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Final Report (Westinghouse Electric Corp.)
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Advanced Energy Systems Division



**Design, Fabrication and Bench Testing of a
SOLAR CHEMICAL RECEIVER**

FINAL REPORT

prepared for the
JET PROPULSION LABORATORY
Contract No. 955639



**Westinghouse Electric Corporation
Advanced Energy Systems Division
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Pittsburgh, Pennsylvania 15236**

DESIGN, FABRICATION, AND BENCH TESTING
OF A SOLAR CHEMICAL RECEIVER MODULE

Final Report Prepared For

Jet Propulsion Laboratory

Contract No. 955639

January, 1981

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ABSTRACT

Solar thermal energy can be effectively collected, transported, stored, and utilized by means of a chemical storage and transport system employing the reversible SO_2 oxidation reaction. A solar chemical receiver for SO_3 thermal decomposition to SO_2 and oxygen has been analyzed and a prototype module of the receiver has been manufactured and tested. Bench tests of a ten-foot section of a receiver module were conducted with dissociated sulfuric acid (SO_3 and H_2O) in an electrical furnace. Measured percent conversion of SO_3 was 85% of the equilibrium value. Testing was terminated prematurely due to a failure in an auxiliary component. Methods were developed to fabricate and assemble a complete receiver module. These methods included applying an aluminide coating to certain exposed surfaces, assembling concentric tubes with a wire spacer, applying a platinum catalyst to the tubing wall, and coiling the entire assembly into the desired configuration. Problems were encountered with the coiling of alonized and platinized tubes, and further work is required to overcome this problem.

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SUMMARY

The purpose of this investigation was to analyze, manufacture, and test a representative module of an SO_3 solar chemical receiver. The work was divided into three main tasks:

- Receiver Concept Analysis
- Receiver Module Design Development
- Receiver Module Fabrication and Functional Performance Testing

A summary of each main task is as follows:

Receiver Concept Analysis

A single module of a solar chemical receiver was analyzed. The module was representative of one of 20 modules designed for a 8 kW_t receiver. Since the heat flux distribution within the receiver cavity was not specified, an average heat input to the module was taken as 400 watts. The size and materials of construction for the module were specified operating conditions, the module capacity (gas flowrate) and performance were calculated. At a mass flowrate of 5.0 pounds per hour all functional requirements were met. The calculated maximum tube wall temperature in the flux zone was 860°C with an extent of reaction (percent conversion of SO_3) of 51.7%. Gas pressure drop was calculated to be 3.0 psi, and heat exchanger effectiveness was 94%, with a 110°C outlet temperature for a 90°C gas inlet temperature. Structural/mechanical aspects of the selected design configuration were found to be satisfactory.

Receiver Module Design Development

This task involved the finalization of module design and the specification of test apparatus and plans. An eight coil module design was prepared utilizing a $5/8" \times .035"$ outer tube and a $3/8" \times .035"$ inner tube. The coiled module has an inner cavity diameter of 11" and an outer coil diameter of 21". An

arrangement drawing of the module was prepared. A test plan and required equipment for the bench tests and functional performance tests were identified.

Receiver Module Fabrication and Functional Performance Testing

This task involved the following:

- Development and verification of fabrication techniques
- Manufacture of SCR bench modules and full-scale modules
- Bench testing of straight modules
- Functional performance testing of a complete module

The fabrication verification included investigation of "aloning", catalyst placement methods, module assembly, and hot bending of the assembled module. Several tubes were alonized by Alon, Inc. and metallographic analysis was conducted. Various methods of applying a platinum catalyst coating to the alonized surfaces were tested. A technique for applying platinum ink, followed by thermal decomposition was developed. Assembly procedures for wrapping the wire spacer and assembling the concentric tubes were established. Methods were developed for welding alonized tubing.

The final and most difficult procedure in module fabrication is the coiling of the concentric tubes. The alonized portion requires hot bending to prevent cracking. A bending apparatus was prepared and bend tests at various temperatures and heating rates were conducted. A bending temperature of 1250°F, maintained by acetylene torches, was selected.

After establishment of the fabrication procedures, the bench test modules were fabricated. This did not require coiling and no problems were encountered. A full-scale bare 316SS module was then assembled and coiled without incident. This was followed by fabrication of an alonized module, also deemed acceptable. However, when alonized and platinized tubes were coiled, severe cracking of the outer tube was discovered in the innermost coil. The cracking attributed to the heat treating that occurs during platinizing. Several other bends were attempted with various changes in platinum application

method and bending techniques. Cracking in the inner tube was eliminated, but cracking in the outer tube persisted. Further work is required to eliminate this problem. In order to circumvent the cracking, a module was fabricated with an alonized and platinized inner tube, but a bare outer tube.

A bench test apparatus was constructed and checked out. A single bench test on a ten foot straight module was conducted using dissociated sulfuric acid (SO_3 and H_2O). Anhydrous SO_3 could not be used due to a failure of the SO_3 delivery pump. The test was terminated prematurely due to a pressure gauge rupture. Results from the foreshortened test showed 34% SO_3 conversion at an average temperature of 670°C . This represents 85% of equilibrium conversion.

The pressure gauge failure resulted in the termination of testing and no functional performance test was conducted on the fabricated full-scale module (bare outer tube).

1.0 INTRODUCTION

The efficient and economical use of solar energy is receiving a major emphasis in the United States. At present, only low temperature solar collectors for space heating or domestic hot water have been commercialized. Considerable research and development is being conducted on medium and high temperature solar systems. The high temperature (500° F-2500° F) systems tend to either be large centralized plants using the central receiver technology or small distributed systems using trough-type or parabolic dish collectors. In all cases, a major consideration is the diurnal nature of solar energy and the requirement for efficient energy storage and transfer.

A very attractive solar energy concept is a chemical storage and transport system which can act, in effect, as a chemical heat pipe. This concept is based on the use of solar heat to drive the endothermic reduction of SO_3 to SO_2 and O_2 . The SO_2/O_2 mixture, along with residual unreacted SO_3 , can be regeneratively cooled to a low temperature and piped to a location for storage and/or ultimate conversion to useful energy to meet "consumer" requirements. The energy contained in the process stream can be recovered by the exothermic recombination of SO_2 and O_2 to form SO_3 . Both the reduction and recombination reaction take place only in the presence of a catalyst. The attractiveness of this system is that the energy can be transported long distances and stored for extended periods of time at ambient conditions with minimal energy losses to meet discontinuities in supply and demand from a logistics and timing standpoint.

The first step in demonstrating effective use of the SO_3 chemical heat pipe concept can be achieved experimentally by developing a solar chemical receiver (SCR) to be used in conjunction with a point focusing distributed solar collector. Tests with this solar chemical receiver will demonstrate the effectiveness to capture solar energy, concentrate the energy inside a chemical reactor, decompose SO_3 into SO_2 and O_2 and cool the SO_2 , O_2 , SO_3 mixture to a stable state for transportation and storage.

Westinghouse Electric Corporation has designed a unique SCR that utilizes metallic components and internal regenerative heat exchange in a compact cavity arrangement. The design, shown in Figure 1-1, consists of a number of spiral tubing modules assembled together to form a parallel flow circuit. Each module consists of concentric stainless steel tubes held apart by a spirally wrapped wire spacer. The innermost coil of the module contains a platinum catalyst and acts as the chemical reactor. The remaining coils serve as regenerative heat exchangers.

The purpose of the work described herein was to perform design point analysis, design and fabrication development, prototype fabrication, and functional performance testing of a representative SCR module. Baseline design parameters, operating conditions, and functional requirements were provided by JPL and were based on the capabilities of the parabolic dish collector being developed at JPL.

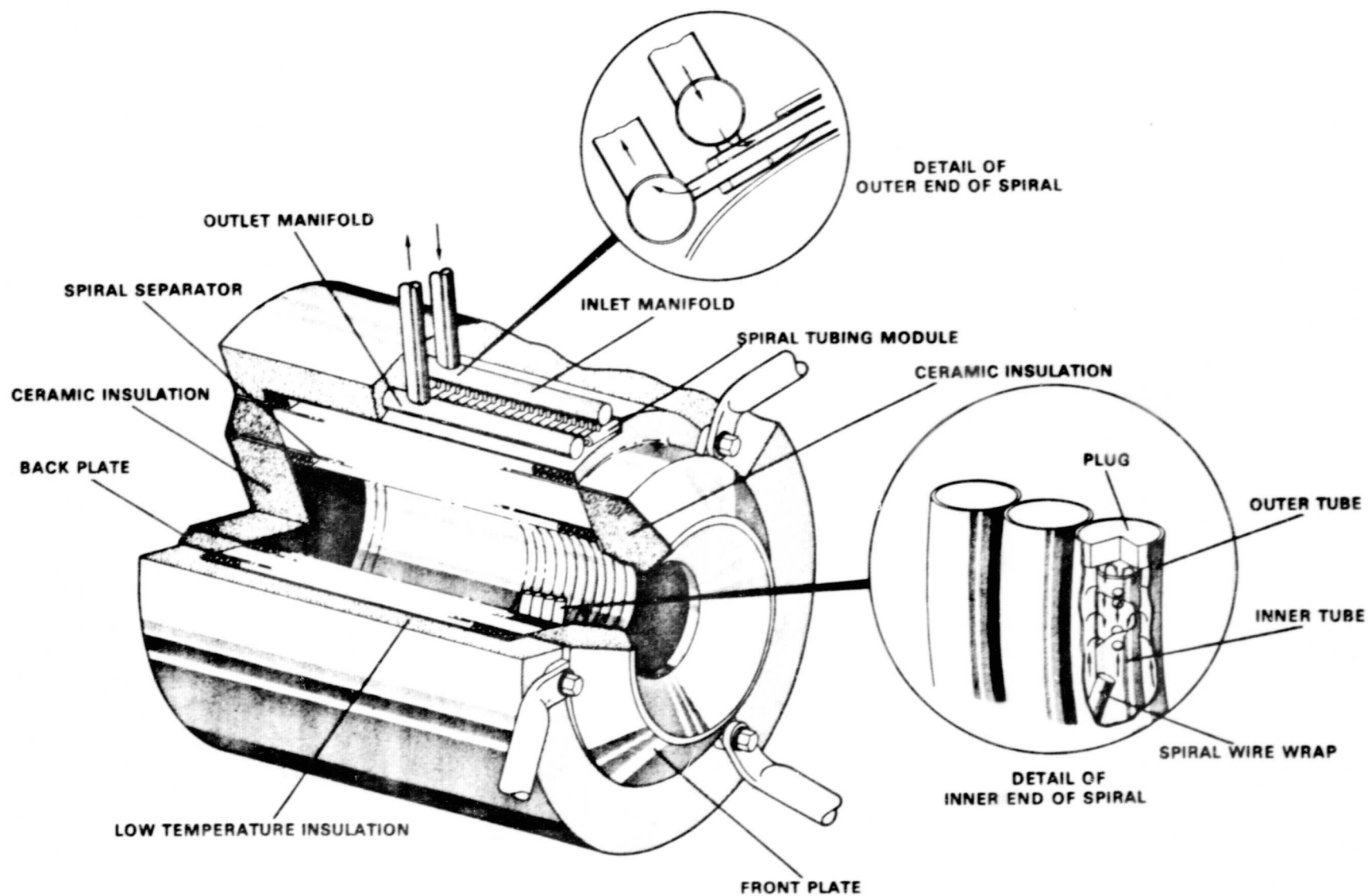


Figure 1-1. Solar Chemical Receiver

2.0 RECEIVER CONCEPT ANALYSIS

The purpose of this task was to perform a design point analysis of a single SCR module. The baseline design parameters, operating conditions, and functional requirements were provided by JPL and are shown in Figures 2-1, 2-2, and 2-3. The functional requirements were performance goals, since the specified design parameters and operating conditions are sufficient to determine the level of performance.

The module analysis was conducted by considering the SO_3 chemical reactor (innermost module coil) and regenerative heat exchanger (remaining coils) separately. Separate mathematical models were developed for each section and then coupled to calculate the overall system performance. Other work performed during the analysis work consisted of a structural/mechanical analysis and an evaluation of catalyst type and placement methods.

2.1 CHEMICAL REACTOR ANALYSIS

The objective of this subtask was to characterize the expected performance of the proposed SO_3 reduction reactor. This consisted of developing the controlling equations to describe mass transfer, reaction kinetics, chemical equilibria, and heat transfer for a single concentric tube module. The tube dimensions, mass flowrates, and heat flux were as specified by JPL. With the use of the controlling equations, the module performance was calculated assuming the tubular reactor behaves as an integral plug-flow reactor. The performance calculations allow prediction of temperature profiles and percent SO_3 conversion.

The SO_3 reaction kinetics depend on mass transfer of reactants and products to the catalyst surface and the surface chemical reaction rate. Both mass transfer rate and chemical reaction rate are functions of temperature and vary along the length of the reactor.

