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Transpiring Wall Supercritical Water Oxidation Test Reactor Design Report

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**TRANSPIRING WALL SUPERCRITICAL WATER OXIDATION TEST
REACTOR DESIGN REPORT**

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ABSTRACT

Sandia National Laboratories is working with GenCorp, Aerojet and Foster Wheeler Development Corporation to develop a transpiring wall supercritical water oxidation reactor. The transpiring wall reactor promises to mitigate problems of salt deposition and corrosion by forming a protective boundary layer of pure supercritical water. A laboratory scale test reactor has been assembled to demonstrate the concept. A 1/4 scale transpiring wall reactor was designed and fabricated by Aerojet using their platelet technology. Sandia's Engineering Evaluation Reactor serves as a test bed to supply, pressurize and heat the waste; collect, measure and analyze the effluent; and control operation of the system. This report describes the design, test capabilities, and operation of this versatile and unique test system with the transpiring wall reactor.

*This work was supported by the DOE/EM-50 Office of Technology Development; the U.S. Army Armament Research, Development, and Engineering Center, Picatinny Arsenal, NJ; and the DOE-DP/DoD Office of Munitions Memorandum of Understanding.

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Transpiring Wall Supercritical Water Oxidation Test Reactor Design Report

Introduction

Laboratory and pilot scale work has shown the efficacy of supercritical water oxidation (SCWO) for safe, clean, and efficient destruction of a variety of organic wastes such as pyrotechnic colored smoke and dye compositions, explosives, propellants, and miscellaneous petroleum-based materials (ref. 1). However, these studies have also identified two major technical issues: corrosion and salt plugging. During the oxidation process, heteroatoms in the wastes form acids or salts, depending on the availability of counter-ions. The acids at high temperature and pressure, combined with excess oxidizer, create a highly corrosive environment (ref. 2). Salts, which are normally soluble in water, become insoluble above the critical point. Deposition of the insoluble salts on the reactor walls leads to reactor plugging (ref. 3). Salt deposition and corrosion have been identified as the two major technical problems for commercialization of SCWO (ref. 4).

Under joint sponsorship by U.S. Army Armament Research, Development, and Engineering Center (ARDEC) and the Department of Energy (DOE), Sandia National Laboratories is working with Foster Wheeler Development Corp. and GenCorp, Aerojet to develop a pilot plant scale, transpiring wall, SCWO reactor. The transpiring wall reactor promises to mitigate both corrosion and salt deposition (ref. 5) by forming a protective boundary layer of pure supercritical water along the wall. The concept is based on Aerojet's platelet technology, which was developed for aerospace applications such as cooling rocket nozzles and nose cones (ref. 6,7,8).

A major aspect of the project has been fabrication and testing of a laboratory scale reactor to validate the transpiring wall reactor concept. The test reactor, which was designed and fabricated by Aerojet, is a 1/4 scale version a pilot plant size unit (80 pounds of waste per hour capacity). The testing was done on Sandia's Engineering Evaluation Reactor (EER). This report describes the design, test capabilities, and operation of the EER as configured for testing the transpiring wall reactor. Test results will be published in a separate report. This work was funded jointly by the DOE/EM-50 Office of Technology Development and the DOE-DP/DoD Office of Munitions Memorandum of Understanding.

Design and Capabilities

Sandia's EER is a second generation, laboratory scale reactor system designed specifically for evaluating engineering aspects of SCWO technology. Its modular design facilitates different test configurations and its computer based control system allows maximum flexibility in operating conditions and data acquisition. It has a maximum operating temperature of 650 °C (1202 °F) at an operating pressure of 345 bar (5000 psi). Experiments are typically carried out at 276 bar (4000 psi). Total flow capacity is about 30 cc / sec (28.5 gallons / hour).

Inconel 625 was used for all high temperature sections of the system. The lines entering and leaving the reactor are 1.4 cm (9/16-inch) outside diameter (OD), 0.48 cm (3/16-inch) inside diameter (ID) Inconel 625 tubing. Inconel 625 T-unions and crosses accommodate pressure transducers and thermocouples at various locations. The fluid streams are heated with 1 kW cable heaters wrapped around the tubing. The heaters are about 60 % efficient in this configuration. Both 304 and 316 stainless steel are used in other parts of the reactor where the high temperature properties of Inconel are not required. The stainless steel is never exposed to temperatures greater than 300 °C (572 °F). Plastic tubing is used in the low pressure supply and effluent manifolds. The system is insulated with Fiberfrax® insulation.

Various features ensure safe operation. The entire system is located in an explosion-proof test cell rated for 1 lb. equivalent TNT. It is remotely operated from an adjacent control room. Burst disks or pressure relief valves are installed at all critical locations. Two air operated vent valves, one in the waste feed line and one in the effluent line, allow the system pressure to be vented remotely. The vent valve in the effluent line is in series with a remotely controlled metering valve to control how quickly the pressure is released. Seven video cameras, located throughout the test cell, allow continuous observation of the system during its remote operation.

The EER was originally configured as a simple, single tube, plug flow reactor. It has been significantly modified and expanded for the transpiring wall reactor tests. The waste stream temperature entering the transpiring wall reactor must be less than about 300 °C (572 °F) to prevent corrosion and deposition in the unprotected supply line. With the wastes of interest to this project (ref. 1), salts precipitate between about 360 °C and 400 °C (682 °F and 752 °F) and corrosion is most severe in the range of 300 °C to 400 °C (572 °F to 752 °F). Once inside the reactor, the temperature must be increased in order to initiate the oxidation reaction. This is accomplished by mixing the waste stream with hot water at the reactor entrance. Similarly, the effluent is cooled to less than 300 °C before exiting the protected reactor zone by mixing with cold water.

For the transpiring wall reactor tests, the EER consisted of the transpiring wall reactor, a waste supply manifold, supply lines for the transpiring wall, heating and cooling water streams, an effluent manifold, diagnostic instrumentation, and the control system. Each of these subsystems is described below. The system is shown schematically in Figures 1 and 2 and pictorially in Figure 3.

Transpiring Wall Reactor Description - The transpiring wall reactor, shown in Figure 4, was fabricated by GenCorp, Aerojet. It is mounted vertically in the EER with the waste inlet at the top. It is made from two sections that are mechanically joined with a Grayloc® flange. Grayloc® flanges also connect the reactor to the waste feed and effluent lines and provide other assembly joints. This modular design provides the option of adding sections in the future to increase length and residence time. The two reactor sections are identical, but are assembled as mirror images so the inlet of the top section is identical to the exit of the bottom

section. The inside diameter is 2.8 cm (1.1 inch), the outside diameter is 6.35 cm (2.5 inches), and the total length is 91.4 cm (36 inches).

The reactor sections consist of an outer pressure wall of Inconel 625 and an inner liner of 304 stainless steel with a distribution plenum in between. The liner has a complex system of internal manifolding and metering channels that distribute water uniformly to small transpiration pores along the inner surface. This was accomplished using Aerojet's platelet technology. Platelet fluid management devices are created by bonding together thin metal sheets, called platelets, which contain chemically etched fluid passages. Prior to etching, the flow passage pattern is precisely located by the use of photographic negatives. After etching, the platelets are stacked in a prescribed sequence and are diffusion bonded to form a monolithic structure with the desired internal fluid flow passages. This bonded structure is then formed and welded into the tubular reactor shape. The platelet liner design concept is illustrated in Figures 5 and 6. Hereafter, the liner will be referred to as a platelet.

