3.0 RESULTS AND DISCUSSION

3.1 Design Basis

Four product yield cases were considered in designing the BFTR. The first two cases are directly scaled-up from the best laboratory runs with catalyst "A" (coprecipitated Fe/Cn/K) and catalyst "B" (cobalt carbonyl on zirconated alumina; see Task 2 report). In the remaining two cases, increased activity of these catalysts and attainment of water-gas shift equilibrium in the reactor were assumed. These latter cases were examined to determine how well the BFTR could handle improved catalyst formulations which are expected to result from laboratory reearch efforts. Design temperature and pressure match the optimum laboratory conditions of 482-500°F and 317-322 psia, respectively. The synthesis gas inlet velocity is 0.3 ft/sec.

3.2 Process Description

Figure 1 is a simplified process flowsheet of the BFTR, showing the 15 heat and material balance points selected for this design study. Portions of the flowsheet will be discussed in detail later in this report. The following summary description is provided to aid the reader in reviewing the heat and material balances.

3.2.1 Bubble-Column Reactor

Gas (stream 1) consisting of carbon monoxide and hydrogen at a molar CO/H_2 ratio of 1.55:1 to 1.98:1 is sparged into the base of the bubble-column reaction vessel, V-1, at 325 psia and ambient temperature. The feed gas is heated by the ba k-mixed molten slurry, providing part of the required reaction cooling duty.

The synthesis gas superficial velocity of 0.30 ft/sec produces a churn-turbulent flow regime. A thermosyphon loop around the reactor provides an upward slurry velocity of up to 0.1 ft/sec for most of

the vessel. Reactor V-1 is maintained at a constant operating temperature by means of the internal boiling water heat exchanger, E-101. The reactor expanded bed height is 30 feet with a slurry of roughly 20 wt% catalyst in a molten hydrocarbon wax, comprising primarily C_{20} -C_{35} alkanes, with 6.6 mole % of heavier components in the wax produced during the laboratory experiment that is the basis for case 1. At steady-state, the catalyst particles are in the 1-2 micron diameter size range.

3.2.2 Packed Column

Unreacted synthesis gas and the volatile reaction products leave the top of the reactor and enter the packed distillation column, V-201. This column is equivalent to four theoretical distillation trays. Its function is to improve the separation of wax and distillate, such that the C_{27}+ content of the product wax stream can be increased to 35-48 mole%. The vapor (stream 2) leaving the top of the packed column is cooled to 400-480°F in condenser E-301. The cooled stream leaving the condenser is phase separated in vessel V-301. All of the condensed liquid (stream 3) is refluxed to the packed column, while the uncondensed gas (stream 4) is sent to the water-cooled heat exchanger, E-102.

3.2.3 Distillate Product Recovery

The uncondensed gas is cooled to 100°F in Exchanger E-102 to condense most of the distillate product. In half the cases considered, the two-phase stream (5) leaving E-102 is further cooled to 42°F in the glycol-cooled exchanger E-202. The resulting two-phase stream 6 flows to the vapor-liquid separator, V-2, where phase separation occurs at reactor pressure. The gas (stream 7) is let down in pressure to 45 psia by the system pressure control valve. The liquid (stream 8) is also let-down from system pressure to 45 psia by a constant liquid level control valve in the

vapor-liquid separator. As the liquid is depressurized, dissolved gases and light hydrocarbon liquids flash from solution. This secondary product gas (stream 9) is combined with the primary product gas (stream 7) and sent to the product gas train for flow measurement and analysis. The flashed product liquid is collected in the distillate receiver, V-102, which is mounted on a load cell. The distillate product (stream 10) is withdrawn from this vessel at the end of each balance period. In cases 1 and 2, the distillate product is two-phase, with an oil phase and an aqueous phase. For continuous product collection, an oil/water separator would be installed in the distillate product line. This separator has not been included, since the material balance accuracy should be higher if all of the product distillate from the material balance period is collected as a batch and fractionated in the lab.

3.2.4 Wax Withdrawal

In the schematic flowsheet the hydrocarbon wax is separated from the catalyst by filtration. As will be discussed later in this report, the detailed flowsheet also provides gravity sedimentation vessels as an alternative means of separating the wax from the catalyst.

The filter, V-401, is a single-element candle filter. The reactor thermosyphon loop passes through the shell of the filter. This circulating slurry loop serves as the feed to the filter and also carries the filter cake back to the reactor. Molten wax (stream 11) is drawn through the candle at a controlled rate to maintain a constant slurry height in the reactor. On a timer sequence, a pulse of high-pressure syngas applied to the wax withdrawal line cracks the filter cake. The cake falls into the filter shell and is transported by the thermosyphon to the reactor. The ability to operate in this cyclic manner is a major advantage of the candle filter. Candle filters worked better than other filters tested with high solids content coal liquefaction slurries.

The filtered wax is let down in pressure across the filter candle and the flow control valve, such that the wax receiver, V-601, is operated at 45 psia. Any flashed gas (stream 12) is combined with the other product gas (streams 7 and 9). The wax receiver is mounted on a load cell and accumulates wax during an entire material balance period. At the end of each period, the product wax (stream 13) is drained from the receiver.

3.2.5 Reactor Temperature Control

The outer surfaces of reactor V-1, the packed column, the filter and the thermosyphon loop are heated to prevent heat loss. Thus, the entire reaction exotherm is removed predominantly by Heat Exchanger E-101 and partly by Condenser V-301. Exchanger E-101 is cooled by boiling water, whose temperature is set by the back pressure maintained on a water reservoir, V-101 (not shown in Figure 1). This pressure is adjusted by controlling the quantity of steam allowed to leave V-101. The overall rate of reaction can be assessed by measuring this steam flow rate. The flow of makeup water (stream 15) is controlled in order to maintain a fixed level in V-101.

3 3 Heat and Material Balance Procedures

Heat and material balances around the major process elements are required to translate the observed laboratory yields into flow rates and compositions of the product and intermediate streams and to properly size the equipment. These balances consist primarily of vapor-liquid equilibrium and enthalpy calculations, simulated using the distillation and flash blocks of Air Products' proprietary CYCSYN process simulation computer program. The thermodynamic calculations within CYCSYN were performed using Air Products' version of the Natural Gas Processors Association (NGPA) data base, using the Chao-Seader correlation. The NGPA and Air Products proprietary data

bases contain substantial experimental vapor-liquid equilibrium data on the components of interest.

In order to simplify the calculations, similar components were combined to reduce the number of chemical species to the 27-28 components listed in Table 1. Since pentacosane is the highest boiling alkane available on NGPA/CYCSYN, an 800-900°F fraction of Iranian crude was used to simulate the $C_{27}+$ wax components. This should be satisfactory because of the very low vapor pressure of these components at 500°F.

3.3.1 Method of Calculation

The first step in these calculations was to model the reactor, packed column, condenser, and condensate receiver as a distillation column of five theoretical plates, in which the total net product slate and unreacted synthesis gas were taken as feed to the reboiler. The wax product was withdrawn from the reboiler, and the condenser was operated at total reflux. The vapor leaving the condenser was sent to the water-cooled exchanger E-102 in the second half of the flowsheet. All of the C_5- light components were combined and treated as methane. This simplication does not significantly affect the accuracy of the material balance and is necessary, since the NGPA program cannot handle the concentration of hydrogen present at these conditions of temperature and pressure. Thus, the distillation calculation was performed on an 18-component system.

