Tangential stress} = p \frac{a^2}{b^2 - a^2} \left( 1 + \frac{b^2}{r^2} \right)
\]
Stress at outside of cylinder \( = \frac{P \cdot a^2}{b^2 - a^2} \times 2 = 14,800 \text{ p.s.i.} \)

Stress at inside of cylinder \( = \frac{P \cdot a^2}{b^2 - a^2} \times \left( 1 + \frac{b^2}{a^2} \right) = 25,100 \text{ p.s.i.} \)

Stress at center of shell \( = \frac{P}{2} \cdot \frac{a^2}{b} \left[ 1 + \left( \frac{2b}{a} \right)^2 \right] = 18,300 \text{ p.s.i.} \)

Where \( p = 10,300 \text{ p.s.i.} \) internal pressure

\( a = 16 \text{ inch internal radius} \)

\( b = 24\frac{3}{4} \text{-inch outside radius} \)

The graphical plot of these results is illustrated in figure 16. The same figure shows the tangential stress distribution of a spirally wound vessel with a predetermined stress condition assumed to be 12,000 p.s.i. on the inside fibers caused by shrinking on the successive layers that form the wall. Obviously, this will permit thinner wall sections, because the internal tensile stress is in the range of 13,000 p.s.i. on the inside diameter, compared to 25,000 p.s.i. for the forged vessel. Also, it places the outside layers under higher stresses, allowing them to stretch uniformly, thereby permitting visual inspection of amount of growth of the outside layers and detection of possible failure.

The "Nicolofen" or wound vessel is fabricated essentially as follows:

The thickness of the inner tube, which forms the core, may range from 7/8 inch for 30-inch inside diameter to 1-1/4 inch for a 48-inch inside diameter vessel. The inner tube must be resistant to corrosion and hydrogen attack at high temperature and pressure. A 3 percent chrome steel with 0.2 percent C, 0.4 percent Mn, 0.3 percent Si, and 0.15 percent V was normally used by the Germans for this purpose. A plate of commercial width is rolled to the desired diameter and butt-welded longitudinally. As many of these cylinders as are necessary for the desired length of the vessel are butt-welded together. Dummy ends of plain carbon steel are welded to the ends of the inner tube. These dummy ends, to which the strip ends are welded, are later cut off when the vessel is finished.

The strip may vary in thickness from 1/4 inch to 3/8 inch, and the width is usually about 10 times the thickness. It may be of plain carbon steel for the lower pressure and temperature ranges or low alloy steel for higher temperatures and pressures, depending on operating conditions or strength of material desired. As shown in figure 13, the strip is rolled to a tongue-and-groove profile, preferably three or more grooves in width, so that the upper layer of strip can overlap by at least one-third its width and still interlock amply for axial strength.
Figure 16. - Graph of forged and wrapped vessel wall stresses.
Figure 17. - Flanged end of Wickelofen vessel.
The wrapping lathe can be the same type used for machining solid wall vessels (see fig. 15). The tool carriage can be altered to run on a special track and accommodate a reel of strip, the strip heating and cooling equipment, and a profiled back-up roller. The strips, as received in mill lengths, are butt-welded, end to end, to a length required to wind a complete layer in one run.

The inner tube, with its dummy ends, is set on the lathe, and cooling water is turned on inside the tube. Spiral grooves are then cut into the outside face of the tube to match the tongues on the strip at a pitch equal to the width of the strip plus a slight clearance.

Before wrapping is started, the end of the strip is welded to the dummy end of the inner tube at an angle corresponding to the pitch. The strip passes between guide rollers and is pressed against the vessel by two 6-inch-diameter profiled rollers diametrically opposite each other, to prevent distortion of the tube. The load on these rollers is about 1 ton. The guide rollers act as the connection for the electric current, which heats the strip as it unwinds from the reel onto the vessel. The power required to heat the strip to between 600° and 850° C. is furnished at 30 to 40 volts and 4,000 to 6,000 amps.

Immediately after passing under the profiled roller the strip is quenched by compressed air jets and about 5 or 6 turns later by cold-water jets. The speed of wrapping is about 15 feet per minute.

After the first layer of strip is completely wrapped, the end is welded to the dummy end of the inner tube, and the carriage is returned to the other end of the lathe. The next strip is then welded to the first-layer strip (see fig. 13) so that it will overlap the joints of the underlying layer by one-third of the width of the strip. The succeeding strips are then wrapped exactly like the first and second, until the required wall thickness is obtained.

To finish the ends of the vessel, a flange is formed by the same wound method, a steel ring is shrunk over the strip ends, and the dummy inner tube ends are cut off to the proper length (see fig. 14). Instead of a wound flange of strips, a solid forged-steel flange may be screwed or shrunk on, if desired. Figure 17 shows a German Wickelofen of the type described.

Advantages claimed for the wound vessel are these:

a. Economy in corrosion and hydrogen-resistant material, because only the inner tube needs these properties.

b. Because of the thin strip, the heat treatment applied to it penetrates to the innermost crystals. Thus, the safety factor for nonhomogeneous material in solid-wall vessels can be reduced.

c. Method of construction gives more uniform stress distribution throughout the wall thickness, allowing use of thinner walls.

d. Further reduced wall thickness results from increased tensile strength, caused by controlled heating and cooling of the strip.
e. Tests showed that wound vessels will stand a pressure 1.25 to 1.4 times that of solid-wall vessels of comparable material and diameter ratio.

f. The material lost in machining is only 1/6 that of solid-wall vessels.

g. Any wound vessel designed for a lower pressure can be made suitable for a higher pressure by simply adding more layers.

h. The use of the Wickel-lathe developed in Oppau by I. G. Farben makes the Wickel process so simple that only three operators are required, and the manufacturing time is reduced to one-fifth that required for a solid-wall vessel, with a consequent saving in cost of 20 to 30 percent.

i. Wound vessels can be made at the plant site shop, thus simplifying handling and transportation problems.

j. Heavy forging equipment can be eliminated, as the heaviest forgings are the comparatively light heads. Thus, the size of the vessels is limited only by the capacity of cranes or other handling equipment in the field, and larger vessels can be built than any so far constructed. This is of particular importance to modern commercial plants.

For further particulars on the construction of the "Wickelofen", the reader is referred to "Engineering in Hydrogenation Plants in Germany", by J. F. Ellis, published by the British Intelligence Objectives Subcommittee, 32 Bryanston Square, London, W. I., or the Office of Technical Services, U. S. Department of Commerce, Washington, D. C.

PREHEATERS

a. Liquid-Phase Paste Preheater F-1

The function of the liquid-phase preheater is to raise the temperature of the coal paste-hydrogen mixture from the 250° F. inlet temperature to 815° F., or near the reaction temperature. The heater operates under 10,300 p.s.i. working pressure. In European practice the convection-type preheater was universally accepted. In that case tubes are not exposed to radiation, and the temperature of the combustion gases is regulated by recirculation. The possibility of dangerous overheating is remote, but to provide the necessary surface on the gas side finned tubes are used for the hairpins. The application of fins presents considerable welding difficulties, and American manufacturers hesitated to bid on a convection-type heater for the demonstration plant. They all advocated the simpler oil-refining type radiant heater with low heat densities corresponding to the high viscosity of the paste.

Comparing the two methods, it was found that the permissible over-all heat-transfer rate is approximately the same in both cases, and it is governed by the film coefficient of the paste, which depends primarily on the viscosity. Owing to temperature changes, the viscosity of coal pastes varies within
Figure 18. - Preheater heat-transfer curve.
extremely wide ranges. The approximate behavior of coal paste, based on German information, is illustrated in figure 18. With higher temperatures, the viscosity drops and the film coefficient increases to a point where the vaporization of the pasting oil and the swelling of the coal are beginning to show their effects. From here on the paste tends to dry, the coal gels, and the film coefficient drops very rapidly. Above a certain temperature, dependent on the composition of the coal and the coal-to-oil ratio in the paste, liquefaction will increase heat-transfer rates with gradually decreasing viscosities. From this it was evident that the different stages of the preheating process have to be performed in separate sections of the preheater, each under carefully controlled optimum temperature conditions.

After accepting the radiant-type preheater principle, it was decided that very low heat densities will be used, and for additional protection against overheating of the high-pressure preheater tubes a thin layer of highly turbulent superheated steam was provided by jacketing the tubes with 4 to 6 chrome-molybdenum pipe.

Without reproducing the extensive and complex thermodynamic computations, the basis of the heater calculations can be summarized as follows:

Total preheater duty is the sum of the duty of the four preheater sections.

In each case: \[ Q = A H_o \Delta T \]

Where \[ Q = \text{Total heat input in B.t.u./hr}. \]

\[ A = \text{Area in sq. ft. based on the inside surface of high-pressure tubes} \]

\[ \Delta T = \text{Mean temperature difference in °F}. \]

\[ H_o = \text{Overall heat transfer rate in B.t.u./sq. ft. based on inside tube surface /°F temperature difference/hr}. \]

\[ H_o \Delta T = \text{Heat density in B.t.u./sq. ft. based on the inside of tube surface} \]

The over-all heat-transfer value depends on the film coefficients and wall conductivities:

\[ H_o = \frac{1}{R_p + R_m + R_{s1} + R_{s2} + R_m2} \]

Where \[ R_p = \text{Heat resistance of paste film, including the fouling effect of a permissible coke layer} \]

\[ R_m = \text{Heat resistance of high-pressure tube wall} \]

\[ R_{s1} = \text{Heat resistance of steam film on outside of high-pressure tube} \]

\[ R_{s2} = \text{Heat resistance of film on inside of jacket tube} \]

\[ R_m2 = \text{Heat resistance of jacket-tube wall} \]
All heat-resistance values are based upon the inside surface of the high-pressure tube. The film resistances are reciprocals of the film coefficients, which, in turn, are functions of the flow characteristics set by temperature and viscosity.

The heat density was selected upon the basis of German laboratory tests and safe commercial data, the governing critical temperature is the tube metal temperature at the paste outlet, and this was kept below 1,000°F. for safety. Figure 19 is a heat-transfer diagram showing temperature distribution at this critical point. It may be noted that the uniformity of the annular space between the two tubes is obtained with a helical metal rib of 24-inch pitch with a resultant spiral steam motion.

Calculations indicate that the over-all heat-transfer rate of the last cell is 17.9 B.t.u./sq.ft./°F./hr., whereas the paste film coefficient is 19.85. It is evident that the heat-transfer loss through the two tubes and the steam jacket amounts to only 1.95 B.t.u., or 11.0 percent, and the heat-transfer rate is governed overwhelmingly by the paste film. In a similar calculation made for a convection-type preheater, where the ratio of the inside tube wall surface to the finned outside surface was 1:20, the over-all heat-transfer rate was 17.15 B.t.u. with the same paste coefficient.