The volume between the platelet and the Inconel wall is a manifold to feed water through the platelet. Each platelet is designed for 5.25 gm / sec (5 gallons per hour) of transpiration water at 450 °C (842 °F). At this flow rate, the pressure drop through the internal channels of the platelet is about 0.9 MPa (130 psi).

The platelet is attached to the Inconel wall by a welded ring. This ring also divides the annular volume between the platelet and the Inconel wall into two, unequal volumes. Each volume is fed by a separate feed line resulting in a total of four lines for the two reactor sections. The larger volume in each section, 33 cm (13 inches) in length, feeds the transpiration pores. The smaller volume feeds several larger, diamond-shaped holes that are interspersed among the transpiration pores in the first 12.7 cm (5 inches) of the platelet. The diamond-shaped holes in the first reactor section are at the waste inlet end and are used to inject heating water. Those in the second section are near the exit and are used to inject cooling water. The reactor length between the heating zone and the cooling zone is 66 cm (26 inches).

Because of differences in thermal expansion, the platelet and the Inconel housing are not welded together at the ends; the welded ring mentioned above is the only point of attachment. Consequently, the annular volume around the platelet is open at the ends of each reactor section and a seal is needed to prevent bypass flow of the transpiration water. This is accomplished with a gold-plated copper gasket that seals the small gap between the two platelets. This design relies on thermal expansion of the platelets to compress the gasket. There is no seal until the system reaches about 320 °C (608 °F). The gasket, which is plastically deformed by the ends of the platelets, is replaced after each test. The gasket prevents direct flow between the plenum and the inside of the reactor (bypassing the platelet) but does not prevent flow of the feed water from one reactor section to the other. Therefore, even though each reactor section has its own transpiration water feed line, the plenum volume is common to both and the pressure drop through both platelets is equalized.