3.4 Product Yield Bases

Four product yield cases were considered in the BFTR design. The net yields (or consumptions) of the 28 components used in the process simulation are reported in Table 2. The yields for Cases 1 and 2 are based upon actual laboratory data (see Table 17), but cases 1A and 2A are hypothetical extrapolations from the laboratory data.

3.4.1 Case 1

Case 1 is based upon Run 7516-30-1.2 which used a 19.8 wt% slurry of catalyst "A" in a laboratory slurry reactor. A hydrogen conversion of about 74% was achieved in the laboratory reactor at reaction conditions of 500°F, 322 psia, and a CO/H_2 molar feed ratio of 1.55. Based upon the observed catalyst activity of 0.0312 pound-moles of syngas consumed per pound of catalyst per hour, the Deckwer computer model predicted a hydrogen conversion of about 43% for the BFTR bubble-column reactor. All product yields were scaled directly from the laboratory data.

Note that catalyst "A" has substantial water-gas shift activity. The water-gas shift equilibrium constant has a value of 74 at 500°F and is not a function of pressure.

$$K = 74 = \frac{(CO_2) \times (H_2)}{(H_2O) \times (CO)}$$

For case 1, this ratio is 5.5 at the exit from the bubble-column.

3.4.2 Case 1A

This product slate is based upon case 1. The reaction temperature and pressure for this case are identical to case 1, but the inlet CO/H_2 ratio was increased to equal the usage ratio of 1.78. The calculation assumes the activity of catalyst "A" will be sufficiently enhanced that hydrogen conversion in the bubble-column reactor will be increased to 90% and the reactor effluent will be in water-gas shift equilibrium.

The relative proportions of the various hydrocarbon components in the product are assumed to be the same as for case 1. The usage ratio and the yields of carbon dioxide, water, and total hydrocarbons were calculated from reaction stoichiometry, elemental balances and the water-gas shift equilibrium equation.

3.4.3 Case 2

Case 2 is based upon the laboratory results from Run 7077-75-C48.4-29, which used a 15.1 wt% slurry of catalyst "B." Closure of the elemental material balance for this experimental run was not sufficiently precise for this mathematical simulation. Therefore, the experimental data were used to determine the CO/H_2 usage ratio, the relative yields of the various hydrocarbon product species, and the catalyst activity. However, the reaction stoichiometry and elemental balances were used to calculate the absolute yields of total hydrocarbons, carbon dioxide, and water. Based upon the experimentally determined catalyst activity of 0.0306 pound-moles of syngas consumed per pound of catalyst per hour, the Deckwer model predicts a hydrogen conversion of about 49% for the BFTR bubble-column. This catalyst has no shift activity as can be seen from the value of 0.07 for the ratio

$$0.07 = \frac{(CO_2) \times (H_2)}{(H_2O) \times (CO)} << K_{eq} = 74$$

3.4.4 Case 2A

This product slate is based upon future improvement to catalyst "B" of case 2. It was assumed that the feed and usage ratios of CO/H_2 were the same, that the hydrogen conversion level was 90%, and that the reactor effluent was in shift equilibrium. The relative distribution of hydrocarbon products was the same as for case 2, but the reactor temperature was increased to 500°F to match case 1A. The usage ratio, and the yields of total hydrocarbons, carbon dioxide, and water were calculated. As in case 1A, the calculated usage ratio is 1.78.

3.4.5 Yield Comparisons

A comparison of the yields for cases 1 and 2 reveals that Catalyst "B," case 2, has significantly greater selectivity for the C_{10}-C_{20} intermediate boiling range products than does catalyst "A," Case 1. At the same time, Catalyst "B" produces considerably less wax and hydrocarbon gas. The quantity of C_{27}^{+} wax produced is only 40% as large as with catalyst "A." This is beneficial because it reduces the quantity of wax which must be separated and removed from the catalyst slurry in order to maintain a constant level in the reactor.

Further, catalyst "A" produces dimethylether, while catalyst "B" does not. In general, catalyst "A" makes much more oxygenates than catalyst "B" does; about half of these oxygenates are alcohols (mainly ethanol) and the other half dimethyl ether.

3.5 Heat and Material Balance Results

The heat and material balance points for the four product yield cases are presented in Tables 3-6. The material balance point numbers refer to locations on the flowsheet, Figure 1.

3.5.1 Flow Rates

Table 7 summarizes the major flow rates for the BFTR. In general, equipment was designed to handle the most demanding of the four cases. The sedimentation vessels are the exception. They were designed to handle case 1 as the extreme, since the very large wax flow rate for cases 1A and 2A would have resulted in much larger vessels. The large catalyst inventory in these sedimentation vessels would increase the time required to measure catalyst deactivation and cause substantial uncertainty and time variability in the quantity of catalyst in the reactor. The filtration system

is designed to handle the wax yield in the extreme case, 1A. The wax rate is much larger than expected for case 2A because a reasonably high $C_{27}+$ concentration in the product wax could not be achieved for this case. This is detailed in Table 8, which gives the product wax compositions for each of the four cases. The desired 45 mole% concentration of $C_{27}+$ waxes in the wax product stream could be attained in cases 1 and 2. However, at 90% conversion and a 500°F reaction temperature, the product gas flow rate is too low to adequately strip the distillate components from the wax. Thus, in case 1A, only a 35 mole% concentration of $C_{27}+$ in the wax product could be achieved. Catalyst B has a significantly lower yield of $C_{27}+$ waxes. Therefore in case 2A the $C_{27}+$ concentration in the wax product could not be increased beyond about 23 mole%. In a full-scale plant, a separate distillation column, independent of the reactor, would be used to strip distillate retained in the wax. A separate distillation column is not justified for the BFTR.

The distillate recovery system does a good job of separating distillate from the product gas in all cases. Tables 9A-D, which show the distribution of each distillate range component between wax, distillate, and gas products, indicate that at least 90% of the Cq is recovered as distillate in all cases. The boiling point distribution of the Case 1 distillate is shown in Figure 2.

3.5.2 Heat Duties

The major heat exchanger duties are listed in Table 10. The duty for Exchanger E-101, the cooling tube inside the reactor, varies considerably among the four cases, from 10,400 to 216,000 Btu/hr. The surface area of this exchanger is dictated by reactor geometry requirements, i.e. the cooling tube must have a 2.25 inch outside diameter to give an effective reactor diameter of 5 inches. Therefore, this wide range of heat duties is compensated for by

varying the temperature difference across this exchanger. This is reasonable for cases 1 and 2; however, for cases 1A and 2A, this produces a temperature difference of more than 80°F. This is probably too large a temperature difference to give effective reactor temperature control. Therefore, cases 1A and 2A will utilize either additional heat removal external to the reactor, e.g. a heat exchanger in the thermosyphon loop, or they must only be operated when the reactor is configured with the larger diameter cooling tube and an effective reactor cross section of four inches. The BFTR design provides an external heat exchanger, E-401, which can assume 100,000 Btu/hr of the reactor cooling duty.