The mass transfer was approximated by similarity with heat transfer in an annulus with only the outside tube heated. Since the Lewis number (ratio of thermal

FIGURE 2-1

BASELINE DESIGN PARAMETERS

CAVITY DIAMETER	10 INCHES
NUMBER OF MODULES	20
OUTER TUBE DIAMETER AND WALL	.625 x .035 INCHES
INNER TUBE DIAMETER AND WALL	.375 x .035 INCHES
SPIRAL WIRE WRAP DIAMETER AND PITCH	.09 x 1.00 INCHES
FLOW CONFIGURATION	INLET FLOW IN ANNULUS

FIGURE 2-2
OPERATING CONDITIONS

TOTAL THERMAL POWER INTO RECEIVER	12.5 kWt
NET THERMAL POWER INTO RECEIVER	8 kWt
NET THERMAL POWER PER MODULE	400 WATTS
GAS PRESSURE ENTERING REACTOR	3 ATM
REACTANT INLET TEMPERATURE	90° C
PRODUCT GAS OUTLET TEMPERATURE	150° C
INLET GAS COMPOSITION	SO ₃ = 91.5%
	SO ₂ = 5.6%
	O ₂ = 2.8%

FIGURE 2-3
FUNCTIONAL REQUIREMENTS

MAXIMUM CAVITY WALL TEMPERATURE	900° C
EXTENT OF REACTION	$\geq 90\%$ OF EQUIL. (800° C)
GAS PRESSURE DROP	$\leq 10\%$ OF INLET PRESS.
HEAT EXCHANGER EFFECTIVENESS	$\geq 80\%$

diffusivity to mass diffusivity) approximately equals one, the mass-transfer Nusselt number or Sherwood number (Sh) can be taken as equal to the heat-transfer Nusselt number⁽¹⁾. Therefore,

$$\text{Nu} = \text{Sh} = \frac{k_c d_h}{D_{1,m}} \quad (2.1)$$

For the tube sizes and gas compositions specified, this reduces approximately to:

$$k_g \left(\frac{\text{lit. gas}}{\text{cm}^2\text{-hr}} \right) = 3.1 \times 10^{-4} T^{3/2} \quad (2.2)$$

The surface reaction rate for SO_3 decomposition catalyzed by platinum was estimated from Westinghouse experimental data⁽²⁾ with a supported-platinum catalyst on alumina. Since the present design calls for platinum deposited on the tubing walls, the experimentally determined rate constant had to be adjusted to account for the differences between wall placement of the catalyst and a porous pellet support. The resulting reaction rate constant was found to be:

$$k_r \left(\frac{\text{lit. gas}}{\text{cm}^2\text{-hr}} \right) = 3.18 \times 10^5 \exp \left(\frac{-21,000}{RT} \right) \quad (2.3)$$

This can be combined with the mass transfer coefficient to give the overall rate constant:

$$k = \frac{1}{\frac{1}{k_g} + \frac{1}{k_r}} \quad (2.4)$$

The rate expression for SO_3 decomposition was modelled as a pseudo first-order reversible reaction with the following equation:

$$-r_{\text{SO}_3} = k \left[C_{\text{SO}_3} - C_{\text{SO}_3, \text{eq}} \right] \quad (2.5)$$

where:

$$k = \text{overall rate constant, } \frac{\text{lit. gas}}{\text{cm}^2 \cdot \text{hr}}$$

$$C_{\text{SO}_3} = \text{concentration of } \text{SO}_3, \frac{\text{moles}}{\text{lit.}}$$

$$C_{\text{SO}_3, \text{eq.}} = \text{concentration of } \text{SO}_3, \text{ at equilibrium, } \frac{\text{moles}}{\text{lit.}}$$

The tubular reactor was assumed to be an integral plug-flow reactor for which the following design relationship applies⁽³⁾:

$$F C_{\text{ao}} dx_a = -r_a dS \quad (2.6)$$

where:

$$F = \text{volumetric flow rate, } \frac{\text{lit.}}{\text{hr}}$$

$$C_{\text{ao}} = \text{initial } \text{SO}_3 \text{ concentration, } \frac{\text{moles}}{\text{lit}}$$

$$S = \text{surface area, cm}^2$$

$$x_a = \text{conversion} = \frac{\text{moles } \text{SO}_3 \text{ reacted}}{\text{moles } \text{SO}_3 \text{ initial}}$$

This equation can be solved to give the following:

$$\frac{kS}{F} = \frac{bX}{b'} + \frac{ab' - a'b}{b'^2} \ln (a' + b'X) \Big|_{x_0}^x \quad (2.7)$$

where a , b , a' , b' - constants depending on the equilibrium conversion and C_{ao} .

Since the temperature varies along the length of the reactor, the mass transfer coefficient (k_g), the reaction rate constant (k_r), and the equilibrium SO_3 concentration ($C_{\text{SO}_3, \text{eq}}$) will also vary along the reactor length. The controlling equations describing each variable as a function of temperature are summarized in Figure 2-4. By the use of these equations and design equation 2.7, the reactor performance at any given constant temperature can be calculated. However, in order to calculate the performance along the length of the reactor, a heat balance must be determined. The kinetic expressions and the heat balances must then be solved simultaneously. This can be done by either writing partial differential equations and solving simultaneously with a numerical technique, or by using an integral method as described below.

Reactor Modelling

The reaction zone model was developed by subdividing the reaction zone into discrete isothermal nodes, then performing a heat balance on each node separately. Figure 2-5 shows the thermal equations used in the analysis of the reactor zone. The primed variables indicate the node exit variables, while the unprimed variables indicate the node inlet conditions. For this analysis, the assumption was made that the two walls are at the same temperature. This was done due to the difficulty of determining the fraction of the annular flow which would react at each wall, and recognizing that radiant heat exchange would tend to reduce the temperature difference between each wall.

The heat transfer coefficients were based upon standard laminar and turbulent flow correlations with a multiplier to account for the effects of the spiralling flow. In the annular supply region, the flow appeared to be laminar through the entire module length. The standard heat transfer correlation is: ⁽⁴⁾

$$h \text{ De}/K = 3.66 K_1 \quad (2.8)$$

where K_1 is a multiplier to account for the spiral annular flow. Data on values for this multiplier were lacking for spiraling laminar flow in an annulus, so the multiplier was developed based upon values stated for turbulent flow in an annulus, ⁽⁵⁾ and for laminar and turbulent flow in curved

FIGURE 2-4
CONTROLLING EQUATIONS

$$K_P = \text{EXP} \left[11.1 - \frac{11,700}{T} \right] \quad (1)$$

$$K_P = \frac{\left[\frac{2}{3} (1 - Y_0) + X_E Y_0 \right]^{3/2}}{Y_0 (1 - X_E) \left(1 + \frac{X_E Y_0}{2} \right)^{1/2}} \quad (2)$$

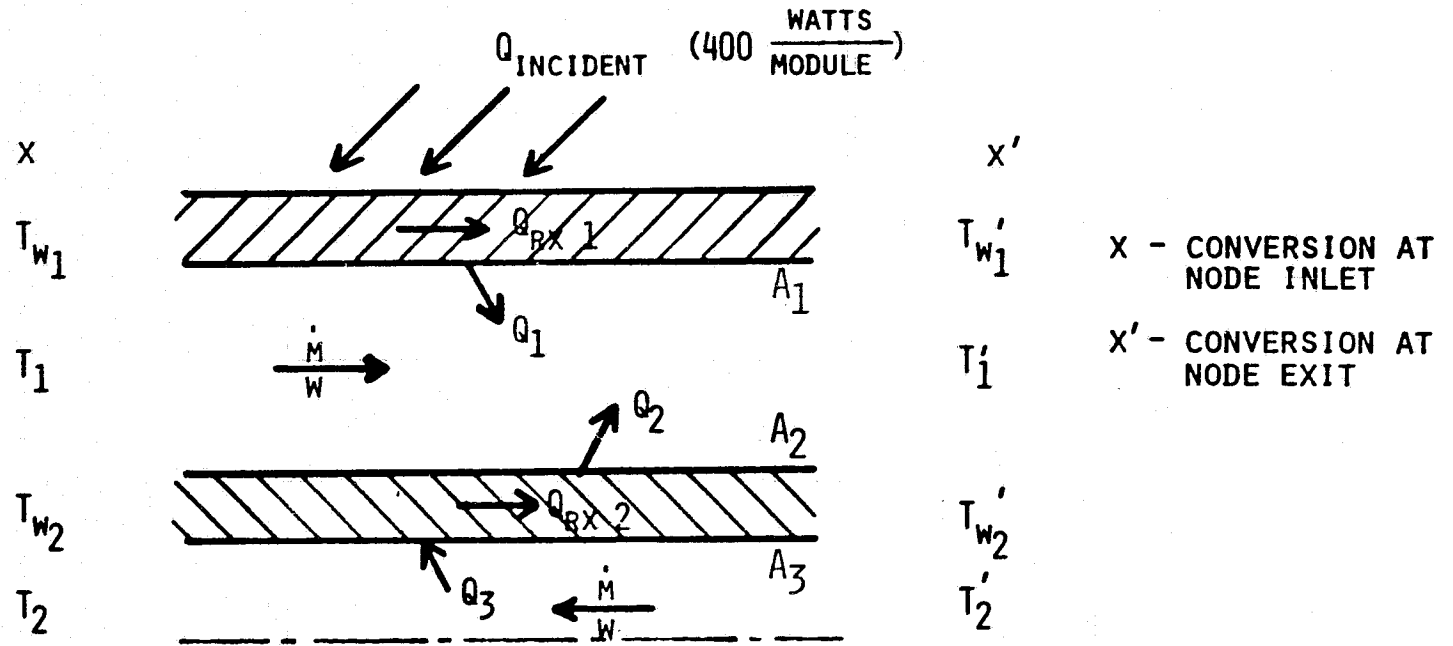
$$K_G = 3.1 \times 10^{-4} T^{3/2} \quad (3)$$

$$K_R = 3.18 \times 10^5 \text{EXP} \left[\frac{-21,000}{RT} \right] \quad (4)$$

$$K_{OV} = \frac{1}{\frac{1}{K_G} + \frac{1}{K_R}} \quad (5)$$

$$\frac{K_{OV} S}{F} = \left| \frac{BX}{B'} + \frac{AB' - A'B}{B'^2} \text{LN} (A' + B'X) \right|_{X_I}^X \quad (6)$$

FIGURE 2-5. REACTOR ZONE ANALYSIS



2-9

SIMPLIFYING ASSUMPTIONS:

$$T_{w1} = T_{w2}, Q_3 = Q_2$$

HEAT ENTERING FLUID 1:

$$Q_1 = Q_{\text{INCIDENT}} + Q_2 - Q_{\text{RX1}} - Q_{\text{RX2}} = \dot{M} C_{p1} (T_1' - T_1)$$

WALL TEMPERATURE:

$$T_{w1} = (T_1 + T_1')/2 + \frac{Q_1}{H_1(A_1 + A_2)}$$

HEAT LEAVING FLUID 2:

$$Q_3 = \frac{1}{\frac{1}{H_2 A_2} + \frac{1}{H_3 A_3}} \left(\frac{T_2 + T_2'}{2} - \frac{T_1 + T_1'}{2} \right) = \dot{M} C_{p2} (T_2' - T_2)$$

HEAT OF REACTION:

$$Q_{\text{RX1}} + Q_{\text{RX2}} = K_1 w (x' - x)$$

K_1 - MOLAL HEAT OF CONVERSION (WATTS/MOLE)

w - MOLAL FLOW OF SO_3 ENTERING REACTOR

ITERATE UNTIL GET HEAT BALANCE

pipes (6,7). For flow in curved pipes, the multipliers ranged from 1.1 to 1.5 for turbulent flow and from 1.3 to over 10 for laminar flow depending on curvature and Reynolds number.

For turbulent flow in an annulus, the multiplier for the wire wrap pitch chosen for this design (one inch, resulting in a P/D ratio of about 1.8) varied from about 1.4 to about 1.8 depending on Reynolds number. Lacking further data, a value of 1.5 for a multiplier was chosen.

For the return flow in the inner tube, the flow is in the transition region at the beginning of the tube, and becomes increasingly turbulent as the fluid is cooled. The standard Dittus-Boelter correlation was used with a multiplier (K_1) to account for the spiralling outward flow in the inner tube:

$$h D/K = 0.023 (K_1) (GD/\mu)^{0.8} (Cp\mu/K)^{0.4} \quad (2.9)$$

Based upon Reference 7, a multiplier of 1.2 appeared reasonable for the flow and geometry for the reference configuration.

The heat balances were then combined with the reaction kinetic expressions and a computer code was developed to calculate the reactor zone performance. Several of the simplifying assumptions used in developing this code are shown in Figure 2-6. A flowsheet of the calculation procedure is given in Figure 2-7.

2.2 HEAT EXCHANGER ANALYSIS

The regenerative heat exchanger, from the gas inlet to the reactor inlet at the beginning of the inner coil, was analyzed separately. It was assumed that the concentric tube heat exchanger was adiabatic. That is, no heat losses occurred either to the surrounding air or adjacent heat exchanger coils.

The heat exchanger was modeled using the TRUMP code.⁽⁸⁾ The nodal network is shown in Figure 2-8. The heat transfer coefficients on the annulus and tube sides were input as a table of equivalent conductivities ($K = h \times \text{distance from node center to boundary}$) versus temperature. Two different cases were modeled.

FIGURE 2-6
SIMPLIFYING ASSUMPTIONS

1. UNIFORM HEAT FLUX AROUND REACTOR TUBE
2. ENERGY BALANCES BASED ON AVERAGE NODE TEMPERATURES
3. INNER AND OUTER WALL TEMPERATURES ARE EQUAL
4. ADIABATIC MODULE ENCLOSURE
5. FULLY-DEVELOPED FLOW CORRELATIONS USED.