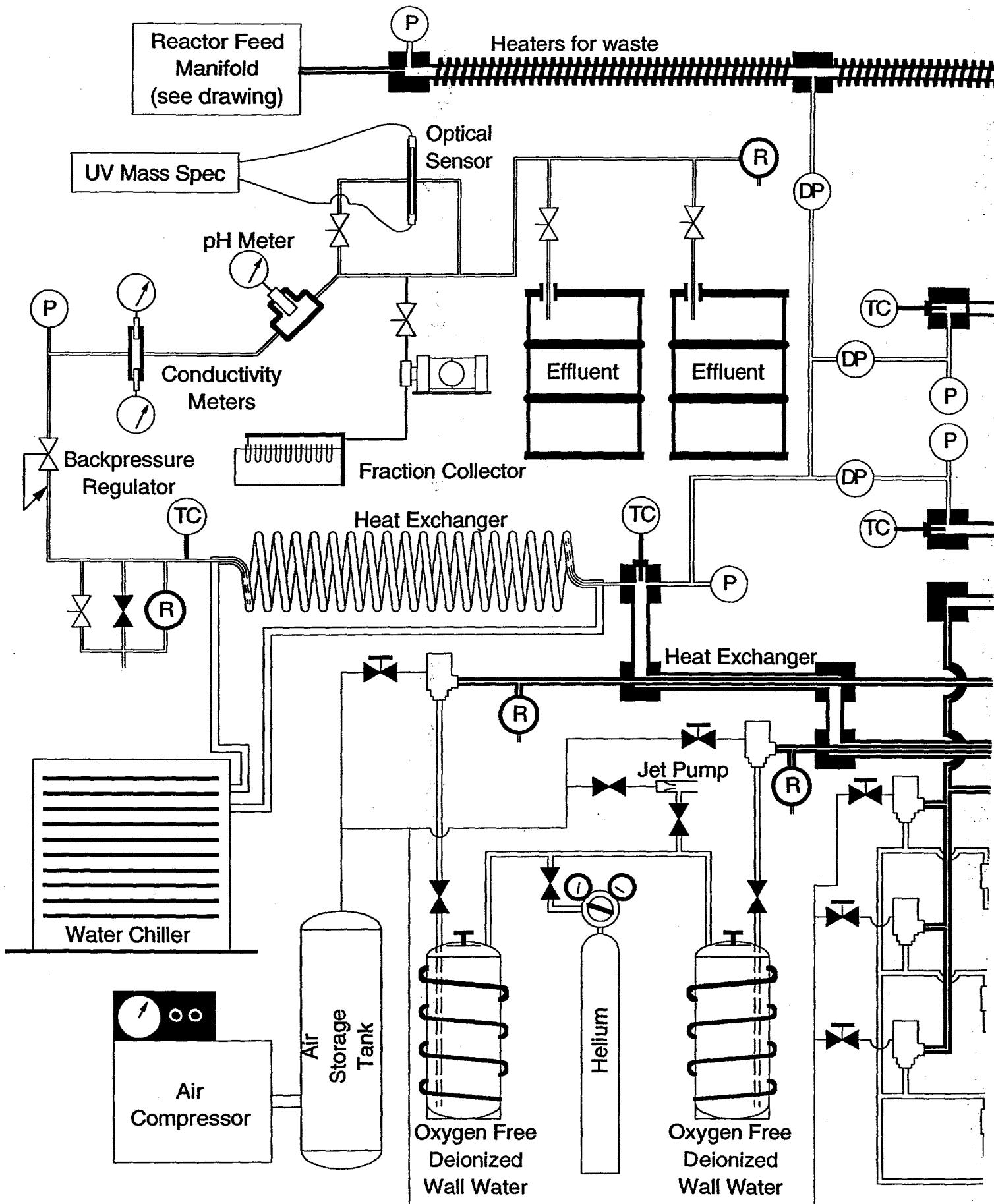
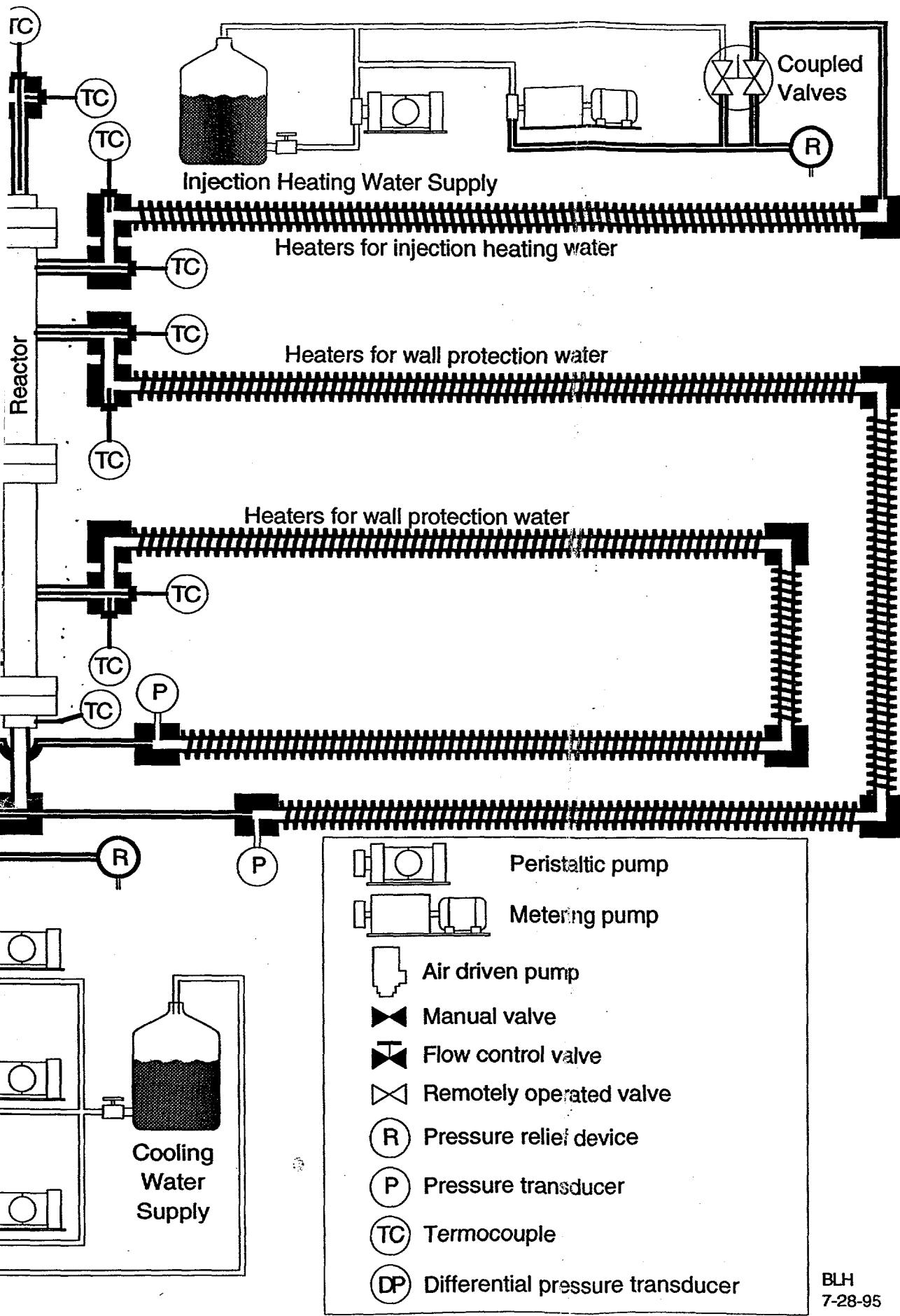
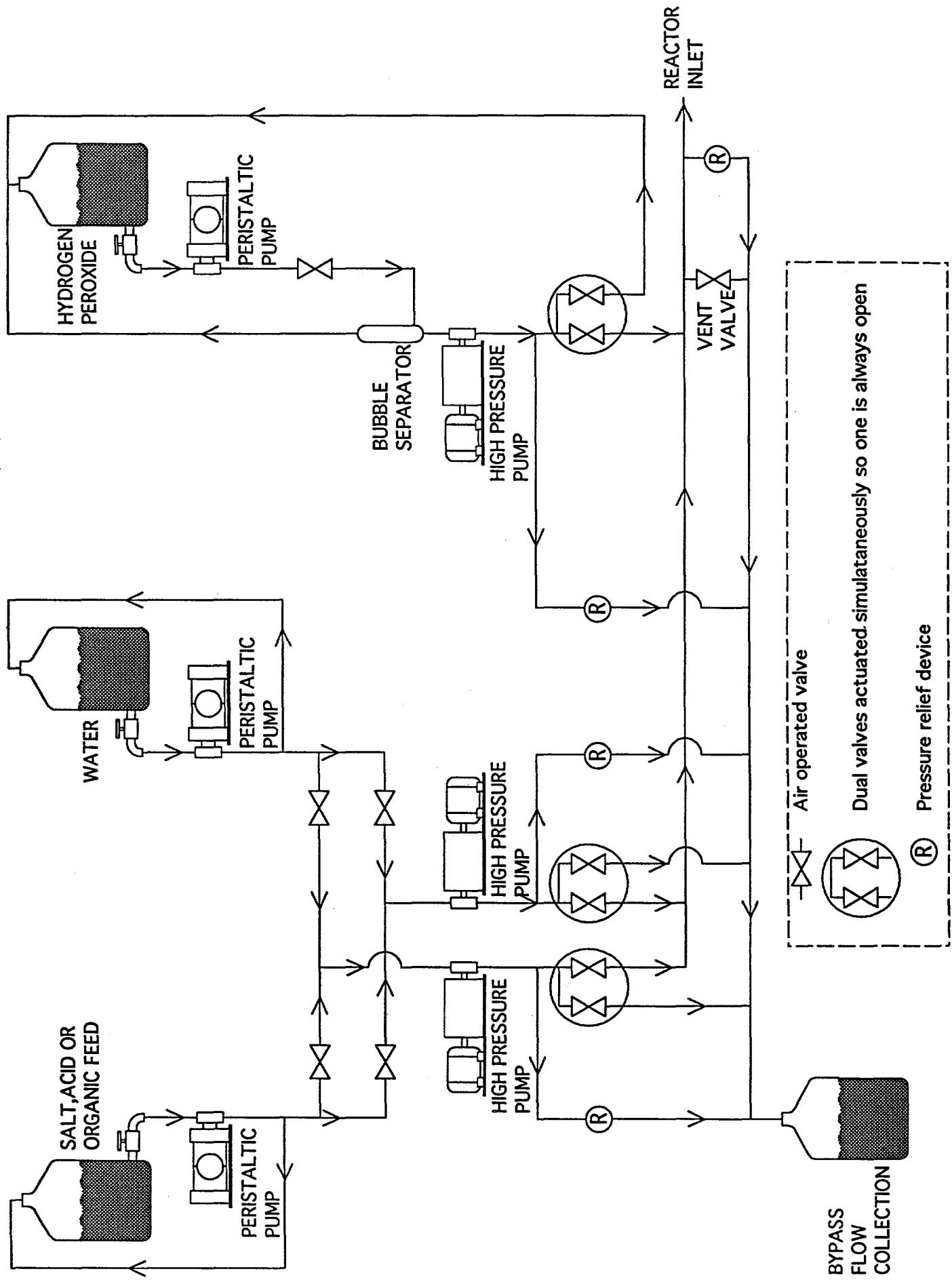


Figure 1.