A typical calculation of tube-wall temperature follows:

In cell 4 the predetermined heat density is 2,730 B.t.u./sq. ft. inside tube area.

\[
\begin{align*}
\text{Paste film drop} & \quad \frac{2730}{19.8} = 138^\circ \text{F.} \\
\text{Drop through tube} & \quad \frac{2730}{185} = 15^\circ \text{F.} \\
\text{(K = 185 based on inside area)} & \\
\text{Drop through inside steam film} & \quad \frac{2730 \times 2.443}{100 \times 4.78} = 14^\circ \text{F.} \\
\text{Drop through outside steam film} & \quad \frac{2730 \times 2.443}{100 \times 5.047} = 13^\circ \text{F.} \\
\text{Drop through outside tube} & \quad \frac{2730 \times 2.443}{962 \times 5.306} = 2^\circ \text{F.} \\
\text{Maximum paste tube wall temperature} & \quad = 815 / 138 / 15 = 968^\circ \text{F.} \\
\text{Maximum steam temperature} & \quad = 815 / 138 / 15 / 14 = 982^\circ \text{F.} \\
\text{Maximum steam tube temperature} & \quad = 815 / 138 / 15 / 14 / 13 / 2 = 997^\circ \text{F.}
\end{align*}
\]
EFFECT OF INCRUSTATION ON THE WALL TEMPERATURES—4th PASS REPRESENTING ABOUT A 5mm (3/16") INCRUSTATION

Figure 19. - Temperature gradient through tube wall in last cell of paste preheater.
**FURNACE DATA SHEET**

**SERVICE**

<table>
<thead>
<tr>
<th>LIQUID PHASE PASTE PREHEATER</th>
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<tr>
<td><strong>NET HEAT</strong></td>
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**PERFORMANCE DATA**

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<td>Heavy Oil Laidown</td>
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<td>Net Paste Gas</td>
<td>1,450</td>
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<td>Total</td>
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| STEAM INTO JACKET | 1,450 | 1,950 |

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<tr>
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<th>P.S.I.G.</th>
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<td>Maximum</td>
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<table>
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<tr>
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**HEATER SECTIONS**

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<th>NO. 3</th>
<th>NO. 4</th>
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<tr>
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<td>1,970,000</td>
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<td>Gas</td>
<td>728,000</td>
<td>662,000</td>
<td>530,000</td>
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<tr>
<td>Total</td>
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<td>728,000</td>
<td>662,000</td>
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<td>900</td>
<td>968</td>
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<tr>
<td>990</td>
<td>990</td>
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<table>
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<tr>
<th>HEAT DENSITY</th>
<th>B.T.U./HR./SQ.FT./°F.</th>
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<th>FIREBOX TEMPERATURE</th>
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<tr>
<td>AVERAGE VELOCITY</td>
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<td>FLUE GAS TEMPERATURE</td>
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**CONSTRUCTION DATA**

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<th>Side Walls</th>
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<tr>
<td>BRIDGE WALL</td>
<td>FLOOR</td>
<td>STACK</td>
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<td>CONNECTING</td>
<td>DRAFT CONTROL</td>
<td>HEADERS</td>
</tr>
<tr>
<td>SHEATHING</td>
<td>OVERALL DIMENSIONS</td>
<td>WEIGHT - Tons</td>
</tr>
<tr>
<td>SUSPENDED B &amp; W, K=20 (9/16&quot; THICK) COVERED WITH 1/4&quot; PLASTIC INSULATION</td>
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<td></td>
</tr>
<tr>
<td>SUSPENDED B &amp; W, K=20 (4/5&quot; THICK) COVERED WITH 1/4&quot; PLASTIC INSULATION</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SAND AND BRICK FILL</td>
<td>30&quot; O.D. X 60&quot;</td>
<td></td>
</tr>
<tr>
<td>8&quot; FABER - TYPE RH AIR REGISTER WITH TYPE RH-IC GAS BURNER</td>
<td>A1, S1, - Type 316</td>
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</tr>
<tr>
<td>MANUAL DAMPER</td>
<td>CORR. ASBESTOS BOARD</td>
<td></td>
</tr>
<tr>
<td>A1, S1, - Type 316</td>
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<table>
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<tr>
<th>TUBE SCHEDULE</th>
<th>HIGH PRESSURE TUBES</th>
<th>TUBE (JACKET) TUBES</th>
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<tbody>
<tr>
<td>Number of Tubes</td>
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<td>64</td>
</tr>
<tr>
<td>Outside Diameter - Inches</td>
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<td>5.565</td>
</tr>
<tr>
<td>Wall Thickness - Inches</td>
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<td>2.508</td>
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<tr>
<td>Inside Diameter - Inches</td>
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<td>5.047</td>
</tr>
<tr>
<td>Overall Length - Feet</td>
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<td>18 1/8&quot;</td>
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<tr>
<td>Exposed Length - Feet</td>
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<td>17 7/8&quot;</td>
</tr>
<tr>
<td>Exposed Surface - Sq. Feet</td>
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<td>866</td>
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<tr>
<td>Material - Tubes</td>
<td>A1, S1, - Type 316</td>
<td>40-50 CHROME - 70 MOLY</td>
</tr>
<tr>
<td>A1, S1, - Type 316</td>
<td>A1, S1, - Type 316</td>
<td></td>
</tr>
<tr>
<td>Volume - Cu. Ft. (Net)</td>
<td>1.75</td>
<td>1.75</td>
</tr>
<tr>
<td>Each Header</td>
<td>A1, S1, - Type 316</td>
<td></td>
</tr>
<tr>
<td>Total Tubes and Headers</td>
<td>44.0</td>
<td>44.0</td>
</tr>
<tr>
<td>Flow</td>
<td>Paste continuous - through 62 tubes</td>
<td></td>
</tr>
<tr>
<td>Paste gas - through 2 tubes - thence through 48 regular tubes</td>
<td></td>
<td></td>
</tr>
<tr>
<td>H.O.L.O. - through 48 tubes (enters at 17th tube)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Purchased Order No.**

| F-451 |
| Manufacturer |
| ALCORN COMBUSTION CO. |
| 6451 |
| M-230 |

<table>
<thead>
<tr>
<th>Furnace Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>F-1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Rechel Corporation Job</th>
<th>Oct. 8, 1948</th>
</tr>
</thead>
</table>

**Figure 20.** Design data on paste preheater.
The four cells of the preheater are enclosed in a refractory walled furnace 29 feet long, 9 feet deep, by 19 feet high. Each cell is gas-fired separately by two vertically arranged burners. The jacketed hairpin tubes are arranged vertically on 15-inch centers along the side walls of the cells.

Inside of the heavy-walled inner tube the paste flows in series through the coils of the four cells. Superheated steam at 400 p.s.i. and 750°F. enters the jacket near the paste inlet and flows concurrent with the paste. Numerous thermocouples are located in the paste tube, the steam jacket at critical tube-wall temperature-reading points, and in the furnace to provide accurate readings and accommodate precise firing control.

Figure 20 is a complete tabulation of the design and construction data for this preheater.

b. Vapor-Phase Preheater E-502

In European practice, the vapor-phase preheater was also of the convection type, but because of the nature of the feed stock a simple radiant-type preheater was selected for the demonstration plant. It consists of a single-cell furnace 10 feet wide, 19 feet 3 inches long, and 11 feet 6 inches high, inside of the walls. It is fired by three gas burners arranged vertically. The two sets of welded hairpin coils, 9 inches on centers, are arranged horizontally along the two side walls of the furnace. They are made of 2-1/2-inch outside diameter by 1-1/4-inch inside diameter tubing of A.S.T.M.-A271-316 specifications. The vapor-phase charging stock is split and flows in two symmetrical streams through the furnace. Preheater duty is 1,890,000 B.t.u./hr. The 30 tubes have 378 square feet of surface based on the outside diameter. Heat density is 5,000 B.t.u./sq. ft. Both liquid and vapor-phase preheaters were constructed by the Alcorn Combustion Co., Philadelphia, Pa.

HEAT EXCHANGERS

High reaction temperatures in the hydrogenation process make the economical utilization of heat important. Heat exchangers, therefore, play a very important part in a coal-hydrogenation plant. Owing to the high pressures, extremely efficient heat-transfer rates are realized in the gas phase. Only exchangers operating at 10,300 p.s.i. will be discussed in this report.

a. Liquid-Phase Exchangers, E-1, E-5, and E-2

All three of these exchangers are in the vapor line between the hot and cold catchpots and are of the double-tube design.

In E-1, the product from the hot catchpot overhead gives up some of its heat to the recycle hydrogen before its injection into the hydrogen coil of the paste preheater. The hot catchpot overhead enters at 867°F. and leaves at 760°F. in heating the hydrogen from 125°F. to 725°F. E-1 is in a vertical position in the liquid-phase hot stall and consists of two jacketed hairpin coils of 81-foot length each, with 52 square feet of heat-transfer surface and 1,470,000 B.t.u. duty. The recycle gas flows through the annular space counter-current to the product. The inner tubes are 1-1/4 inches outside diameter by
8-gage wall thickness, and the outer tube is 4-1/32 inches outside diameter by 1.005 inches wall thickness. Both tubes are of 8 to 10 percent chrome, 1 percent molybdenum steel (B&W Croloy 9M). The operating pressure in both tubes is 10,300 p.s.i., and, as the inside-tube wall thickness indicates, the exchanger is designed to hold the differential pressure only. Testing and starting has to be done with both tubes under pressure.

In E-5, the same overhead line from the hot catchpot heats the wash oil from 140° to 400° F., thereby reducing its own temperature from about 760° F. to about 600° F. E-5 is in the liquid-phase cold stall and consists of one vertical, jacketed, hairpin coil about 40 ft. 9 inches over-all length. Total heat-transfer surface is 84 square feet, and the duty is 1,954,000 B.t.u./hr. The wash-oil stream flows countercurrent to the product through the annular space. The inner tube is the same dimension and material as the jacket of E-1. The outer tube is a 5-inch-diameter, schedule-40, carbon-steel pipe.

E-2 is a cooler. It is also in the liquid-phase cold stall and reduces the catchpot overhead temperature from about 600° F. to about 120° F. by exchange with water in the jacket running countercurrent to the product flow. It consists of 16 vertical, jacketed, hairpin coils, 12-inch tube centers, about 42 feet over-all length. The tubes are the same as for E-5, and the jackets are 6-inch-diameter, schedule-40, carbon-steel pipe. Total heat-transfer surface is 1,344 square feet with a duty of 6,100,000 B.t.u./hr.

As an alternate to cooler E-2, it is proposed to build a shell and tube bundle-type cooler similar to the high-pressure heat exchangers in use in German commercial hydrogenation plants. Such a cooler has been designed and is intended to replace E-2 for experimental purposes. The tube bundle consists of 54 tubes 1-3/8 inches outside diameter, 3-4-inch inside diameter, and 50 feet long spaced 1-3/4 inches on centers, all enclosed in a 14-1/2-inch inside-diameter water shell. The tubes are 4 to 6 percent chrome steel rolled into S.A.E.-4130 forged-steel tube sheets. There are no baffles to impede the flow of water or lateral supports for the tubes along their entire length.

Advantages claimed for this type of cooler are, first, much smaller stall floor space requirements, because it is installed vertically, and, second, the weight is only about half that of a jacketed tube-type cooler of the same heat-exchange surface. These advantages and consequent savings are of more importance in a commercial-size plant.

b. Hot Catchpot Bottoms Cooler E-6

Hot catchpot bottoms or heavy-oil letdown must be cooled to approximately 375° F. before letdown to vessels V-14 and V-26. The heavy oil leaves the hot catchpot at a rate of 3,821 pounds per hour at 765° F., requiring a duty of 715,000 B.t.u./hr. Proper temperature control is of importance to prevent the heavy oil from solidifying. The rate of letdown is fluctuating on account of the hot catchpot liquid level-control arrangement. Under these conditions water cooling would result in steam formation or over-chilling; therefore, a forced-draft air cooler was selected for the service.