FIGURE 2-7
REACTOR ZONE PERFORMANCE CALCULATION

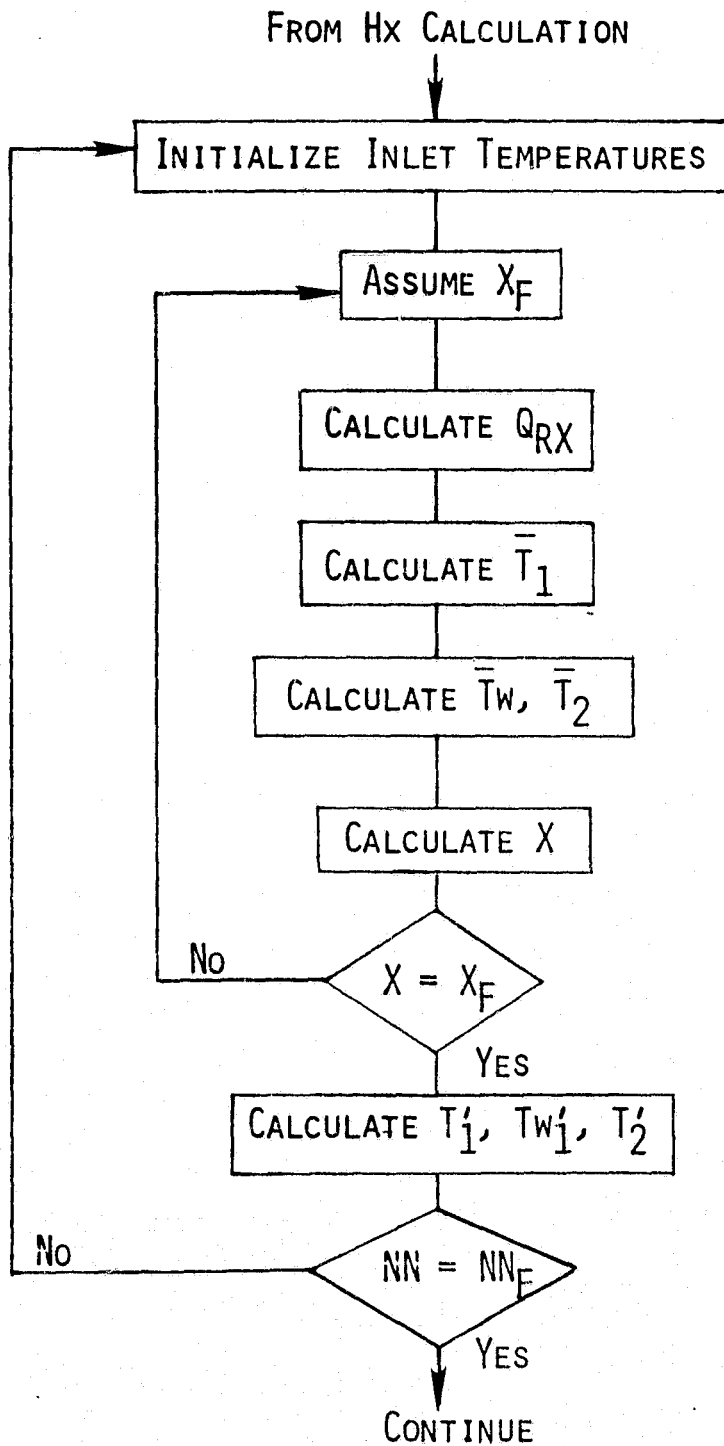
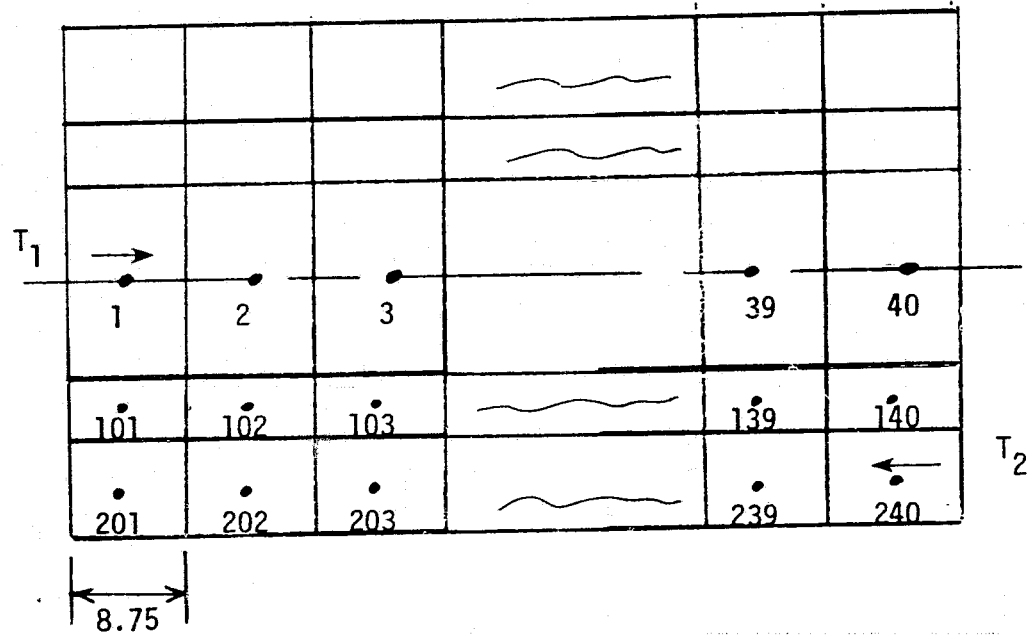
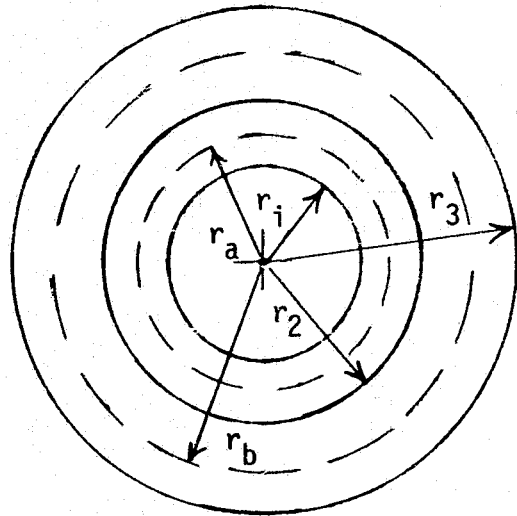


Figure 2-8



2-13

Physical dimensions: $r_1 = 0.1525$ in. $r_2 = 0.1875$ in $r_3 = 0.2775$ in

Nodal positions:
 1 - 40 (Return flow) along centerline
 101-140 (Inner tube), $r_a = 0.170$ in
 201-240 (Supply flow), $r_b = 0.2296$ in

Boundary conditions:
 T_1 - Reactor exit temperature (from reactor calculation)
 T_2 - Heat exchanger inlet temperature (90°C)
 Case 1: Adiabatic boundary at $r_3 = 0.2775$ in
 Case 2: Heat flow from spiral to spiral - (201 to 203, 202 to 204, etc.)

Figure 2-8. TRUMP Heat Exchanger Model

The first case was just a simple counterflow heat exchanger with an adiabatic boundary at the outer tube wall. The second case modeled up the heat flow that would occur radially outward from the receiver cavity through succeeding turns of each spiral module.

The pressure drop was calculated separately for the annular and tube side flows. Friction factors were calculated based upon laminar flow in the annular region and turbulent flow in the return tube. The effect of the spiral wire wrap in the annular region was accounted for by increasing the friction factor calculated for straight laminar flow in an annulus by a factor of 2.0^(5,6,7).

For the return flow in the inner tube, a multiplier of 1.1 was used on the friction factor calculated for straight tube flow, to account for the spiralling outward arrangement of the module.

2.3 MODEL COUPLING

Since each section of the receiver (reaction zone and heat exchanger) were modeled up separately, it was necessary to couple the two models at their boundary. Basically, the temperature of the reactant products leaving the reactor zone were needed as input for the heat exchanger calculation, and the temperature of the SO₃ reactant fluid leaving the heat exchanger section was needed as input for the reactor zone calculation. An iteration process was used, shown in Figure 2-9, to couple the analysis of the two models. This iterative process was continued until the temperatures in the annulus leaving the heat exchanger and entering the reactor matched, and the temperatures in the inner tube leaving the reactor and entering the heat exchanger matched.

2.4 PERFORMANCE ANALYSIS RESULTS

The final results of the modeling analysis are shown in Figures 2.10, 2.11, and 2.12. The flow rate that finally resulted from the analysis was 5.0 lb/hr. The heat exchanger performance is shown in Figure 2.10 for this flowrate. With a total heat exchanger length of 350 inches and a flowrate of 5.0 lb/hr, the heat exchanger had an effectiveness of about 94%. The reactant products entered

