CANADA

PILOT PLANT FOR LOW AND HIGH PRESSURE FLUID CATALYST BED REACTIONS

J. P. MOGAN, R. W. TAYLOR, AND F. L BOOTH

EPARTMENT OF MINES AND
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Mines Branch Technical Bulletin TB-78

PILOT PLANT FOR LOW AND HIGH-PRESSURE FLUID-CATALYST-BED REACTIONS

by

J.P. Mogan[1], R.W. Taylor[2], and F.L. Booth[3]

ABSTRACT

The construction details of a high-pressure fluid-catalyst pilot-plant are described in this report. This versatile unit will be used to study a variety of fluid-bed reaction schemes (cracking, hydrofining, ...) for the beneficiation of crude oils and bitumens. Of side-by-side configuration, the plant can operate from 15 to 1500 psi at up to 1200°F. The charge oil rate can be varied from 0.08 to 1.75 Imp gph The oil reacts in the presence of a continuously regenerated recirculating catalyst flow of 7 to 25 lb per hour from a 6-lb inventory. An alternate once-through catalyst system circulates 32 lb of catalyst through the reactor from pressure storage vessels.

The ancillary equipment--connecting lines, heaters, safety equipment, supports, and insulation--is also described.

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Direction des mines

Bulletin technique TB-78

USINE-PILOTE POUR L'ÉTUDE DES RÉACTIONS EN LIT À CATALYSEUR FLUIDE SOUS BASSE ET HAUTE PRESSION

par

J.P. Mogan[1], R.W. Taylor[2], et F.L. Booth[3]

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RÉSUMÉ

Les auteurs du rapport décrivent en détail la construction d'une usine-pilote à haute pression utilisant un catalyseur fluide. Cette unité versatile servira à étudier une variété de réactions en lit fluidisé (craquage, hydroraffinage, etc.) pour le raffinage des pétroles bruts et des bitumes. D'une configuration en côte-à-côte, cette unité peut fonctionner de 15 à 1500 livres par pouce carré et jusqu'à 1200°F. Le débit d'alimentation en pétrole peut être modifié de 0.08 à 1.75 gallon impérial à l'heure. Le pétrole réagit en présence d'un catalyseur continuellement régénéré et recyclé qui coule à raison de 7 à 25 livres par heure à partir d'un inventaire de 6 livres. Dans le cas d'une marche sans recyclage, 32 livres de catalyseur circulent à travers le réacteur à partir de réservoirs sous pression.

Les auteurs décrivent aussi l'équipement auxiliaire: conduites de raccordement, réchauffeurs, équipement de sécurité, supports, et isolement.

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INTRODUCTION

"Fluid bed processing" refers to that operating technique in which a solid particulate material is suspended in a stream of fluid flowing between the interstices of the solid particles. This "fluidized bed" then behaves as a true liquid. That is it exerts pressure on the walls of the container, floats buoyant objects on its surface, and is transferable through pipe lines.

The solids-liquid fluidized bed is mainly used in the mineral separation and water treatment industries. The solids-gas bed finds its application in gas-solids reactions, or gas phase reactions with a solid catalyst. The solids-gas bed features intimate gas-solids contact, extremely uniform temperature profile, and the option of easy transfer of the bed from vessel to vessel.

The fluid catalytic petroleum cracker, circa 1940, was the first large-scale commercial use of a solids-gas fluid bed as a reaction device. Earlier catalytic crackers split oil molecules in a reaction cycle and either switched to air feed to burn deposited coke from the fixed catalyst, or moved the catalyst through a regeneration area with a bucket elevator. The advent of the fluid bed cracker permitted continuous interchange of catalyst between reaction and regeneration vessels through connecting pipe lines.

The original units used a side-by-side reactor-regenerator configuration with catalyst flow controlled by slide valves. Subsequent developments placed the reactor-stripper-regenerator combinations in a vertical line with plug valves for catalyst control. These modifications were based on mechanical energy, heat, and structural economics. The fluid catalyst unit described in this report, however, was designed with the classic side-by-side configuration. This assembly more readily permits separation of the reaction and regeneration phases--a desirable option in some high-pressure studies.

While the unit was conceived primarily to study cracking reaction conditions, catalysts and feed stocks, it will not be limited to cracking operations. Any reaction amenable to fluidized-bed techniques may be programmed. In some reaction schemes (such as fluid hydrofining in a static bed, two-stage reactions, etc.), the side-by-side configuration will provide added versatility.

DESCRIPTION OF THE UNIT

The Mines Branch's Fluid Catalyst Unit is a side-by-side reactor-regenerator combination with a stripping vessel mounted below the reactor (Figures 1 and 2).

This configuration is similar to the 40 psi unit built by the Gulf Research and Development Company at Harmarville, Pennsylvania (1). The reactor system is designed to operate at 2500 psi and 1200°F, with the maximum pressure currently restricted to 1500 psi by the instrumentation.

In this first stage of construction, the regenerator system has been limited to 150 psi rating. However, the most complex component, the regenerator itself, was constructed to 2500-psi standards, in order that two-stage reaction or reaction-regeneration systems could subsequently be studied at high pressure.

When the unit is operating as a conventional fluid catalytic cracker, the spent powdered catalyst drops from the reactor to the stripper and thence to an air lift which brings it back to the regenerator. Regenerated catalyst falls back to the reactor, contacting the supplementary fluidizing gas and hot oil feed spray in the transfer line.

(An alternative non-regenerating catalyst circulating system is provided for simplified high-pressure operation in experiments of conparatively short duration. Catalyst from a fresh catalyst drum is gas-lifted to the reactor-regenerator transfer line. Spent catalyst falls from the stripper to a similar drum. This mode of operation yields many advantages such as the following: 1. reactor upsets that may result from catalyst irregularities caused by regenerator fluctuations are eliminated, 2. there is no risk of mixing regeneration air with combustible hydrocarbon vapours, 3. full operator attention can be applied to the reaction system.)

In the normal operating cycle combustion gases from the regenerator pass through a cyclone for catalyst fines removal, and are filtered, cooled, measured, sampled, and vented.

The product vapours and supplementary fluidizing gas from the reactor are routed through a catalyst fines filter, a condenser, and a cyclone for separating the product mist. Separated liquid product goes to storage. The contaminated gas is treated for H_2S removal, water-washed, and dried. A portion of this clean gas is normally recirculated as fluidizing gas. The remainder is measured, sampled, and vented as excess gas. The system may, however, be operated with total venting (no recirculation), with fluidizing gas supplied continuously from pressure vessels or commercial cylinders. A fractionating unit is presently being designed to continuously separate the reactor total output into miscellaneous refinery products.