The condenser (E-301) heat duty varies from 600 to 4100 btu/hr, with case 1 being the limiting case. The duties for E-601 to cool the wax are all very small, varying from 140-1000 Btu/hr Therefore, this exchanger will be deleted. The main product cooler, E-102, has a fairly constant duty of 10,100-15,700 Btu/hr, with the limiting case being case 2. This exchanger cools the product gas and distillate to 100°F, to condense the bulk of the distillate. In cases 1 and 2, the product gas rates are too large to permit the light distillate compounds to be adequately recovered at 100°F. For these cases, further cooling to 42°F in the glycol/water exchanger, E-202, is required. The duty for this exchanger is 2100-2500 Btu/hr for cases 1 and 2 and zero for the other two cases.

3.6 BFTR Design

Figure 3 is a detailed process flowsheet of the BFTR, showing all of the controllers, control valves, and major manual valves in addition to all vessels and heat exchangers. The synthesis gas metering section has not been included, since the methanol unit's feed gas system is assumed to be used. Table II lists the equipment from the LaPorte Methanol unit which will be incorporated into the BFTR as well as the equipment from the present Air Products laboratory Fischer-Tropsch project which will be utilized. A process design of the product gas metering/analysis train has not been undertaken.

3.7 Reactor Design

The reactor design was influenced by three goals:

- data must be collected in the churn-turbulent flow regime
- the reactor diameter should be minimized to reduce expensive syngas consumption
- the reactor height must be sufficient to produce syngas conversion levels of about 50% with the present catalysts.

3.7.1 Choice of Flow Regime

Cocurrent up-flow bubble columns can operate in any one of three flow regimes[1] (see Figure 4).

The bubbly flow or quiescent regime occurs at velocities less than typically 0.15 ft/sec. Bubbles are uniform in size and do not interact or coalesce to any substantial degree. For a given three phase system the bubble size is influenced by the sparger hole size and hole spacing. At higher gas velocities, the bubbles can no longer rise uninfluenced by neighboring bubbles and the churn turbulent regime is entered. Coalescence occurs yielding larger bubbles with faster rise velocities. For a given gas flow rate, higher bubble rise velocities result in a decrease in gas holdup or gas volume fraction. Because of coalescence, gas holdup does not increase as quickly with gas flow rate in the churn turbulent regime as in the bubbly flow regime.

Where the diameter of the coalesced bubble would be greater than the diameter of the vessel, the slug flow regime is reached. Slug flow is undesirable because of the low gas/liquid interfacial area that results and the tendency for slurry to be lifted out of the reactor along with the gas.

The choice of flow regime is dictated by two economic factors. For a given reactor volume:

- The highest space time yield (STY) or production rate per unit volume is optimal.

- A tall column is preferred to a short one.

The higher the STY, the smaller the required reactor volume. Numerous bubble column computer simulations have been run to determine the STY for various flow regimes and reactor conditions. These simulations used the model proposed by Deckwer[2]. Kinetic parameters obtained from the autoclave studies were incorporated into this model along with hydrodynamic correlations derived in Task 3. For a given catalyst loading there exists an optimum gas velocity for a maximum space time yield. Above this velocity, the reaction rate becomes limiting as the total amount of catalyst is reduced by the greater gas holdup. In every case, this optimum is in the churn turbulent regime. The value of 9.1 cm/sec (0.3 ft/sec) although slightly below that optimum would still allow a reasonable conversion for the reactor height chosen.

For a commercial plant with multiple trains, it is advantageous to have as few reactor vessels as possible. In the quiescent regime only about 15' of reactor height is required to achieve 90+% conversion of syngas. By increasing the gas velocity into the churn turbulent regime, 90+% conversion of syngas is not achieved until a height of 60'. For equivalent STY's, it is more economical to have N vessels at 60' than to have four times that number of vessels, each with a height of 15'.

3.7.2 Choice of Reactor Diameter

Syngas cost should be minimized. The minimum reactor diameter which allows operation in the churn turbulent regime is optimum. Air Products' cold-flow model experiments, performed under this contract, indicate that the churn turbulent regime is present in 5" ID reactors, confirming the flow regime chart (Figure 4). However, the exact diameter of that transition is an unknown function of the slurry viscosity and surface tension.

Both objectives were achieved by designing the reaction vessel with an inside diameter of 5.5 inches and interchangeable internal cooling tubes having outside diameters of 2.25 inches and 3.75 inches, to give effective reactor diameters of 5 inches and 4 inches, respectively. In this usage, effective diameter is defined as the diameter of an open pipe having the same cross-section flow area as the annular flow area of the BFTR reactor. Operation with the 3.75 inch O.D. cooling tube will reduce syngas consumption by 36% compared to operation with the 2.25 inch O.D. tube and will also permit the 90% syngas conversion cases (1A and 2A) to be run with a reasonable temperature difference of about 30°F instead of 80°F, without resorting to an external heat exchanger.

3.7.3 Heat Transfer: Internal vs. External

The rate at which heat can be removed from the reactor is given by the product of (heat transfer coefficient) X (surface area) X (temperature difference). The reactor temperature will be more stable (and a commercial process will have improved thermal efficiency) if the temperature difference between the reactor contents and the cooling medium is minimized. Since the overall heat transfer coefficient is set by system hydrodynamics, the temperature difference is essentially a function of the ratio of (reaction volume) / (heat transfer area).

For a small laboratory or pilot-plant reactor, in which a single heat exchanger tube would be used for internal heat transfer, a smaller (reaction volume) / (heat transfer area) ratio can be achieved by simple jacketing the reactor and cooling externally. This is probably the easiest and most-efficient approach to use with the 5-inch diameter reactor planned for this pilot plant. The disadvantage of this approach is that it is not suitable for larger diameter reactors; the ratio (reaction volume) / (reactor surface area) increases linearly with reactor diameter. Thus a commercial-scale reactor has only two viable choices for removing the heat of reaction:

(1) use a large number of internal heat transfer tubes in order to achieve a reasonable (reactor volume) / (heat transfer area)

(2) remove a slurry stream from the reactor, pump it through external heat exchangers and back into the reactor.

However, it is believed that heat transfer internals will be the mode of operation of choice in commercial reactors. Internal heat transfer precludes the need for 1) an external heat exchanger, 2) a large (and expensive) slurry pump, and 3) much larger slurry piping for the entire slurry circuit. Also operational difficulties involved with the erosion and plugging caused by slurry circulation are minimized. Further, heat transfer internals offer a more uniform temperature in the reactor. Thus, it is important to gather basic engineering data on the hydrodynamics of an actual Fischer-Tropsch reaction in a bubble column with heat transfer internals.

Most experimental work to date does not include heat transfer internals. Therefore, experimental measurement of the heat transfer coefficient for internal heat exchange tubes in a Fischer-Tropsch reactor operating in the same hydrodynamic flow regime as a

commercial-scale reactor will be extremely beneficial to future design and cost estimation work. For this reason, internal cooling was elected for the BFTR, even though an external jacket on the reactor would be a simpler and more efficient cooling system at 5-inch diameter.