SCR MODELING FLOWSHEET

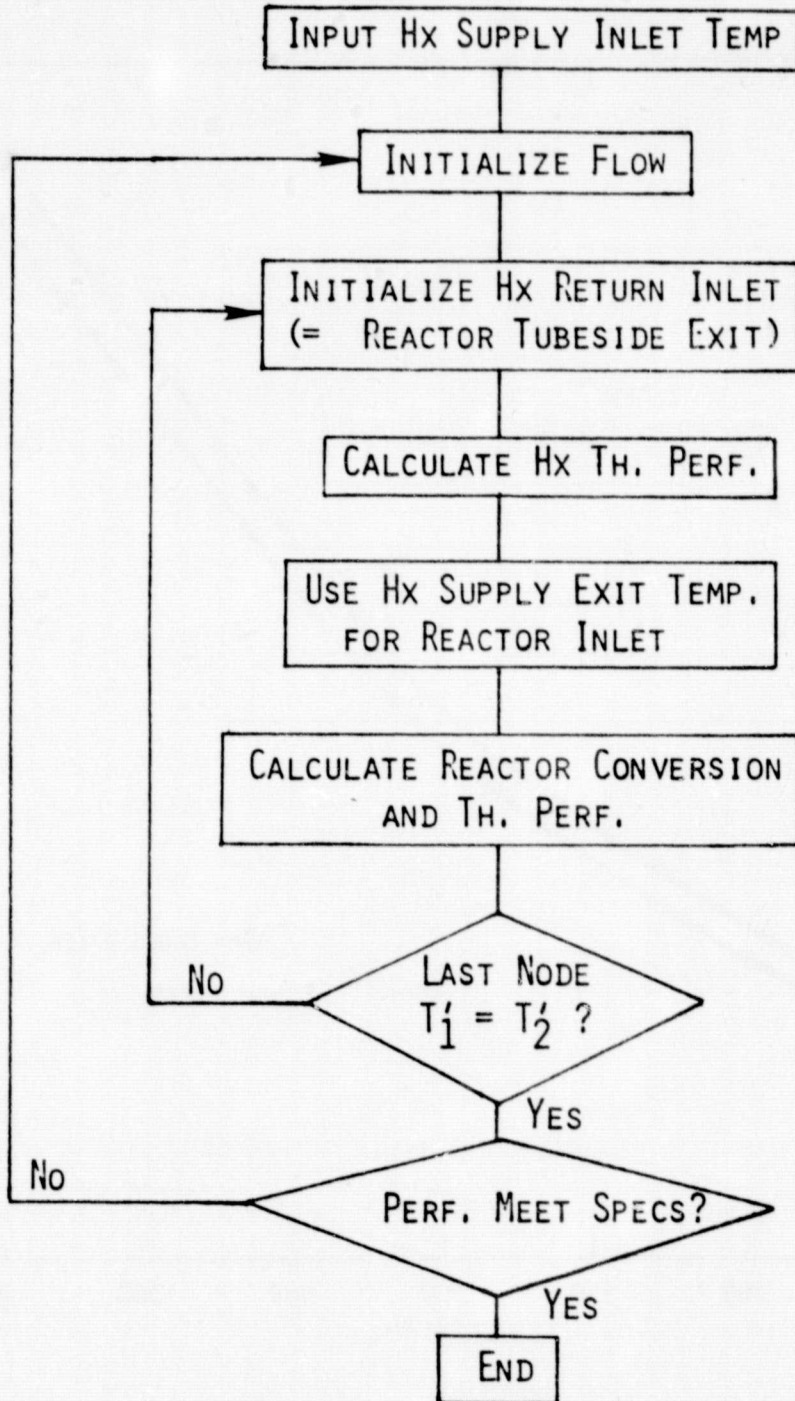
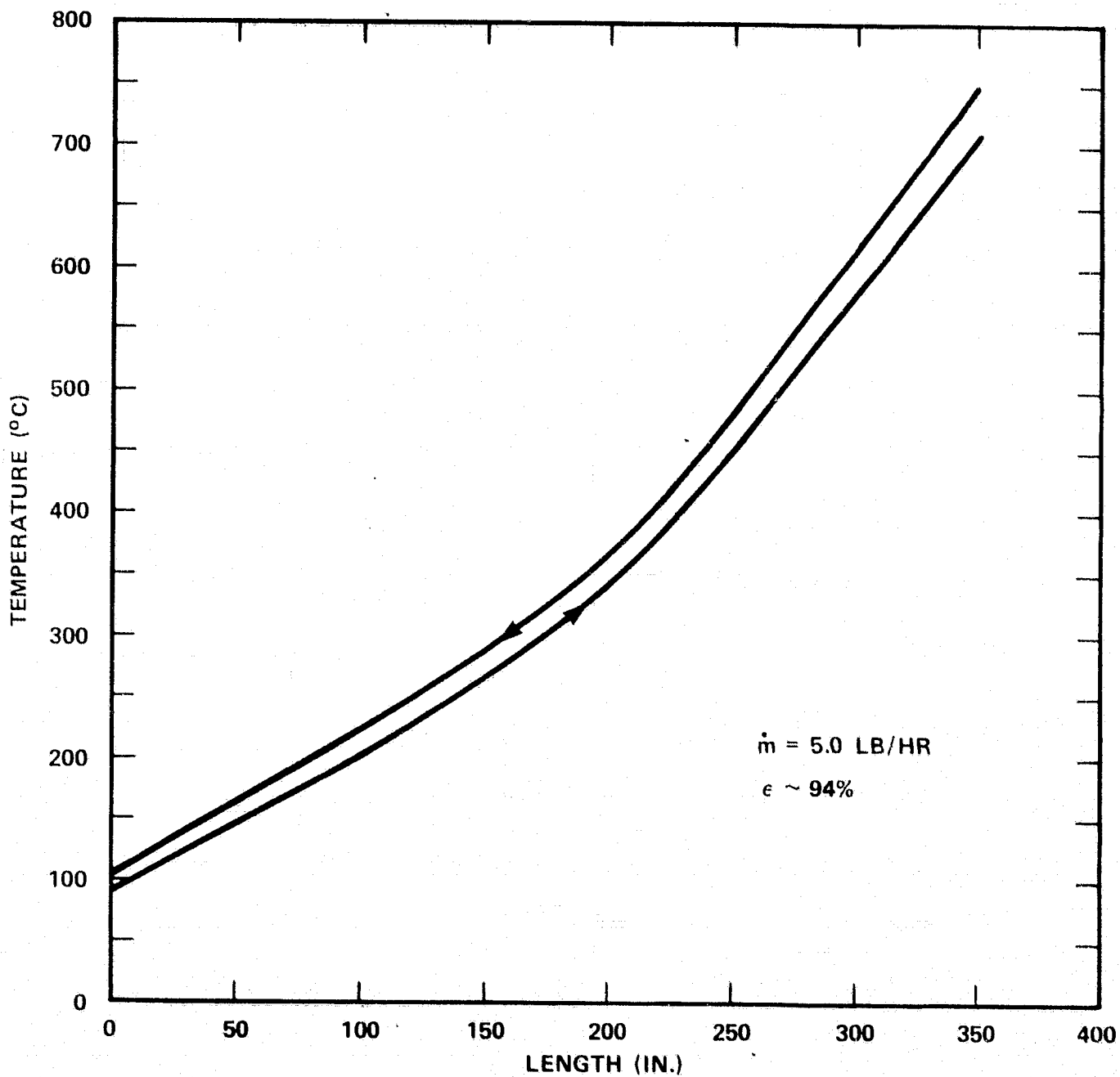
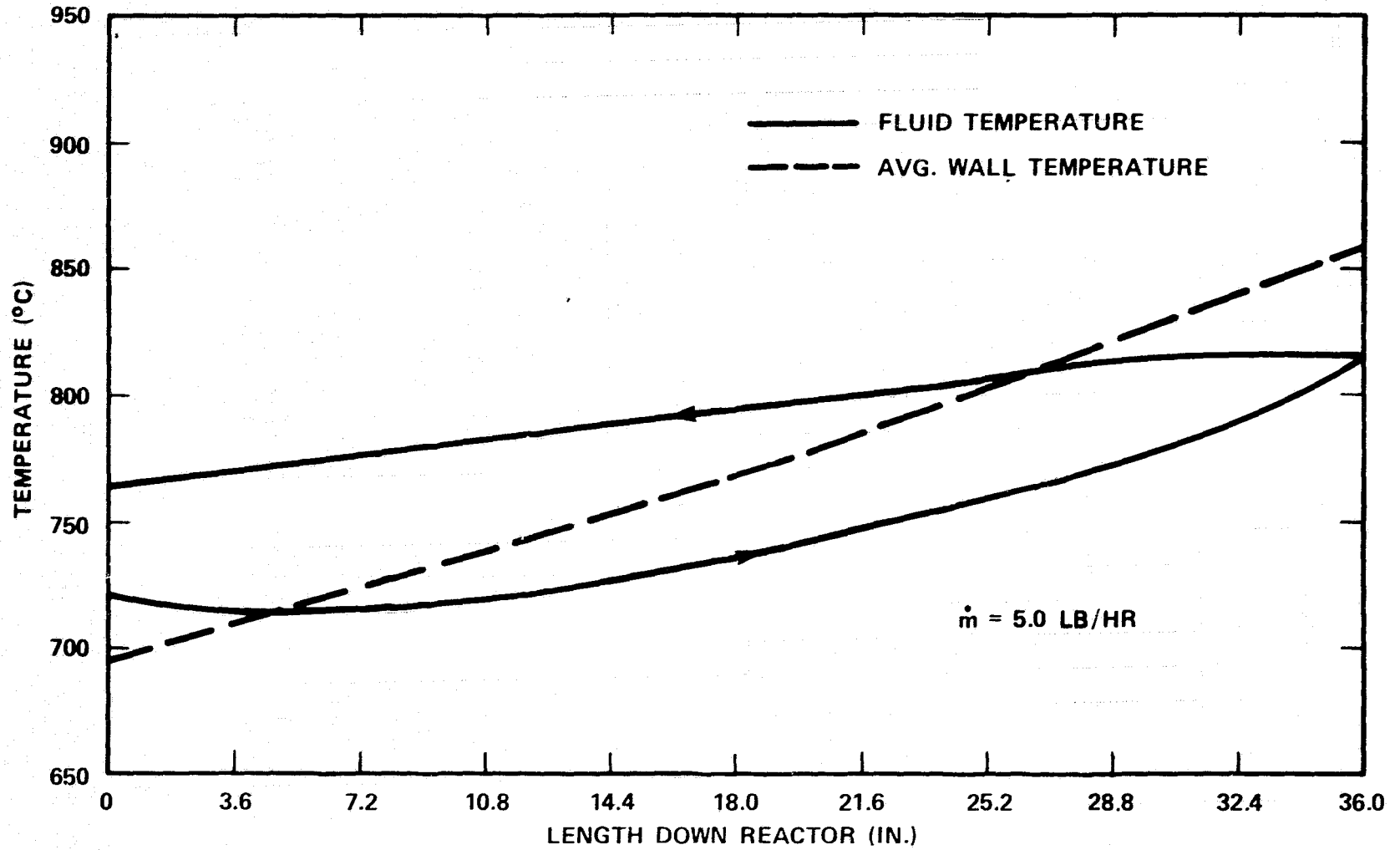


Figure 2-9. Coupling of Reactor Zone and Heat Exchanger Zone Calculations



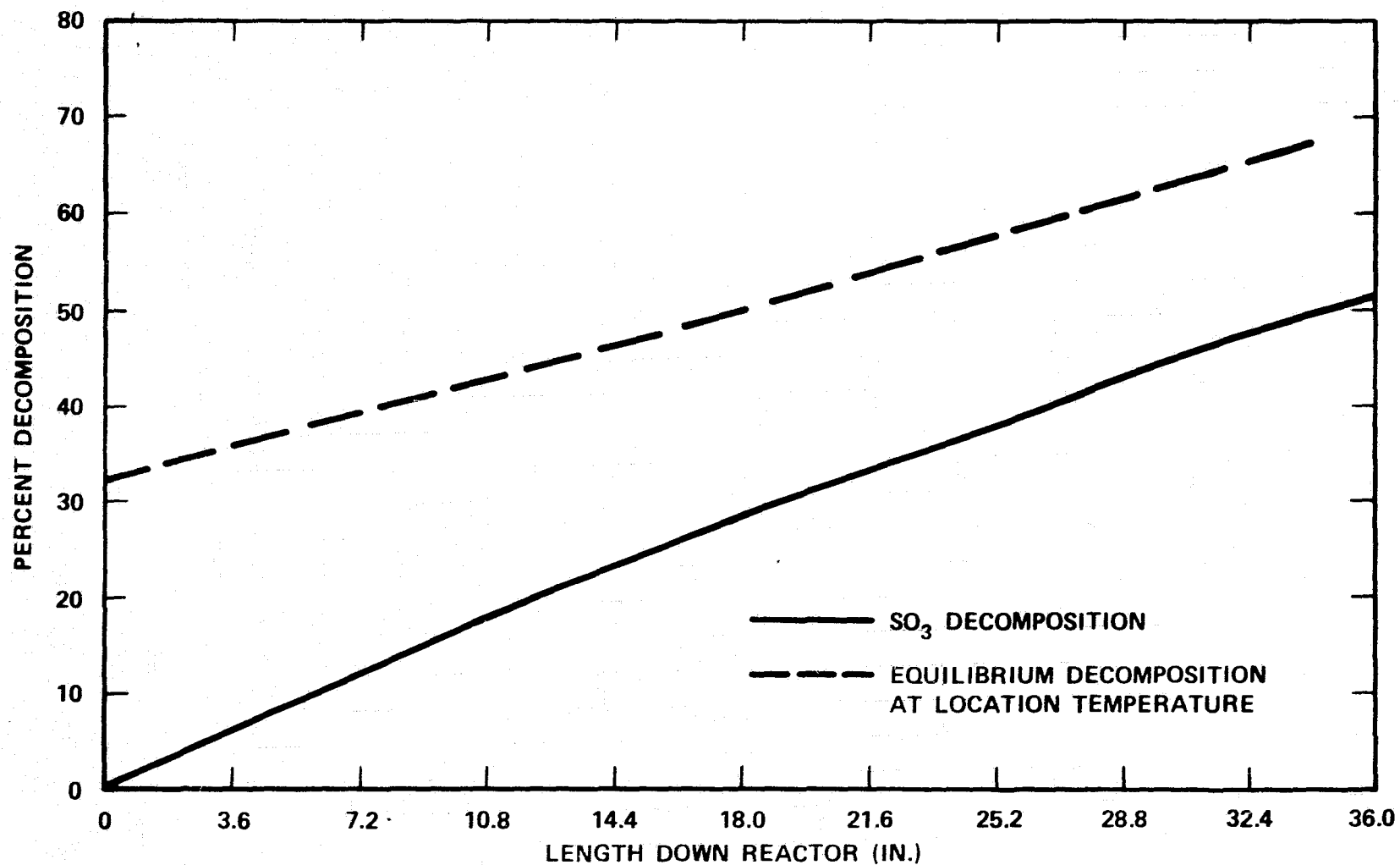
615625-2

Figure 2-10. Heat Exchanger Thermal Performance



615625-3

Figure 2-11. Reactor Zone Thermal Performance



615625-1

Figure 2-12. SO₃ Decomposition Versus Length

the heat exchanger at a temperature of about 760°C and was cooled down to 110°C. This 110°C outlet temperature was well below the requirement of a maximum of 150°C. Meanwhile, the SO₃ reactant entered at 90°C and was heated up to 720°C.

The heat exchanger curve shown in Figure 2-10 is for an adiabatic outer wall surface. Including the effects of radially outward heat flow from one turn of the spiral module to another did not change the heat exchanger temperatures by more than 5°C at any position.

The reactor zone thermal performance is shown in Figure 2-11, while the amount of chemical conversion of SO₃ is shown in Figure 2-12. At the inlet the reaction is consuming more heat than is being supplied by solar energy and from heat transfer from the inner fluid, so the reaction removes heat from the entering fluid and causes a reduction in the gas temperature. As the reaction proceeds toward equilibrium, the rate is reduced and there is excess heat that goes into heatup of the gas, as shown after a distance of about 4 inches. The gas continues to be heated for the rest of the reaction zone, and reaches an upper limit of 820°C. The flow then exits the annular reaction zone and returns through the inner tube. Heat is transferred from this returning fluid to the reaction annulus, thereby lowering the temperature of the return flow. The reactant products are cooled to a final temperature of 760°C by the end of the reaction zone. The peak tube wall temperature is approximately 860°C, which is below the maximum of 900°C set forth in the functional requirements.

The amount of chemical conversion is shown in Figure 2-12. At the beginning of the reaction zone there is a large difference between the degree of conversion and the equilibrium conversion at the tube wall temperature for this position. This resulted in a rate of reaction that exceeds the heat transfer rate, and causes a reduction in the reactant fluid temperature at this location. As the reaction proceeds toward equilibrium, the rate is reduced so that some heat contributes to an increase in the tube temperature, and a resulting increase in the equilibrium decomposition. This rise of the equilibrium decomposition tends to result in essentially an even rate of decomposition of SO₃ to occur, with a resulting fairly steady rise in the reactant temperature.

The final degree of decomposition is approximately 51.7%, which is just equal to the amount of conversion set forth in the functional requirements.

The pressure drop was calculated based upon a flow rate of 5.0 lb/hr. In the annular side, the total pressure drop was approximately 1.6 psi, while for the flow returning in the inner tube the pressure drop was approximately 1.36 psi. This resulted in a total drop of approximately 3.0 psi, which is approximately 0.2 atmospheres. This is in comparison with a requirement of a total pressure drop of less than 0.3 atmospheres.