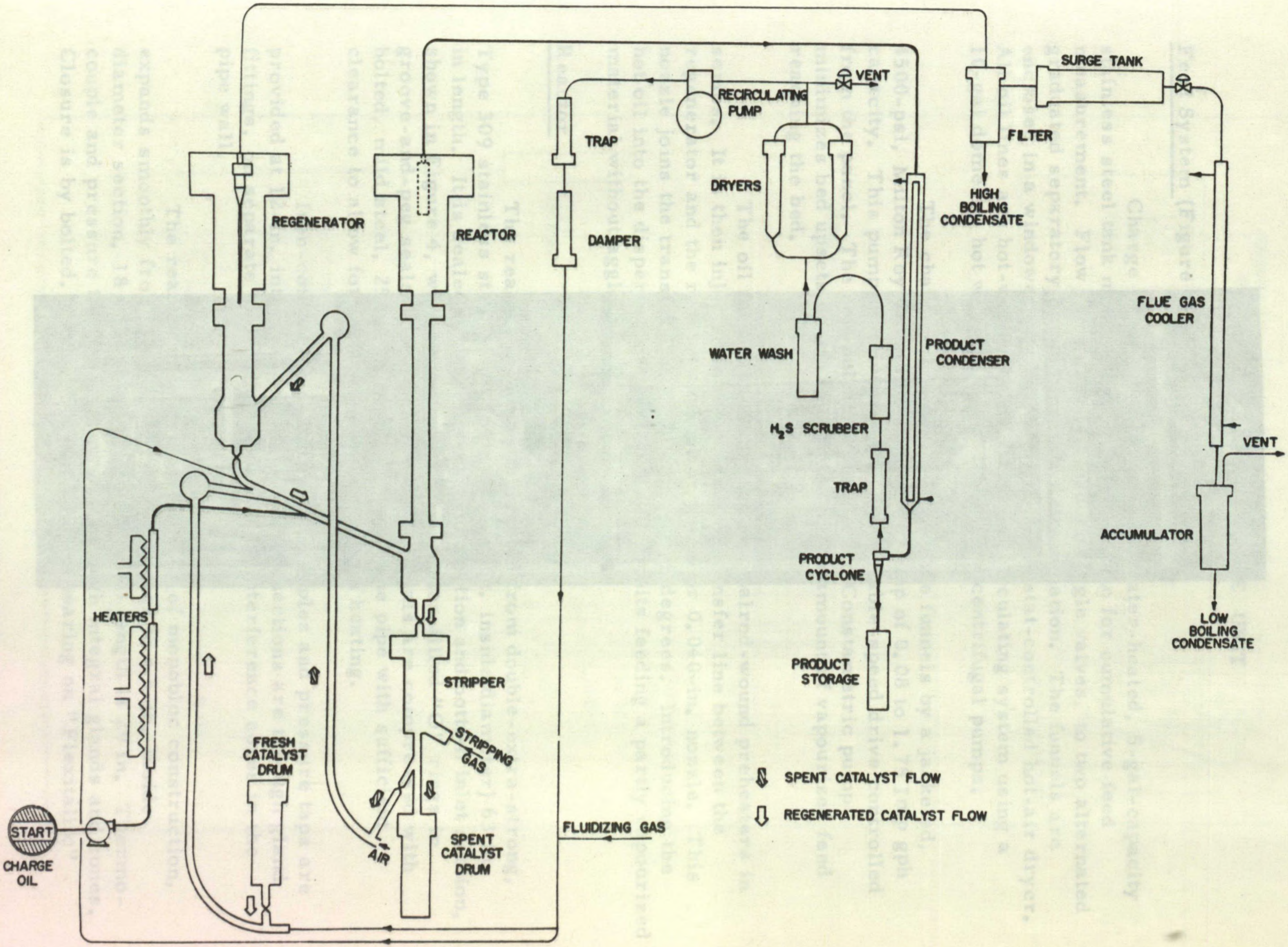

Figure 1. Schematic Layout

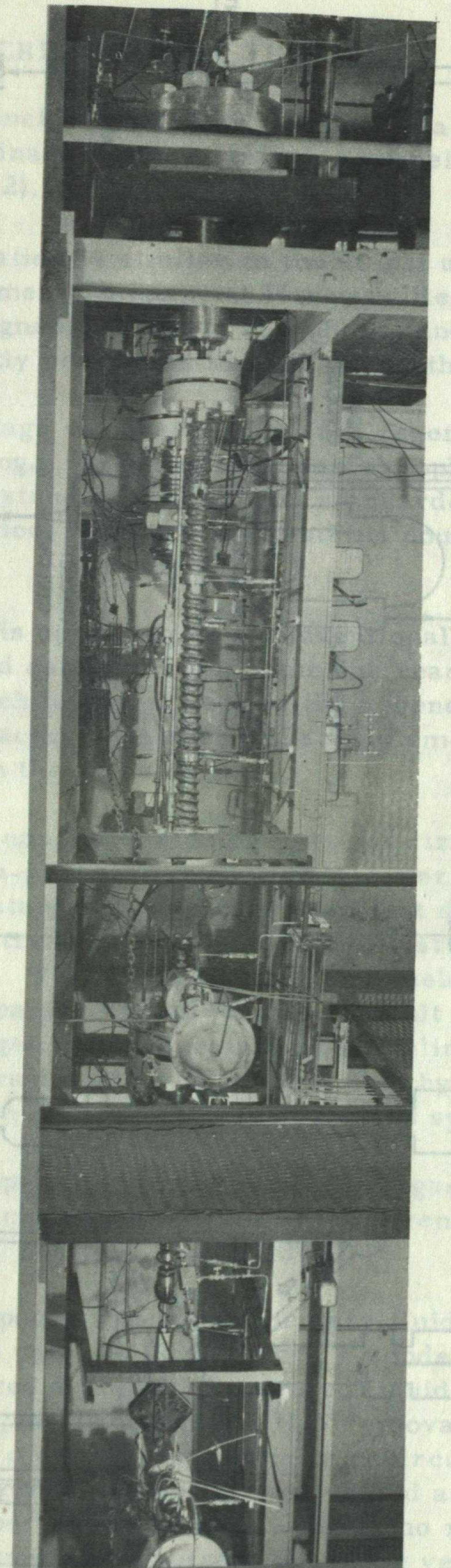

Figure 2. The Main Process Vessels.

COMPONENTS OF THE UNIT

Feed System (Figure 3)

Charge stock is stored in a hot-water-heated, 5-gal-capacity stainless steel tank mounted on a platform scale for cumulative feed measurement. Flow is by gravity, through toggle valves, to two alternated graduated separatory funnels for rate determination. The funnels are enclosed in a windowed box heated by a thermostat-controlled hot-air dryer. All oil lines are hot-water-traced from a recirculating system using a 10-gal domestic hot water tank and laboratory centrifugal pumps.

The charge oil is pumped from the funnels by a jacketed, 4500-psi, Milton Roy Constametric duplex pump of 0.08 to 1.75 Imp gph capacity. This pump is driven by a D.C. variable-speed drive controlled from the panel. The non-pulsating flow of the Constametric pump minimizes bed upsets due to fluctuation of the amount of vapourized feed reaching the bed.

The oil feed passes through two Calrod-wound preheaters in series. It is then injected into the catalyst transfer line between the regenerator and the reactor through an 0.035 or 0.040-in. nozzle. This nozzle joins the transfer line at an angle of 15 degrees. Introducing the hot oil into the dispersed catalyst stream permits feeding a partly vapourized material without agglomeration of the catalyst.

Reactor

The reactor body was fabricated from double-extra-strong, Type 309 stainless steel, 2-in. pipe (1 1/2-in. inside diameter) 63 in. in length. It is sealed to a top disengaging section and bottom inlet section, shown in Figure 4, with stainless steel, asbestos-filled "O" rings in groove-and-peg seals in the pipe ends. The seals are compressed with bolted, mild steel, 2500-psi flanges fitted to the pipe with sufficient clearance to allow for differential expansion on heating.

Iron-constantan pencil thermocouples and pressure taps are provided at 12-in. intervals on the body. Connections are through gland fittings, on separate loose rings, bearing on interference cones in the pipe wall.

The reactor disengaging section, of monobloc construction, expands smoothly from the reactor pipe diameter to a 5-in. inside diameter section, 18 in. in length. The overall length is 29 in. Thermocouple and pressure tap connections are through integral glands and cones. Closure is by bolted, 2500-psi, 6-in. flanges bearing on "Flexitallic"

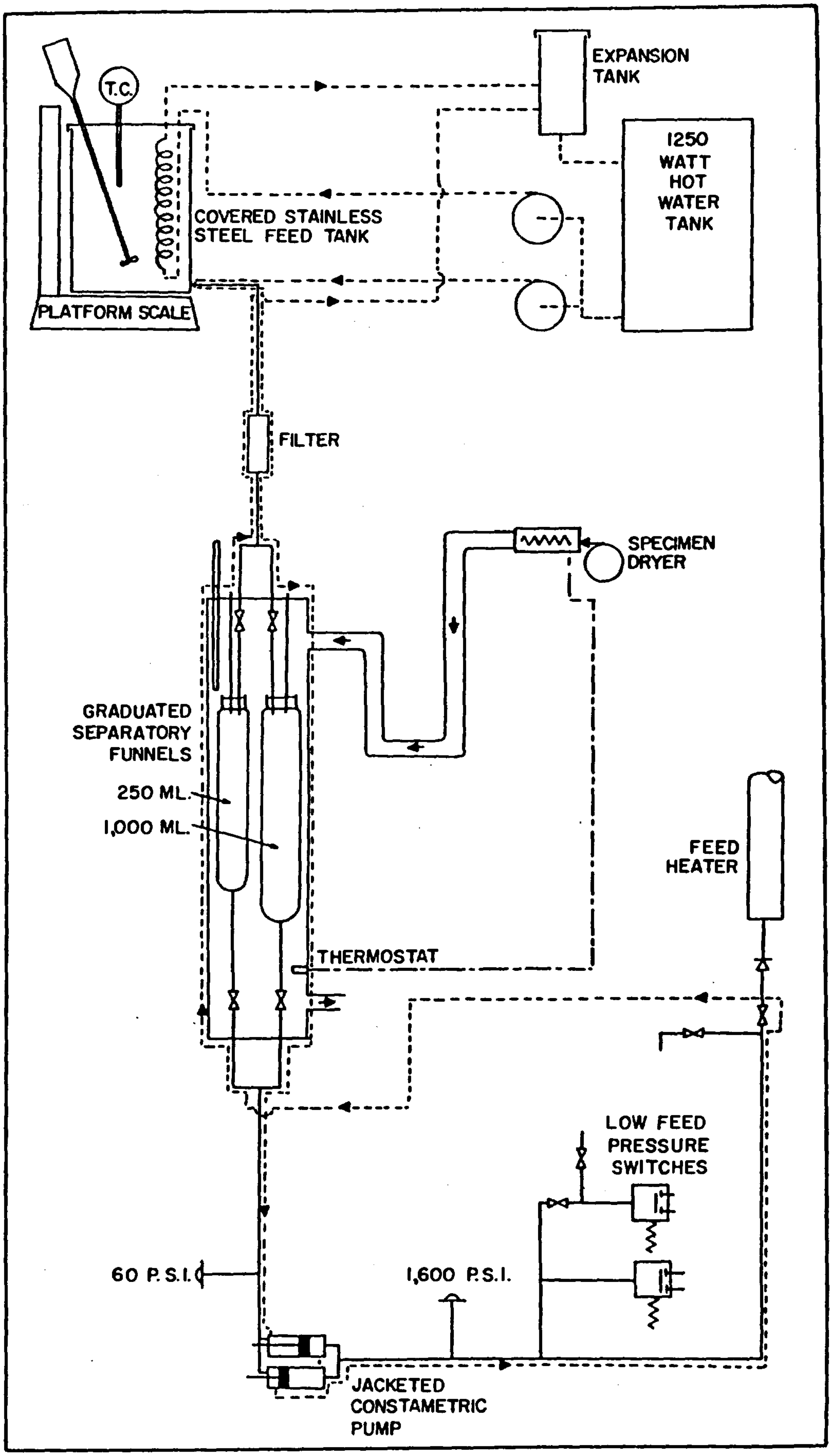

Figure 3. The Feed System.