An incidental reason for using heat transfer internals is the cost savings on the gas consumption, mentioned above, although this advantage could also be obtained by installing a dummy tube.

The boiling water internal heat exchanger selected for the BFTR can be operated on either a closed or open water system. In a closed system, all of the steam produced in the internal heat exchanger (E-101) is condensed against cooling water in a heat exchanger and returned to the water reservoir (V-101). Since steam does not leave the system, a continuous water make-up system is not needed. The temperature of the boiling water in E-101 is controlled by the pressure of the water reservoir; an applied nitrogen pressure or a dead-weight valve is used to control reservoir pressure. The advantage of this system is that make-up water flow control is not required.

In the open system, the steam generated in E-101 is allowed to leave the water reservoir; the steam release rate is measured and this value is used to calculate the heat generation rate within the reactor. The reservoir pressure is governed by controlling the flow rate of steam from the system. The water level in the reservoir must be monitored continuously and make-up water pumped to the reservoir at a controlled rate to maintain a fixed level. Thus, this system has the disadvantage that it is more complex, requiring pressure and level control loops. The open system was selected for the BFTR in order to provide a measure of heat production during the reaction.

3.7.4 Choice of Reactor Height

The Deckwer computer model of Task 3 indicates that a slurry height of 30 feet is required to achieve a conversion level of 50% with the best laboratory catalysts tested to date.

3.7.5 Mode of Operation

The slurry reactor is operated in a fluidized-bed mode. In this mode of operation, slurry is initially charged to the system while nitrogen is sparged to the reactor. The nitrogen provides an inert atmosphere for the BFTR and keeps the slurry suspended. Once charged, slurry will not be added or removed from the slurry reactor except for a small stream, to remove high molecular weight reactor wax, from the wax withdrawal loop, that has accumulated as a result of the Fischer-Tropsch synthesis and a smaller stream required for catalyst regeneration. Gas enters the bottom of the reactor and is distributed by a ring sparger. Other gas distribution methods, including plain tubing discharge, should be tested during the course of the experimental program with the BFTR. A ring sparger is relatively easy to scale up. As gas flow is increased, holes can be added to the sparger. Sparger diameter is increased along with reactor diameter, and sparger rings can be added. The velocity of gas leaving the sparger holes and the superficial gas velocity in the reactor are the primary considerations. Gas flows up the column and exits out the column top.

During reactor upsets, nitrogen will be sparged through the slurry reactor whenever syngas cannot be. This will be done in order to prevent overheating and oxidation of the catalyst.

3.7.6 Hydrodynamic Considerations

The prime consideration for optimum reactor performance is to have neither kinetics nor transfer resistance controlling. If the kinetic reaction rate were limiting, then catalyst loading in the reactor would be increased to increase STY. For 0.5-5 micron particles, the catalyst remains well suspended at the concentrations required with our present catalysts. If mass transfer is limiting, then catalyst loading can be decreased without adversely affecting STY.

Heat transfer does not control the production rate either. The Rheinpruessen reactor operated the Fischer-Tropsch synthesis in the slurry phase in the 1950s[3]. About 6% of the 1.5 m ID x 8 m reactor was taken up by heat transfer internals. The 25-cm-diameter reactor operated by the British at Warren Spring laboratory had 4% of the reactor volume occupied by 4 cooling tubes. Thus, increasing the space time yield to that of a commercial Synthol reactor would not require a substantial increase in heat transfer volume.

Liquid dispersion, from the cold flow studies, also appears to be quite adequate to allow for close to isothermal operation.

3.7.7 Details of the Reactor Design

Details of the reactor design are shown in Figure 5. The reactor is constructed entirely of type 304 stainless steel and will be an ASME code-stamped vessel. The lowest pressure rated components are the class 400# flanges, which have a pressure rating of 555 psig at 600°F. This provides a sufficient margin of safety and operating flexibility beyond the 307 psig, 500°F normal operating conditions. Flanges are used in construction of the reactor to facilitate changes in cooling tube diameter and sparger design.

The reactor vessel has five connections:

- A 3-inch flange connection between the internal heat exchanger and the water reservoir (V-101).

- A 3-inch weld connection to the packed distillation column (V-201).

- A 3-inch weld connection to Vessel V-801, which houses the capacitance probe that measures slurry level in the reactor and also feeds the thermosyphon recycle loop.

- A 3-inch weld connection to the Filter Vessel V-401, through which the thermosyphon recycle stream flows back to the reactor.

- A 1-inch pipe connection through which the synthesis gas enters the base of the reactor. The gas flows through a 2-inch diameter sintered-metal sparger to produce fine bubbles. The reactor has a 6" x 3" concentric reducer at the bottom to help prevent catalyst settling in this region of the reactor, where the net liquid flow rate is zero.

The internal heat exchanger tube is welded to a 6-inch, 600# blind flange and is flanged to the top of the reactor to permit relatively simple changeover (with a crane) between the 2.25-inch and 3.75-inch diameter cooling tubes. A number of specially machined socket-weld flanges for 6-inch tubing are required to permit the transition between 6-inch tubing and 6-inch pipe and pipe fittings in the construction of the reactor.

A six-foot head space is provided between the desired slurry level and the 3-inch flange connection to the packed distillation column. Although the Air Products laboratory Fischer-Tropsch experimental

program has not indicated that foaming would be a problem in the BFTR, a spiral froth-breaker will be installed in the headspace, based upon the experience of Farley and Ray[4].

3.8 Wax Withdrawal System

Since the Fischer-Tropsch Process produces high-boiling waxes which cannot be removed as vapor at the 500°F reaction temperature, liquid wax must be withdrawn to maintain a fixed slurry level in the reactor. Catalyst "A" requires a larger wax purge flow than does catalyst "B." Removing the wax product is difficult because the 1-2 micron catalyst particles must be separated from the wax and returned to the reactor.

The BFTR design provides equipment to test both filtration and gravity sedimentation for catalyst removal from the wax. During operation of the BFTR, testing of other catalyst removal systems may be desirable. For example, if an iron catalyst is the choice for future development, testing a magnetic separator should be considered. The wax removal section of the BFTR is highlighted in Figure 6.

3.8.1 Thermosyphon

A thermosyphon loop will circulate wax slurry from the reactor, at a point about 1 foot below the normal slurry level, through V-801 and the shell of the filter vessel, V-401, and back to the reactor at a point about 1 foot above the base. The driving force for the thermosyphon is the difference in density between the reactor contents and the slurry in the thermosyphon leg. The Deckwer model indicates that the BFTR reactor, at its design conditions, will have a gas holdup of 50%. The results of Task 3 indicate a much lower gas hold-up of 25%, which was used in designing the thermosyphon loop. Since the slurry in the thermosyphon leg has almost no gas

holdup, this provides a differential liquid head of almost 3 psi to circulate slurry through the thermosyphon loop. Using 1-inch pipe for the thermosyphon loop, the slurry flow rate can be up to 9 gpm. Flow Control Valve FCV-1401 is provided to regulate the flow rate through the thermosyphon loop. The circulation rate is measured by a venturi meter, FT-1401.