Based upon the results of this analysis, the JPL chemical receiver configuration appeared able to meet or exceed all of the functional requirements. A summary of required versus calculated performance is given in Figure 2-13. The tube temperature was desired to stay below 900°C, while the analysis showed a maximum temperature of 860°C. The heat exchanger exit fluid temperature was desired to be below 150°C, while the analysis resulted in a temperature of 110°C. The extent of reaction (or percent of SO_3 decomposed) was desired to be 51.7% or better, while the analysis just met this requirement with a final conversion of 51.7% for the chosen flow rate. As a result the design appears able to satisfy all of the functional requirements.

2.5 STRUCTURAL/MECHANICAL ANALYSIS

The primary hoop stress in the .625 O.D. x .035 thick outer tube resulting from the expected operating internal pressure of 3 atmospheres is approximately 300 psi. The strength of 316 SS for rupture in 10^5 hours at 800°C (1471°F) is approximately 2500 psi. At 900°C (1651°F) this extrapolates to approximately 1500 psi. Therefore, the available long time, high temperature strength is at least 5 times that required based on the full .035 in. tube wall thickness. This should provide sufficient margin to accommodate a substantial reduction in strength due to chemical corrosion, Alonizing*, etc.

*Alonizing is a trademark of Alon Inc.

FIGURE 2-13
REQUIRED VS. CALCULATED PERFORMANCE

<u>ITEM</u>	<u>REQUIRED</u>	<u>CALCULATED</u>
HX EFFECTIVENESS	$\geq 80\%$	94%
HX OUTLET TEMPERATURE	$\leq 150^{\circ} \text{ C}$	110° C
SO ₃ CONVERSION	$\geq 90\%$ OF EQUILIBRIUM AT 800° C (=51.7%)	51.7%
MAXIMUM WALL TEMPERATURE	$\leq 900^{\circ} \text{ C}$	860° C
FLUID PRESSURE DROP	$\leq 10\%$ INLET PRESSURE (= .3 ATM)	0.2 ATM

The linear expansion of the 386 inch long inner tube due to thermal expansion, assuming 1600°F operating temperature at the reactor section, is approximately 3.75 inches. The outer tube expands approximately 0.2 inches less than the inner tube due to the somewhat lower temperature of the outlet gas to which it is exposed. An axial clearance substantially greater than this amount can be provided between the end of the inner tube and the outer tube end plug to accommodate transient effects in addition to the 0.2 inch differential growth of design conditions. Since the tubes are both fixed at their relatively cool outer extremities to the manifolds and casing structures, the thermal expansion of the tubing must be accommodated by a rotation of the innermost ends of the tubes around the cavity periphery. The amount of this rotation is estimated to be approximately 47° for the 386 inch long tube module. The tubing expansion in the inward direction also results in a small reduction in the cavity diameter to 9.93 in from the original 10.00 in value as a result, imposes a bending stress in the tubing as it conforms to the smaller radius.

The bending stress imposed on the innermost turn of the tubing module as a result of its being forced to conform to a reduced radius of curvature is estimated to be 8,180 psi. This value assumes a value of 20×10^6 for Youngs Modulus at the 1600°F (approx) operating temperature of the innermost turn and, in addition, assumes that the module is initially wound, together with the separator spiral, into a completely tight configuration and is free from residual stress in this configuration.

The 8,180 psi bending stress, although somewhat below the typical short time yield strength at 1600°F of approximately 15,000 psi, will be subject to relaxation due to high temperature creep. It can be inferred from the low value of the 1% creep strength in 10,000 hours of less than 3000 psi that most of the 8,180 psi bending strength will creep out early in the operating lifetime leaving the module inner turns in a relaxed condition at the smaller operating radius. Having achieved this state, cooling of the module, due to removal of the source of heat, will result in thermal contraction of the tubing and rotation of the inner turns but at smaller radii than in the initial cold condition. Thus clearance will have been introduced between the turns of the module as a result of the creep relaxation and resulting reforming of the tube radii.

Subsequent reheating of the module will cause the tubing to re-expand and absorb the relaxation clearance, returning the tubing to its partly relaxed bending condition. Creep relaxation then continues for the remainder of the operating life. It should be observed that the bending stresses in the tubing are not considered as primary stresses, being self limiting (load is removed as a result of small amounts of strain due to creep). As a result, these stresses and the resulting creep relaxation do not reduce the operating lifetime in any way. This is in contrast to the hoop stress in the tubing due to internal gas pressure, discussed earlier, which is primary in nature and does not experience any relief of load as a result of creep.

The foregoing discussion ignores the effects of friction between the tubes during sliding. In an initially tightly wound assembly, the buildup of frictional force along the tube from its innermost end could conceivably result in significant stress in the tubing. This may be somewhat academic, since an initially tightly wound assembly will probably be impractical from manufacturing and assembly considerations. The clearance between tubes and separator which will have to be provided to facilitate manufacture and assembly will probably be sufficient to accommodate the thermal expansion motion without significant sliding forces.

The tubes, in addition to expanding linearly, also expand radially. As a result, a stack of 20 tube modules expands approximately 0.2 inches across the hot cavity periphery region. This expansion can be easily accommodated by providing clearance between the adjacent modules. A total axial clearance of substantially greater than this value will probably be appropriate to accommodate manufacturing tolerances in the tubing modules in addition to the thermal expansion. Some springiness in the stacked tubing modules can be expected as a result of tolerance effects. The springiness will then provide adequate location for the tubing modules while accommodating thermal expansion.

The tubing modules will be welded at their outer ends to the outlet and inlet manifolds and to the outer edge of the spiral separator. The outermost turn of the separator is welded, in turn, to the periphery of the back and front plates forming a stiff cylindrical support structure for the tubing modules. The

width of the spiral separator is reduced towards the hot cavity periphery to provide axial clearance at the end plates to accommodate thermal expansion of the separator in the axial direction. A possible approach to preventing air circulation in the radial direction at the ends of the spiral separator is to employ ceramic fiber insulation in the back and front plates and permit the separator to compress the insulation locally as it expands.

The inner tube of the coaxial tube spiral module is centered in the outer tube by means of a spiral wire wrap. As previously mentioned the inner tube is entirely free to expand and contract independently of the outer tube. The hoop stress in the inner tube is insignificant, resulting from the gas pressure drop only. The gas pressure drop increases from essentially zero at the hot end, where the tube is weakest to its maximum value at the inlet and outlet manifolds where the tube is cool and therefore much stronger. The structural/mechanical analysis of the proposed module arrangement are summarized in Figure 2-14.

2.6 CATALYST TYPE AND PLACEMENT METHODS

In the preferred approach, catalyst in the form of platinum metal will be applied to the Alonized stainless steel inner surface of the outer tube and the outer surface of the inner tube in the innermost turn of the tube modules. In this approach no provision will be made for catalyst replacement since it is envisaged that the platinum will be bonded thermally before the tubing is bent into its spiral configuration. The separation of the catalyst treated portions of the inner and outer tubes from the heat exchanger portions by cutting through the tubing could possibly be considered but the replacement of the inner turn sections by welding to the heat exchanger portion would be difficult to achieve and would result in a vulnerable section of unprotected weld in the flowing gas stream.

The alternative pellet catalyst approach is probably more amenable to catalyst replacement by removing the end plug from the outer tube and replacing it by welding after replacement of the catalyst. The use of a tightly fitting plug could probably afford some protection to the weld area at the tube end where Alonizing would not be possible. In this approach, the spiral wire wrapping

FIGURE 2-14
STRUCTURAL/MECHANICAL ASPECTS

HOOP STRESS IN OUTER TUBE AT 3 ATM	300 PSI
316 SS STRENGTH (RUPTURE IN 10^5 HRS. AT 900° C)	1500 PSI
LINEAR EXPANSION OF INNER TUBE	3.75 INS.
LINEAR EXPANSION OF OUTER TUBE	3.55 INS.

on the inner tube would probably have to be eliminated in the inner turn of the module and the inner tube allowed to contact the outer tube wall. Such an eccentric location of the inner tube would allow the accommodation of larger size catalyst beads than would otherwise be possible. Using this approach, the end of the inner tube would probably be plugged and the gas admitted to the inner tube through many small drilled holes to minimize the possibility of catalyst being admitted to the inner flow passage.

In summary, platinum applied to the channel walls is judged the most desirable approach. Platinum replacement will be difficult to justify as a result of the compromises in the design simplicity and structural integrity incurred in its achievement. Further discussion of catalyst placement is given in Section 4.2.

3.0 RECEIVER MODULE DESIGN DEVELOPMENT

The objectives of this task were the following:

- To design a solar chemical receiver module based on the results of the design analysis.
- To specify and design the bench-test apparatus and associated hardware required for the module fabrication and testing.
- To prepare an outline of the experiments required to conduct the bench tests and module functional performance test.

A drawing of the conceived solar chemical receiver module is shown in Figure 3-1. It consists of a concentric tube arrangement, with a 3/8" x .035 wall inner tube contained inside a 5/8" x .035 wall outer tube. A .090" stainless steel wire is spirally wrapped with a 1" pitch around the inner tube to maintain the spacing between tubes. The inner tube, outer tube, and spiral wrap are all constructed of 316SS. The concentric tubing is spirally wound into eight coils, with an inner-coil diameter of approximately 11" and an outer coil diameter of approximately of 21". Total length of the tubing is approximately 33 feet. The final 10 feet of the module, consisting of the innermost 2-1/2 coils, is coated with an Alonized* aluminide coating on both sides of the annulus space (i.e., the outer tube ID and the inner tube OD). The final three feet of the module, consisting of the innermost coil, is coated with a platinum catalyst on top of the Alonized surface. A end plug is welded to the reactor end of the module and an inlet adapter is welded to the inlet end. A cutaway of the reactor end of the module is shown in Figure 3-2.

Bench tests were planned to measure module performance at various mass flowrates and heat fluxes. They consisted of testing ten foot long straight concentric tubes simulating the innermost 2-1/2 coils of a module. The tubing was full-scale and the final three feet (i.e., innermost coil) were subjected to a

*Alonized is a trademark of Alon Incorporated.

radiation flux in a radiant furnace. The bench test apparatus design and test plan are discussed further in Section 5.0 of this report.