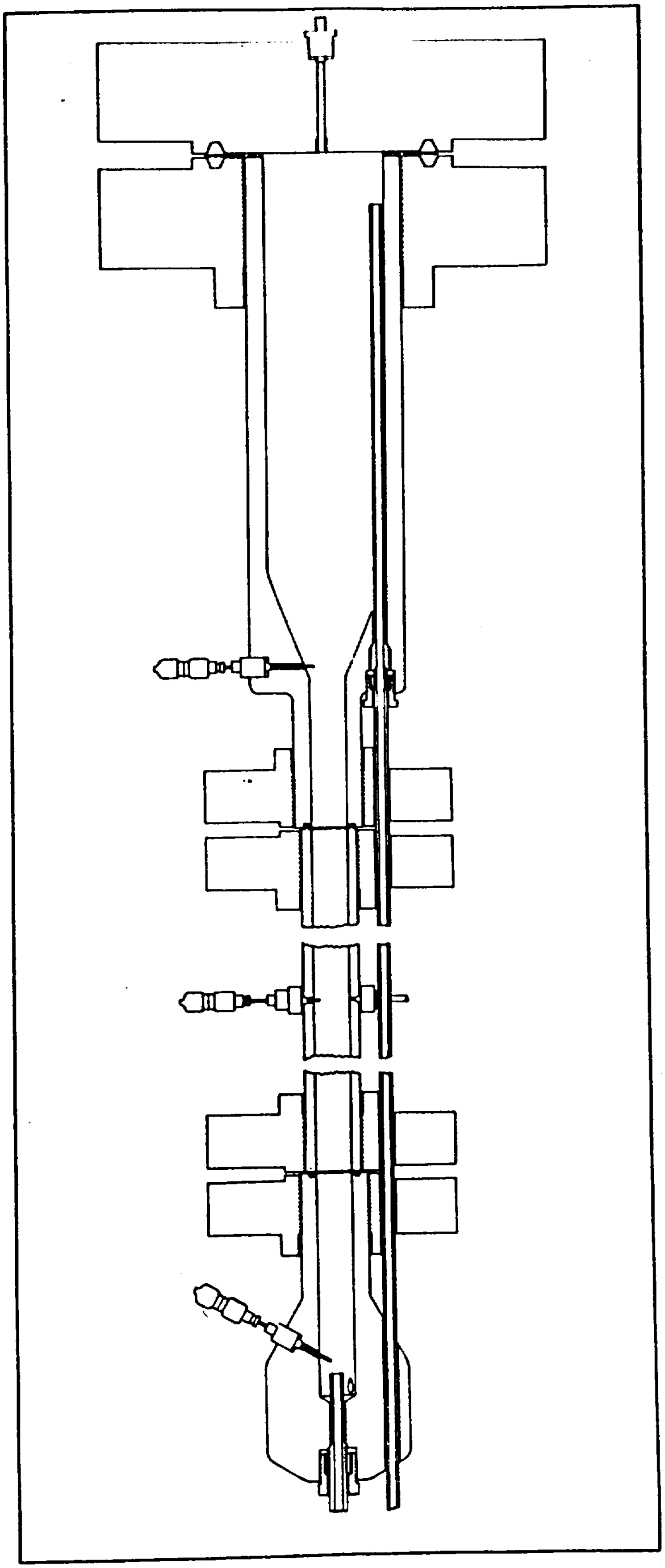

Figure 4. The Reactor

gaskets sealing on the monobloc. Stainless steel was specified for the upper flange, which is in contact with the process fluid. The mating mild steel screwed flange is fitted to the monobloc with sufficient clearance to allow for differential thermal expansion. A bolting alloy steel with an expansion coefficient intermediate between stainless steel and mild steel was chosen, to prevent changes in gasket loading while heating. A cylindrical, 20-micron, sintered stainless steel filter (6 by 1 1/2 in.) catches the entrained catalyst fines at the product outlet from the reactor.

The reactor inlet section is a 9 1/4 in.-deep monobloc. It includes a tangential 5/16-in. diameter fresh-catalyst and feed inlet, a shrouded spent-catalyst outlet, a pressure tap, and a thermocouple connection. All of these are of the interference cone and integral gland type. The overall internal length of the reactor is 101 1/4 in., with 72 1/4 in. devoted to reaction space. This provides an L/D ratio similar to that found satisfactory by Gulf Research and Development.

Slide Valves

The slide valves, Figure 5, control catalyst flow from the reactor to the stripper, from the stripper or fresh catalyst drum to the air lift, and from the regenerator to the reactor.

Stock, high-pressure, finned, control-valve bonnets (Honeywell 800-9D) were used as the actuators and top works. The bodies, machined from Type 309 stainless hexagon bar, contain stellite (to resist catalyst erosion) sleeves and plugs ground to produce an expanding diamond-shaped passage as the valve stem is raised. The plugs were shrunk on Haynes 25 alloy extensions (a machinable alloy with the same coefficient of expansion as stellite). The extensions were bored concentrically and pinned to the valve stems. Radial location of the plugs is by squared, hardened, Type 410 stainless steel guides and by set screws for the sleeves.

The bonnets and end plugs are sealed with monel delta rings, and the inlet and outlet piping by interference cones. A purge inlet on the body prevents catalyst build-up between the plug and sleeve with consequent jamming.

Stripper

The stripper (see Figure 6) was machined from a Type 309 stainless circular bar. It includes a 3 by 15-in. disengaging section, blending smoothly into a 1 3/8 by 7-in. catalyst section with an oblique stripping-gas inlet. Stripped catalyst overflows through an eccentric 5/8-in. diameter duct that runs parallel to, and originates at the same level as, the catalyst section. A gas purge prevents bridging of the

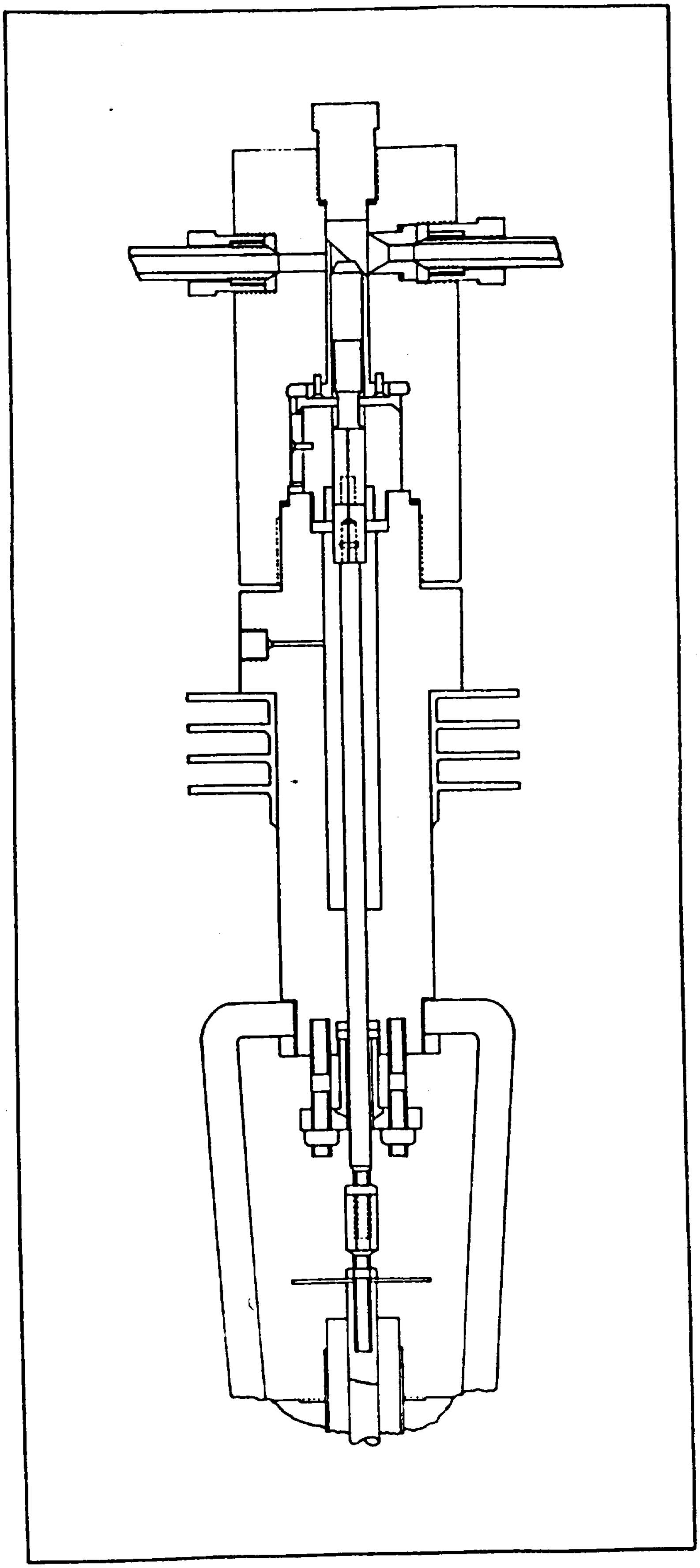

Figure 5. The Slide Valve.

catalyst at this junction. Pencil thermocouples monitor the bed and disengaging space temperatures. Closure is by bolted 4-in. flange and "Flexitallic" gasket as in the reactor. All other connections are of the interference cone type.

Stripping gas, preheated by proximity to the vessel wall, enters the offset inlet. This gas, plus hydrocarbons stripped from the spent catalyst, passes through the top closure and through a line running under the insulation of the slide valve and reactor (to prevent cooling) to the reactor disengaging space. A double interference-cone seal to the disengaging space permits these gases and vapours to be introduced at the level of the reactor filter. Catalyst remaining in the stripper at the end of a run may be taken off through a plug valve at the bottom of the stripping gas inlet.

Regenerator

For normal operation of the unit, catalyst is conveyed from the stripper to the regenerator (Figure 7) for carbon burn-off.