The cooler has eight 30-foot-long, 1-inch Croloy 9M hairpin coils connected for series flow. The cooling air steam passes downward at 58 feet per
second through a 64-inch by 10-inch rectangular duct that encloses the coil. The air is supplied by a 10-h.p. motor-driven fan at 90\(^\circ\) F. and leaves at 134\(^\circ\) F. The cooler was supplied by Industrial Engineering Co. of Philadelphia, Pa.

c. **Vapor-Phase Exchangers E-503 and E-504**

Both of these exchangers are in the product line between the converter and the cold catchpot in the order named.

In E-503 the product from the converter gives up most of its heat to the vapor-phase feed stock just ahead of the preheater. It is in the vapor-phase hot stall and consists of 28 vertical, jacketed, hairpin coils spaced at 12-inch tube centers 43 feet in length. The size and dimensions of tubes and jackets, their material specifications, and design pressure and the same as for E-1. However, the product flows through the jacket in this exchanger countercurrent to the vapor-phase feed in the tubes. The duty of the exchanger is 9,350,000 B.t.u. per hour, and the total heat-transfer surface is 728 square feet.

In E-504 the product flows through the tubes and is further cooled from 350 F. to about 120 F. by circulating water in the jackets in countercurrent flow to the product. It is also located in the vapor-phase hot stall and consists of eight vertical, jacketed, hairpin coils about 42 feet over-all length. The sizes and dimensions of tubes and jackets as well as their arrangement are the same as for E-2. However, instead of using Croloy 9M for the tubes, carbon steel is used for both tubes and jackets. The duty of the cooler is 3,560,000 B.t.u. per hour, and the total heat transfer surface is 672 square feet.

d. **Recycle Gas Coolers E-13 and E-505**

E-13 is the liquid-phase, compressor, recycle cooler and E-505 the vapor-phase, recycle-gas cooler. Part of the recompressed circulating gas is by-passed into the suction line of the recirculating compressors for operational safety. The heat of compression in the by-passed stream is removed in these coolers by water jackets. Design data of the two coolers is listed below:

<table>
<thead>
<tr>
<th>Service</th>
<th>E-13</th>
<th>E-505</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid-phase compressor recycle cooler</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Duty - B.t.u./hr</td>
<td>166,400</td>
<td>102,000</td>
</tr>
<tr>
<td>Heat-transfer surface, sq. ft.</td>
<td>23.8</td>
<td>23.8</td>
</tr>
<tr>
<td>Transfer rate, B.t.u./hr./sq. ft./F.</td>
<td>120</td>
<td>159</td>
</tr>
<tr>
<td>Type</td>
<td>15 ft. long, vertical, double-tube, single hairpin</td>
<td>15 ft. long, vertical, double-tube, single hairpin</td>
</tr>
</tbody>
</table>

3270
Size jacket 4-inch I.P.S. 4-inch I.P.S.
Size high-pressure tube 3-1/32-inch O.D. x 0.765-inch wall 3-1/32-inch O.D. x 0.765-inch wall
Inner tube H₂, N₂, and light H₂, N₂, and light
 Fluids H₂, N₂, and light H₂, N₂, and light
Jacket gas gas
Temperature in, °F. 153 85 153 85
Temperature out, °F. 140 91 140 89
Quantities entering, lbs./hr. 12,800 25,900 6,370 25,900

All of the above-mentioned high-pressure heat exchangers are of welded construction with flanged connections and return bonds and are furnished by Industrial Engineers, Inc., of Los Angeles, Calif.

HYDROGEN PRODUCTION AND COMPRESSION

a. Hydrogen Production

The hydrogen production unit is one of the former ammonia lines available from the Missouri Ordinance Works. It consists of two H₂-cracking furnaces, two CO converters, a mixed-gas cooler, a waste-heat boiler, a cooling water pump, and a cooling tower. Two 300,000 cubic foot gasometers are hooked up to handle the production of any one line or the combined load of the hydrogen unit. The plant was built and erected by the Hercules Powder Co. for the Government.

The furnace charge stock is natural gas and steam. The natural gas contains about 79 percent CH₄, 9.4 percent N₂, 5.9 percent C₂H₂, and 3.3 percent C₃H₈. The reaction takes place in the presence of steam and a catalyst, at a temperature of 1,700°F to 1,900°F, in the cracking furnaces. The mixed gas leaving the furnace contains about 60.1 percent H₂, 2.3 percent N₂, 4.6 percent CO₂, 23.6 percent CO, and 0.4 percent CH₄. It is cooled by steam to about 800°F before entering the CO-converters, where the CO is converted to H₂ and CO₂ in the presence of steam and a catalyst. The mixed gas leaving the converter contains about 77 percent H₂, 18 percent CO₂, and 1.5 percent CO. This gas passes through the mixed gas cooler to the 300,000 cubic foot gas holders for use in hydrogenation as required.

b. Hydrogen Compression

The hydrogen is compressed to 11,800 p.s.i. by one of the compressors formerly used in the Missouri Ordnance Works. Its availability, together with the hydrogen-manufacturing unit and power plant, contributed largely to the selection of this site for the demonstration plant.
The hyper is a 7-stage Ingersoll-Rand compressor. When operating at nominal capacity of 200-barrels-per-day aviation gasoline, 88,000 to 110,000 cubic feet per hour of 97 percent pure H₂ will be required in the liquid phase and 35,000 to 55,000 cubic feet per hour in the vapor phase, or a total of 123,000 to 165,000 cubic feet per hour, depending on the kind of coal used for hydrogenation. The compressor has a capacity of 210,000 cubic feet per hour cracked gas, or about 125,000 to 145,000 cubic feet per hour pure H₂. The operating pressure is 11,000 p.s.i.g., though it was originally designed for higher pressure.

The gas is scrubbed between the third and fourth stages. The scrubbing tower is designed to wash out 17.5 percent CO₂ when operating at capacity. Additional details concerning hydrogen production and compression should be obtained through the Hercules Powder Co. of Wilmington, Del.

c. Hydrogen Recirculation

Hydrogen is required in the hydrogenation process for four distinct purposes:

1. For the hydrogenation of the coal and oil feeds. This is the make-up H₂ produced in the H₂-cracking furnaces.

2. To promote the flow of the feed stock through the preheater and converter.

3. To take away part of the heat of reaction in the converters and offer a means of effecting temperature control in these vessels.

4. To maintain an H₂ partial pressure of at least 75 percent of the total pressure.

To perform these functions, an approximate 1:5 make-up to recycle hydrogen ratio is required. There are four recycle compressors, two each for the liquid and vapor phase. They are so interconnected that any one or all of them can serve either phase. Although the capacity of each compressor is ample to recirculate all the hydrogen required for either the liquid- or vapor-phase system, the continuity of supply is so important that it is intended to operate both compressors of any one of the two systems at the same time at reduced rates.

These are Ingersoll-Rand type AL, single-stage, double-acting compressors. The cylinder dimensions are 3-3/4-inch and 3-3/4-inch bore by 6-3/4-inch stroke. Belt-driven at 150 r.p.m., they are capable of recirculating 400,000 c.f.h. of H₂ at a stall pressure of 10,300 p.s.i.g. Two are driven by 150 h.p., 600-r.p.m. induction motors, whereas the other two are driven by 150-h.p., Westinghouse type C-1/4, impulse, steam turbines at 4,000 r.p.m. with a gear reduction to 600 r.p.m. A by-pass is provided from the compressor discharge to the suction line, which will be opened automatically by a regulating valve if the differential pressure between discharge and suction exceeds 750 p.s.i.g.
NITROGEN GAS PRODUCTION AND COMPRESSION

a. Nitrogen Production

The nitrogen production may be subdivided into two categories - low and high pressure. The low-pressure nitrogen is used for general blanketing and purging in the coal-paste preparation and distillation sections; the high pressure, for purging the hydrogenation section.

The low-pressure nitrogen or inert flue gas is taken from the stack of the waste-heat boiler in the hydrogen-production unit. The products of combustion, mostly $N_2$ and $CO_2$ from the $H_2$-cracking furnaces, give up most of their heat in the waste-heat boiler before passing up the stack. The stack is tapped about 20 feet above the base by a 10-inch-diameter pipe, which takes some of this inert gas mixture through a water cooler to the $N_2$ compressor inlet.

The high-pressure nitrogen must not contain over 0.5 percent $O_2$ and more than 5 percent combustible gases; therefore, the inert gas used in the low-pressure system cannot be used for the high-pressure work. The high-pressure nitrogen is generated from natural gas in a unit furnished by the C. M. Kemp Manufacturing Co. of Baltimore, Md. This unit has a capacity of 15,000 c.f.h. at 15-inch water gage, of an inert gas containing less than 0.6 percent $O_2$, about 11 percent $CO_2$, and 89 percent $N_2$. A 10 by 10 Roots-Comersville blower of 560 cu. ft. per minute capacity, discharging at 5-1/2 p.s.i., transfers this gas from the producer to a 50,000 cu. ft. inert-gas holder, from which it may be drawn for use in either the high- or low-pressure $N_2$ systems.

b. Nitrogen Compression

The low-pressure nitrogen compressor is a conventional, steam-driven, simplex compressor discharging at about 30 p.s.i. through a receiver and separator to the low-pressure $N_2$ piping system.

The high-pressure nitrogen compressor is a 6-stage, Worthington ODC-6, horizontal, duplex, opposed-type machine. It has a capacity of 15,000 c.f.h. at 14,000 p.s.i. There is an intercooler between each stage, and the $CO_2$ is scrubbed out between the 3rd and 4th stages. The demand on this compressor will be irregular and intermittent. To regulate the quantity of nitrogen fed into the system the compressor is provided with an automatic Hammel-Dahl regulating valve, which controls the pressure between 11,000 and 14,400 p.s.i. and regulates the by-pass from the discharge to the suction.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Bore, inches</th>
<th>Stroke, inches</th>
<th>Type</th>
<th>Approximate discharge pressure, p.s.i.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>12</td>
<td>Double-acting</td>
<td>40</td>
</tr>
<tr>
<td>2</td>
<td>8-1/4</td>
<td>12</td>
<td>Single-acting</td>
<td>175</td>
</tr>
<tr>
<td>3</td>
<td>4-1/4</td>
<td>12</td>
<td>do.</td>
<td>550</td>
</tr>
<tr>
<td>4</td>
<td>2-5/8</td>
<td>10</td>
<td>do.</td>
<td>1,935</td>
</tr>
<tr>
<td>5</td>
<td>1-7/16</td>
<td>10</td>
<td>do.</td>
<td>5,785</td>
</tr>
<tr>
<td>6</td>
<td>7/8</td>
<td>10</td>
<td>do.</td>
<td>14,400</td>
</tr>
</tbody>
</table>
Figure 21. - Paste injection pump.
The compressor drive is a 250-h.p., 300-r.p.m., synchronous motor with flywheel direct-connected to the compressor crankshaft.

The high-pressure nitrogen-purging system is tied in with the high-pressure flushing-oil system, so that both use V-35 as a combined accumulator in which the nitrogen forms a cushion on top of the oil and maintains the pressure at 11,000 p.s.i.g.