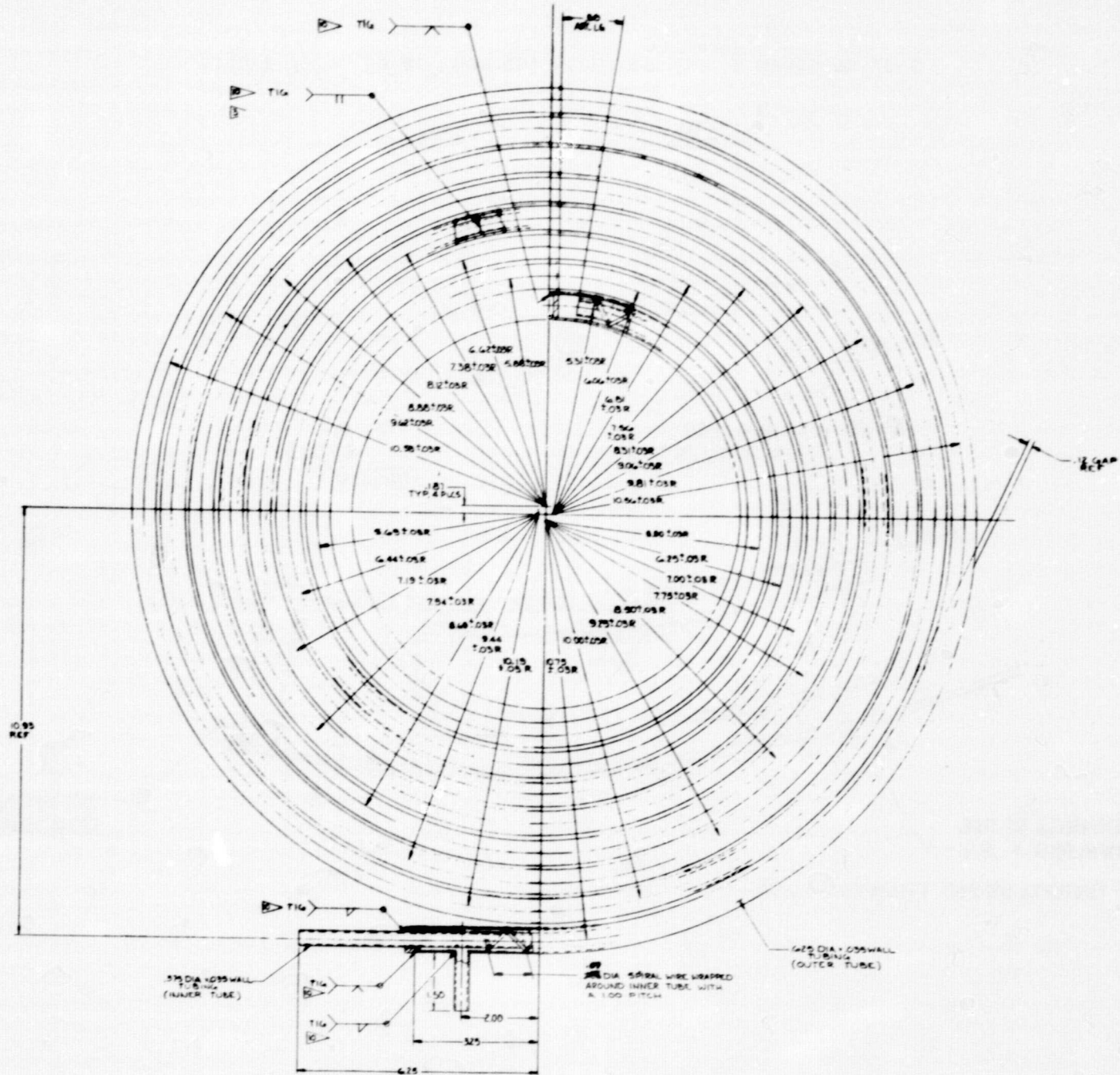


Figure 3-1. Solar Chemical Receiver Module

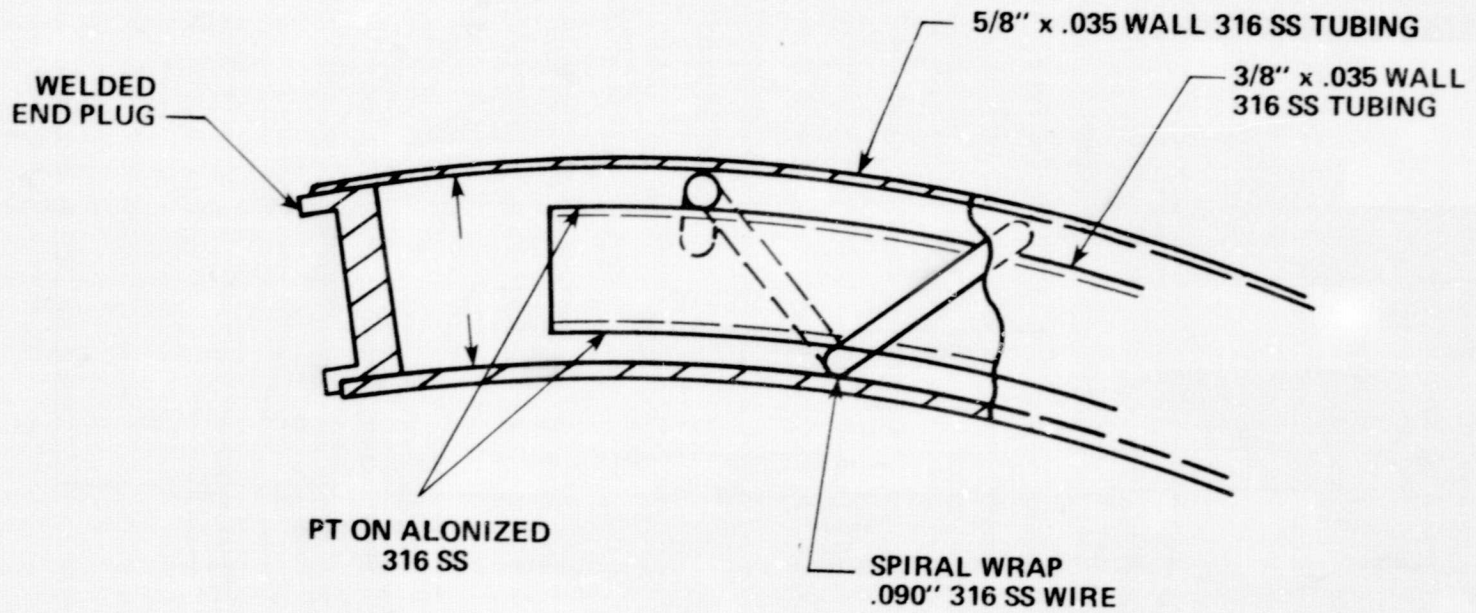


Figure 3-2. Solar Chemical Receiver Module Reaction Zone

4.0 RECEIVER MODULE FABRICATION

The purpose of this task was to experimentally resolve several areas of uncertainty associated with the design and construction of the Solar Chemical Receiver. The areas of uncertainty addressed included joining alonized tubing, catalyst placement, assembly and fabrication of concentric tubes, and coiling alonized tubing.

Several innovative techniques and processes were necessary to fabricate the coiled module. These procedures were developed and successfully demonstrated within the scope of this task.

The following sections present detailed discussions of all the steps, procedures, etc. that were utilized in fabricating a Solar Chemical Receiver Module.

4.1 ALONIZING

Alonizing is a high temperature process that diffuses aluminum into a base metal to form an aluminide surface layer; i.e., Fe-Al in carbon steel. This inter-metallic compound provides both excellent heat and corrosion properties coupled with strength and rigidity.

The alonizing in the critical regions of the Solar Chemical Receiver not only provides oxidation protection but it also helps to prevent the back reaction ($\text{SO}_2 + 1/2 \text{O}_2 \rightarrow \text{SO}_3$), which is catalyzed by iron oxide, from occurring during cool down.

In addition, the alonized surface also provides a suitable substrate for the platinum catalyst. If the Pt catalyst was directly applied to the 316 SS, it would ultimately diffuse into the metal and become ineffective. The alonized surface prevents this from occurring.

The base design of the Solar Chemical Receiver requires that three surfaces be alonized. They are:

- 1) the final three feet of the inside diameter of the outer tube. The alonizing here is used as a catalyst substrate;
- 2) the final three feet of the outside diameter of the inner tube. This surface is also a catalyst surface;
- 3) the final ten feet of the inside diameter of the inner tube. Here the alonizing provides oxidation protection and prevents the reverse reaction. Al_2O_3 has been shown to be a poor catalyst for the back reaction or oxidation of SO_2 to SO_3 .

The total needs of the project (including catalyst placement tests, fabrication verification, module fabrication, etc.) were estimated and sufficient lengths of 316 SS were sent to Alon, Inc., Tarentum, PA., for alonizing. Along with the ten foot lengths, several shorter lengths (~ 2 ft.) were also included. The short lengths were used in the catalyst placement tests.

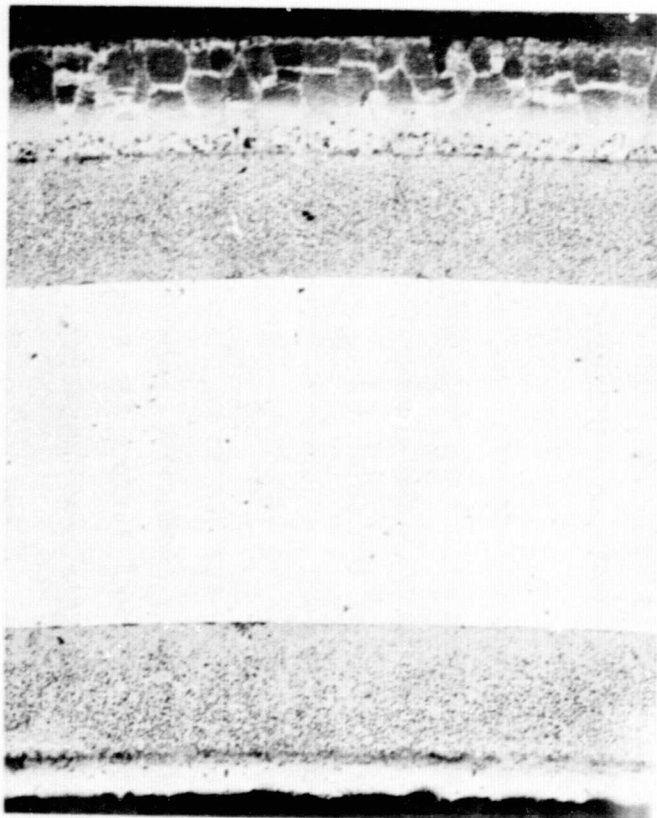
Upon return of the alonized tubes, samples were taken and analyzed. Figure 4-1 presents photomicrographs of these samples. Photo A is a transverse view of a 3/8-inch O.D. x 0.035-inch wall 316 SS tube. Both the I.D. and O.D. are alonized. The diffusion layer on the O.D. is ~ 12 mils (0.012-inch) and the I.D. is ~ 9 mils. Photo B is an alonized 5/8-inch O.D. x 0.035-inch wall 316 SS tube alonized on the I.D. only. Here, the alonizing is ~ 4.5 mils thick.

The alonizing process is proprietary to Alon, Inc., and therefore the variables controlling the depth of aluminum diffusion (i.e., the alonized layer) are unknown.

Although the alonizing provides corrosion resistance and a substrate for the catalyst, it also has its disadvantages. These include:

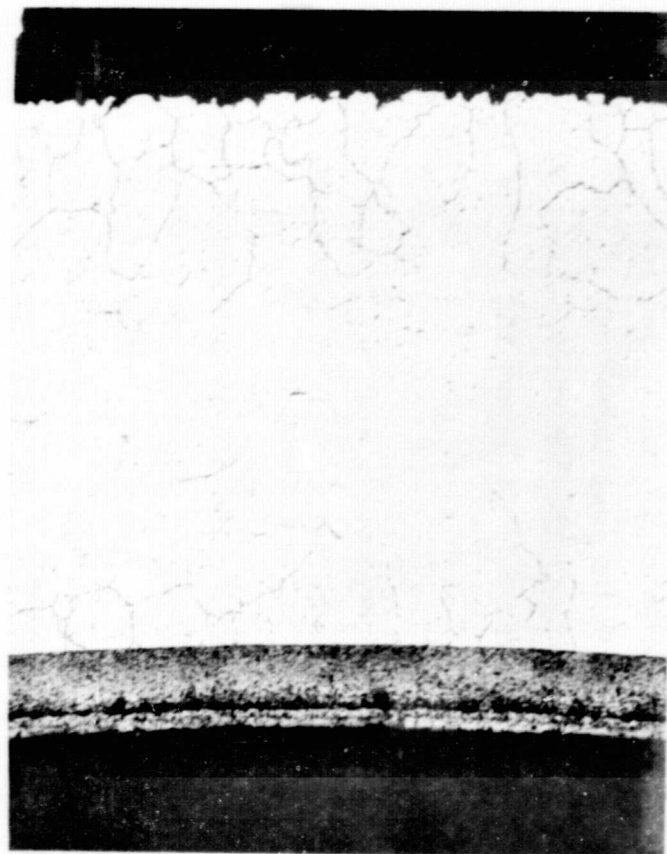
- 1) the alonized tubes are returned with a greasy, gritty surface that requires cleaning;
- 2) alonized surfaces are not conducive to welding;
- 3) aluminide compounds are very brittle and require high temperatures to facilitate bending without cracking.

These problems and the work done to overcome them are addressed in subsequent sections of this report.



(A)

3/8-inch O.D. x 0.035-inch wall
Alonized I.D. and O.D.
Transverse View
100 X



(B)

5/8-inch x 0.035-inch wall
Alonized I.D.
Transverse View
100 X

Figure 4-1. Photomicrographs of Alonized 316 SS Tubing.

4.2 CATALYST PLACEMENT

The objective of the catalyst placement task was to develop a technique for depositing a platinum catalyst on an alonized surface. The platinum catalyst is necessary to accelerate the reduction of SO_3 to SO_2 and oxygen. The reduction reaction occurs primarily in the last three feet of the Solar Chemical Receiver. The concentric tube geometry of the SCR requires that this catalyst be located on the inside surface of the outer tube and the outside surface of the inner tube.

Ideally the platinum would be deposited directly onto the 316 SS. However, due to the thermal exposure the SCR will experience, this is not feasible.

Eventually the platinum would diffuse into the 316 SS and become ineffective. A substrate to hold the catalyst in place is required. An alonized surface proves to be an efficient catalyst support as it possesses little catalytic activity and is compatible with the process environment. However, there was no information available on techniques for depositing platinum on an alonized surface.

Initial efforts included contacting commercial catalyst manufacturers and discussions with various personnel at the Westinghouse Research and Development Center. The commercial vendors were not willing to undertake such a task without a commitment for a large order or a substantial development program.

Personnel at the R&D Center suggested several methods, including chemical vapor deposition, ion implantation, and various electroplating techniques. CVD and ion implantation were not suitable due to the size and geometry of the specimens to be coated, particularly the inside surface of the outer tube, and for economic considerations. Electroplating, therefore, was explored in further detail.

As a prerequisite for electroplating, the surface to be plated (the alonized surface in this case) must be electrically conductive. The alonized surface, composed of alumina (Al_2O_3) was not an ideal surface for electrodeposition. Therefore, a precoat of a conductive material was necessary.