The regenerator disengaging section resembles that of the reactor, but is flanged (3-in., 2500-psi mild steel flanges) to another monobloc which forms the entire lower section. Sealing is also by stainless steel "O" rings and "Flexitallic" gaskets.

The regenerator body is shorter and wider than the reactor, 2 1/4-in. inside diameter by 28 1/2-in. in length. Similar inlet, outlet, thermocouple and pressure tap connections, utilizing integral glands, are provided, with the thermocouples and pressure taps at 3 1/2-in. intervals. Since the flanges make installation of a wall thermocouple awkward, a long thermocouple extends from the top flange to monitor the temperature at this area.

A thin-walled cyclone, made from Type 303 stainless steel bar stock, 8 1/2-in. in length by 1 1/2-in. inside diameter, is threaded to the top flange at the outlet connection and projects down into the regenerator.

A hopper, fabricated from 3-in. pipe and a capped union, is attached to the regenerator top closure flange through a 9/16-in. interference cone adapter. Additional catalyst may be added to the system during operation through the vented, double-valve (3/4-in. gate) lock system which connects the hopper to the regenerator.

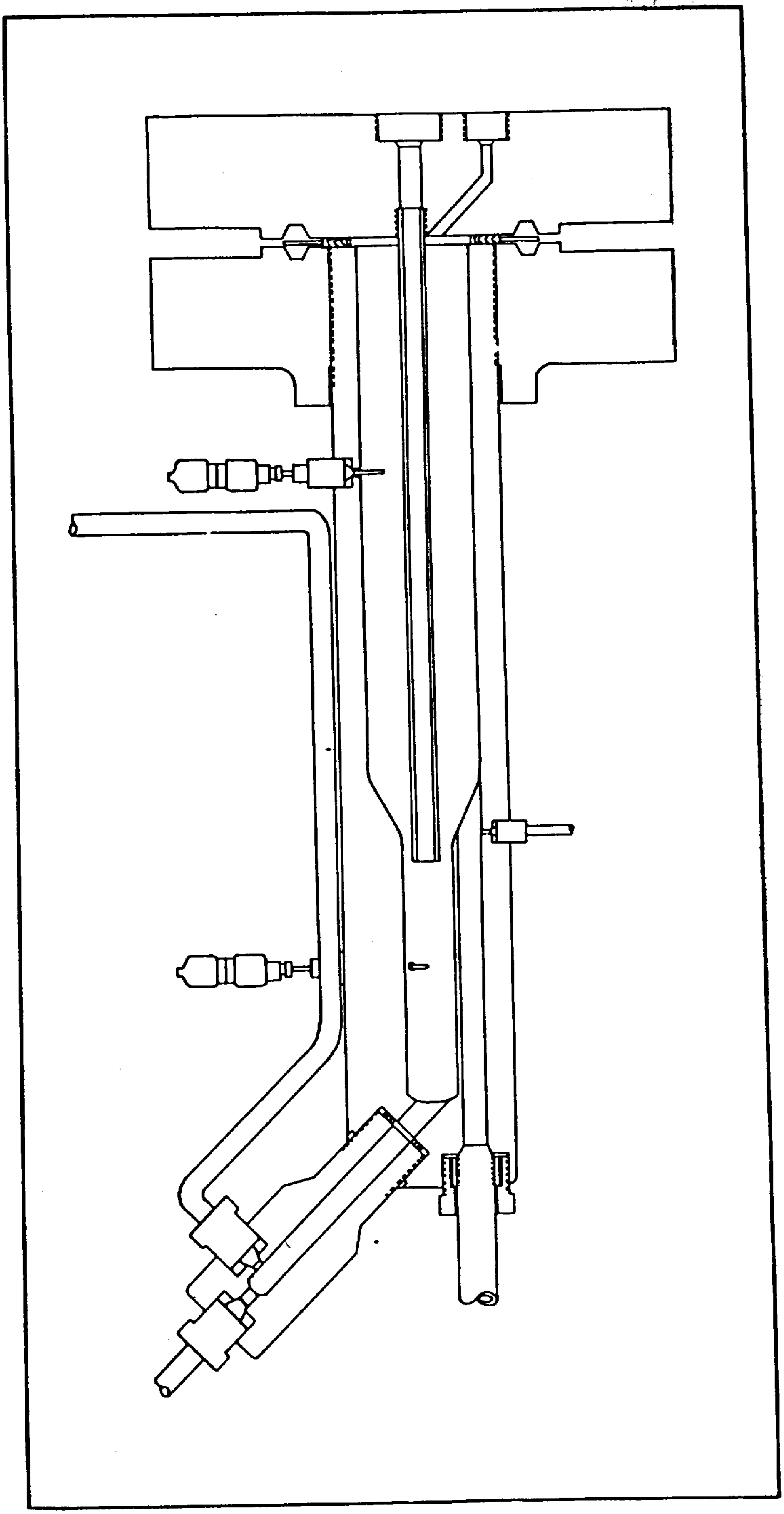

Figure 6. The Stripper

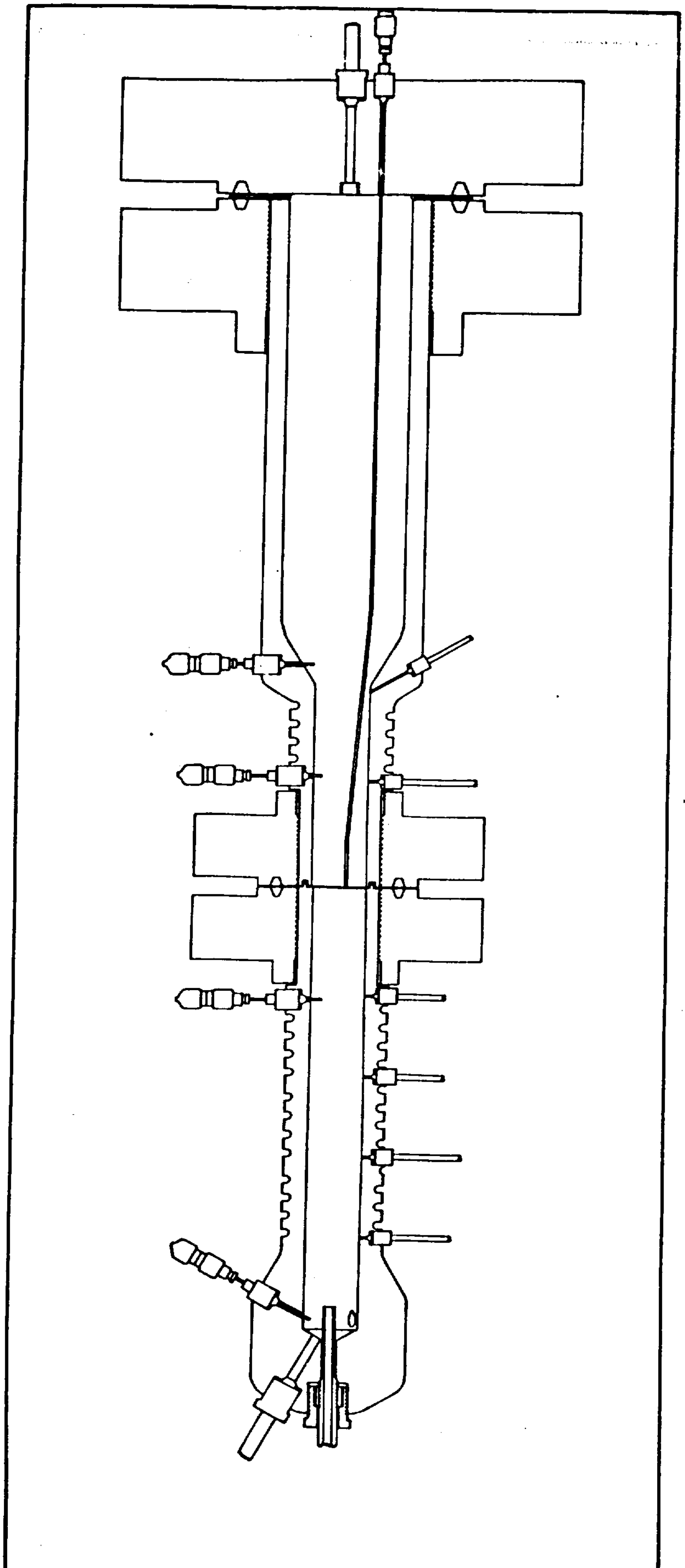

Figure 7. The Regenerator

Catalyst Drums

As an adjunct to the normal integrated operation of the fluid catalyst unit, a separate reactor-stripper catalyst circulation system is provided. This permits the study of reaction schemes in a segmented unit without the complexity of simultaneous regeneration. The catalyst may be regenerated in separate batches between runs, and subsequently well mixed to ensure uniformity during the reactor study. In studies such as hydrofining, the risk of unpredicted combinations of hydrogen, hydrocarbon vapours, and regeneration air is eliminated in this mode of operation. Because the regenerated catalyst is supplied from a pressure vessel and run from the stripper to a similar vessel, only experiments of limited duration are possible with this scheme.

The 1.1-cu ft catalyst drums were fabricated from schedule 80, Type 316 stainless steel, 6-in. pipe, 6 ft in length. The bottom seals are by welded, stainless, concentric reducers screwed (1/2-in. pipe thread) into interference-cone adapters. Top closures are by bolted 1500-psi flanges compressing Flexitallic gaskets on the pipe ends. The top blind flanges are Type 316 stainless steel. The mating screwed flanges are mild steel. All of the vessels built at the Mines Branch for this unit feature similar construction as shown in Figure 8. The closures were either Flexitallic gaskets, or octagonal ring joints in mating stainless steel flanges.