The 1-inch pipe in the thermosyphon loop is jacketed so that the slurry temperature can be maintained at 500°F. Valves HV-61 and HV-71 are provided to permit catalyst slurry to be added to the reactor or withdrawn from the system into sample cylinders, as would be required to regenerate catalyst in small batches. The slurry mixing tank, V-701, can be used to add or remove large quantities of slurry.

3.8.2 Filtration

Filtration is the most convenient means of removing the catalyst from the product wax stream. Unfortunately, filtration is not certain to work. Quantitative data are not available on the filtration characteristics of the Fischer-Tropsch slurry. The Air Products laboratory reactor uses a porous sinter on the reactor wall to filter catalyst from the wax. This sinter is backflushed with gas (but not after each wax withdrawal) to dislodge the filter cake, which falls back into the reactor. This system works reasonably well, although the sinter, on occasion, becomes plugged after several weeks to a month of operation. The filter cake apparently is not unduly compressible but does tend to blind the filter at a slow rate.

The BFTR design provides a single-element sintered-metal candle filter assembly with a surface area of 0.9 ft^2. Since this filter has a fine particle removal rating of 1-micron nominal, 3-micron absolute, it will not be able to achieve the desired 98-99% removal

efficiency. Thus the filter cake itself (and partial blinding of the sinter) must be relied upon to remove the finest catalyst particles. At the end of each cycle the bulk of the filter cake will be discharged from the candle to the thermosyphon recycle stream flowing through the filter housing by shocking the cake with hydraulic/gas backpressure on a cyclic basis. The solids concentration in the slurry is too large to permit liquid backflushing of the filter, so flushing with gas must be relied upon to prevent unacceptable filter blinding over the required several months run duration. Several months life-time for a candle sinter is reasonable based upon operation of the laboratory sinter and the low filter loading rate of only 1.1 gallon/ft^2-hr in the worst case (1A) and 0.4 and 0.16 gallons/ft^2-hr for Cases 1 and 2, respectively. Although no experimental filtration rate data are available for this system, filter loadings of 10 gph/ft^2 are more typical, even for relatively difficult filtrations. The available pressure drop across the filter is about 250 psi; if the filter cake is incompressible, the filtration rate should be linear with pressure drop.

When the filter is operating, valves HV-411A and HV-411B are closed, isolating the sedimentation vessels. The filtration cycle operates as follows. During the roughly 90% of the cycle when clean wax is being withdrawn through the candle, valve FCV-411 is closed and the wax flow rate is controlled by flow control valve FCV-401 (normally 401A; 401B is installed spare). This valve is governed by controller LC-1, which maintains a fixed slurry level in the reactor. The clean wax flows from valve FCV-401 to the wax receiver, V-601, which is at a pressure of 45 psia. Thus a pressure drop of 277 psi is incurred between the shell of the filter vessel and the wax receiver. The proportion of this pressure drop taken across the filter candle and across FCV-401 will vary depending upon the cake thickness and extent of candle blinding at any time. Based on a timing cycle (which will be determined during startup), valve

FCV-401 is closed to end the wax withdrawal portion of the filtration cycle. Valve FCV-411 is then opened for several seconds to apply nitrogen back pressure of 600 psig to the inside of the filter candle. This will crack the filter cake and force some clean wax from the inside of the candle to flow backwards through the candle and filter cake into the shell of the filter. The filter cake falls off the candle and is carried back to the reactor by the flow in the thermosyphon loop. Valve FCV-411 is closed, ending the backflush portion of the cycle. Valve FCV-401 is reopened (with a limit on how far it can open), beginning a new wax withdrawal cycle.

The "clean" wax is expected to contain about 0.3 wt% catalyst in normal operation. An in-line cartridge filter, F-401 or F-501, removes the remaining catalyst from the wax, so that the BFTR can produce wax of satisfactory quality for product testing and so that the catalyst particles that pass through the candle filter can be analyzed. The in-line polishing filter should be able to operate continuously for at least 8 and probably more than 24 hours before requiring replacement and cleaning. The filtrate piping is steam traced and 300 psi steam is used to control the temperature of the product wax before it reaches receiver, V-601.

An extensive study of commercially available filters and cartridges for the primary filter or the in-line polishing filter has not been performed. The cost estimate is based upon telephone quotations from Pall Filter Company. The primary filter candle is a 2-3/8 inch diameter by 18 inch long grade H sintered powder cartridge that is backflushable by liquid pulse and suitable for intermediate-term cyclic operation. The 304 stainless steel housing for the filter would be custom-built because a jacket for heat transfer fluid is required and because the diameter of the housing is set by operating requirements of the sedimentation system rather than the filter. The housing is 6 inches in diameter and two feet long. The candle

will be mounted an inch below the top inner surface of the housing to prevent a gas layer from collecting at the top of the housing and short-circuiting the filter. Gas that does collect at the top of the filter housing is piped to the gaseous products' exit of the reactor.

Farley and Ray[4] tested filtration in their pilot plant for removal of catalyst from wax. Filtration was adequate at some process conditions but was unsatisfactory at high wax yield conditions because wax could not be withdrawn fast enough to control the slurry level in the reactor. Unfortunately they did not publish filtration rates or details of the filter design and operation. However, their partial success is evidence that filter cake compression and filter blinding were not severe problems with a Fischer-Tropsch wax slurry.

3.8.3 Sedimentation

Farley and Ray[4] eventually chose a batch-operated gravity settler for separation of catalyst from the wax. This system could handle higher wax withdrawal rates than the filter. In order to reduce the volume of the sedimentation vessel to a reasonable size, they placed inserts in the vessel to provide additional surface for the particles to settle onto. These surfaces, in the form of stacked cones, bent-elliptical plates, or pipe were spaced such that the catalyst particles only had to settle about an inch to hit a surface. These inserts reduced the required sedimentation time by a factor of 10 to 100. The particles agglomerated to such an extent that only 1% of the particles were below 3.5 micron diameter. This agglomeration is essential to the operation of the sedimentation system, since a 1 micron particle has a terminal settling velocity of only 0.12 inches per hour. The 3.5 micron agglomerates will settle at a rate of 1.5 inches per hour. The agglomerates form a sludge blanket on the surface of the insert. Farley and Ray[4]

did not experience difficulty with flowability of the concentrated solids blanket, which readily slid down the settling surfaces, out of the sedimentation vessel, and down the sedimentation limb between the filter housing and the sedimentation vessel, all of which are mounted at an angle of 60 degrees to the horizontal. The function of the filter is apparently to insure that gas bubbles do not enter the sedimentation limb and that the sedimentation system is isolated from the high flow velocities and turbulence of the thermosyphon loop. Careful temperature control in the whole sedimentation system is mandatory, since convective circulation of the wax would interfere with sedimentation. In long-term operation this system reduced the catalyst concentration in the wax to less than 0.2 wt%. But, until the initial wide particle-size range catalyst charge to the reactor approached its steady-state particle size distribution of 1-3 microns, significantly higher catalyst concentrations in the clean wax were observed for the initial 60 hours of operation. For this reason, the BFTR design includes a filtrate/sedimentator overflow recycle line from valve HV-471 to V-701, which can be used to recover the "dirty" wax produced at the start of the run, for recycling to the reactor.