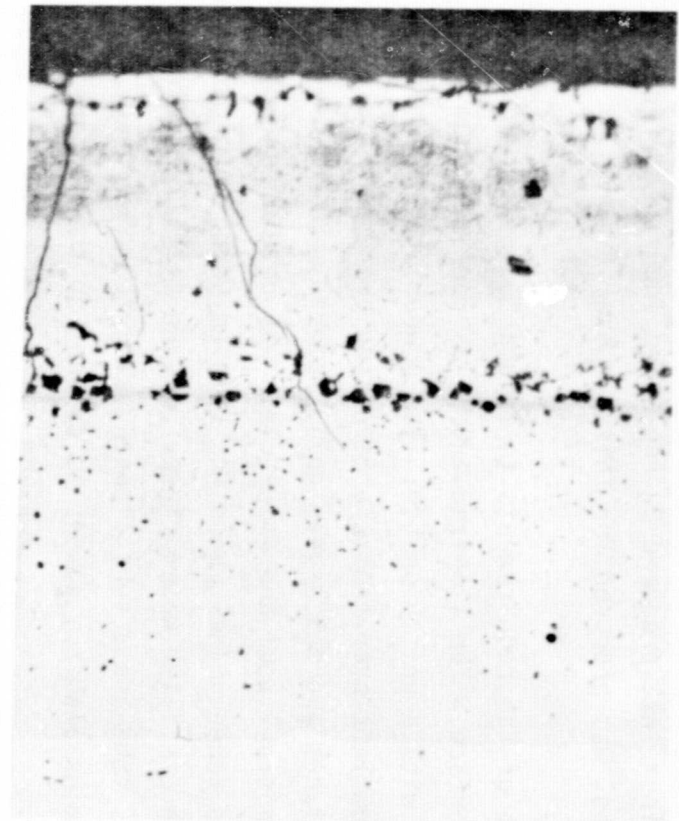
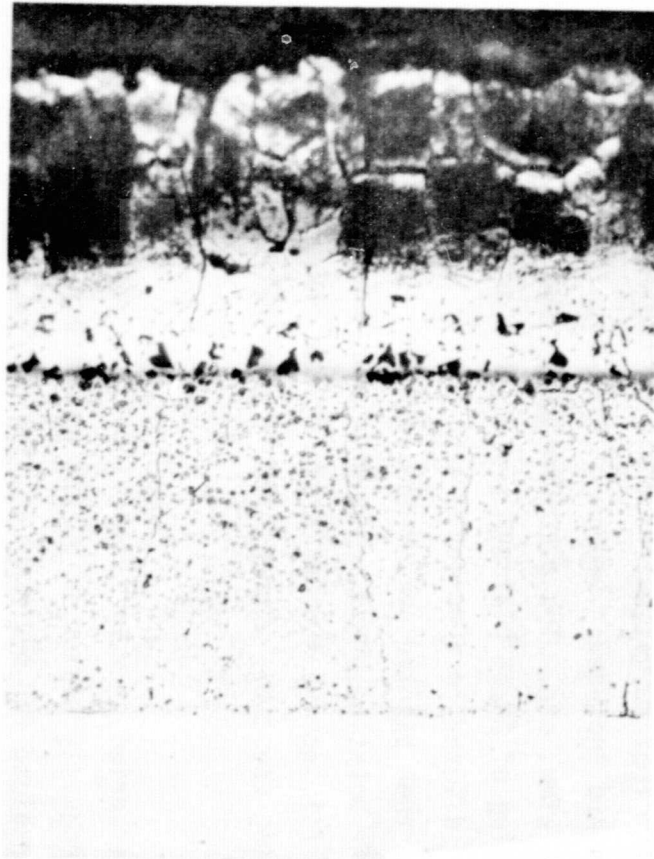
A commercially available platinum ink, produced by Englehard Industries for application on ceramic surfaces, was procured for this purpose.

According to the supplier, one of the requirements to insure successful application is a clean, greaseless surface. The alonized tubes, as received from the vendor, are greasy and coated with a gritty powder. The powder was determined to be residual alumina-aluminum left over from the alonizing process. The powder was not bonded to the tubes and was mechanically removed. A cleaning procedure using extremely fine steel wool and emory cloth was developed and used to clean all the alonized tubes. The grease was removed using an organic solvent followed by a thorough distilled water rinse. Figure 4-2 illustrates a comparison of an "as-received" tube and a tube cleaned using the developed procedures. The residual alumina-aluminum powder is evident on the surface of the as-received alonized tube.

Several sections of tubing (approximately three inches long) were then coated with the platinum ink with varying degrees of success.

After several attempts, an efficient technique for applying the ink was developed. Parameters determined included: degree of dilution of the ink, method of application, drying time, etc. The platinum ink is supplied as a highly viscous liquid consisting of 66.7% platinum (by weight) suspended in an organic vehicle. The ink was diluted to a workable consistency using iso-amyl acetate. The diluted ink was then painted on the alonized outside surface of the 3/8-inch tube using a soft-bristled brush. The alonized inner surface of the outer tube was coated by filling the tube with the dilute ink, stoppering both ends, then slowly rotating the tube. Excess ink is then removed and saved.

The coated sample was then air dried at room temperature for ~18 hours. After air drying, the sample was gradually heated in an air atmosphere to $>1000^{\circ}\text{C}$. The peak temperature was maintained for one hour and the sample then slowly cooled to room temperature. The firing procedure was necessary to decompose the volatile organic binder in the ink and to bond the platinum to the alonized surface. A slow heatup rate was used to prevent the ink from blistering and



(A)
As-Received

(B)
After Cleaning

3/8-inch O.D. x 0.035-inch Wall 316 SS Tube
Alonized O.D.
300 X Magnification
Transverse View

Figure 4-2. Photomicrographs Comparing as-received and Cleaned Alonized Tubing.

peeling. The entire painting and firing sequence was repeated for each sample to insure a uniform platinum surface.

A visual inspection of the tube was conducted to determine if there were any uncoated regions.

To monitor the process, samples were randomly taken for metallographic, EDAX, and SEM analysis. Examination of the samples showed that the platinum layer, ~ 0.5 mils thick, was relatively uniform and covered the entire surface. Figure 4-3 shows the surface of an alonized 316 SS tube prior to platinizing (Photo A) and after platinum has been applied (Photo B). Figure 4-4 is a photomicrograph (transverse view) of a platinized-alonized 316 SS tube. The platinum coating shown here is ~ 0.5 mils thick.

An EDAX scan of the platinized surfaces of the inside of the outer tube and the outside of the inner tube revealed that platinum was the only detectable species.

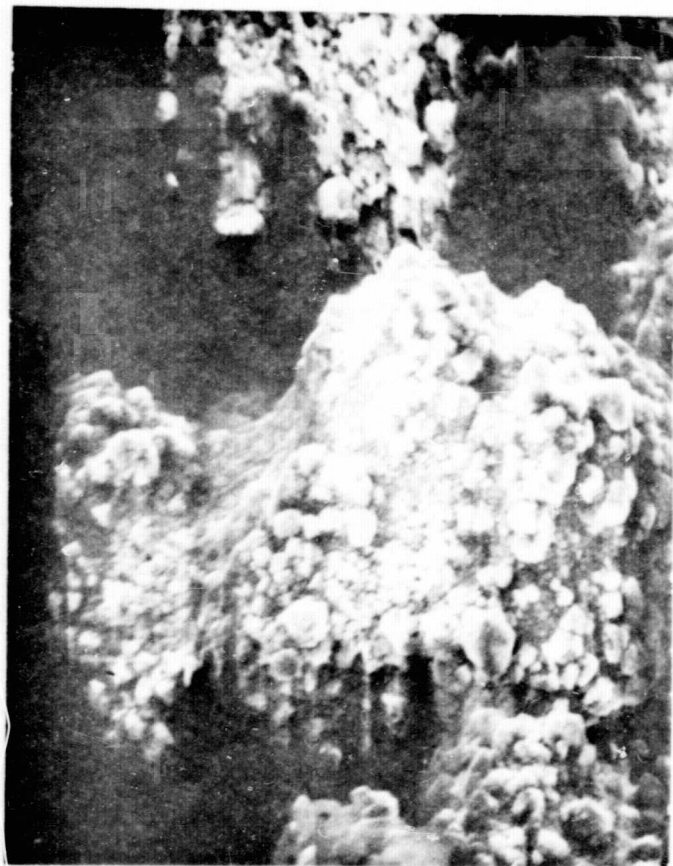
Once the catalyst placement techniques were determined to be successful on small samples, the actual tubes to be used for the module and the bench tests were platinized using the same procedure.

Based on the success with this catalyst placement procedure, efforts on electroplating were discontinued.

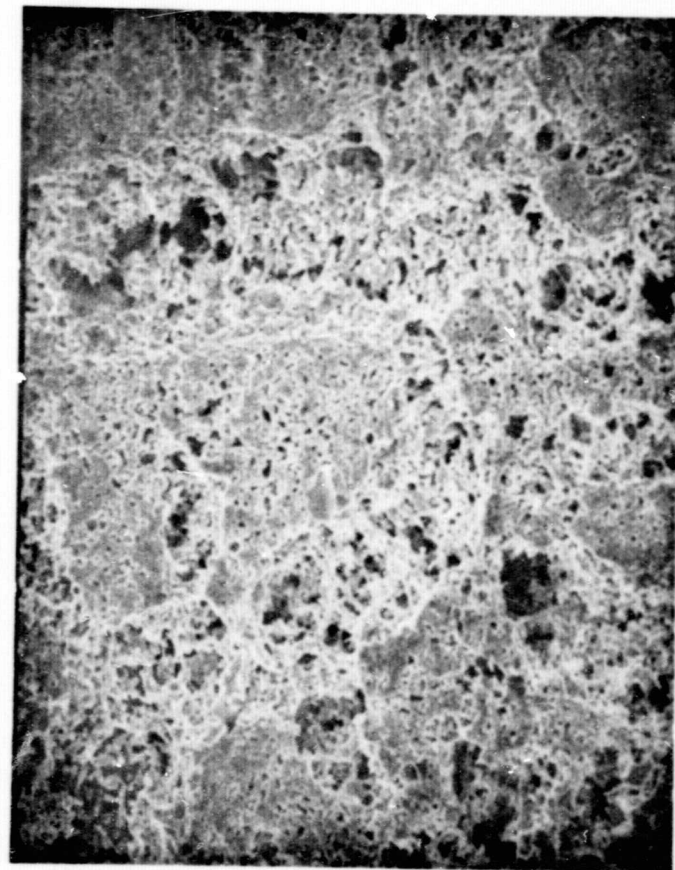
4.3 CONCENTRIC TUBE ASSEMBLY FABRICATION

The concentric tube geometry of the Solar Chemical Receiver, coupled with the use of alonized 316 SS required the development of several unique manufacturing techniques. The objective of this task was to develop and demonstrate the techniques required to assemble a straight concentric tube assembly. These include:

- inserting the inner tube into the outer tube;
- maintaining the concentricity of the two tubes;
- welding alonized tubes.



(A)
750 X
Alonized 316 SS Tube



(B)
800 X
Platinized-Alonized 316 SS Tube

Figure 4-3. Comparison of Alonized Surface and Pt-Coated Alonized Surface.



300 X Magnification
Transverse View
Platinized-Alonized I.D. of a 5/8-inch x 0.035-inch Wall
316 SS Tube

Figure 4-4. Photomicrograph of Platinized-Alonized 316 SS Tube.

The design of the SCR incorporated a spiral wrap around the inner tube to maintain concentricity and to aid in heat transfer.

A wire wrapped on a one-inch pitch around the inner tube served this purpose. A roll of 0.094-inch diameter 304 SS wire was purchased and swaged to a diameter of 0.088 inches (the annular gap is \sim 0.090 inches).

To fabricate the spiral, a ten foot length of wire was wrapped on a 1/2-inch pitch around a 7/16-inch diameter mandrel. The spiral was then stretched to the desired one-inch pitch.

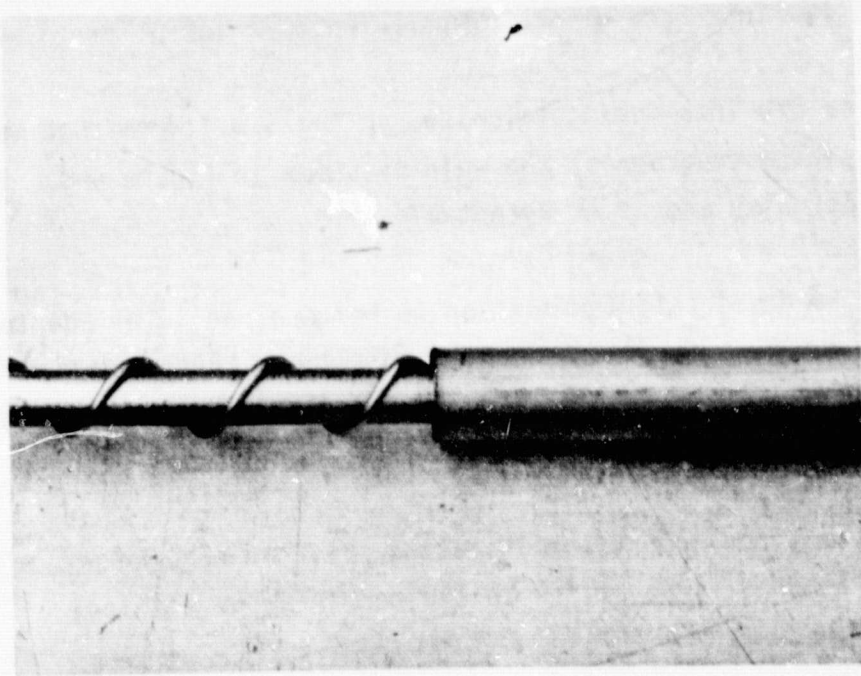
Next, the 3/8-inch tube was inserted into the spiral. One end of the spiral was then welded to the tube, the spiral stretched until it was intimate with the tube, then the loose end of the spiral was welded to the tube. Sections of spiral and tube were assembled until the desired length was attained.

On the first attempt to insert the wrapped assembly into the 5/8-inch tube, the spiral wrap severely scraped the inside of the 5/8-inch tube. This interference fit was not permissible since any abrasive action between the spiral and the outer tube would remove catalyst from the I.D. of the outer tube.

To provide additional clearance, the wire was swaged to a diameter of \sim 0.081 inches. In the swaging process, the 304 SS wire became work hardened and had to be annealed.