The fresh-catalyst vessel supplies hot catalyst to the lift unit to be conveyed to the transfer line between the regenerator and reactor. This lift unit duplicates the air lift to the regenerator to facilitate transfer of the instrument functions from this system to conventional operation.

Flue Gas System

Combustion products (flue gas) from the regenerator, after separation of the entrained catalyst in the cyclone, pass through a filter vessel. Here, the high-boiling hydrocarbon mist and residual catalyst are removed. The cylindrical alumina filters (2 by 12-in., 15-micron) are housed in an 0.9-cu ft Type 347 stainless steel vessel of typical Mines Branch construction. A 15-in. length of schedule 40, 3 1/2-in. pipe was welded at the bottom to a concentric reducer with a 1/2-in. female pipe thread. Top closure is by a 150-psi stainless steel screwed flange sealing on a mating blind flange with an octagonal ring. The screwed flange was welded to the pipe body as difficulty was experienced in making a gas tight seal with the 3 1/2-in. pipe thread.

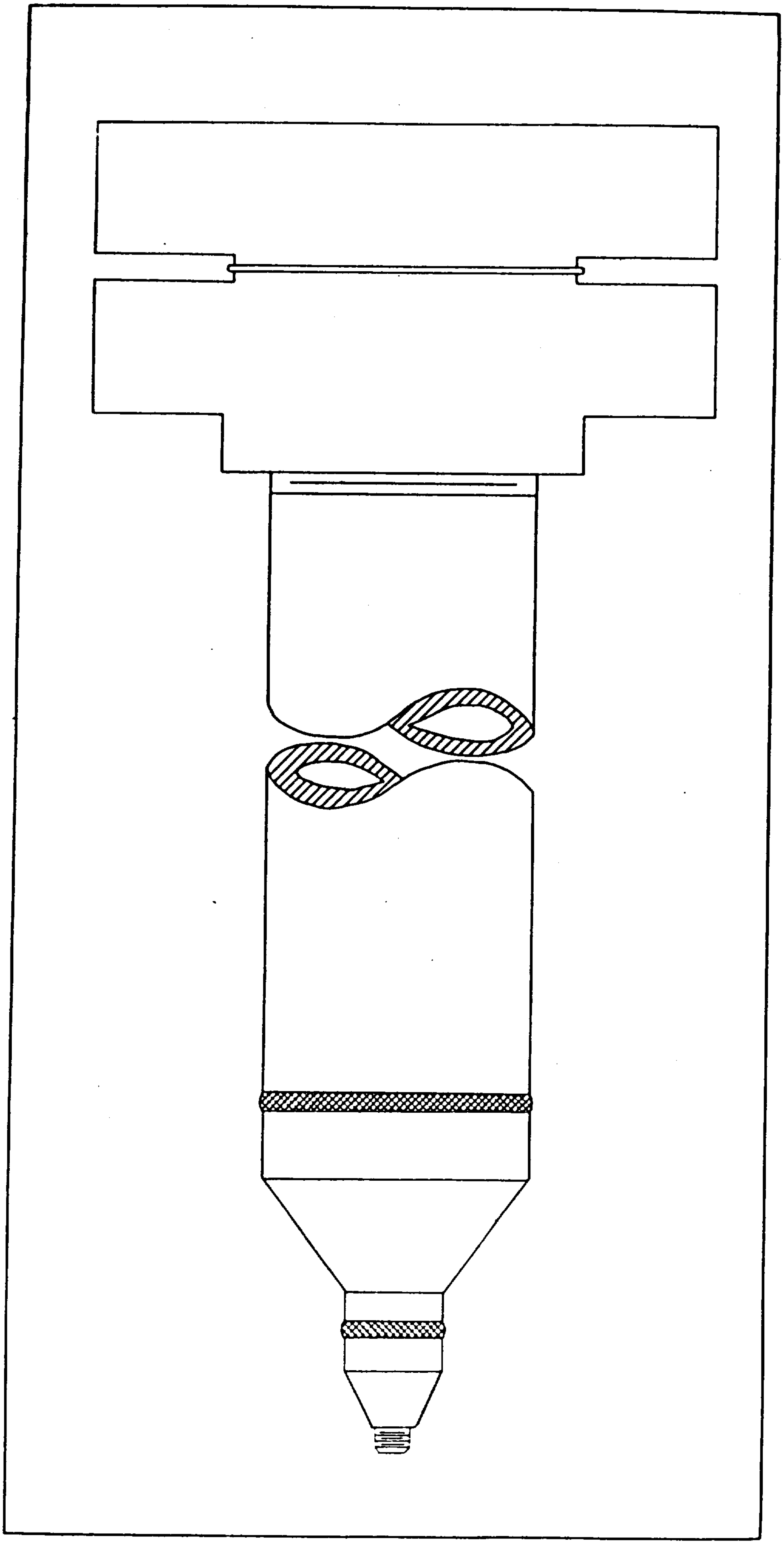

Figure 8. The Catalyst Drum.

The flue gas then flows through an 0.18-cu ft surge tank, of similar construction to the filter, to damp out fluctuations from the action of the control valve, which may disturb the regenerator bed. This valve, known as an Annin "Wee Willie Domotor", was chosen for its versatility, (rangeability, 100,000:1). It relates the regenerator pressure to the reactor pressure by controlling the rate of flue gas venting.

The vent gas, now at atmospheric pressure, is cooled in a "heat exchanger" made from 6 1/2 ft of 5/16-in. inside diameter stainless steel tubing, wound with two cooling coils of 3/16-in, copper tubing. These coils are connected in parallel to the water supply, and potted in "Thermon" (a high-conductivity hardening paste). An 0.07-cu ft vessel similar to the filter and surge tank receives the condensed low-boiling hydrocarbons and water from this exchanger. The cooled flue gas passes through an American Wet Test Meter (150 cfh, Monel, with removable back), is sampled by a Sigma pump, and is vented.

Product System

Overhead vapours and fluidizing gas from the reactor are carried by a sloped line to the product condenser. This condenser utilizes two identical three-component sections, connected in parallel. These are suspended vertically to provide the requisite area while minimizing hold-up.

Each of these identical sections has an atmospheric-air-cooled portion of 5/16-in. inside diameter tubing, 44 in. long. This portion provides the initial moderate cooling to prevent product fog formation due to rapid chilling. A 112-in. water-cooled portion follows. This was fabricated from 0.209-in. inside diameter tubing enclosed in 3/4-in. brass tubing which was silver-soldered to bronze expansion bellows and sealed in Swagelock tees with bored-through reducers. A final 42-in. portion similar to the water-cooled portion contains circulating refrigerant to remove "fixed" hydrocarbon gases from the recirculating gas stream.

The condensed product and gases pass from the condensers to a small cyclone for product separation. This cyclone is of monobloc construction with a threaded closure sealing on a compressible gasket. Removable inserts form an involute entrance. Its capacity may be adjusted to varying product rates by changing inserts.

The separated product receiver system is composed of a 4-gal vessel as a general receiver, plus three sample receivers of 0.05-cu ft capacity. These receivers are continuously vented to the gas system, with a separate manifold provided to permit running the product to any of the four vessels. It was felt that this system would be more trouble-free in operation than one based on continuous product removal. Gas displaced from the receivers will be automatically vented through the system-pressure control valve.

The general receiver was purchased from Autoclave Engineers Incorporated. A modified delta-ring closure sealed by an integral flange is used. The sample receivers were each fabricated from a 28-in. section of schedule 80, Type 316 stainless 2-in. pipe. These sections are screwed and welded at the bottom ends to 2 by 1/2-in. reducers, and are sealed at the tops by 1500-psi stainless and mild steel flanges compressing Flexitallic gaskets.

Recirculation Gas Conditioning

Gas from the product cyclone runs to an 0.03-cu ft trap vessel formed from 8 inches of schedule 160, Type 316 stainless 3-in. pipe sealed at the top with stainless 1500-psi flanges and an octagonal ring, and welded at the bottom to a reducer with an integral 3/8-in. Ermeto connection.

The gas then reaches a 2-gal vessel, similar to the general receiver, containing a stainless steel canister of iron-sponge H_2S absorbant. After this the H_2S-free gas bubbles through an immersed inlet in an 0.08-cu ft water wash vessel and one of two alternate 0.06-cu ft "Drierite" dryers. The water wash vessel and dryers are made from schedule 80, Type 316 stainless 3-in. pipe (22 and 15 in., respectively) with 316 stainless 1500-psi top flanges sealing on 316 stainless octagonal rings. Bottom sealing is by welded 316 stainless reducers threaded (1/4-inch pipe) to 3/8-in. Ermeto adapters.

The excess clean gas is vented through a "Wee Willie Domotor", controlling the reactor pressure. This gas is then measured in an American Wet Test Meter (150 cfh, stainless, with removable back), sampled by Sigma pump, and vented.

Gas Recirculation System

After venting the excess, the remaining clean gas is moved around the system by a reciprocating gas pump. This pump is a single-cylinder "Rix" compressor with a 1/2-in. bore and 3-in. stroke, driven by a Reeves variable-speed drive through a 5 to 1 reduction "Vee" belt.

Gas circulated by the pump passes into a trap to remove any lubricant, etc., added by the pump, and then to a pulsation damper with two adjustable weirs. The trap and pulsation damper (both 0.05 cu ft) are each made from 1 ft of schedule 80, Type 316 stainless steel 3-in. pipe welded at one end to a reducer with an integral 3/8-in. Ermeto connection. The other end closure is by Type 316 stainless, 1500-psi flanges, sealing on an octagonal ring.