PUMPS

More than 80 pumps are built into the demonstration plant. Most of these are low-pressure, conventional, reciprocating or centrifugal pumps and require no explanation. However, some operate at 10,300 p.s.i., or are designed for special services, and these will be described in some detail.

a. Coal Paste Transfer Pumps P-6A, B, and C

A mixture containing 53 percent heavy oil and 47 percent solids, consisting of finely pulverized coal ash and catalyst forms the coal paste. This is a very viscous mixture of 1,285 centipoises at atmospheric pressure and 210° F. and a specific gravity of 1.2 at 60° F. The paste is pumped from storage tank T-26 in the preparation building to the suction side of the injection pumps by transfer pumps 6A, B, and C. These transfer pumps are 2-stage, type B6-6, Robbins & Myers "Myno" pumps with a capacity of 26 g.p.m. at 210° F. and 150 p.s.i. discharge. They are driven by 7-1/2 h.p. gear motors at 640 r.p.m. The "Myno" pump is a positive displacement pump with a hardened tool-steel helical screw rotating in a double helical-thread, cast-iron stator. Both inlet and outlet are 2-1/2 inches diameter. Two of the pumps will be operated continuously, one acting as a spare. To control the quantity delivered to the injection pumps, an automatic, pressure-controlled by-pass is provided from the discharge to the suction line.

b. Coal-Paste Injection Pumps P-7A, B, and C

The severe service conditions of these pumps place them in the critical category. Their function is to inject the coal paste or pasting oil into and force it through the liquid-phase preheater and converters. This paste has a viscosity of 4,750 centipoises at the 200° F. pumping temperature and 10,300 p.s.i.g. discharge pressure. Two pumps operate continuously, and one is a spare. They are 24- by 2-3/4-inch bore, 18-inch stroke, double-acting duplex plunger pumps with forged-steel plungers and pump body. They are designed for a capacity of 25 g.p.m. at 14.6 r.p.m. with a suction of 50 p.c.i. and 10,300 p.s.i. discharge at a pumping temperature of 200° F. The steam inlet pressure is 250 p.s.i. with a 50 p.s.i. exhaust. The hydraulic horsepower developed is 150, and the hourly steam consumption is 17,250 pounds. The plungers, valves, and valve seats are of 12 percent chrome steel, Allegheny L-12, hardened to 450 Brinell. The plunger stuffing box is 8-1/8 inches deep and is provided with a flushing oil connection. The packing is Garlock chevron and consists of one ring each of braided copper wire and braided cotton followed by a bronze lantern ring, bronze base ring, four chevron rings of special cotton duck impregnated with oil-resisting "Neoprene" compound, four bronze spacer rings, and a bronze adapter ring. The steam pump was chosen for this service because of its characteristic cushioned, steady operation and easy speed or rate of feed regulation. Figure 21 shows a paste-injection pump.
c. Heavy-Oil-Let-Down Recycle Pump P-9

The H.O.L.D. or heavy-oil-let-down recycle pump takes 10 to 25 percent of the liquid hot catchpot bottoms containing 20 to 35 percent solids, consisting of ash, catalyst, and unconverted coal particles, and injects it into the paste stream just ahead of the second liquid-phase preheater cell for recycling in the liquid-phase process. The recycled product has a temperature of about 750°F, a viscosity of about 21 centipoises at this temperature, and a specific gravity of about 1.24 at 60°F. The main purpose of recycling heavy oil let-down is to control or prevent the swelling of certain bituminous coals in the preheater and, in turn, control tube temperature for safer operation. Another reason is to return the heavy-oil residue, rich in solids, from the bottom of the hot catchpot to the converters for reprocessing without much loss of heat and pressure.

The H.O.L.D. recirculator is a surge pump with the valve block separated from the pump proper, so that the hot oil does not pass through the pump. The pump is connected to the valve block by two 2-inch pipe lines. In these lines, cool flushing oil surges back and forth to open and close the hot-oil valves in the valve block. The arrangement is shown schematically in figure 22. The pump is a simplex, double-acting, Burnham, forged-steel plunger pump with hardened stainless-steel plungers. It is steam-driven, has 8-inch and 2-1/2-inch bore, and 12-inch stroke. The valve block is a 12-percent chrome-steel forging with 12 percent chrome-steel valve disks and seats, 2-1/2-inch diameter hot-oil inlet, and 2-inch diameter discharge. The pump is designed for a capacity of 5 g.p.m. at 9,500 p.s.i. inlet and 10,300 p.s.i. discharge pressure. Union Steam Co. of Battle Creek, Mich., is the supplier.

d. Oil, Naphtha, and Water Injection Pumps

With minor exceptions, these pumps, which include P-10, P-21, P-22, P-23, P-502 and P-504, are similar to P-7. All except one are reciprocating, duplex, steam pumps furnished by Union Steam Pump Co. of Battle Creek, Mich. The operating and performance features of these pumps are listed in tabular form in figure 23.

P-10B is an Aldrich-Groff, vertical, triplex, controllable-capacity pump with 5/8-inch diameter plungers and stroke adjustable up to 4 inches, herringbone gear-driven at 310 r.p.m. by a 40-h.p., 1,750 r.p.m. motor. The plunger packing is a 6-ring "Teflon", 1-3/8-inch outside diameter made by U.S. Gasket Co., Camden, N.J., with bronze spacer rings and a lantern ring for 1/4-inch lubricating-oil connection. The cylinder block is solid-steel forging of chrome-nickel-molybdenum steel heat-treated to an ultimate tensile strength of 156,000 p.s.i. and a yield point of 119,000 p.s.i. The plungers, valves, and valve seats are of S.A.E. 52 100 steel, normally used in ball bearings for resistance to abrasion.

The unusual feature of this pump is its adjustable stroke, which automatically controls its capacity from 0 to maximum, by an air-operated control valve. This pump was primarily selected to prove its practicability at 10,000 p.s.i. and to gain operating experience. It was felt that in commercial-size operation, a battery of fixed-stroke pumps could work at fairly constant pressure, and a sensitive adjustable-stroke pump could take up the minor variations in feed.
Figure 22. - H. O. L. D. circulating system.
<table>
<thead>
<tr>
<th>PUMP NO.</th>
<th>SERVICE</th>
<th>TYPE &amp; SIZE</th>
<th>CAPACITY &amp; SPEED</th>
<th>HYDRAULIC</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>P-7A &amp; 6</td>
<td>Coal Paste Injection</td>
<td>Duplex, Double-acting, 24&quot; x 2-3/4&quot; x 18&quot;</td>
<td>25 G.P.M. 14.6 R.P.M.</td>
<td>650</td>
<td>1. Paste has 4% solids with viscosity at 4750 centipoises at pumping temperature of 200°F. 2. 65 p.s.i. suction and 10,500 p.s.i. discharge. 3. 50 lb. of Garlock special chervon packing in an 8-1/8&quot; deep stuffing box connected to flushing oil system. 4. 115 lbs./D.H.P. hr. of steam at 250 p.s.i. inlet and 50 p.s.i. exhaust. 5. 12% chrome hardened valves and seats.</td>
</tr>
<tr>
<td>P-21A &amp; B</td>
<td>Wash Oil Injection</td>
<td>Duplex, Double-acting, 24&quot; x 2-3/4&quot; x 18&quot;</td>
<td>34 G.P.M. 19 R.P.M.</td>
<td>203</td>
<td>1. Wash oil has viscosity of 12.6 centipoises at 120°F; pumping temperature. 2. 65 p.s.i. suction and 10,500 p.s.i. discharge. 3. 6 rings of Garlock special chervon packing in an 8-1/8&quot; deep stuffing box. 4. 115 lbs./D.H.P. hr. of steam at 250 p.s.i. inlet and 50 p.s.i. exhaust. 5. 12% chrome hardened seats and valves.</td>
</tr>
<tr>
<td>P-502A &amp; B</td>
<td>Vapor-phase feed Injection</td>
<td>Duplex, Double-acting, 24&quot; x 2-3/4&quot; x 18&quot;</td>
<td>21 G.P.M. 14.6 R.P.M.</td>
<td>126</td>
<td>1. Vapor-phase feed stock has viscosity of 55.4 centipoises at 120°F; pumping temperature. 2. 65 p.s.i. suction and 10,500 p.s.i. discharge. 3. 6 rings of Garlock special chervon packing in an 8-1/8&quot; deep stuffing box. 4. 115 lbs./D.H.P. hr. of steam at 250 p.s.i. inlet and 50 p.s.i. exhaust. 5. 12% chrome hardened seats and valves.</td>
</tr>
<tr>
<td>P-25A &amp; B</td>
<td>Wash oil water Injection for Liquid-phase</td>
<td>Duplex, Double-acting, 18&quot; x 21&quot; x 12&quot;</td>
<td>12 G.P.M. 20 R.P.M.</td>
<td>72</td>
<td>1. Viscosity of wash water = 0.56 centipoises @ 120°F; pumping temperature. 2. 6 rings of Garlock special chervon packing in a 7-1/4&quot; deep stuffing box. 3. 65 p.s.i. suction and 10,500 p.s.i. discharge. 4. 115 lbs./D.H.P. hr. of steam at 250 p.s.i. inlet and 50 p.s.i. exhaust. 5. 12% chrome hardened seats and valves.</td>
</tr>
<tr>
<td>P-504</td>
<td>Vapor-phase water Injection</td>
<td>Duplex, Double-acting, 18&quot; x 21&quot; x 12&quot;</td>
<td>12 G.P.M. 20 R.P.M.</td>
<td>72</td>
<td>1. Viscosity of water = 0.9 centipoises at 120°F; pumping temperature. 2. 6 rings of Garlock special chervon packing in a 7-1/4&quot; deep stuffing box. 3. 65 p.s.i. suction and 10,500 p.s.i. discharge. 4. 115 lbs./D.H.P. hr. of steam at 250 p.s.i. inlet and 50 p.s.i. exhaust. 5. 12% chrome hardened valves and seats.</td>
</tr>
<tr>
<td>P-10A</td>
<td>Flushing oil Injection</td>
<td>Duplex, Double-acting, 12&quot; x 1-1/8&quot; x 12&quot;</td>
<td>4 G.P.M. 20 R.P.M.</td>
<td>25.4</td>
<td>1. Flushing oil has viscosity of 50 centipoises at 250°F; pumping temperature. 2. 6 rings of Garlock special chervon packing in a 7-1/4&quot; deep stuffing box. 3. 65 p.s.i. suction and 10,900 p.s.i. discharge. 4. 145 lbs./D.H.P. hr. of steam at 250 p.s.i. and 50 p.s.i. discharge. 5. 12% hardened chrome valves and seats.</td>
</tr>
<tr>
<td>P-25A &amp; B</td>
<td>Naphtha Injection</td>
<td>Duplex, Double-acting, 12&quot; x 1-1/8&quot; x 12&quot;</td>
<td>4 G.P.M. 20 R.P.M.</td>
<td>25.5</td>
<td>1. Naphtha has a viscosity of 4 centipoises at 120°F; pumping temperature. 2. 6 rings of Garlock special chervon packing in a 7-1/4&quot; deep stuffing box. 3. 65 p.s.i. suction and 10,500 p.s.i. discharge. 4. 145 lbs./D.H.P. hr. of steam at 250 p.s.i. and 50 p.s.i. discharge. 5. 12% hardened chrome valves and seats.</td>
</tr>
<tr>
<td>P-22A &amp; B</td>
<td>Lettead water Injection</td>
<td>Duplex, Double-acting, 12&quot; x 1-1/8&quot; x 12&quot;</td>
<td>4 G.P.M. 20 R.P.M.</td>
<td>25.5</td>
<td>1. Viscosity of letted water = 50 centipoises at 120°F; pumping temperature. 2. 6 rings of Garlock special chervon packing in a 7-1/4&quot; deep stuffing box. 3. 65 p.s.i. suction and 10,500 p.s.i. discharge. 4. 145 lbs./D.H.P. hr. of steam at 250 p.s.i. and 50 p.s.i. discharge. 5. 12% hardened chrome valves and seats.</td>
</tr>
</tbody>
</table>

Figure 23. - High-pressure pump tabulation.
Figure 24. - Graph of pipe stress vs. \( \frac{OD}{ID} \) ratio.
HIGH-PRESSURE (10,300 p.s.i.) TUBING, FLANGES, FITTINGS AND VALVES

Design and tentative standardization of the 10,300 p.s.i. working pressure tubing presented numerous problems. In selecting the standard tube sizes, the corresponding wall thicknesses, and suitable alloy compositions, consideration had to be given to process-flow requirements, prevailing tube-manufacturing practices, permissible working stresses at moderate temperature, creep stresses at high temperatures, and to the effects of corrosion, erosion, and hydrogen attack.