The sedimentation system in the BFTR design is adapted from the approach of Farley and Ray[4]. Two batch sedimentation vessels operate in a matched swing-cycle to provide continuous wax withdrawal at constant rate. These sedimenters can also be tested in continuous parallel operation. Each of the sedimentation vessels is essentially a shell and tube heat exchanger mounted at a 60 degree angle with the horizontal. The catalyst sedimentation from the wax occurs on the tube side; a heat transfer fluid is circulated on the shell side to maintain a fixed and uniform temperature.

The sedimentation cycle operates in the following manner. While vessel V-501A is in the quiescent settling mode, wax is withdrawn from vessel V-501B. Valves HV-411A and B are both open. Valve

FV-501A is closed and valve FV-501B is open, while valve FV-401A is controlling the wax withdrawal rate from sedimentation vessel V-501B, at a rate governed by controller LC-1, to hold a constant slurry level in the reactor. F-501 is an in-line cartridge filter which removes residual catalyst particles from the clean wax. As clean wax is withdrawn from V-501B, 20 wt% catalyst in wax slurry flows in through the sedimentation limb from the filter housing, V-401. The clean wax withdrawal rate is slow enough that the settled 3.5 micron agglomerates cannot be resuspended, and wax withdrawal is halted before the advancing front of catalyst can reach the top of the sedimentation tubes. The wax withdrawal phase of the cycle has a duration of three hours. When this phase is complete, valve FCV-501B is closed. Catalyst particles settle from the wax slurry in the sedimentation tubes forming a sludge at the bottom of the tubes. This sludge slides down the tubes to the bottom tube header of V-501B, resuspending some of the settled particles because of the motion of the sludge, particularly at the point where the sludge drops out of the tube into the header. Because of this resuspension/resettling and particle agglomeration, settling time required to produce clear wax in the tubes cannot be calculated rigorously. Based on Farley and Ray's[4] experience, the three hours provided in this design seems conservative. The greatest settling distance for any particle is 3 inches, requiring 2 hours settling time for a 3.5 micron aggregate. The design settling time provides a factor of 1.5 on this calculated time to allow for resuspension/resettling. Once the catalyst sludge reaches the vessel header, it will slide down the sedimentation limb, which is at a 60 degree angle, to the filter vessel, V-401. The sedimentation limb terminates in the filter vessel at a site where liquid velocity and entrained gas bubbles should be minimized, i.e. near the wall, several inches above the cone section, and at 120 degrees from both the V-501A sedimentation limb and the inflow point from the thermosyphon loop.

During the first hours of operation with a fresh catalyst batch, when the "clean" wax leaving the sedimentation vessel will contain an unusually high concentration of catalyst, valve HV-561 can be closed and HV-551 opened to route the product wax to the slurry mix tank.

Both sedimentation vessels consist of 13 tubes, each having a diameter of 1.5 inches. The total cross-sectional flow area of 0.16 ft^2 results in a wax velocity of nearly 0.5 ft/hr for the case 1 wax withdrawal rate. This conservatively assures that wax withdrawal will not resuspend catalyst; a measure of the degree of conservatism is provided by the fact that this 0.5 ft/hr velocity is only 0.3% of the saltation velocity (which really applies to horizontal pipes) of a 3.5 micron-particle. The length of the tubes is set at 1.9 feet, based upon the required volume for a six hour cycle and the assumption that only 2/3 of the liquid in the tubes can be withdrawn on each cycle without risking catalyst breakthrough. This seems reasonable since the flow will be very laminar and the L/D ratio is 15.

For the case where the two sedimentation vessels are operated continuously in parallel, the vertical component of the velocity through the tubes corresponds to the terminal settling velocity of a 3.5 micron iron catalyst agglomerate. Thus, continuous operation may be possible for cases 1 and 2. The sedimentation system was not designed to handle cases 1A and 2A, where the wax flow is 2.3-2.7 times that for case 1.

3.8.4 Volume of Wax Withdrawal System

The sedimentation system was not designed to handle the limiting flow case, 2A, because this would make the volume of slurry in the wax withdrawal system larger than the volume in the reactor. A major problem with the present design is that the ratio of slurry in the wax withdrawal loop to that in the reactor approaches unity.

For a 30 foot slurry level, the reactor volume is 4.1 ft^3. The Deckwer model[2] predicts a gas holdup of 50%, making the slurry volume in the reactor only 2.05 ft^3. The volume of slurry in the wax withdrawal loop is 1.5 ft^3, which breaks down as follows:

2 Sedimentation Vessels	-	0.8 ft^3
Sedimentation Limbs	-	0.1 ft^3
Filter Vessel	-	0.35 ft^3
Thermosyphon Loop	-	0.22 ft^3

Use of the external heat exchanger, E-401, will increase the slurry volume in the thermosyphon loop. The large slurry volume in the wax withdrawal system is a problem for two reasons. First, the time required to observe any given catalyst age or extent of deactivation is doubled. Second, if catalyst sludge flows from the sedimentation vessels in surges this could cause noticeable fluctuations in the quantity of catalyst in the reactor. The slurry in the filter and thermosyphon loop is well-mixed and should have the same catalyst concentration as the slurry in the reactor. The unmixed slurry in the sedimentation system represents 25% of the total slurry in the BFTR. If 10% of the catalyst in this volume of unmixed slurry entered the filter as a slug, it would increase the catalyst concentration in the reactor by 3%, an increase which might be noticeable in the product yields but certainly would not be drastic. When filtration is used the sedimentation system is valved off and this problem is obviated.

3.8.5 Need for Additional Data

The design of the filtration and sedimentation systems is empirical and requires laboratory data on the specific slurry to be t ted This data was not available to aid in this preliminary BFTR desig ; as a result this design is probably overly conservative, but because of the level of uncertainty we cannot absolutely assure its

adequacy. An experimental program designed to quantify the filtering and sedimentation characteristics of several catalyst slurries should be performed and the results incorporated into the reactor design prior to construction of the BFTR.

3.9 Product Fractionation Design

3.9.1 Packed Column

The packed distillation column, V-201, was added to the design to effect a better separation between the wax and the distillate product. The column was designed to give a fractionation efficiency equivalent to 4 theoretical plates for case 1. The column is 9 feet tall, has a diameter of 3.5 inches, and is packed with 3/8 inch diameter Raschig rings. The unusual feature of the column design is that the gas mass flow rate of 1200 pounds/ft^2-hr is so much larger than the liquid mass flow rate (L) of 50 pounds/ft^2-hr. The gas mass flow rate was fixed at 60% of the flooding rate in sizing the column. The data of Shulman[5], as summarized by Treybal, were conservatively extrapolated from an L' of 500 to an L' of 50 to give an interfacial area of 3 ft^2/ft^3. The overall height of a transfer stage was then calculated to be 1.6-2.2 feet.