REACTOR FEED MANIFOLD SCHEMATIC



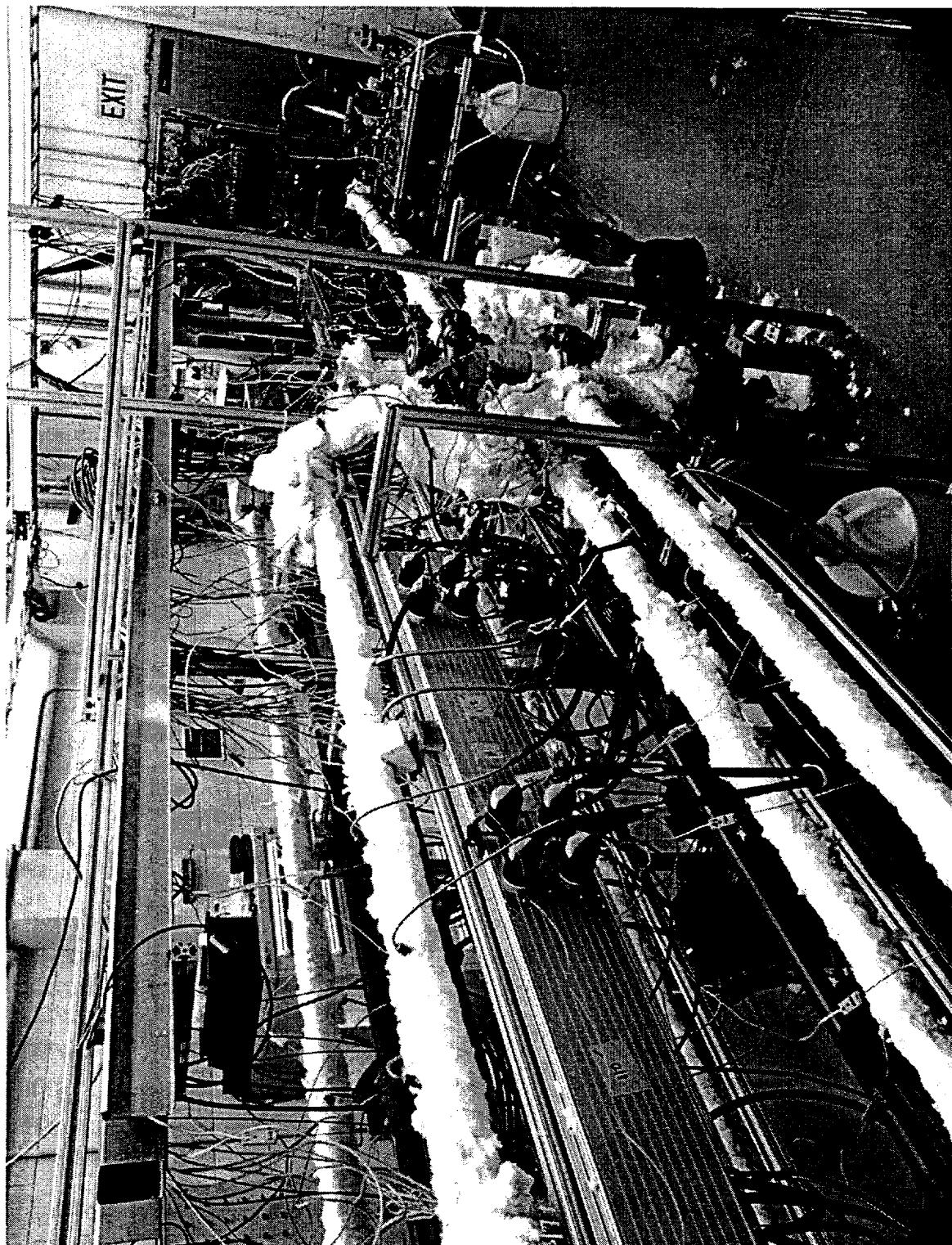
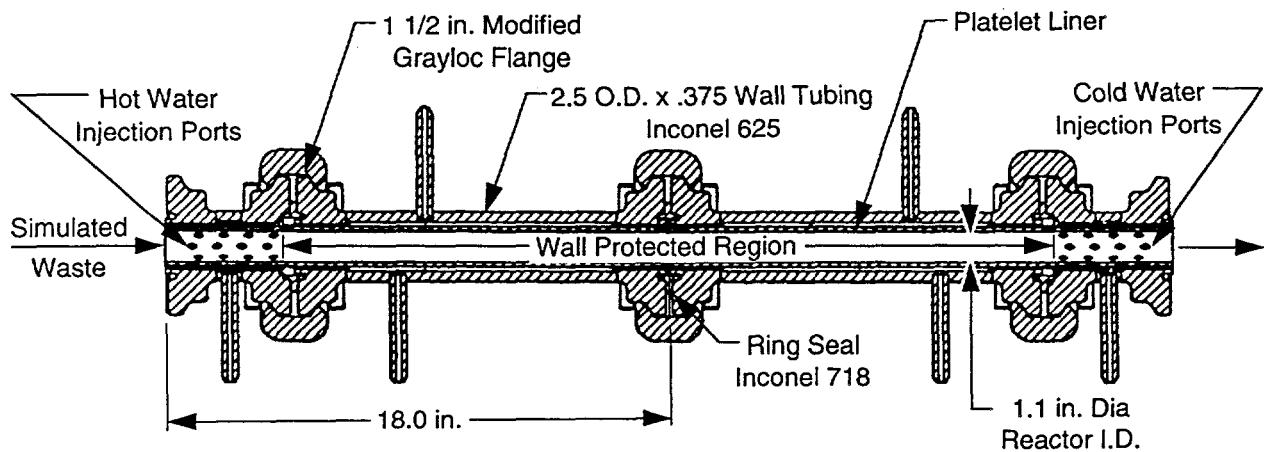


Figure 3.



The Bench Scale Is a Modular Assembly

Figure 4.

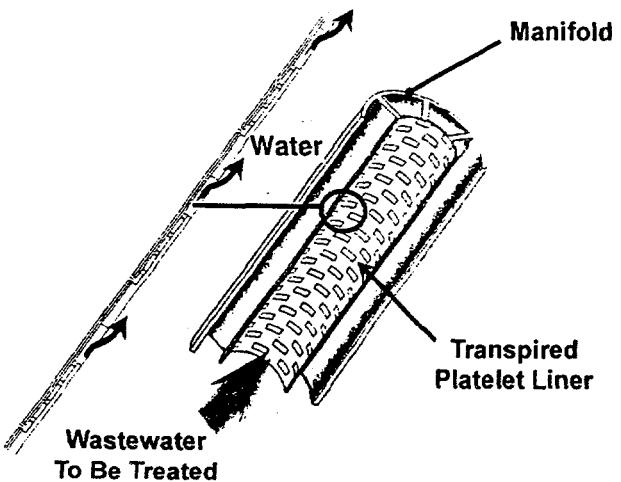
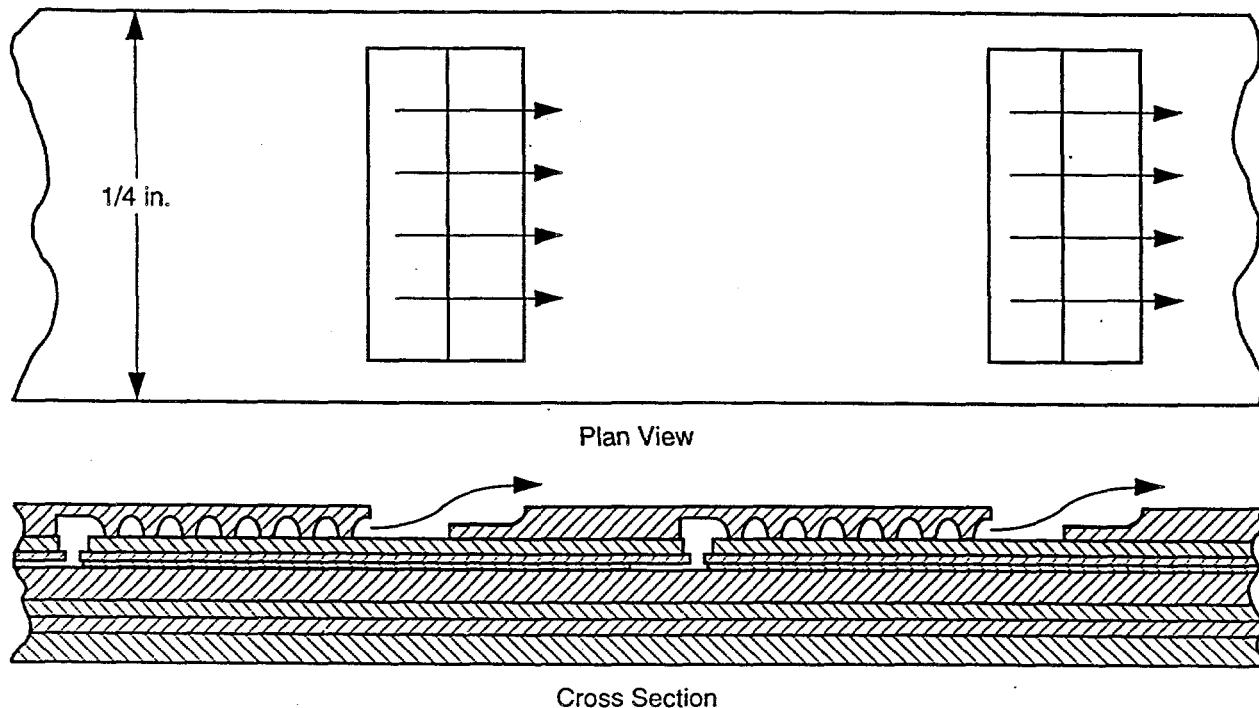