The smooth flow of clean recirculating gas is then measured in a Model 5107 Foxboro recording flowmeter and a Model 1432 Brooks Hi-Pressure Rotameter. At this point, a fresh gas stream from commercial cylinders may be added to supplement or replace the recirculation gas. The total gas flow may be used to pick up catalyst below the fresh catalyst drum slide valve, or part may be added to the regenerator to reactor transfer line. In either case, it serves finally to assist the feed vapours in fluidizing the catalyst in the reactor.

CONNECTING LINES

The fluid catalyst unit was designed to facilitate disassembly, transport, and reassembly. This was necessitated by the proposed relocation of the Department's research facilities to the National Capital Commission's "Greenbelt" area. To this end, many components were connected with gland-type seals rather than welds. This, of course, meant that considerable attention had to be devoted to expansion problems to avoid leakage on heating.

The main catalyst circuit (reactor, regenerator, stripper, catalyst drums, and associated slide valves) is piped with Type 309 stainless steel tubing (9/16 by 5/16 in.) bearing on interference cones with Type 309 stainless glands and collars made by Autoclave Engineers Incorporated. Special 1 by 5/8-in. lines with proportional glands and collars were fabricated from Type 309 stainless bar for areas in which difficulty with the catalyst flow was anticipated. These include reactor to slide valve, slide valve to stripper, below stripper, and regenerator to slide valve. Purge gas connections to these 1-in. lines are by loose gland rings (Type 309 stainless) bearing on interference cones in the pipe walls. Such connections in the 9/16-in. lines are by conventional tees and reducers. "Silver Goop", an anti-galling and sealing compound with a service temperature of 2000°F, was used on all high-temperature lines, bolts, and connections in the unit. All catalyst lines avoid abrupt bends to minimize erosion and reduce the incidence of flow blockage.

The cold gas and product lines of the system were formed from 3/8 by 0.209-in. Type 304 stainless tubing with Type 316 stainless Ermeto sleeves and Type 416 glands.

The high-pressure instrument and purge lines are 1/4 by 1/8-in. Type 304 stainless tubing, or 1/4 by 0.083-in. Type 304 tubing in areas close to the catalyst circuit. It was felt that the higher gas velocities in this smaller inside diameter tubing would reduce the risk of catalyst contamination in the lines. Hoke 10-micron filters (Model 540) are interposed between the instrumentation and the connection to the process vessels, as well as in the dead-end branch lines to the differential pressure converters, as further precautions against catalyst contamination. The instrument lines are connected with Autoclave Engineers' Ermeto seals (416 glands, 316 sleeves), Imperial Hi-Seal fittings (316 glands and sleeves), and Autoclave Engineers' interference cones. The latter are used in locations which must be disassembled for moving, as these fittings may be easily resurfaced if damaged.

All low-pressure instrument lines and supply air lines are piped in 3/8 or 1/4-in. copper tubing with Imperial compression fittings. These lines can be replaced, at minor expense, after the unit is moved.

Autoclave Engineers' 3/8-in. Ermeto "Speed Valves" are used in the process circuit in crucial applications. Otherwise, Crane 1/4-in. pipe valves (3000 psi) or Nordstrom 1/4-in. pipe lubricated plug cocks (3000 psi) were installed with pipe to Ermeto adapters. Instrument line valves in the process area are generally Imperial Hi-Seal, with several Autoclave Engineers' interference-cone valves where disassembly will be required.

INSTRUMENTATION

Panel Boards

The instruments are mounted on a semi-graphic panel (see Figure 9 and 10). The lower section of this panel is made from three 10-gauge steel plates (79 by 95 in., 72 1/2 by 95 in., and 72 x 95 in.) bolted to the catwalk structural steel. This panel section contains rotameters with associated valving, regulators, differential-pressure recorder-controllers, the flow recorder, the temperature indicator and recorder, manometers, temperature controllers, and pressure gauges. The equipment on the three individual plates is assembled so as to permit splitting into three units for moving. The upper section of the panel holds a graphic flow sheet of the main components of the unit. It was made from two plain 12-gauge steel plates (60 by 79 in. and 60 by 74 1/2 in.) located directly above the control section of the panel. The instrumentation on the functional section below is referenced to the flow sheet above by graphic lines or code.

A second 10-gauge panel (75 by 102 in.) is free-standing at right angles to the main panel. This contains the circuit breakers, variable transformers, switches, and ammeters for the heaters, and is portable as a unit.

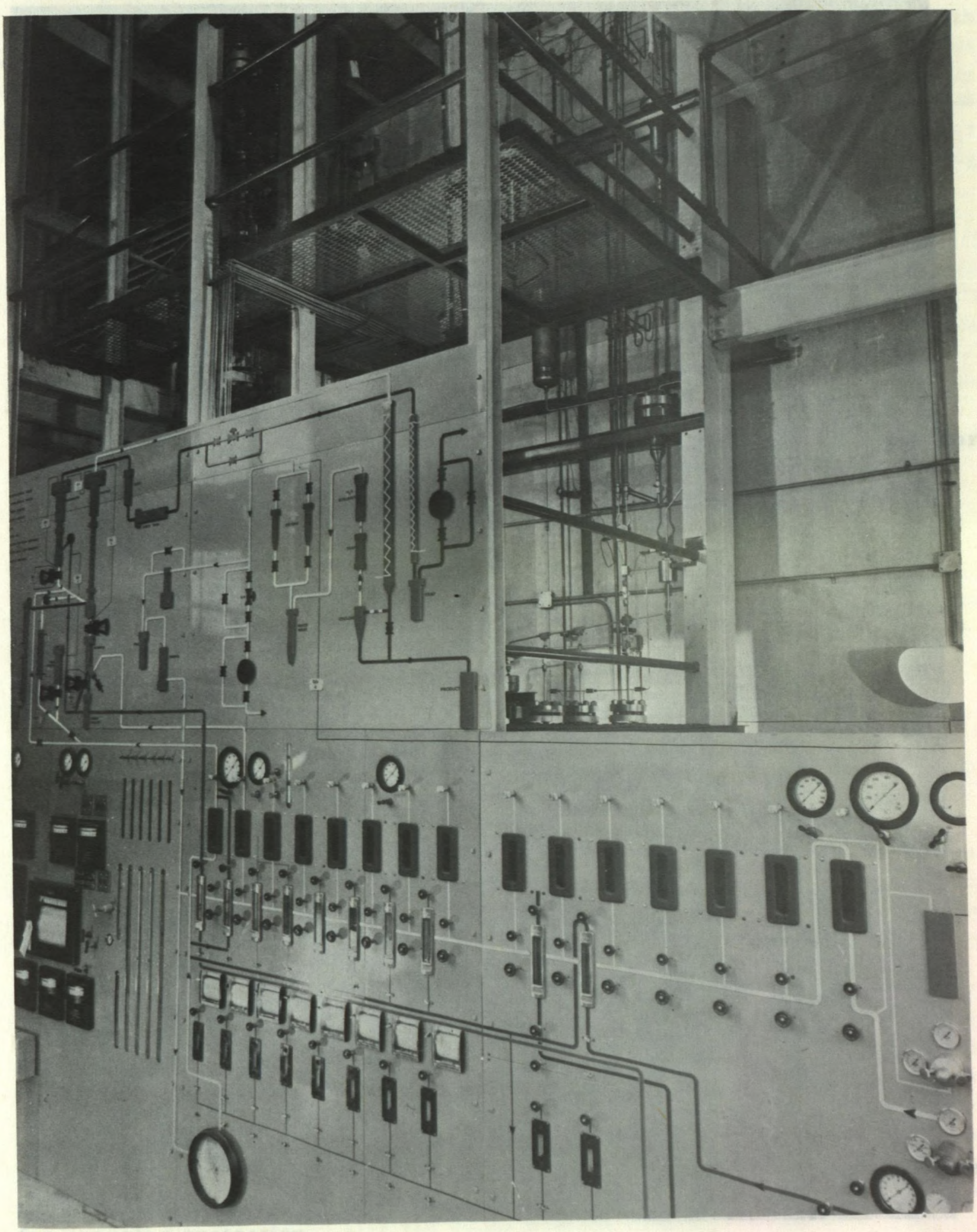

Figure 9. The Control Panel (Front View)