Considering the maximum allowable stress, it was quickly realized that many theories are offered to explain the failure of thick-walled cylinders from excessive internal pressure, and that they give widely different results at the 10,000 p.s.i. design level. The maximum principal stress theory in which the tangential stress alone is considered proved to be the most suitable selection. The A.S.A. code for pressure piping adopts this theory and in paragraph 122B

\[
\tau_m = \frac{D}{2} \left( 1 - \frac{S - P}{S} \right)^{1/2} C
\]

where \( \tau_m = \) Minimum pipe-wall thickness, in inches, not including manufacturing tolerances

\( P = \) Maximum internal service pressure, in p.s.i.

\( D = \) Outside diameter, in inches

\( S = \) Allowable stress in material due to internal pressure at the operating temperature, in p.s.i.

\( C = \) Allowance for threading and corrosion, in inches

A study of equation "i" revealed how various factors affect pipe-wall thickness. By dropping the constant "c" and rearranging the equation, it becomes \( \frac{D}{d} = \sqrt{\frac{S - P}{S}} \), where \( \frac{D}{d} \) = theoretical ratio of outside diameter to inside diameter. Plotting \( \frac{D}{d} \) against allowable stress for the internal pressure of 10,300 p.s.i., the curve shown in figure 24 is obtained. Several significant things are evident from this curve. As the allowable stress approaches 10,300 p.s.i., the \( \frac{D}{d} \) ratio approaches infinity. It is also apparent that at relatively low stress values, small increments cause large changes in the \( \frac{D}{d} \) ratio. The rate of decrease diminishes as the allowable stress increases.

Some idea of weight savings achieved by selecting A.P.I. 5L, grade-C steel rather than more commonly used pipe steels can be obtained by comparing it with A.S.T.M.-A106, grade-B, carbon steel, for which the A.S.A. code of pressure piping stipulates an allowable stress of 18,000 p.s.i. at 375° F. A.P.I. 5L has an allowable stress of 22,900 p.s.i. at the same temperature. The \( \frac{D}{d} \) ratio, including corrosion allowance, for a 2-1/2-inch inside-diameter pipe
made of A106 steel becomes 2.1 and would weigh about 57 pounds per foot. The \( \frac{D}{d} \) ratio for A.P.I. 5L becomes 1.76 and would weigh about 34 pounds per foot.

This theoretical weight saving of 23 pounds per foot, though significant, is not the complete story. Owing to manufacturing tolerances, A106 pipe would actually have a specified wall thickness of 1.875 inches and weigh about 88 pounds per foot. The 2-1/2-inch inside diameter A.P.I. 5L pipe has a wall thickness of 1.169 inches and weighs about 45 pounds per foot. It is apparent that a saving of about 43 pounds per foot was realized by using the higher-strength steel.

Returning to the demonstration-plant tube standards, the pipe ends were threaded for flanging. There are eight American-standard straight threads per inch, and a corresponding allowance of 0.080 inch was made in the wall thickness. With an additional corrosion or erosion allowance of 0.031, the "C" value became 0.111 inch for all pipe sizes having eight threads per inch.

It was contemplated that some of the seamless steel tubing will be finished by the hot-rolling method, and the following tolerances presented by manufacturers had to be considered in calculating the wall thicknesses:

(a) \(-1/32 \leq \frac{d}{D} \leq 1/32\) on sizes below 1-1/2-inch outside diameter.

(b) \(-1/32 \leq \frac{d}{D} \leq 1/32\) on sizes above 1-1/2-inch outside diameter.

(c) \(-\) 12-1/2 percent tolerance on the wall thickness for all sizes.

Applying the above equation "C" factor and adding the proper tolerances, tubing dimensions were established as shown in figure 23. Following German precedents, three different temperature groups were set up for the high-pressure tubing materials.

- Low-temperature service up to 375\(^\circ\) F.
- Medium-temperature service, 376\(^\circ\) to 850\(^\circ\) F.
- High-temperature service, 851\(^\circ\) to 1,000\(^\circ\) F.

It is apparent from the above that the material for the three temperature groups was selected in such manner that, for the sake of standardization, tubing, fittings, etc. would have the same dimensions in all cases. Obviously, the dimensions were chosen for the lowest permissible stress from the three groups, and the stronger materials are thicker than necessary. It turned out that dimensions were finally based upon the properties of A.P.I. 5L, grade C, carbon-steel seamless tubing. According to the A.S.A. code of pressure piping, this material has an allowable stress of 22,900 p.s.i. at 375\(^\circ\) F. temperature.

Other reasons in favor of uniform dimensions for all temperature ranges were:

- Creloy 9M had a higher strength than carbon steel, but its behavior at temperatures near 850\(^\circ\) F. were not known well enough to warrant taking full advantage of its strength.
Though it would appear that safety might be sacrificed by using these dimensions for stainless-steel pipe at temperatures around 1,000° F., the outside diameter to inside diameter ratio of 1.95 was about the optimum, according to German experience. Thicker walls could be expected to give trouble owing to undesirable stresses and because of greater likelihood of internal faults in thick-walled sections.

Specifications of materials selected for the three temperature groups of the demonstration plant are as follows:

**Material Specifications for Piping, Fittings, Valves, etc.**

**Low-temperature service, 0° to 375° F.:**

**Tubing (early design) -** Carbon steel, B&W, MT-1040, equal to A.P.I. 5L, grade C, Y.P. 45,000 p.s.i., U.T.S. 75,000 p.s.i., Brinell hardness 159 min.

**Tubing (new design) -** Carbon steel S.A.E.-4130, Y.P. 60,000 p.s.i., U.T.S. 90,000 p.s.i., Brinell hardness 180 min.

**Fittings -** Carbon steel, S.A.E.-1030, Y.P. 45,000 p.s.i., U.T.S. 75,000 p.s.i., Brinell hardness 163 min.

**Flanges -** Carbon steel, A.S.T.M.-A-105, grade I, Y.P. 36,000 p.s.i., U.T.S. 70,000 p.s.i., Brinell hardness 136 min.

**Blind flanges -** Same as flanges.


**Valves -** Carbon steel bodies, S.A.E.-1030, Y.P. 45,000 p.s.i., U.T.S. 75,000 p.s.i., Brinell hardness 163 min.

**Bolts -** A.S.T.M.-A-193-44, grade B-7, (S.A.E.-4140), min. draw temp. 1100° F. min., Y.P. 105,000 p.s.i. min, U.T.S. 125,000 p.s.i.

**Nuts -** A.S.T.M.-A-194-40 Class 2-h, heavy series.

**Medium temperature, 376° to 850° F.:**


**Fittings -** Type 304 18-8 stainless steel, Y.P. 30,000 p.s.i., min., U.T.S. 75,000 p.s.i., A.S.T.M.-A-182, Gr. F-8, Brinell hardness maximum attainable.
Flanges:

On tubing  

On fittings and valves  
- Alloy steel, A.I.S.I. Type 304, A.S.T.M.-A-182, Gr. F-8, Y.P. 30,000 p.s.i. min, U.T.S. 75,000 p.s.i. min., Brinell hardness 200 max.

Blind flanges  
- Same as flanges on fittings and valves.

Lens gaskets  
- Type 405 chrome steel, Y.P. 40,900 p.s.i., U.T.S. 64,160 p.s.i. annealed, Brinell hardness 121 min., 130 max.

Valves  
- Alloy steel bodies, A.S.T.M.-A-182-44, Type F-8m (A.I.S.I. Type 316), Y.P. 30,000 p.s.i., U.T.S. 75,000 p.s.i., Brinell hardness 190 max.

Bolts  

Nuts  

High-temperature service, 851° to 1000°F:

Tubing  

Fittings  
- A.S.T.M.-A-182-44, Type F-8m (A.I.S.I. Type 316), Y.P. 30,000 p.s.i., U.T.S. 75,000 p.s.i., Brinell hardness maximum attainable.

Flanges  

Blind flanges  
- Same as flanges.

Lens gaskets  
- Type 405 chrome steel, Y.P. 40,900 p.s.i., U.T.S. 64,160 p.s.i. annealed, Brinell hardness 121 min., 130 max.

Valves  
- Alloy steel bodies, A.S.T.M.-A-182-44, Type F-8m, (A.I.S.I. Type 316), Y.P. 30,000 p.s.i., U.T.S. 75,000 p.s.i., Brinell hardness 190 max.

Bolts  
- A.S.T.M.-A-193-44, Grade B-7, (S.A.E.-4140), min. draw temperature 1100°F, min. Y.P. 105,000 p.s.i. min. U.T.S. 125,000 p.s.i.

Nuts  
<table>
<thead>
<tr>
<th>NOM. SIZE</th>
<th>O.D.</th>
<th>I.D.</th>
<th>B</th>
<th>E</th>
<th>F</th>
<th>ALLOWABLE TOLERANCE ON O.D.</th>
<th>ON WALL THICKNESS</th>
</tr>
</thead>
<tbody>
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<td>3/8&quot;</td>
<td>1 1/32&quot;</td>
<td>0.357&quot;</td>
<td>1&quot;</td>
<td>1 1/4&quot;</td>
<td>0.75&quot;</td>
<td>-1/32&quot;+1/64&quot; ± 15 %</td>
<td></td>
</tr>
<tr>
<td>5/8&quot;</td>
<td>1 3/32&quot;</td>
<td>0.637&quot;</td>
<td>1 1/2&quot;</td>
<td>1 3/8&quot;</td>
<td>1.125&quot;</td>
<td>-1/32&quot;+1/64&quot; ± 12 1/2 %</td>
<td></td>
</tr>
<tr>
<td>1&quot;</td>
<td>2 9/32&quot;</td>
<td>1.039&quot;</td>
<td>2 1/4&quot;</td>
<td>1 3/4&quot;</td>
<td>1.75&quot;</td>
<td>± 1/32&quot; ± 12 1/2 %</td>
<td></td>
</tr>
<tr>
<td>1 1/2&quot;</td>
<td>3 1/32&quot;</td>
<td>1.461&quot;</td>
<td>3&quot;</td>
<td>2 1/4&quot;</td>
<td>2.35&quot;</td>
<td>± 1/32&quot; ± 12 1/2 %</td>
<td></td>
</tr>
<tr>
<td>2&quot;</td>
<td>4 1/32&quot;</td>
<td>2.021&quot;</td>
<td>4&quot;</td>
<td>2 7/8&quot;</td>
<td>3.125&quot;</td>
<td>± 1/32&quot; ± 12 1/2 %</td>
<td></td>
</tr>
<tr>
<td>2 1/2&quot;</td>
<td>4 25/32&quot;</td>
<td>2.443&quot;</td>
<td>4 3/4&quot;</td>
<td>3 1/2&quot;</td>
<td>3.75&quot;</td>
<td>+1/32&quot;-3/64&quot; ± 12 1/2 %</td>
<td></td>
</tr>
</tbody>
</table>

NOTES:

THREADS AMERICAN NATIONAL 8 THREADS PER INCH, CLASS 2 FIT.