Figure 5. Tubular SCWO Reactor With Transpired Platelet Liner



Flat Platelet Device Used to Demonstrate Transpired SCWO Reactor Wall Concept

Figure 6.

A similar potential for bypass flow of the heating and cooling water exists. However, the pressure drop through the diamond-shaped holes, at less than 0.07 MPa (10 psi), provides little driving pressure for bypass. Furthermore, some bypass flow will not adversely affect performance because the distribution of heating and cooling water is not critical. Consequently, gaskets are not used at these locations.

Although the test reactor is a 1/4 scale model of a pilot plant size unit, three differences should be noted. First, the test reactor is intended to demonstrate the ability of the transpiring wall to prevent deposition and corrosion, not to destroy waste. The ability of SCWO to destroy wastes has been demonstrated on other reactor systems (ref. 1). Because of capacity limitations of the EER, the test reactor does not provide sufficient temperature or residence time to fully oxidize most organic wastes.

Second, the ratio of waste to transpiration fluid does not scale linearly with reactor diameter. The flow rate of transpiration fluid scales with the surface area which is proportional to diameter. The flow of waste scales roughly with cross sectional area which is proportional to diameter squared. Other fluid dynamics effects increase the scaling exponent to greater than two. Consequently, the ratio of waste to transpiration flow may seem quite small in the laboratory scale reactor, but the analogous full scale conditions are quite reasonable.

Third, heating and cooling will not be accomplished by injection of hot and cold water through diamond-shaped holes in the pilot plant because of the large amount of water that would be needed. Instead, heating will be done with a small amount of hot water and a supplementary fuel. An injector is proposed for mixing the various inlet streams. Cooling will be accomplished with a recirculating quench tank at the reactor exit.

Waste Supply - The waste supply system is designed for flexibility in the composition, flow rate, and temperature of the waste stream. Aqueous solutions of salts, acids, organic compounds, or hydrogen peroxide can be pumped up to 34.5 MPa (5000 psi). Concentration is limited by the solubility of the material since there is no slurry pumping capability. Hydrogen peroxide is used as the oxidizer in the EER, instead of oxygen or air, because, as a liquid, it is easier to pump.

Three separate high pressure, positive displacement, metering pumps permit waste to be supplied at flow rate from 0.013 cc/sec up to 6.5 cc/sec. Two pumps are Milton Roy MilRoyal B pumps, which can each supply 0.3 to 3.2 cc/sec. The axial pump shaft on these pumps is geared to an electric motor and servo-actuator for remote flow control. The third pump is an LDC Analytical minipump metering pump, model 2396-89, with a capacity of 0.013 to 0.25 cc/sec. The flow rate of the minipump is set manually with a micrometer dial so it cannot be changed during a test. Output of the three pumps is mixed in a high pressure manifold before entering the preheat section. Any pressure surge from the piston stroke of the pumps appears to be completely damped out once the system reaches supercritical conditions.

A schematic of the inlet manifold was shown in Figure 2. The wastes are stored in plastic carboys and are fed to the pumps through plastic tubing. The feed to the LDC Analytical and one of the Milton Roy pumps can be switched remotely from one carboy to another while the pumps continue to run. This allows the system to be started and stopped with water and switched to waste only when stable operating conditions are established. The flow from each of the three pumps can be diverted to a vent line instead of going to the manifold. This allows the flow into the reactor to be stopped without stopping the pumps. This is primarily used for rapid shutdown since the pumps take several minutes to wind down.

Peristaltic pumps ensure that the high pressure pumps remain primed. They recirculate the feed stream at several times the pump's flow requirements and pressurize the suction side of the high pressure pumps to 69 kPa (10 psig). The pressure is regulated with an in-line relief valve. This feed system is particularly useful for the hydrogen peroxide where oxygen bubbles, formed by chemical dissociation, can vapor lock the pump. The recirculation line coupled with a bubble separator at the high pressure pump inlet directs the bubbles back to the feed carboy. Up to 10 weight percent hydrogen peroxide has been successfully pumped.

The inlet manifold is made of 0.63 cm (1/4 inch) OD, 0.21 cm (0.083 inch) ID, 304 stainless steel tubing. From the manifold, fluid enters the Inconel 625 preheat section where it is heated with five, 1 kW cable heaters. Because the waste stream is cooler than the reactor, conductive heat transfer through the inlet tubing heats the waste as it approaches the reactor. Consequently, the temperature leaving the preheat section must be set less than the desired waste inlet temperature to avoid salt deposition in the preheat line. This concern is greatest when the flow rate is low.

At the reactor inlet, where the inside diameter increases from 0.48 cm (3/16 inch) in the tubing to 2.8 cm (1.1 inch) in the reactor, a diffuser plate is used to promote uniform flow. The diffuser has eighty-eight, 0.13 mm (0.005 inch) diameter holes.

Transpiration Water - To prevent corrosion in the small internal platelet passages, deionized, oxygen-free water is used for the transpiration. It is supplied from four, 60 liter (16 gallon), stainless steel tanks, which is sufficient for about 6 hours of operation at normal conditions. The water is deionized before entering the tanks. The oxygen is removed by heating the water to above 95 °C (203 °F) and evacuating the tank head space for about 15 seconds. Two, 1.5 kW, external band heaters take 4 to 5 hours to heat each tank. An air-driven ejector is used to evacuate the overpressure. The head space in the tanks is then backfilled with helium to prevent air leakage into the tanks. A helium supply tank with a pressure regulator maintains the overpressure at about 0.2 MPa (30 psig) throughout the test. The overpressure is sufficient for the high pressure pump inlet so peristaltic pumps were not needed on these lines.