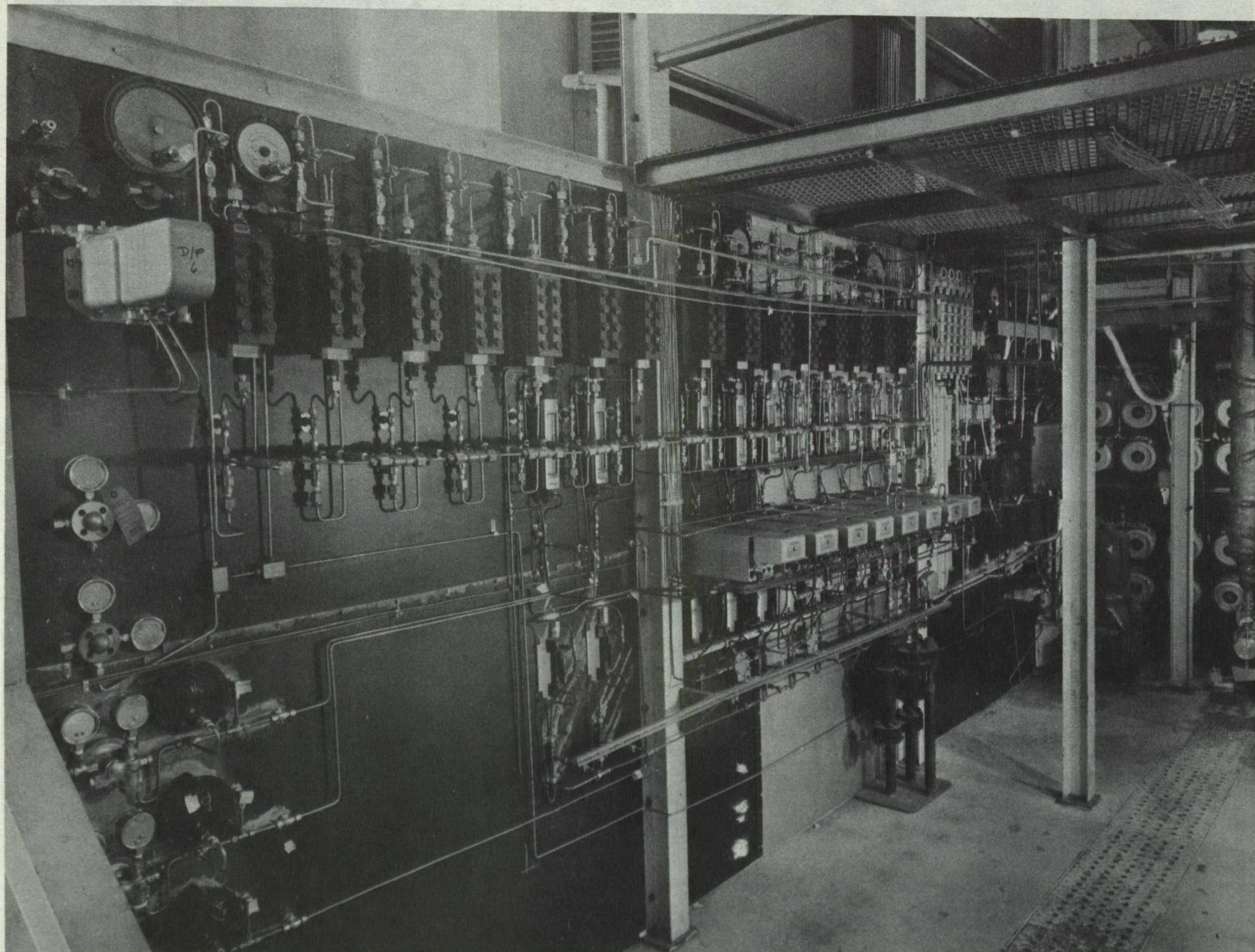

Figure 10. The Control Panel (Rear View)

Temperature

Temperatures throughout the unit (refrigerant excepted) are determined with iron-constantan thermocouples. In the process vessels and lines, 1/8-in. diameter Autoclave Engineers' pencil thermocouples are used. Thermo Electric pencil thermocouples, silver soldered to 1/2-in. brass pipe plugs, are employed in the cooling water and refrigerant systems. The numerous skin thermocouples were fabricated from fused thermocouple wire and applied with "Thermon" and hose clamps.

Reactor and regenerator temperatures are monitored continuously on a Honeywell "Electronik" 12-point recorder. Other non-control temperatures throughout the unit are checked at intervals with a Honeywell "Electronik" 48-point indicator.

Differential Pressure

The differential pressures across the reactor bed, regenerator bed, spent-catalyst lift unit, and fresh-catalyst lift unit are measured by Honeywell differential converters (Model N 7). Bleed gas flows keep the instrument taps free of catalyst. These differential pressures are also monitored by mercury manometers. The high-pressure manometers were built from Penberthy reflex gauges (Model W 18), while Meriam "Standard Cleanout Manometers" are used in the low-pressure applications. Mercury traps are provided in all cases.

The 3-15 psi signals from the differential converters are fed to Foxboro Model M-53 Consotrol Recorders with Model M-58 proportional-plus-reset controllers. Two reverse-acting controllers operate slide valves below the reactor and regenerator to control their bed levels. A third direct-acting controller regulates the catalyst flow rate in the air lift to the regenerator, or the gas lift to the reactor, by operating the slide valves below the stripper and catalyst drum, respectively.

Pressure

Gas streams to the unit are supplied from banks of commercial gas cylinders, or from the High Pressure Chemistry Section's storage vessels. The high-pressure gas is regulated by panel-mounted, 3,000-psi, Hoke Model 521 regulators. The low-pressure requirements are supplied from National Cylinder Gas Company regulators, also panel mounted.

The gas supply pressures and the system pressures are indicated by panel-mounted 4-in. and 6-in. gauges. All of the high-pressure gauges are equipped with blow-out backs, and are mounted above head level for operator protection.

The absolute pressure of the reactor is set by comparing it with that of a preloaded 0.046-cu ft vessel with a Honeywell Model N-7 differential converter. The signal from the converter is sent to a direct-acting Foxboro recorder-controller (M-53, M-58). This controls the rate of venting excess gas from the reactor circuit through an "air-to-open" Wee Willie Domotor control valve. An identical control loop relates the reactor-regenerator differential pressure by controlling the rate of flue gas venting.

Bleed and process gas flows are metered through thirteen Brooks Model 1432 Hi-Pressure Rotameters, two Schutte and Koerting Model 1822-A Armoured Rotameters, ten Fischer and Porter Model 10A2700 Flowrators, and ten Fischer and Porter 10A3135 Purge Meters. The flows are manually controlled by Hoke Model 280 metering valves with vernier handwheels, placed downstream from the rotameters. By-pass and shut-off valves for each rotameter are also mounted on the panel.

Recirculating Gas

As indicated previously, the recirculating gas rate is recorded with a Foxboro recording flowmeter and indicated on a Brooks Hi-Pressure rotameter. This value is used to reset the 3-15 psi air signal to the "AIR-trol" which controls the speed of the recirculating pump's Reeves drive. Closed loop control may be fitted as a future refinement.

Fresh gas to the unit is run through the Hi-Pressure rotameter, with the rate manually set by the metering valve.

Catalyst Rate

The rate of catalyst transfer from the stripper to the regenerator, or to the reactor from the fresh catalyst drum, is continuously and automatically controlled. If the bed inventories are held constant, this same catalyst flow rate must be obtained throughout the unit.

At a constant gas rate, the pressure drop in a transfer line will be proportional to the catalyst rate. Accordingly, the pressure drop over a specified length of spent-catalyst transfer line to the regenerator is monitored by a Honeywell differential converter. The drop in an identical section of transfer line from the fresh catalyst drum is measured by a similar converter for the alternate system. The signal from either converter energizes a common Foxboro recorder-controller (M-53, M-58, proportional-plus-reset with an anti-windup autoselector feature). The use of identical sensing loops permits catalyst flows to be compared directly in the two systems. The direct-acting controller operates the slide valve below the stripper or the fresh catalyst drum to maintain a constant catalyst inventory in the measured section of the transfer line.

The actual catalyst flow in the transfer lines is checked periodically by means of 4-way valves (appendix 1) and rate boots. The 4-way valve diverts the catalyst flow into a removable chamber (rate boot) for a specified interval, allowing the filtered gas to continue through the transfer line. After the normal catalyst flow has been restored, the lower section of the rate boot is removed and weighed.

The rate boots were constructed from two Type 316 stainless steel monoblocs fitted to Type 316, 3000 psi, 1 1/2-in. pipe unions. The lower monobloc, which is 7 5/8 in. long, is removable. The upper, 3 1/4 in. in length, contains a 10-micron filter on the outlet. It is connected, through Hoke 3/8-in. "Rotoball" valves, to the 4-way valve. In this way, a tight shut-off is unnecessary in the 4-way valve. The rate boots are re-pressurized with inert gas after each catalyst-rate determination.

HEATERS AND CONTROLS

The vessels and piping of the catalyst circuit are heated with 220-volt Inconel-sheathed General Electric "Calrods" or stainless-steel-sheathed Pyrotenax heating cable. The heaters are imbedded in Thermon heat transfer mud to improve heat conduction to the process components. Fifteen-amp circuit breakers, Powerstat Model 236 variable transformers, toggle switches, and 15-amp Simpson Model 157 ammeters are mounted on a single panel for each of the thirty-four heaters. As the variable transformers have a 9-amp maximum-rated-current output, separate fuses are provided to protect them from overload.

Eight heaters in critical areas throughout the unit are connected through panel-mounted Honeywell Model 105 C 4 PYR-O-VANE indicating temperature controllers (time proportioning). These heaters (two reactor, two regenerator, fresh catalyst to reactor, spent catalyst to regenerator, product from reactor, and high-temperature feed preheater) are controlled from process or skin thermocouples in their immediate vicinities. The remaining twenty-six are manually adjusted by the variable transformers according to the temperatures shown on the indicator and recorder.

SAFETY

Safety Heads

The high-pressure vessels, circulating pump, rotameter manifolds, differential converters, regulators, and manometers are protected by several Autoclave Engineers Incorporated safety heads of 1600-psi burst rating. These heads are placed so that at least one will be connected to each subdivision in the circuit which might be isolated by valves or blanks. Calculations indicated that pressures of 300+ psi may be reached in the safety head blowdown line, and pipe and tubing of consistent ratings were therefore used. A spoiler plate across the outlet of the blowdown line prevents the reaction of the expanding gas from damaging the line supports.

The low-pressure equipment is protected with 60-psi safety heads at all points where the high-pressure gas might inadvertently be admitted. A separate blowdown system is, of course, provided for these safety heads.