SMOOTH MACHINE SURFACE MARKED $f$ TO BE USED ON ALL H.P. PIPING SPECS.

Figure 25. - High-pressure pipe standards.
Figure 26. - High-pressure tubing and gasket sections.
During the construction of the plant, primarily for better weldability, the low-temperature carbon-steel tubing was replaced by a low chromium-molybdenum steel designated the S.A.E.-4130.

The specifications shall be used with the attached design standards. Piping design data on figure 25 include nominal sizes, wall thickness, end finish, threads, and allowable tolerances.

All pipe ends are beveled for lens ring gaskets and threaded for flanged connections. Lengths of sections between flanges is limited only by considerations for handling and fabrication. Welded joints are used wherever practical, though held to a minimum on Croloy 9M. All connections to headers or equipment are flanged. Welding is done by the electric arc method in accordance with the A.S.M.E. code for fusion-welded pressure piping. No backing rings are used at welds, but adjoining pieces are aligned on the outside diameter and taper-bored for offsets owing to possible eccentricity of the bores. All carbon-steel tubing is preheated before welding to a minimum of 200° F. and stress-relieved without loss of preheat at 1,200° F. maximum for about 1 hour per inch of metal thickness and then cooled in still air. Croloy 9M tubing is preheated to 500° F., stress-relieved at 1,200° F. for 4 hours, and air-cooled. The heat treatment recommended for the type 316 preheater tubes with the Croloy 5 jacket consists of preheating to 300° to 400° F., stress-relieving at about 1,350° F. for 1 hour, followed by cooling in air. Electrodes used for welding the jacketed tubes were stabilized 25-20 Cr-Ni steel. Other welding rods are of the same or similar material as the base metal.

Fabricated pipe spools, with fittings, were hydrostatically tested to 15,450 p.s.i.

The liquid-phase preheater presented the most difficult tubing material problem on account of the 1,000° F. operating temperature where the tube steel must resist hydrogen penetration, causing carbide precipitation and embrittlement. The material selected is stainless steel, type 316, B&W Croloy 16-13-3. This alloy contains no stabilizing agent but was considered to be the best compromise, because it has a higher creep strength than the stabilized stainless steels. In order to keep the ratio of outside to inside diameter to not more than 2, it was necessary to use a maximum stress value approaching the creep strength at 1,000° F. Greater wall thickness would give unfavorable stress conditions. This is particularly true for stainless steel, because of its high coefficient of expansion and low thermal conductivity.

Croloy 9M was selected for the medium temperature range. This steel, when properly heat-treated, has excellent physical and corrosion-resistance properties up to 900° F. Although close control is necessary when welding, it was considered the optimum selection for services where temperature shocks might occur - e.g., in emergency let-down line, etc.

All high-pressure tubing was supplied by Babcock & Wilcox Co., Beaver Falls, Pa. It was at first intended to hot-roll the carbon steel and Croloy 9M tubing, but so much difficulty was had in holding specified tolerances of roundness, wall thickness, and concentricity of bore that it was finally decided to cold-draw all high-pressure tubing. Figure 26 shows, on the left, a Croloy 9-tube section obtained by the hot-rolling method and, at the right, a well-shaped cold-drawn tube.
For a commercial-size coal-hydrogenation plant, serious consideration will no doubt be given to the use of S.A.E.-4130 steel pipe, water-quenched and drawn to give a yield strength about equal to the 82,000 p.s.i. of the normalized Croloy 9M pipe now in use.

As manufacture of Croloy 9M piping has been completed for the demonstration plant, enough additional information has been obtained to indicate that an allowable stress of around 40,000 p.s.i. is entirely practicable. It is quite possible that both low- and medium-temperature piping could be designed using this stress. In that event, a 2-1/2-inch inside-diameter pipe would have a wall thickness of about 0.659 inch and would weigh about 22 pounds per foot. This would represent 50 percent reduction in weight. Of course, strength can not be increased by heat treatment at the expense of ductility beyond certain limits. Mill-tension test reports of Croloy 9M show a minimum of about 35 percent elongation in a 2-inch length. Probably as low as 25 percent would not be undesirable. This same figure should be obtainable in S.A.E.-4130 steel after it has been quenched and drawn to give ultimate and yield strengths equal to Croloy 9M. Difficulty could be expected in getting uniform results in the heat treatment of heavy sections of S.A.E.-4130, but the reduced wall thickness of higher design stresses may well eliminate this trouble.

The development of stainless-steel pipe with higher creep strength than that of the 316 type is quite possible. The American Rolling Mill Co. has developed a stainless steel called "Armco GT-45", with superior qualities for high-pressure and high-temperature work. Composition of this steel is 2.80 to 3.50 percent Cu to 15.75 to 16.75 percent Cr, 13.40 to 14.40 percent Ni, and 2.00 to 2.75 percent Mo, with small amounts of the stabilizing elements columbium and titanium. The comparable yield point and creep strength at 1,200° F. are claimed to be:

<table>
<thead>
<tr>
<th>At 1,200° F. and 0.1 percent elongation in 1,000 hours</th>
<th>B&amp;W 16-13-3</th>
<th>Armco GT-45</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield point, p.s.i.</td>
<td>22,600</td>
<td>27,000</td>
</tr>
<tr>
<td>Creep strength, p.s.i., 0.1 percent in 1,000 hours</td>
<td>8,200</td>
<td>24,000</td>
</tr>
</tbody>
</table>

It is apparent that where the design must be based on creep strength substantial savings in material could be effected by substitution of some such new material as this. Real savings, however, would depend on the comparative costs of the materials considered.

It is interesting to note that the most popular German steel in use for high-pressure, high-temperature tubing was a ferritic steel designated N10 by I.G. Farbenindustrie, with the following analysis:

C = 0.18 to 0.22 percent, lower limit for heat treatment, upper limit for welding.
Cr = 3.0 to 3.6 percent, lower limit for H2 resistance, upper limit for creep strength.
Mo = 0.5 to 0.75 percent, necessary for minimum mechanical properties.
V = 0.75 to 1.00 percent, necessary for creep strength.
I.G. FARBEN FLANGE & TUBE.

BUREAU OF MINES SPECIFICATIONS; TUBE, FLANGE & GASKET.

Figure 27. - High-pressure flanged joint.
MACHINE ONE \( \frac{1}{16} \)" RADIUS IDENTIFICATION GROOVE ON MEDIUM TEMPERATURE FLANGES.
MACHINE TWO \( \frac{1}{16} \)" RADIUS IDENTIFICATION GROOVE ON HIGH TEMPERATURE FLANGES.

<table>
<thead>
<tr>
<th>NOM. SIZE</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>K</th>
<th>M</th>
<th>NO. HOLES</th>
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<td>7/8&quot;</td>
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<td>10 3/16&quot;</td>
<td>7 7/16&quot;</td>
<td>1 1/2&quot;</td>
<td>6</td>
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</table>

NOTES:
TO BE USED ON ALL H.P. PIPING SPECS.
THREADS AMERICAN NATIONAL 8 THDS. PER INCH, CLASS 2 FIT.
HOLES TO BE EQUALLY SPACED & DRILLED.
ROUGH MACHINE ALL OVER.

Figure 28. - High-pressure flange standards.
G = 2R \sin 20°
G = R \times 0.684
\sin 20° = \frac{G}{2R}

<table>
<thead>
<tr>
<th>NOM. SIZE</th>
<th>INSIDE DIAMETER</th>
<th>I.D.</th>
<th>d</th>
<th>d₁</th>
<th>L</th>
<th>r</th>
<th>e</th>
<th>SPHER RAD.</th>
<th>CONTACT CIRCLE</th>
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<td>3.284&quot;</td>
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NOTES:

TO BE USED ON H.P. PIPING SPECS. "AA" & "DD"
GRIND OR POLISH, SURFACE MARKED
SMOOTH MACHINE SURFACE MARKED
ROUGH MACHINE ALL OVER.

Figure 29. - High-pressure solid-lens gasket standard.
<table>
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<th>NOM. SIZE</th>
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<th>O.D. D₁</th>
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<th>D</th>
<th>WIDTH S</th>
<th>S₁</th>
<th>SPHER. RAD. R</th>
<th>DIA. OF 20° C.S.K. ON PIPE</th>
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<td>3&quot;</td>
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<td>1½/8&quot;</td>
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<td>7.375&quot;</td>
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</tbody>
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NOTES:
SMOOTH MACHINE SURFACES MARKED /
GRIND OR POLISH SURFACES MARKED */
ROUGH MACHINE ALL OVER.
TO BE USED ON H.P. PIPING SPECS. "BB", "CC", "EE", & "FF."

Figure 30. - High-pressure bellows gasket standards.
\[ W = 0.3 \text{ to } 0.5 \text{ percent, necessary for creep strength.} \]
\[ Mn = 0.3 \text{ to } 0.5 \text{ percent.} \]
\[ Si = 0.4 \text{ percent.} \]

The ultimate tensile strength of this steel was reported to be 105,000 to 125,000 p.s.i.; elastic limit = 70,000 to 75,000 p.s.i.; creep strength at 1022 F. = 22,000 p.s.i. To obtain this creep strength, the steel was subjected to the following heat treatment: Heat to 1,920° F. and cool at 45° F. per minute in air through range of 1,470° and 1,110° F.

**Flanged Joints**

As flanged joints rated at 10,300 p.s.i. operating pressure are beyond the scope of existing A.S.A. standards, it became necessary to select a design and set up standards for this work. Fortunately, data on German-type flanges were available for guidance. A comparison was therefore made between the German type of joint, as exemplified in figure 27, and conventional American joints. The study resulted in the decision to use the German lens-ring type of joint for the following reasons:

1. The gasket seating surface is closer to the center line of the pipe, which tends to reduce bolt load and, consequently, flange thickness.

2. Internal pressure working on the inner surface of the lens ring tends to make the joint self-sealing, whereas on conventional American joints the internal pressure tends only to reduce the gasket seating surface unit pressure.

3. The hard, spherical-surfaced, lens ring bearing on the harder conical surface of the pipe end results in a very narrow contact width, so that high unit seating pressures are achieved with relatively low bolt loads. This also tends to decrease flange thickness. German information indicates that joint tightness can be achieved with a total bolt load only about 2-1/2 times the total fluid load based on the area within the lens seating surface.

4. The "ball and cone" seat allows some misalignment in erection without adverse effects.

Some idea of the weight reductions achieved by use of the 10,300 p.s.i. type lens joint can be obtained by comparing the weight of a flange for 2-1/2-inch nominal-size pipe with the weight of an A.S.A. 2,500-pound screwed flange for the same pipe size. Each weighs about 50 pounds. Figure 28 shows the high-pressure flange standards for all sizes used in the demonstration plant.

All high-pressure gaskets for flange connections are of the lens-ring type with spherical seating surfaces. The solid ring gasket shown on figure 29 is used for all pipe sizes in the low-temperature range and for the 3/8-inch and 5/8-inch sizes in the high-temperature ranges, also. Figure 30 shows the bellows lens gasket used for 1-inch size and above in the medium- and high-temperature range. Bellows lenses are not made for the 3/8-inch and 5/8-inch pipe sizes. It may be noted that all gaskets bear directly on the beveled pipe ends and not on the flanges, as is usual in moderate-pressure work, and that the inside diameter equals the inside diameter of the pipe. The bellows type gives greater flexibility required in expansion at high temperatures.
The bellows effect is obtained by cutting a radial groove on the inside of the semifinished gasket and drilling two small holes from the inside gasket face to the bottom of the groove. The gasket is then pressed together, leaving a small hollow space at the bottom of the groove, in which the pressure is equalized with the pressure in the tube by the two drilled holes. Figure 26 illustrates a solid lens ring, a bellows-type lens, and the conical tube end of the threaded pipe that contacts the lens gasket. As may be seen from the material specifications, the gasket material is softer than the tube on which it bears. As the pressure of the product in the tube is directly transmitted to the inner face of the gasket, it has a self-sealing tendency.