The vapor leaving the fractionation column is cooled in Exchanger E-301 to 400-480°F. The condensed liquid is separated in V-301 and totally refluxed to the fractionation column. Thus the reactor, packed column, and condenser provide a reaction product fractionation equivalent to 6 theoretical plates. Exchanger E-301 has a surface area of 11 square feet, based on the calculated requirement for 9 square feet of surface area with an overall heat transfer coefficient of 10 Btu/ft^2-hr-°F and a log-mean temperature difference of 53°F. This is a shell-and-tube heat exchanger with boiling water as the shell-side transfer fluid. The temperature of the product leaving E-301 is controlled by varying the pressure of the water reservoir V-103 which fixes the temperature of the boiling water.

3.9.2 Distillate Recovery

The product vapors leaving V-301 are indirectly cooled against cooling water to a temperature of 100°F in exchanger E-102, in order to condense the distillate product. Like E-301, exchanger E-102 is an American Standard shell and tube exchanger having 14 tubes of 3/8" O.D. and 0.02 inch wall. Based upon an overall heat transfer coefficient of 10 Btu/ft^2-hr-°F and a log-mean temperature difference of 87°F, the limiting case 1 requires 18 ft^2 of heat transfer area. The BFTR design provides for two-11 ft^2 exchangers to be operated in series. The gas flow is too high to permit adequate recovery of the distillate at 100°F in cases 1 and 2. Therefore, a glycol-water cooled exchanger, E-202, has been provided to further cool the process stream to 42°F. case 1 is also the limiting case for exchanger E-202, requiring a cooling duty of 2600 Btu/hr. For the overall heat transfer coefficient of 10 Btu/ft^2-hr-°F and the log-mean temperature difference of 28°F (assuming a 32°F glycol inlet temperature), the required heat transfer surface area is 9-1/2 ft^2. An 11-square foot American Standard shell-and-tube heat exchanger is provided. Assuming a heat leak of 2000 Btu/hr into the refrigerant loop, a glycol-water circulation rate of 1 gpm is required. Further, a refrigeration system with a capacity of at least 4600 Btu/hr at 25°F must be provided. The BFTR design includes an RC-100 glycol-water refrigeration system manufactured by FTS systems, which provides a glycol-water circulation rate of 2.5 gpm and a refrigeration duty of 6400 Btu/hr at 14°F.

3.9.3 Pressure Let Down

The overhead reactor products are maintained at reactor pressure through the vapor-liquid separator, V-2. The system pressure is regulated by controller PC-2, which governs the rate at which product gas is allowed to flow through Valve FCV-12A or B from the

vapor-liquid separator to the gas analysis train. Valve FCV-12B provides an installed spare in parallel to FCV-12A and is used only when maintenance is being performed on FCV-12A. Both valves are Badger Research Control valves, installed with a valve stem positioner for air-to-close operation. The valve trim must cover the Cv range 0.18-0.25 in normal (50-70% open) operation. The distillate is let down in pressure from roughly 320 psia to 45 psia across valve FCV-2A or B. FCV-2B is an installed spare for FCV-2A. These valves are controlled by LC-2, which maintains a constant distillate level in V-2, based upon the signal from the dielectic conductivity probe, Lt-2. Both valves are Badger Research Control valves equipped for air-to-open operation. These valves must cover the 0.0004-0.0011 Cv range in normal operation.

The product distillate, after pressure letdown, is collected in receiver V-102, which is operated at 45 psia. A small quantity of dissolved gas will flash from the liquid; this gas is combined with the main product gas stream and piped to the gas analysis train. The liquid level in V-102 is not controlled; instead, a load cell measures the rate at which distillate accumulates during an entire material balance period. At the end of the balance period the distillate is drained either to a sample container or to the underground storage tank.

3.10 Ancillary Vessel Specifications

All vessels in the BFTR will be constructed of type 304 stainless steel. They will be painted with a high temperature paint to protect against chloride stress corrosion cracking caused by chloride leaking from the wet insulation. They will all be fabricated by Air Products' CRSD or manufacturing shops; only the reactor and the sedimentation vessels will be ASME-coded vessels. Most vessels are flanged to facilitate cleaning and inspection; weld construction could be substituted and would not affect the cost estimate.

3.10.1 The Heat Exchanger Reservoir

V-101 consists of a 6-foot length of 6-inch schedule 40 pipe welded to a 6-inch 600# socket-weld flange with a mating blind flange at the top and to a 6" x 3" reducing tee, then a 6" x 3" concentric reducer and then to a 3-inch, 600# socket-weld flange at the bottom. The 3" flange will connect to the 3" pipe flange on the cooling-water feed tube which is welded to the top flange of the reactor. The top flange has the following fittings: a 1/2-inch female National Pipe Thread (FNPT) fitting for rupture disc PSE-101 (700 psig @ 600°F), a 1/2-inch FNPT fitting for the make-up cooling water line; a 1/2-inch FNPT fitting for the effluent steam line, and a 1-1/2 inch FNPT fitting for the level probe. The make-up cooling water tubing extends the entire length of V-101, passes out the bottom 3-inch flange and extends 35 feet into the reactor's internal cooling tube. The 3-inch side arm from the reducing tee is the water feed and steam return from E-401. V-101 holds 0.8 ft^3 of water when 2/3 full; coupled with the 0.4 ft^3 of water inventory (assumes 50% volume) in the heat exchanger tube, this provides enough reserve cooling capacity for about 20-minutes operation in the limiting case 1A, in the event that the make-up cooling water supply is lost. This vessel has a design pressure rating of 800 psig @ 600°F. If the temperature rises to the normal reactor operating temperature of 500°F, the pressure would be 666 psig. The rupture disc would fail if the temperature reached 505°F, at which point the pressure would be 700 psig. A redundant steam flow control valve, PCV-101B, is provided to minimize the likelihood of V-101 overpressuring because of a failure of the steam discharge valve.

3.10.2 Condensate Receiver

The condensate receiver, V-301, is a 2-foot length of 8-inch schedule 40 pipe, with a flanged-top (300# flanges) and a pipe cap

for the bottom. This vessel has an internal volume of 0.7 ft^3, but is operated essentially empty; a trap sets the liquid level, and any condensate simply flows out the bottom and back to the packed column. The purpose of this vessel is to prevent the condensate from being entrained in the gas flow.

3.10.3 Wax Receiver

The wax receiver, V-601, is fabricated from schedule 10 pipe and 150# flanges, since its operating pressure is only 45 psia. This vessel is 4-feet long and 6 inches in diameter and has a volume of 0.8 ft^3. Thus it can accommodate 4 hours wax product for the limiting case 1A, where the wax rate is about 1.2 gallon per hour. This vessel is protected from overpressuring by the 1-inch pressure-relief valve, SV-60. The vessel is mounted on a Revere USP-1 load cell, having a maximum load capability of 200 pounds and a manufacturer's stated accuracy of 0.02%. The empty tank has a weight of about 130 pounds. The schedule 10 pipe was chosen to minimize tank weight; it is rated at 390 psig at 400°F.