Anti-Flow-Reversal System (2)

In normal operation, the reactor and stripper contain combustible hydrocarbon vapours, while the air lift and regenerator contain air. Intermixing of the two systems would therefore lead to fires or explosions. This intermixing could occur if the air to the air lift travelled up through the slide valve below the stripper, instead of the spent catalyst travelling down. Similarly, hydrocarbon vapours could pass up through the regenerator slide valve, rather than the regenerated catalyst passing down. In either case (increasing regenerator bed height or reduced inventory in the air lift), the normal signal would tend to decrease to open the slide valve. This, of course, would favour the process upset that was causing the reversal of flow to take place. For flow reversal to occur, however, the pressure drop across the particular slide valve must pass through zero. These pressure drops are monitored by differential converters (Foxboro Model 13A5) and fed to reverse-acting proportional recorder-controllers (Foxboro M-53, M-58). The respective controller sends out an increasing signal as flow reversal becomes imminent. This signal and that from the normal control loop are fed to a Moore Model 58S auto-selector relay which transmits the higher of the two input signals. Thus, an upset tending to flow reversal causes the alternate control loop to take over and close the slide valve. This prevents flow reversal and may tend to alleviate the factors causing the upset. This alleviation could cause the pressure drop across the slide valve to be re-established in the normal direction, resulting in a decay of the anti-flow-reversal loop signal and a consequent takeover by the normal control loop.

In order to prevent windup (in the controller reset bellows), which would result in "bumping" on the re-establishment of normal control, a special auto-selector controller is used. This controller takes its reset signal externally from the auto-selector relay output rather than from the normal internal connection to the controller output. A proportional-only controller is used for the anit-flow-reversal function to eliminate this difficulty in the pressure-drop loop.

Explosion Wall

For the study of unknown reactions at high pressures, it was decided to make provision for an explosion wall around the heated segment of the unit. This wall, cut from 3/8-in. mild steel plate, is fitted into the catwalk support that surrounds the unit on three levels. The structural steel of the catwalk supports the outside edges of the plates. Access doors which overlap the plate so as to be similarly supported are provided on each level. Non-walled areas will be covered with heavy wire mesh belting, to limit projectile travel in the case of equipment rupture.

Interlocks

The feed oil flow is monitored by pressure switches (failure of the flow automatically shuts down the feed preheaters and the oil-catalyst transfer line heater). A micro-switch operated by a decrease in flow of the recirculating gas to the recording flowmeter shuts down the feed pump (and as a consequence, the three heaters on its interlock), the fresh catalyst to reactor heater or the recirculating gas to the oil-catalyst transfer line heater, and three reactor heaters.

Pressure Test

The vessels, lines, compressors, valves, and other parts of the unit were pressure-tested to 1975 psi with commercial cylinder gas (the Autoclave Engineers' vessels excepted). The catalyst drums and the product condensers were tested hydraulically to 3900 and 4000 psi respectively, because of the nature of the welds that were required in these components.

. The various welded sections of the unit were x-rayed on a spot-check basis before installation and pressure-testing. The thicknesses of critical vessels and lines were checked with an ultrasonic device for subsequent determination of corrosion and erosion rates.

Blanketing Gas

A solenoid operated 3-way valve is provided in the supply manifold to the gas regulators. If the system is operating with a combustible gas such as hydrogen, this valve will permit the introduction of inert gas through all of the system bleeds to smother an uncontrolled combustion.

SUPPORTS

Because the catalyst lines connecting the various components of the fluid catalyst unit must be smooth-walled and have no sharp bends (to facilitate catalyst flow and minimize erosion), conventional expansion joints and bends may not be used. Instead, the components were allowed to move relative to each other to compensate for thermal expansion.

The main process circuit (reactor, regenerator, stripper, slide valves, and catalyst drums) is supported on two 15-in. channels 32 in. apart. These channels were existing framework from a previous experimental program. They were adapted by welding an additional 32 in. of 12-in. channel to the top. Two 15-in. I-beams, located 39 in. away from the channels, and 62 in. apart, serve as support for the ancillary equipment (product separation and gas conditioning).

The reactor and the regenerator are counterbalanced on a rocking platform in the manner of an analytical balance. Hardened tool steel (Ultimo 4) knife edges and plates permit the vessels to move relative to one another with a minimum of force. All slide valves in the unit are cable-suspended at their balance points over pulleys to counterweights. Both the stripper and the spent catalyst vessel rest on 3 by 1 1/2-in. channels bolted between the 15-in. channels. Partly compressed springs between the vessel closure flanges and the supports provide sufficient freedom of movement to allow expansion.

The product recovery and gas conditioning components are carried on various 3 by 1 1/2-in. channels bolted between the two 15-in. I beams. Right angle bends in the associated piping compensate for differential contraction in the cooled parts of this system, so the vessels rest directly on the supporting channels. Expansion of the product condenser is taken up in a spring-loaded clevis at the top.

INSULATION

The hot process vessels and piping are insulated with 1 1/2-in. of hydrous calcium silicate pipe insulation. The regenerator, because of its complexity, is enclosed in an expanded mica-filled aluminum casing. The low-temperature product separation section is insulated with 2 1/2 in. of cellular glass.

DISCUSSION

Most of the significant features of the fluid catalyst unit have been described in this report. The calculations and other design criteria, on which the design details are based, are on file in the Petroleum Process Engineering Section. Subsequent reports will deal with the operating experience with this unit, and any detail modifications which may be required.

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APPENDIX

The 4-Way Valve

The 4-way valve (see Figure 11) has developed through several evolutionary changes which are described in this appendix. This valve is a plug-type rotating valve with four tubing connections at 90°. Rotating the stem through 90° connects adjacent taps so that a catalyst-gas suspension may pass through one corner of the valve, or be redirected into the rate boot, with the gas return from the rate boot following the normal circulating line.

The 4-way valve body and cap were machined from Type 309 stainless steel 4-in. hexagon bar stock. Closure is by Monel delta ring, a Viton S "O" ring seals the stem, and tubing connections are 9/16-in. interference cone.

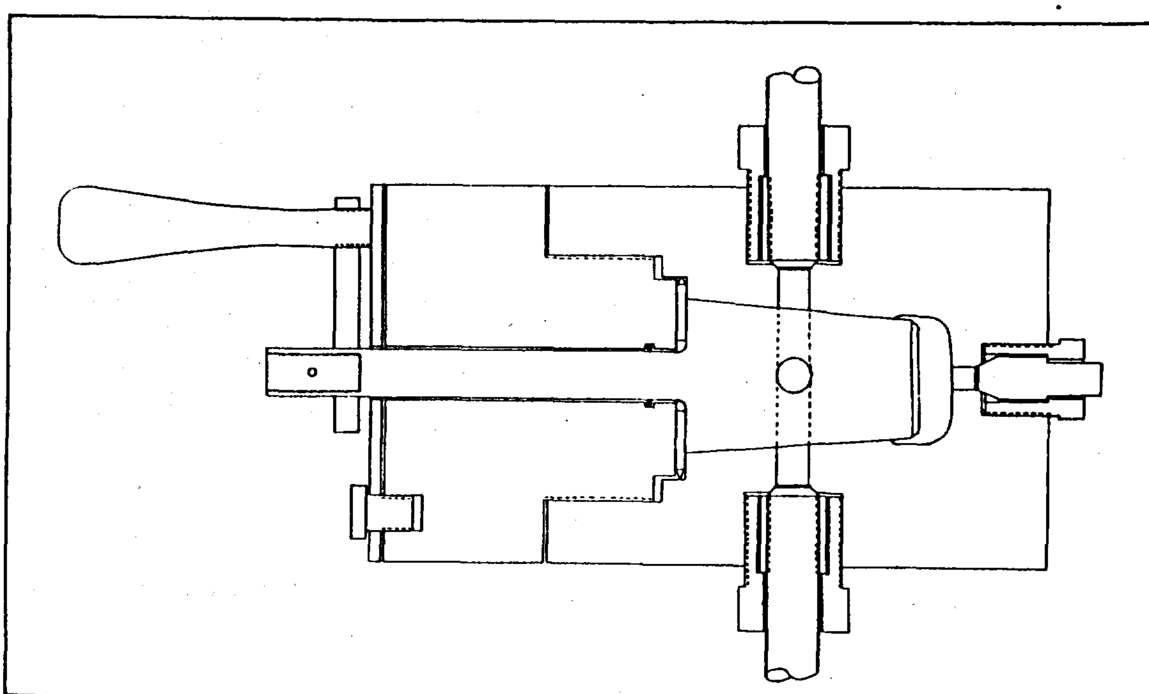

Figure 11. The 4-Way Valve.

Initial testing of the valve at 1000 psi, disclosed that gas was leaking to the surface above the plug, but not below it. This resulted in a force in excess of 1000 lb seating the plug on the taper, thus immobilizing it. A balancing passage was drilled to connect the spaces above and below the plug to eliminate this problem.

It was also found, during preliminary testing, that galling was likely to be a problem in service. Because a fluid or semi-fluid lubricant would likely cause catalyst agglomeration in the valve, several solid lubricants (colloidal graphite, molybdenum disulphide, the catalyst itself) were tried. When these proved unsuccessful, it was decided to hard-surface the valve bodies. A patented chrome-alloy coating 0.0004 in. thick was applied to the taper seats by the "Electrolizing Company" of Providence, Rhode Island.

The coating solved the galling problem but reduced the effectiveness of the plug seal. If the plug does not seal on the taper, the differential area between the top of the plug (less the stem) and the projected area of the bottom causes a force imbalance which lifts the plug off the taper and forces it against the cap. To combat this, a Teflon thrust washer was fitted between the cap and the plug. This re-established the seal, and the slight differential area favours a net force away from the cap if the plug is sealed. This reduces the load on the washer, so that the valve operates more readily at 1000 psi than at atmospheric pressure.

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