**Fittings**

All fittings are forgeries and are limited to tees, 90° ells, and reducers. The solid forged tee blanks are bored to the required inside diameter after forging, whereas ells are bored before bending. All medium- and high-temperature fittings carry permanent identification marks. Figures 31, 32, 33, and 34 illustrate the flanged-type fittings used for all temperature ranges. In the low-temperature range, welded manifolds were used where possible, and the corresponding welding-type fittings are shown on figures 35, 36, and 37. Figure 38 shows a typical 2-1/2-inch high-pressure line with flanges and fittings. All pipe fittings were furnished by the Hawley Forge Co., San Francisco, Calif. Return bends and ells for preheater and heat-exchanger coils were furnished by Tube-Turns, Inc., and Midwest Piping & Supply Co.

**Valves for Operation Above 10,000 p.s.i.**

Generally speaking, valves fall into these classifications:

- **Class I**: Shut-off valves.
- **Class II**: Check valves.
- **Class III**: Throttling valves.
- **Class IV**: Relief valves.

All of these are angle valves, except for a few vertical-lift check valves, which are the straight-through type. The smaller sizes of shut-off and throttling valves are direct hand-wheel operated and have screwed packing gland nuts, whereas the larger sizes, 1-1/2-inch and above, have bolted, flanged packing glands and are spur-gear and hand-wheel operated. Valves are so arranged that the pressure will be above the disk when closed.

The most intricate valve group in the plant is shown in figure 39. This figure shows the heavy-oil let-down system from the hot catchpot. The line from E-6 is divided into three parallel branches. The two outside streams are normally used to let down the hot catchpot heavy oil to either V-14 or V-26 from the 10,000 p.s.i. operating pressure to 100 p.s.i. These two lines are automatically controlled through the severe throttling-type diaphragm valves "A" by recording-level controller RLC-116, which controls the liquid level in the hot catchpot. The center stream is hand-controlled by severe throttling
STAMP MEDIUM TEMPERATURE FITTINGS THUS —
STAMP HIGH TEMPERATURE FITTINGS THUS —

SECTION "A-A"

PIPE I.D.

PIPE O.D.

BEVEL DETAIL

NOTE:
TO BE USED ON
ALL H.P. PIPING SPECS.

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<th>O. D.</th>
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<td>1 1/4&quot;</td>
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NOTES:
THREADS AMERICAN NATIONAL 8 THDS
PER INCH. CLASS 2 FIT.
SMOOTH MACHINE SURFACE MARKED "¢"
A" — THIS DIMENSION DESIGNATES SQ. BODY.

Figure 31. — High-pressure flanged tee.
STAMP MEDIUM TEMP. FITTINGS THUS—A
STAMP HIGH TEMP. FITTINGS THUS—B

NOTE: TO BE USED ON ALL H.P. PIPING SPECS.

ALL DIMENSIONS ARE IN INCHES.

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NOTES: THDS. AMERICAN NATIONAL STD. 8 THDS. PER INCH, CLASS 2 FIT, SMOOTH MACHINE. SURFACE MARKED "A"—THIS DIMENSION DESIGNATES SQ. BODY.

Figure 32. – High-pressure flanged reducing tee.
PIPE

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<th>O.D.</th>
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</tr>
<tr>
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<td>1.039&quot;</td>
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</tr>
<tr>
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<td>2.021&quot;</td>
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ELBOW

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| 5/8"      | 1 5/8" | 1 1/2" | 1 3/8" | 1.125" | 2 5/8" | 5" | 2 3/8"
| 1"        | 2 7/16" | 2 1/4" | 1 3/4" | 1.75" | 3 1/8" | 6 3/8" | 3 1/4" |
| 1 1/2"    | 3 3/16" | 3" | 2 1/4" | 2.35" | 3 7/8" | 7 3/4" | 3 7/8" |
| 2"        | 4 1/4" | 4" | 2 7/8" | 3.125" | 5" | 9 3/4" | 4 3/4" |
| 2 1/2"    | 5" | 4 3/4" | 3 1/2" | 3.75" | 6 1/8" | 11 3/4" | 5 5/8" |

NOTES: TO BE USED ON ALL H.P. PIPING SPECS.
THDS. AM. NATIONAL 8 THDS PER INCH, CLASS 2 FIT SMOOTH MACHINE SURFACE MARKED /

Figure 33. - High-pressure flanged 90° elbow.
REJECTER SIZES

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<th>Y</th>
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PIPE

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STANDARD REDUCER DIMS.

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NOTES: THREADS WILL BE AMER. NATIONAL 8 THDS. PER INCH, CLASS 2 FIT. SMOOTH MACHINE SURFACE MARKED X

Figure 34. - High-pressure flanged reducer.
NOTE:
FOR 1" & SMALLER USE 37½° STRAIGHT BEVEL.
FOR 1½" & LARGER USE 20° "U" BEVEL.

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NOTE:
WELDING TEE TO BE USED FOR PIPING SPECIFICATION "AA" & "DD"

Figure 35. - High-pressure welding tee.
NOTE:

WELDING TEE TO BE USED FOR PIPING SPECS. - "AA" & "DD"

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<td>2.021&quot;</td>
</tr>
<tr>
<td>2 ½&quot;</td>
<td>2.443&quot;</td>
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</tbody>
</table>

Figure 36. - High-pressure welding reducing tee.
**Note:** For 1" & smaller use 37 1/2° straight bevel. For 1 1/2" & larger use 20° "U" bevel.

<table>
<thead>
<tr>
<th>PIPE</th>
<th>ELBOW</th>
</tr>
</thead>
<tbody>
<tr>
<td>NOM. SIZE</td>
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<tr>
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<tr>
<td>5/8&quot;</td>
<td>0.637&quot;</td>
</tr>
<tr>
<td>1&quot;</td>
<td>1.039&quot;</td>
</tr>
<tr>
<td>1 1/2&quot;</td>
<td>1.461&quot;</td>
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<tr>
<td>2&quot;</td>
<td>2.021&quot;</td>
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<td>2 1/2&quot;</td>
<td>2.443&quot;</td>
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</tbody>
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*Figure 37. - High-pressure welding 90° ell.*
Figure 38. - High-pressure flanged ell and tee assembly.
Figure 39. - Let-down valve group.
Figure 40. - High-pressure shut-off valve assembly.
Figure 41. - 2-inch high-pressure shut-off valve.
Figure 42. - High-pressure vertical check valve.
valve No. 114 and is used only in an emergency. All three streams are so
valved that the heavy oil let-down can be diverted to either one of the two
let-down receivers. It may be noted that, because of the extremely severe
service of handling hot heavy oil containing solids and gases at 10,300 p.s.i.
around each one of the severe throttling valves, there are three shut-off
valves upstream and one downstream, numbered 1, 2, 3, and 4. To shut off any
one of the three streams the valves are closed in the following order: "A"
or 114, 4, 3, and 2. Valve 1 is left open, except when valve 3 must be replaced.
To start a stream, valves 4, 3, 2, and "A" or 114 are opened in the order named.

Shut-off Valves

Figure 40 shows a typical shut-off valve. The hard-metal insert on the
bearing surfaces of both seat and disk is characteristic of all high-pressure
valves. Stellite is used in this case. The Exelloy disk is integral with the
stem, and the seat is made removable. Note the self-sealing feature of seat-
ring closure, which permits easy disassembly for replacing valve seats. The
packing is 7-ring type 220 Duralmetallic with a Nitralloy base ring and gland.
Figure 41 shows a gear-operated 2-inch shut-off valve with a 2-inch, 150 p.s.i.,
screwed valve placed on the gear for comparison.

Shut-off and check valves are shop-tested hydrostatically to 15,450
p.s.i.g. and with nitrogen under water at 100 p.s.i.g.

Check Valves

Both angle and straight-through type check valves are used. The angle
piston-type check valve is built and functions somewhat like a spring-loaded
relief valve. Seat and disk are Stellite-faced. The seat in this valve is not
removable. The Exelloy disk is integral with the stem and is held against the
seat by a small stainless-steel spring. The spring space behind the stem is
connected with the pressure space by a drilled hole for pressure equalization.
This type of valve is used for gases or liquids free of solids.

The straight-way, vertical-lift, check valve is of a novel design and is
shown in figure 42. It is used for slurries or liquids containing solids. It
functions like a ball-check valve, but the hollow disk is "tear-drop" shaped
and is guided in its vertical movement by three vanes 120° apart and looks like
a small, streamlined, aerial bomb. The guide vanes, their guiding surface, and
the disk and seat are Stellite-faced on their contact surfaces. The seat is
made removable.

Throttling Valves

The severe throttling valve is the most unusual. This valve is used at
the critical pressure let-down services, where the 10,300 p.s.i. operating
pressure of the liquid, solid, and gas mixtures of the hydrogenation system
must be reduced to low or moderate pressures in the distillation section or
H.O.I.D. storage. In case of an emergency let-down, pressure is reduced to
atmospheric. The absorbed gases expand 700-fold, increasing the velocity of
the whole mixture, and the abrasive effect of solids is highly intensified.
The severe throttling valve is built in two forms: hand-operated or automatic-diaphragm type. The hand-controlled type is shown in figure 43. In construction, it is similar to the German "Patzlennventil" or cartridge valve. As the name indicates, the target plate is removable, somewhat like a rifle cartridge. Parts subjected to severe abrasion are made of hardened material: Kennametal disk and seat insert, Excolloy disk holder hardened to 400 Brinell, and Stellite-lined target plate. The removable cartridge disk is attached to the bottom of the valve stem by the screwed-on disk holder and is backed up by a chrome-vanadium steel spring to protect the brittle Kennametal tip and seat from damage by excessive pressure applied to the stem. The lock nuts on the stem above the yoke bushing serve a similar purpose. The Kennametal outlet nozzle forming the seat is of the venturi type to minimize cavitation and erosion at high velocities. The outlet nozzle discharges into an expansion chamber, the removable bottom of which, called "target plate", is made integral with the outlet flange lens gasket. The jet from the outlet nozzle impinges upon the Stellite-lined, concave, spherical surface of the target and is discharged through the target outlet, drilled eccentrically to the target face. Most of the gas entrained in the liquid is liberated in this expansion chamber and eventually separated from it in the let-down vessel or receiver.

The hand-operated mild throttling valve used at less critical points is similar, with only slight differences in construction. It has no expansion chamber and outlet target, and the venturi-type outlet nozzle is not Kennametal-lined, though the seat is Kennametal-faced. The cartridge-type disk is also of Kennametal, but, instead of being backed-up by a spring, it is rigidly fastened to the bottom of the valve stem by the disk holder. All throttling valves are shop-tested hydrostatically to 15,450 p.s.i.g. and with nitrogen under water at 100 p.s.i.g.

Relief Valves

All high-pressure relief valves are of the spring-loaded type shown in figure 44. The removable Stellite-faced seat is similar to the mild throttling valve, except that the pressure is under the seat. The Stellite-tipped needle is integral with the stem, which extends through the spring and is guided near its lower and upper ends.