The transpiration water is supplied to the two reactor sections through separate supply lines with Autoclave Maximator model PPSF72 or PPSF111 air-driven piston pumps. The flow rate is controlled by manually opening a valve on the air supply line to each pump. These valves are in the control room, allowing the flow to be varied during the test. Although crude, this method of control proved adequate. Once the correct flow is established, it is typically not changed until shutdown.

A single air compressor supplies air for all air-driven pumps. The air compressor cycles between 0.62 and 0.76 MPa (90 and 110 psi) about every 3 to 5 minutes. These fluctuations lead to small changes in the pumping rate, which lead to cyclic fluctuation in the reactor pressure as large as 0.69 MPa (100 psi). This fluctuation has caused no operational difficulties.

The transpiration water is heated in two stages. First it is preheated by the effluent water in two counterflow heat exchangers to between 200 and 300 °C (392 and 572 °F). Twenty-three, 1 kW cable heaters on each line then heat it to the desired temperature. The electric heaters have enough capacity to heat the water to 450 °C (842 °F) without the preheaters, which are primarily needed to cool the effluent. The preheaters, shown in Figure 7, are three foot, counterflow, concentric tube heat exchangers. The outer tube, which contains the effluent, is

1.4 cm (9/16 inch) OD, 0.48 cm (3/16 inch) ID Inconel tubing. The inner tube is 0.32 cm (1/8 inch) OD, 1.8 mm (0.070 inch) ID 304L stainless steel tubing.

Heating Water - The flow rate of injection heating water is typically two to four times that of the waste, depending on the relative temperatures. It is pressurized with a Milton Roy metering pump like that used for the waste. An Autoclave air-driven pump is substituted if a greater flow rate is required. A peristaltic pump feeds the high pressure pump. Distilled water is normally used for the injection heating.

The heating section has twenty-three, 1 kW heaters. The peak temperature depends on the flow rate and may be limited by either the capacity of the heaters or the heat transfer rate. The control system will not allow the temperature of the Inconel tubing to exceed 650 °C (1202 °F) so the heaters may operate below capacity even if the water temperature is less than desired. The water temperature entering the reactor is typically about 600 °C (1112 °F), but has been as high as 640 °C (1184 °F).

Cooling Water - Room temperature distilled water is supplied for the injection cooling by three air-driven Autoclave pumps through 0.63 cm (1/4 inch) OD, 304 stainless steel tubing. Total capacity is about 15 cc/sec. As with the other air driven pumps, these pumps are controlled from inside the control room by changing the air flow.

Effluent System - After the cold water injection, the temperature of the reactor effluent is between 300 °C and 350 °C (572 °F and 662 °F). It immediately goes through the two preheaters which reduces the temperature by 70 °C to 100 °C (126 °F to 180 °F). Finally it is cooled to about 10 °C (50 °F) in a counter flow, concentric tube heat exchanger that is cooled by a 23.5 kW (80,000 BTU/hr) chiller. The chiller typically runs near capacity and is the limiting element preventing tests at a higher reactor temperature.

The cooled flow is discharged through a Tescom liquid back pressure regulator. This is the only pressure control in the system. The pressure at all other points, such as the wall protection feed lines, is determined by the flow rates and flow resistance in the system. System pressure fluctuates during the test by as much as 0.7 MPa (100 psi). The biggest source of pressure variation is the cycling of the air compressor discussed previously. A secondary source is gas bubbles passing through the regulator.

The low pressure effluent flows through the on-line diagnostics and then into waste collection barrels for disposal.

Instrumentation and Diagnostics - Instrumentation includes fluid temperatures, tubing wall temperatures, pressures, flow rates and on-line analysis of the effluent.

Temperature is measured at 88 locations with 1.6 mm (1/16 inch), Inconel 600 sheathed, Chromel-Alumel (type K) thermocouples. Twenty-nine of these

thermocouples are inserted directly into the high pressure fluid using 1.6 mm (1/6 inch) HIP swage fittings. The others measure wall temperatures and are clamped to the tubing with hose clamps. The location of each thermocouple is shown on Figure 8. Thermocouple accuracy is the less accurate of ± 2.2 °C (4 °F) or ± 0.75 %. Temperature is measured inside the reactor by inserting long thermocouples from either end to the desired location. These thermocouples provide an unprotected surface for salt deposition and are removed for some tests. Typically the waste inlet temperature is measured 1.3 cm (1/2 inch) above the diffuser plate, the mixed temperature 2.5 cm (1 inch) below the hot water injection zone, and the effluent temperature at the reactor exit. Five thermocouples measure the temperature at different locations in the annular volume between the platelets and the Inconel wall: two where the transpiration water enters from the two feed lines, two diametrically opposite from them, and one where the heating water enters.

Pressure in each reactor feed line and the reactor exit are measured with six Teledyne-Taber transducers (models 2107 or 2105) with full scale range of 68.9 MPa (10,000 psi). A 3.4 MPa (500 psi) Teledyne-Taber model 206 transducer measures pressure below the pressure regulator. Accuracy is ± 2 % of full scale. Despite the inaccuracy in the pressure measurements, fluctuations as small as 0.14 MPa (20 psi) can be reproducibly detected. For example, even though two transducers at the same location may differ by 1.4 MPa (200 psi), they will both show the same small fluctuations due to pump variation. This was important on these tests because the magnitude of the pressure was not as significant as the magnitude of the fluctuations. Three Teledyne-Taber model 2104 differential pressure transducers are also used. They measure the differential pressure between the reactor inlet and outlet and between the two transpiration water feed lines and the reactor outlet. They are used to detect plugging from salt deposition and to verify that the gasket between the two platelets seals correctly. Each differential transducer has full scale range of 3.4 MPa (500 psid) and is accurate to $\pm .07$ MPa (10 psi).

The flow rate of each fluid stream is determined by measuring the weight loss of the feed carboy. The waste and hot water carboys are measured with 45 kg (100 lb.) scales. The others use 227 kg (500 lb.) scales. Because of noise in the measurement, the flow must be averaged over time. Over a half hour, the average flow rates are accurate to within about ± 3 %. Small fluctuations in flow, such as those from cycling of the air compressor or intermittent plugging, cannot be resolved in the data.

On-line conductivity and pH meters provide real-time data on the reactor effluent. The data from these meters are recorded and displayed graphically during the test on a Macintosh Quadra 700 computer using LABview software. Two Myron L Co. 758-29 conductivity meters cover the low and high ranges. The first uses an S51-s sensor with a range of 0 to 20 mS/cm. The second uses an S52A sensor with a range of 0 to 200 mS/cm. Accuracy is ± 1 % of full scale. A roughly linear relationship exists between the concentration of an ionic species in solution and its conductivity. There is some variation of conductivity between ions, but in general they are similar. The exceptions are solutions with H⁺ or OH⁻, which

have conductivities about four times or two times greater than usual, respectively. However, when the conductivity meter is used in conjunction with the pH meter, the effect of either of these ions can be removed. The conductivity measurements are particular useful for salt deposition tests. A salt solution, typically sodium sulfate, is fed through the reactor at subcritical temperature where no deposition will occur and then again at supercritical temperature. The conductivity of the effluent for each condition is then compared to determine what percentage of the salt was deposited at supercritical temperature.