3.10.4 Slurry Mix Tank

V-701 is a 65 gallon, 316 SS tank built by Distillation Engineering. It is 24-inches diameter by 38-inches long and has an ASME code-stamp rating of 150 psig at 500°F. The vessel has a 6" flanged top in order to accommodate the agitator, which is a 5-1/2 inch dual blade mixer built by Cleveland Mixer to operate at a fixed 420 rpm speed with a 1/4 horsepower, explosion-proof motor. The mixer can handle slurry viscosities of up to 1000 centipoise. This tank and mixer are used to prepare the initial reactor charge and as a vessel into which the reactor contents may be drained and stored.

3.10.5 Capacitance Probe Housing

Vessel V-801 is provided primarily as a housing for the reactor liquid level probe but also to aid in separating the entrained gas bubbles from the slurry feeding the thermosyphon loop. This vessel is built around an 8-inch by 3-inch schedule 40 reducing tee. The side-arm of the tee is welded to a 3-inch, 40#, socket-weld flange which attaches to the 3-inch pipe from the 3-inch diameter side-draw at the normal slurry-level location of the reactor. The top of the tee is welded to a 1-foot length of 8-inch schedule 40 pipe which terminates in an 8-inch, 400#, socket-weld flange and matching blind flange. The bottom of the tee is welded to an 8-inch by 4-inch concentric reducer, which terminates in a 4-inch by 2-inch reducer and a 2-inch 400# socket-weld flange. The fittings for V-801 consist of a 1-1/2-inch FNPT fitting for the level probe and a 1-inch FNPT fitting for a connection to the reactor vapor effluent line, both on the top flange. The bottom 2-inch flange connects directly to the top of the thermosyphon loop. Because of the high temperature, the capacitance probe must be bare or coated with ceramic rather than teflon. Ceramic probes of more than a few feet length are costly and require larger diameter fittings. A test was run by Drexelbrook Engineering with iron oxide in wax to assure that the conductivity probe would function properly with catalyst "A" slurry. Also, Air Products has used ceramic-sheathed conductivity probes in coal liquefaction gas-liquid separators at 2000 psig and 400°F.

3.10.6 Vapor-Liquid Separator

V-2 is 3-feet long by 6-inches diameter and has a volume of 0.5 ft^3. The vessel consists of a 28-inch length of 6-inch diameter schedule 40 pipe, welded to a 6-inch by 3-inch reducer, with 400# flanges at top and bottom. As with most vessels, the top flange system consists of a socket-weld flange welded to the pipe and a

mating blind flange which can be removed to permit easy inspection and cleaning. The bottom blind flange is welded directly to the reducer. The top flange has a 1-1/2-inch FNPT fitting for the dielectric conductivity liquid level probe, a 1/2-inch FNPT fitting for the product inlet pipe, and 1/2-inch FNPT fittings for the distillate withdrawal pipes feeding valves FCV-2A and FCV-2B.

3.10.7 Distillate Reservoir

V-102 has an operating pressure of only 45 psia and is constructed of schedule 10 pipe and 150# flanges. This vessel is 5-feet long and 6-inches in diameter and has a volume of 1.0 ft^3, which is adequate to hold all of the distillate produced in a 4-hour balance period for the limiting case 2A. The vessel weighs 120 pounds empty and 160 pounds with its maximum distillate inventory and is mounted on a Revere load cell.

3.10.8 Water Reservoir

The pressurized hot-water reservoir, V-103, which feeds the shell side of exchanger E-101 must operate at temperatures of up to 500°F and pressures of up to 666 psia. It is constructed of 6-inch schedule 40 pipe and pipe caps and a 6-inch by 3-inch reducing tee to permit a magnetic-float level transmitter to be installed. The total volume of this vessel is 0.8 ft^3.

3.11 Temperature Control

Most vessels will be enclosed in Watlow ceramic fiber heaters, in order to maintain adiabatic conditions during steady-state operation and to provide reasonably rapid heat-up to operating temperature during start-up. The reaction vessel, which is 40-feet long, will be divided into four independent temperature control zones. Each zone will be controlled by a separate relay and Watlow series 808

digital temperature controller and will consist of multiple 2-foot long by 8-inch diameter hemicylindrical heating elements. The reactor will be wrapped with a single layer of insulation inside the heaters, and the heaters will be operated to hold the insulation temperature at 500±5°F, i.e., simply to balance the heat leak. The heat transfer coefficient is 0.2/Btu/ft^2-hr-°F of outer-heater surface. The 8-inch heaters have an outer diameter of 12 inches, so for a 500°F reactor temperature and 70°F ambient temperature, the heat leak is only 271 Btu/hr-ft of reactor length, or per hemicylindrical heater module. If the cold reactor vessel is to be heated to reaction temperature in 2 hours, the heat load would be 950 Btu/hr-ft of reactor. Thus the maximum heater duty is 1220 Btu/hr or 360 watts per hemicylindrical element. This is a very low heater duty which permits construction of lower-cost heating elements containing less heater wire. The heaters will be operated in groups of six to permit use of three-phase power supply.

Ceramic fiber heaters will also be used for temperature maintenance on vessels V-201, V-301, V-601, and V-801. The mixing tank, V-701, will be heated by strip heaters and covered with insulating cement.

The precise temperature control required in the sedimentation loop requires that the sedimentation vessels, transfer piping between the sedimentation vessels and the filter, the filter itself, and the piping for the thermosyphon loop be jacketed and that circulating heat transfer fluid be used for temperature control. The jacketed piping is 1-inch schedule 40, 304 SS inner pipe with a 2-inch schedule 40 carbon-steel shell. About 40 feet of this jacketed pipe will be required for the thermosyphon loop and for the sedimentation lines between the filter and the sedimentation vessels Assuming that the vessel shells and the piping are covered with a 2-inch thick insulation blanket and that the overall heat transfer coefficient is 0.2 Btu/ft^2-°F-hr (on outer insulation surface area), the total heat leak from the heat transfer fluid-jacketed

system will be 7100 Btu/hr. If the heat transfer fluid enters the jacketed system at 505°F and exists at 490°F, a circulation rate of 4 gpm is required. The recycle pump, P-11, has been sized for a continuous flow rate of 5 gpm. The product wax lines also must be heated. This can be done electrically or with 300 psig steam.

3.12 Process Controllers

The cost estimate is based upon using the International Products and Technologies Model 811 industrial controller for the pressure, level and flow controllers and Watlow series 808-1/4 DIN-digital temperature controllers. The Model 811 controller's setpoint can be updated by the computer with a 0-5 volt signal. Instrument signals of 4-20 milliamp, 0-5 volt, or 1-5 volt can be handled and output signals to the control valves of 0-5 volt or 4-20 milliamp are available. This is a three-mode (proportional, integral, and derivative) controller which can be equipped with alarm switches.

The Watlow controller is thermocouple compatible and provides proportioning with auto reset and rate as the controlling mode. These controllers must be coupled with a relay to handle the large electrical duties of the hemicylindrical heaters, especially in the case of the reactor temperature controllers which will control up to 12 heater elements per controller.

Emergency shutdown will be triggered either manually, by high alarm on the system pressure controller, or by the computer, which is monitoring all instrument readings for out-of-limit values.