All relief-valve bodies are S.A.E.-1030 steel forgings. The valves are designed for a set pressure of 10,900 p.s.i.g. and 300°F. They are hydrostatically shop-tested at the set pressure and with nitrogen or helium under water at 100 p.s.i.g. The diaphragm control valves are furnished by Climax Engineering Co. of Tulsa, Okla. All other high-pressure valves were furnished by the Crane Co., Chicago, Ill.

High-Pressure Instrument Lines

All tubing for these services is 3/16-inch inside diameter by 9/16-inch outside diameter, type 304 stainless steel. All valves and fittings are stainless-steel cinch-joint type, straight-way needle stop valves and angle check valves, Autoclave Engineers 15,000 series 101 and 202, respectively. Tubing, valves, and fittings were furnished by Autoclave Engineers, Inc., Chicago, Ill., with one exception. The tubing from the hydrogen recycle lines to the hydrogen recording analyzers and specific gravity recorders is 1/16-inch
Figure 43. - High-pressure severe throttling valve.
SLOT FOR HOLDING STEM WHEN CHANGING PRESSURE ADJUSTMENT TO MAINTAIN TRUE SEAT BEARING

WELD

SPRING IS SHOWN COMPRESSED TO SET PRESSURE OF 10,900 P.S.I.

STELLITE FACED SEAT AND DISC.

DETAILS TO BE FURNISHED BY U.S. BUREAU OF MINES UPON AWARD OF BID.

\( \frac{1}{2} \)-8N-2

Figure 44. - High-pressure relief valve.
inside diameter by 1/4-inch outside diameter chrome-molybdenum steel tubing furnished by American Instrument Co., Silver Springs, Md. This smaller size was chosen for the service to obtain more pressure drop between the high-pressure hydrogen lines and the instruments.

LOW-PRESSURE AUXILIARY OPERATIONS

Auxiliary operations conducted at conventional pressure and temperature ranges presented few extraordinary problems. Therefore, a very brief summary will be presented in this paper, and only unusual features are described. The most important of these supplementary operations are coal preparation, paste preparation, heavy-oil centrifuging, flash distillation, product recovery, tankage, and power generation.

Coal Preparation and Pasting

The coal is received in hopper-bottom railroad cars. It is unloaded into a track hopper through an apron feeder and flight conveyor; it reaches a hammer mill, where it is crushed to 3/4-inch maximum; and is conveyed to an 80-ton storage bin by a bucket elevator. Coal handling and crushing is designed for 75 tons per hour operation; though the actual raw-coal consumption, based upon a nominal plant capacity of 200 barrels per day of gasoline plus byproducts, is only about 75 tons per day, varying somewhat with the kind of coal. The moisture- and ash-free coal consumption is about 53-1/2 tons per day.

From the crushed-coal storage bin the coal is conveyed to the pulverizer and dryer. This is a Kennedy-Van Saun 7-foot diameter, 9-foot long, horizontal ball mill with a capacity of 7-1/2 tons per hour of pulverized coal ground to 100 percent minus 60-mesh and not over 15 percent minus 200-mesh. The pulverizing is done in an inert or flue gas atmosphere at about 300°F., which reduces the moisture content to 1-1/2 percent. The dryer furnace is gas-fired and is designed for duty of 6,800,000 B.t.u. per hour. The hot gas flows through the pulverizer at such velocity that it carries the fines to a classifier, cyclone separator, and through a Redler conveyor into the 60-ton pulverized-coal storage bin.

It is considered detrimental to have a large percentage of minus 200-mesh coal; as this would increase the viscosity of the coal paste. It has also been found that some of the fines are carried over into the product, thereby increasing the solids content of the heavy oil and even the pasting oil from the centrifuges, all of which reduces the over-all efficiency of the hydrogenation process.

The pulverized coal from the 60-ton bin is mixed into a paste containing 47 percent solids, with pasting oil from tanks T-3 or T-4 and with some catalyst in the 820-gallon steam-jacketed batch mixer T-15. The amount of catalyst added may range between 0.2 and 3.0 percent of the coal, depending on the nature and composition of the coal as well as that of the catalyst. Provision is made to add Bayer messe, FeSO\(_4\)\(_3\), and Na\(_2\)S. The Bayer messe, which is the "red mud" residue from aluminum ores containing considerable Fe\(_2\)O\(_3\) and Si, and the FeSO\(_4\)\(_3\) are added separately in powdered form to the coal in T-15, whereas the Na\(_2\)S is added in a premixed slurry with pasting oil. The pasting
oil is a blend of heavy-oil let-down, centrifuge filtrate or flash distillate, and light-oil bottoms and contains about 11 percent solids. The mixed paste is discharged at about 210° F. into the 14,000-gallon, steam-heated, and agitated paste-feed storage tank, T-26, and it is kept in circulation by paste-transfer pumps, P-6A, B, or C. Paste-injection pumps P-7A, B, and C take suction from this recirculating line.

**Heavy-Oil Centrifuging and Flash Distillation**

The liquid-phase heavy oil in the bottom of the hot catchpot, V-5, is let down from 10,300 to 100 p.s.i. through the severe throttling valves to the 4-foot diameter by 6-foot-long, vertical let-down receivers, V-14 or V-26, where the gas entrained in the H.O.L.D. is liberated and conveyed to fuel surge drum V-727 in the distillation area to be used as fuel, whereas the heavy oil goes to the steam-heated and agitated storage tanks, T-10 or T-11. It may contain 20 to 35 percent by weight of solids, consisting of ash, catalyst, and unconverted coal particles. Its weight per cubic foot at 300° F. is 73.4 pounds, its viscosity at this temperature is 73 centipoise, initial boiling point 482° F., and softening point 53° F.

From T-10 or T-11, the H.O.L.D. is pumped at 300° F. to the steam-heated and agitated centrifuge feed-storage tanks, T-1 or T-2, where it is blended with light oil bottoms from distillation with a density of 0.965 and viscosity of 1.7 centipoise at 300° F. German experience has shown that the separation of solids in the centrifuges is facilitated by diluting the feed to a solids content of about 20 percent maximum. This mixture is then centrifuged in one of two centrifuges, CF-1A or CF-1B. CF-1A is a Sharples type H-2 vertical centrifuge driven by a 15-horsepower motor through a gear and worm drive at 6,000 r.p.m. spindle speed. CF-1B is a Bird 18-inch by 28-inch, continuous, horizontal centrifuge driven by a 10-horsepower motor and V-belt drive at 1,400 r.p.m. Both centrifuges are designed to separate 7.2 tons per 24 hours of solids from the feed, leaving a filtrate containing not over 5 percent solids and a concentrate of about 40 percent minimum solids when operating at a temperature of 300° F. The filtrate is mixed with H.O.L.D. and light-oil bottoms to make pasting oil, and the concentrate is wasted. German commercial practice was to extract the residual oil in the centrifuge concentrate in a low-temperature carbonization process and use the solids residual as boiler fuel. The Germans had also experimented with filtering the H.O.L.D. directly, instead of centrifuging, though not very successfully because of high viscosity and lack of suitable filter media.

Both centrifuges are in the paste-preparation building, together with the catalyst and paste-mixing equipment.

An alternate method to separate the solids from the H.O.L.D. and recover the oil is provided by flash distillation. In this case, the heavy oil from the bottom of the hot catchpot, V-5, is diverted to steam flash drum V-27. After pressure reduction, the H.O.L.D. is injected with superheated steam at 1,100° F. and 20 p.s.i. at a steam-to-oil ratio of 2:1 into the top of the drum. V-27 is a vertical drum 5 feet in diameter by 18 feet high. Here the oil is vaporized at about 750° F., and the vapor passes into the quench tower, V-18, whereas the liquid and solid constituents drop to the bottom of the flash drum.
Figure 45. - Distillation area, looking southwest.
The hot bottoms are pumped through extrusion nozzles and are solidified by cold-water sprays. The resulting pitch, containing about 70 percent by weight of solids, is collected in containers and hauled away as waste. In a large-scale plant this material could be utilized as fuel.

The steam and oil vapors are quenched in V-18 by recycled heavy-oil-distillate at about 180°F. The quench tower is 3 feet in diameter by 31 feet 9 inches high, has 19 trays, and is mounted on the top of a 6-foot-diameter by 12 foot 7-inch surge tank. The distillate is pumped from the surge tank to storage tanks T-5 and T-6 and from there to posting oil-blending tanks T-3 and T-4. The steam and noncondensable hydrocarbons are vented to the atmosphere through a 12-inch exhaust head.

**Distillation and Tankage**

Figure 2 is the process flow diagram of the complete distillation plant; figure 45 is a photographic view looking southwest, and the plot plan of the process area shows the relative location and extent of the distillation plant, product tankage, and pump house on the south side of the plot and heavy-oil pump house and tankage area east of the coal hydrogenation plant. The major parts of the distillation plant are the liquid-phase still, vapor-phase still, wash oil-distillation unit, gas-absorption system, gasoline stabilizer, caustic-treating plant, and the liquor-treating unit. The equipment is quite conventional and similar to that used in oil-refining practice. A detailed description is not considered necessary.

**Power Generation**

The total electrical-power demand in the hydrogenation demonstration plant, including gas generation and compression, is about 4,000 kw. The steam requirement for process or power, such as for reciprocating or turbine-driven pumps and compressors, is about 100,000 pounds per hour. Steam and power are generated at the former Missouri Ordnance Works power plant. There are three pulverized coal-fired Combustion Engineering steam generators each capable of producing 145,000 pounds steam per hour at 415 p.s.i., superheated to 750°F. The three 7,500-kw. steam turbo-generators operate at 400 p.s.i., 745°F., with a back-pressure of 50 p.s.i. Power is generated at 13,800 volts and distributed to the substations. The plant has its own service-water pumping and treating system.

**CONCLUSIONS**

During construction it was found that neither the average American manufacturer nor the construction forces realized fully the severity of the service in a coal-hydrogenation plant. After the design was completed in painstaking detail and reasonably severe specifications were drawn, repeated difficulties were experienced in their enforcement. The importance of careful supervision, inspection, and testing during every step of the fabrication and installation became evident. Standards of excellence applicable up to 2,500 p.s.i. service conditions were not necessarily good enough for 10,000 p.s.i. work. In numerous instances, flaws in materials, such as porous forgings and surface cracks, and unsatisfactory workmanship, particularly in welding, were not brought to light until after shop tests or even after delivery to the job site. In such cases it was not always easy to fix responsibility, and good judgment had to be exercised in correcting the shortcomings at a reasonable cost.
It became evident at an early stage that the demonstration-plant program was fulfilling several of its main functions. During the construction and break-in periods, many improvements in process and design were developed. In addition, manufacturers gained considerable knowledge in high-pressure design and fabrication methods - a long step toward preparing them for the conversion necessary in building full-scale plant equipment.

The second phase of the work will be the demonstration of the operation of an integrated plant producing synthetic liquid fuels from coal. During this period operating data will be collected, analyzed, and interpreted in order to determine the effects of operating variables upon the processes and products. This will point the way to further improvements and refinements. Studies will then be made of the economics of the process to determine the cost of products and the investment required for commercial-scale operation.

Finally, engineers and operators will be trained in the required skills of the new industry, and reports will be written for the purpose of disseminating all the technical and economic information developed in the demonstration-plant program. Thus, the road toward an economically sound synthetic-fuel industry must lead through the actual construction, operation, and improvement of demonstration and commercial-size plants.