The pH meter is a Hach model EC1000. It is designed for use in harsh process environments and is able to withstand constant or periodic use over a wide range of pH. The meter is calibrated with standard buffer solutions to an accuracy of better than 0.01 pH units. The pH data are of most use for corrosion testing. Corrosion and acid concentration have been shown to be directly correlated.

A Guided Wave model 200 Ultraviolet / Visible spectrometer collects continuous on-line spectra from the liquid effluent. The spectrometer measures the absorption of visible and ultraviolet light by compounds in the effluent. Post-test analyses of these spectra provide data on the level of unreacted waste and corrosion products. Chromium was shown to be detectable to 10 parts per billion.

An Astro Inc. 2001 Total Organic Carbon (TOC) analyzer collects samples of the effluent periodically to provide near real-time data on both organic and inorganic carbon. This equipment works on the principle of chemical oxidation of the sample at 50 °C by persulfate ion in the presence of ultraviolet light. The CO₂ produced is sparged from the solution and carried to a carbon dioxide analyzer. Samples are collected at roughly 2.5 minute intervals. Alternate samples are used to measure inorganic carbon then total carbon.

A small fraction of the effluent stream is diverted through a peristaltic pump to a Biorad 2128 fraction collector for post test chemical analysis. Typically a 10 cc sample is collected every 10 minutes, but the rate can be varied.

Control System -- The EER control system was developed using a graphical programming language, LABview. LABview programs are coded as icon based functional block diagrams. The control system, which runs on a Macintosh Quadra 950 computer, forms a virtual instrument by incorporating all reactor controls and data displays into a graphical user interface. In the interface, the controls and displays overlay a schematic of the EER. Various pop-up menus allow access to other features. The operator remotely controls the reactor with a mouse and observes the reactor status from on-screen displays. Valves are opened and closed with mouse clicks on their schematic representation. Pumps and temperatures are controlled with virtual dials. The combination of system schematic with real time displays and controls creates a familiar and intuitive user interface.

The main control parameters are temperature set points, data sampling rate, and flow rates of the Milton Roy pumps. A feedback control system using tubing surface temperature maintains stable operating temperatures and prevents

overheating of the tubing. Sixteen independent temperature control channels allow the temperature profile to be tailored for each test. Fluid temperatures are measured, but are not used in the control algorithm. The locations of the sixteen temperature control channels on the reactor are shown in Figure 8.

Critical temperatures, pressures, and flow rates are displayed continuously in digital and graphical format. All other measured temperatures and pressures can be viewed at any time by scrolling to the desired data channel on the display. Control parameter settings and all measured test data are continuously logged by the computer for subsequent analysis. The sampling rate can be specified down to five second increments. The collected data can be plotted using Kaleidagraph software without interrupting the computer control or data acquisition (a few sample points may be missed while Kaleidagraph opens the data file).

The computer sounds an alarm and prints a warning on the monitor if the temperature at any point exceeds 650 °C (1202 °F). A similar warning is given if the differential pressure across the platelet becomes negative. This is to prevent reverse flow that could damage the transpiring wall. The system is designed to be fail safe; if the computer control system fails, the heaters and pumps turn off.

Operation

The EER is an experimental test facility with operating conditions that vary from test to test. In general, water is used during the startup phase which last 60 to 90 minutes. The normal startup sequence is:

1. Start the computer control and data acquisition systems.
2. Start the on-line diagnostic systems.
3. Start the waste feed pumps using water and adjust the flow rate.
4. Start the wall protection pumps and set the flow rate.
5. Start all other pumps.
6. Increase the system pressure to 26.2 MPa (3800 psi).
7. Switch the waste feed from water to waste to test the pumps and the supply system and to calibrate conductivity and pH measurements.
8. Switch back to water and flush the waste out of the system.
9. Increase the temperature of the transpiration water and the heating water.
10. Increase the temperature of the waste feed.
11. Allow the system to stabilize.

Once the system is stabilized at the desired temperature, the feed to the waste pump is switched from water to waste. Temperatures, pressures and effluent conductivity are monitored closely for evidence of plugging which could force a reactor shutdown. Data acquisition continues throughout the test. The sampling frequency is typically reduced during startup to minimize the size of the data file, then increased once the system is at temperature and ready to process waste.

The system can be run at the same conditions for three to four hours or several different conditions may be tested during one experiment. Total test duration

including startup and shutdown is limited to about six hours by the supply of transpiration water. If different conditions are tested, the feed to the waste pump is switched back to water and the waste is flushed from the system before the conditions are changed. Salt that has deposited on the walls will remain in place. Generally the lowest temperature conditions are tested first so that the reactor temperature is either stable or increasing throughout the test. This minimizes the possibility of leaks in the high pressure fittings that can occur when the Inconel tubing cools and contracts more rapidly than the larger fitting it is screwed into. Cooling can result either from turning down a heater or increasing a flow rate.

Before shutting down, the feed is switched to water to flush the waste out of the system, then one of two shutdown modes is used: 1) The temperature is slowly decreased to dissolve any salt deposits and reach closure of the feed salt. This is a slow process and is frequently unsuccessful due to leaks. 2) The system is shut down and vented quickly. This eliminates the possibility of leaks and, more importantly, it leaves any salt deposits in place for later observation. The rapid shut down procedure is:

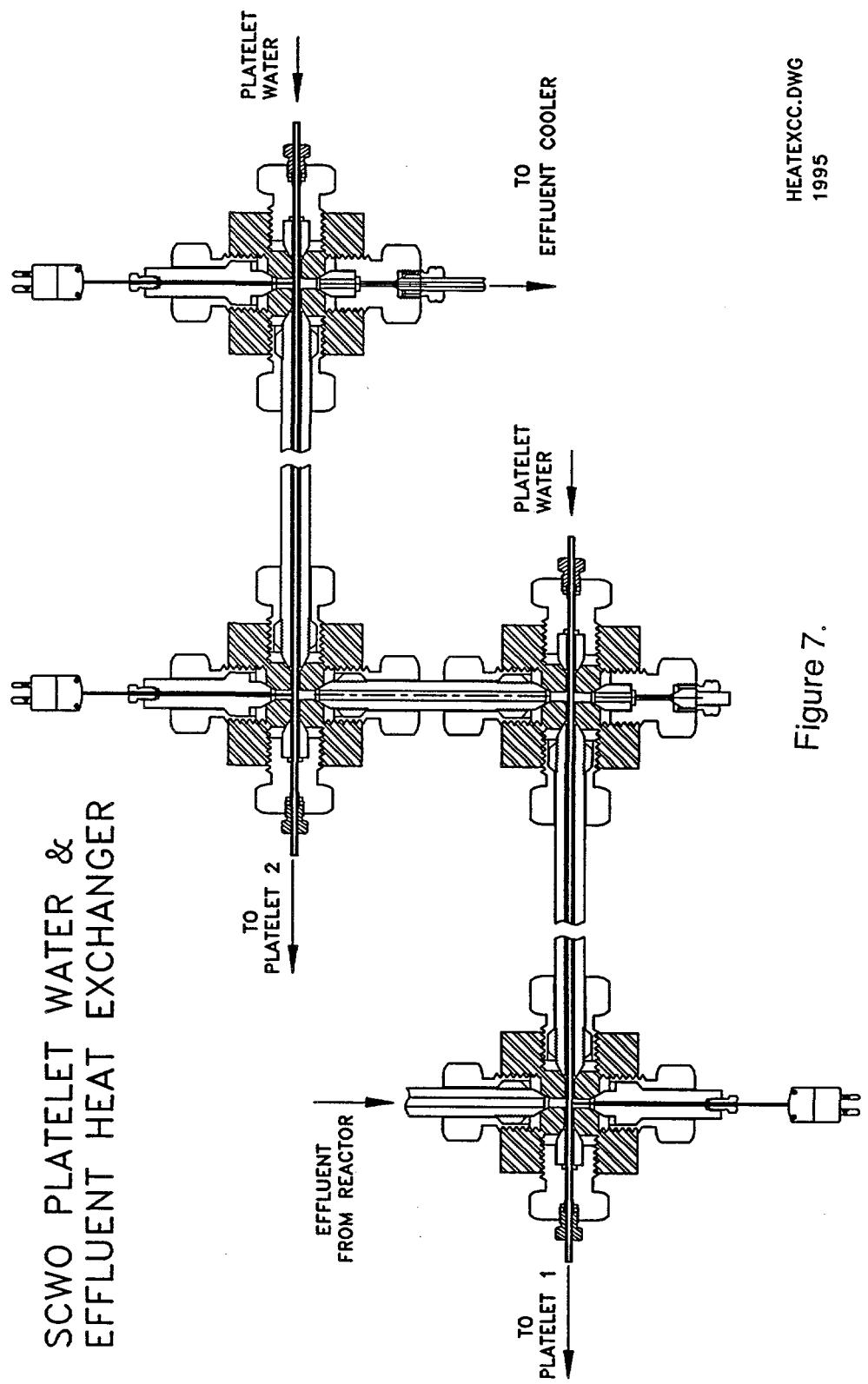
1. Flush the waste from the system.
2. Turn off all heaters.
3. Shut off all the air-driven pumps.
4. Stop the flow from the metering pumps to the system. (These pumps take minutes to wind down so the flow is simply diverted.)
5. Open the vent valve to release pressure. (A metering valve is used to control the pressure release.)
6. Flush the system with dry nitrogen or helium to remove any remaining water.
7. Allow the system to cool overnight.

Steps 2 through 5 all occur in about 15 to 20 seconds. The reactor can be disassembled the following day for observation. It retains enough heat to prevent grain growth in deliquescent salt crystals

Conclusion

Installation of Aerojet's 1/4 scale transpiring wall reactor in Sandia's EER has been completed and the system has been successfully operated. The modular design and flexible control system of the EER allow a unique opportunity to test and demonstrate this innovative concept for mitigating salt deposition and corrosion. The system meets all original design requirements and is sufficiently versatile to accommodate all tests needed to fully characterize reactor performance. New on-line diagnostics allow real time monitoring of effluent conductivity, pH, and TOC. The only significant limitation of the system is the peak operating temperature which precludes full oxidation of complex organic compounds. Testing is underway with various salts, acids, and organic compounds to characterize the performance of the reactor. Test results will be published separately.

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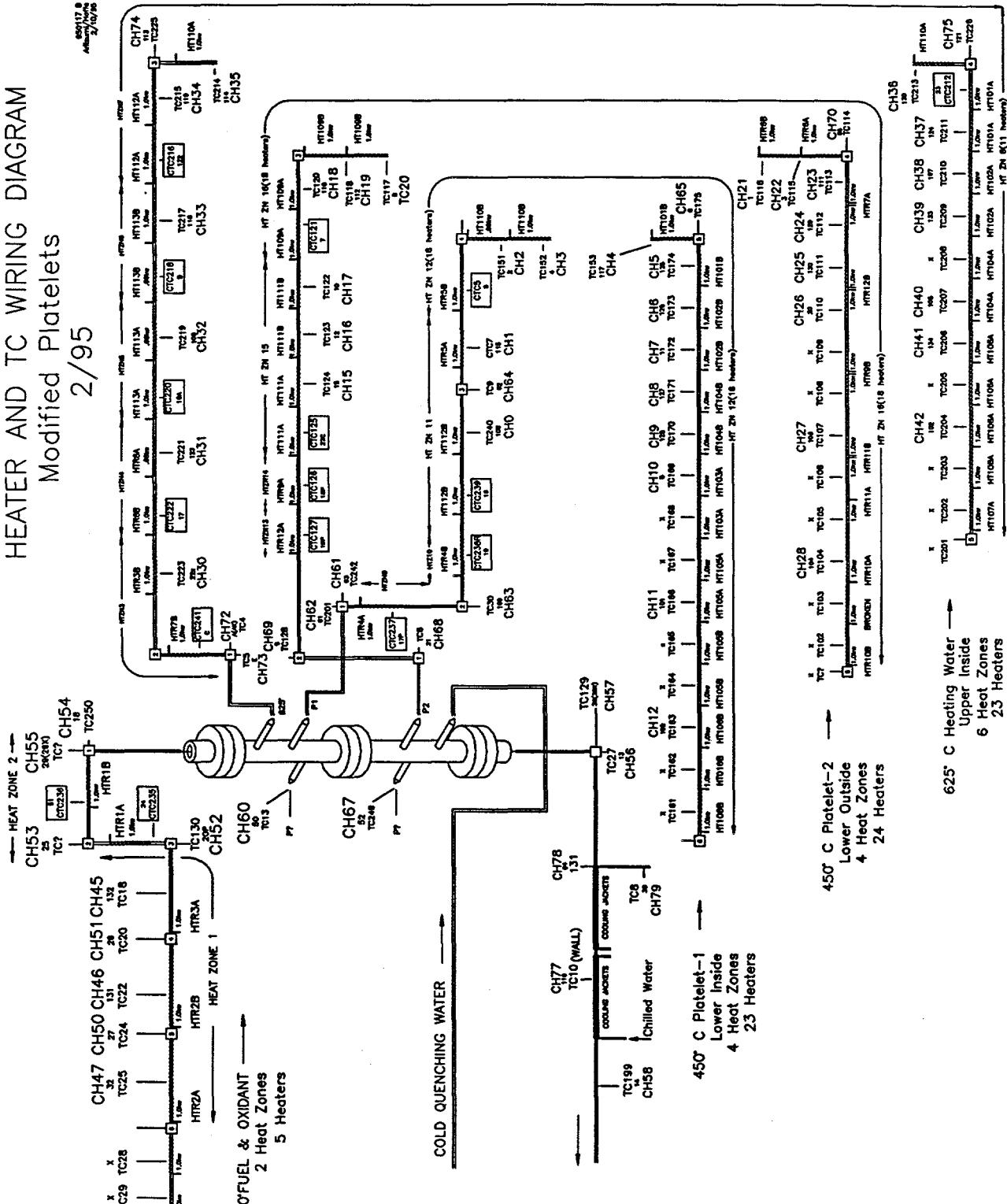
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Figure 7.

Figure 8.

HEATER AND TC WIRING DIAGRAM Modified Platelets

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