A second attempt to insert a wrapped 3/8-inch tube into the 5/8-inch was successful. A small, approximately six feet long, module of bare 316 SS was then built. Figure 4-5 shows the wrapped inner tube being inserted into the outer tube.

A thirty-three foot bare 316 SS module was then built and coiled. (A description of the coiling procedure and apparatus is detailed in the following section.) Construction of the bare module involved welding the necessary lengths of inner and outer tubes together to achieve the desired length.



**ORIGINAL PAGE IS
OF POOR QUALITY**

Figure 4-5. Photograph of Spiralled Assembly
Being Inserted Into 5/8" Tube

Upon successful construction of a bare 316 SS module the next step was to build an alonized (but non-platinized) module.

To build this alonized module, it was necessary to develop a technique for removing the alonized surfaces at the weld joints. If the alonizing is not removed, residual aluminum-alumina would vaporize during welding and be entrapped in the welded region. The entrapped porosity would reduce the integrity of the weld. To insure the quality of the weld, the alonized surface was machined off. Figure 4-6 shows an alonized surface before and after weld preparation. Figure 4-7 illustrates the interface of the prepared weld region prior to welding.

Two pieces of 5/8-inch tubing (alonized on the I.D.) were then machined and welded. A photomicrograph of the weld is shown in Figure 4-8. This photo shows no sign of porosity and is an acceptable weld.

In addition to developing fabrication techniques, an inlet adapter and an end plug of the module were designed under this task. Both items were machined from 316 SS. Photographs of the inlet adapter and the end plug are shown in Figures 4-9 and 4-10.

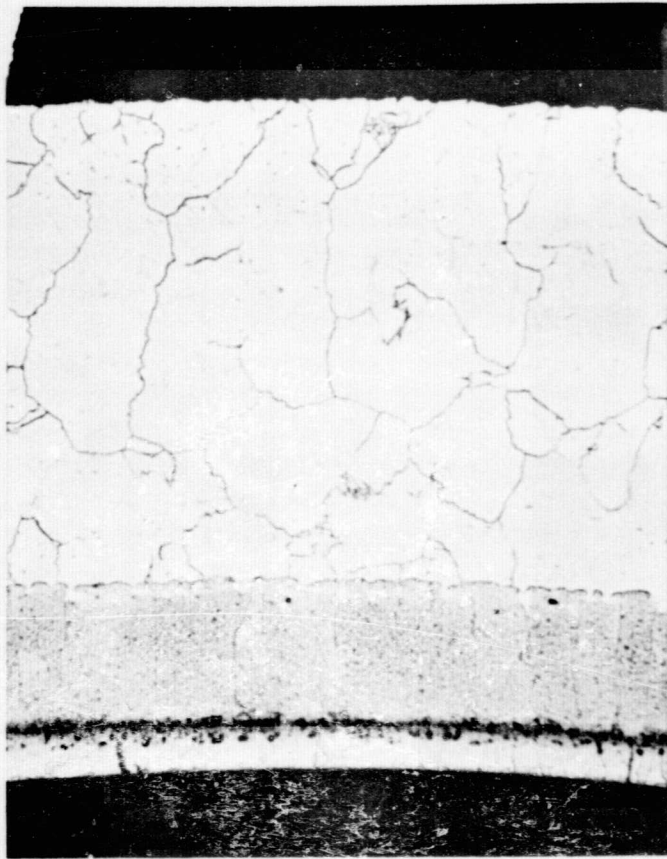
The end plug was designed to be installed prior to coiling while the inlet adapter was planned to be welded to the module after coiling.

The inlet adapter was installed by slipping it over the end of the module until it contacted the outer tube. This joint was then sealed by welding. The inner tube extends through the adapter and out the opposite end. A 316 SS collar was fitted between the tube and adapter. The joints were then welded.

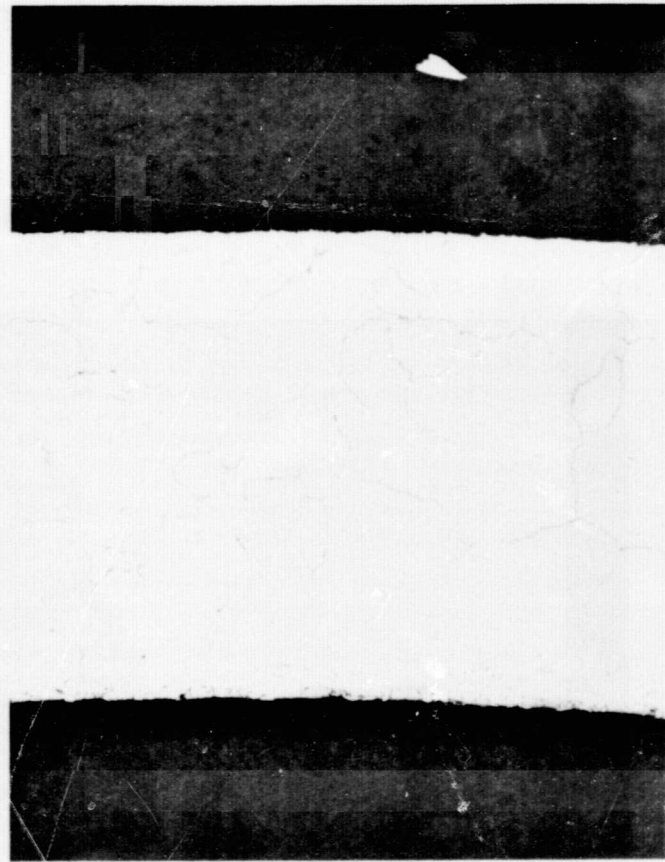
The inlet gas was delivered through the opening in the top of the T-shaped adapter and flows through the annular gap. The return gas flows through the inside of the inner tube.

The end plug was simply inserted into the end of the outer tube (top photo Figure 4-9) and butt welded.

4-13



100x
Before Weld prep



100x
After Weld Prep

Figure 4-6. Samples of Alonized 316 SS Tubes before and After Weld Prep



100x

Figure 4-7. Interface of Prepared Weld Region



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Figure 4-8. Photomicrograph of Welded 5/8" 316 SS Tubing Alonized ID Only

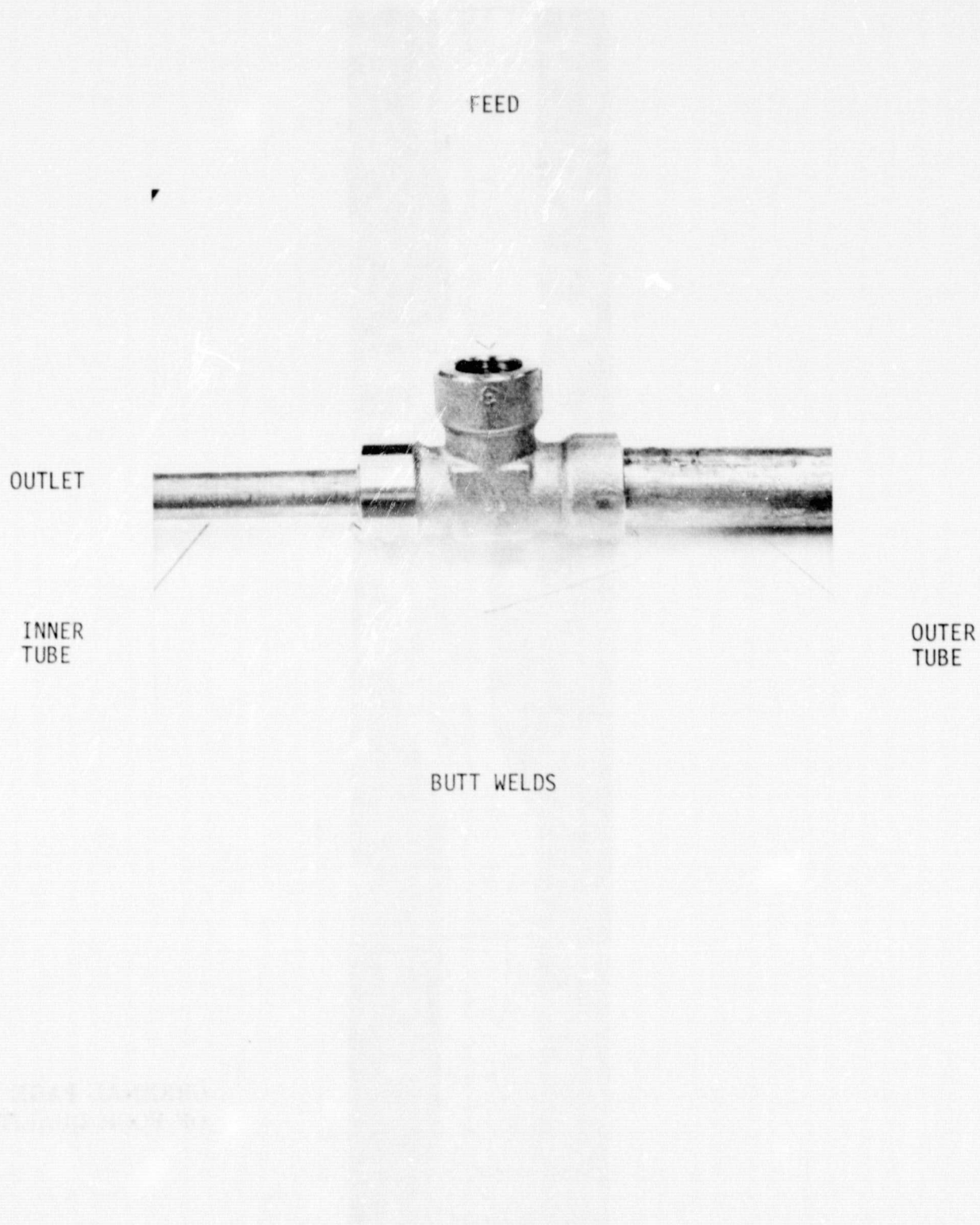


Figure 4-9. SCR Module Inlet Adapter.

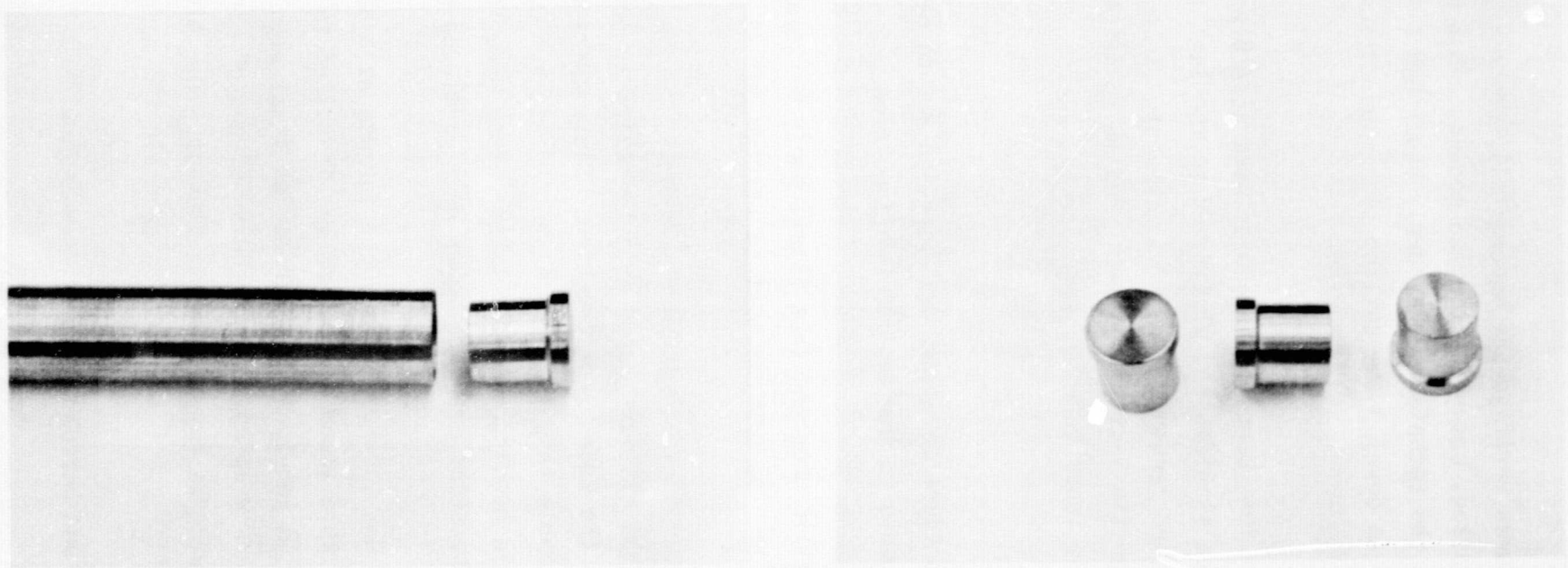


Figure 4-10. End Plugs for SCR Module.

With the assembly procedures verified and successfully demonstrated, the next step was to build an alonized, but nonplatinized module. This mockup module was successfully built using the techniques previously described. Platinum was not used because of the need to develop the coiling technique (described in the following section).

Additionally, the two ten foot bench test modules were assembled. Module #1 consisted of an inner tube alonized and platinized on the last three feet of its O.D. and alonized the entire length of its I.D. The outer tube was alonized and platinized on its I.D. for the last three feet. Module #2 differed only in that the inside diameter of the inner tube was bare 316 SS.

4.4 COILING

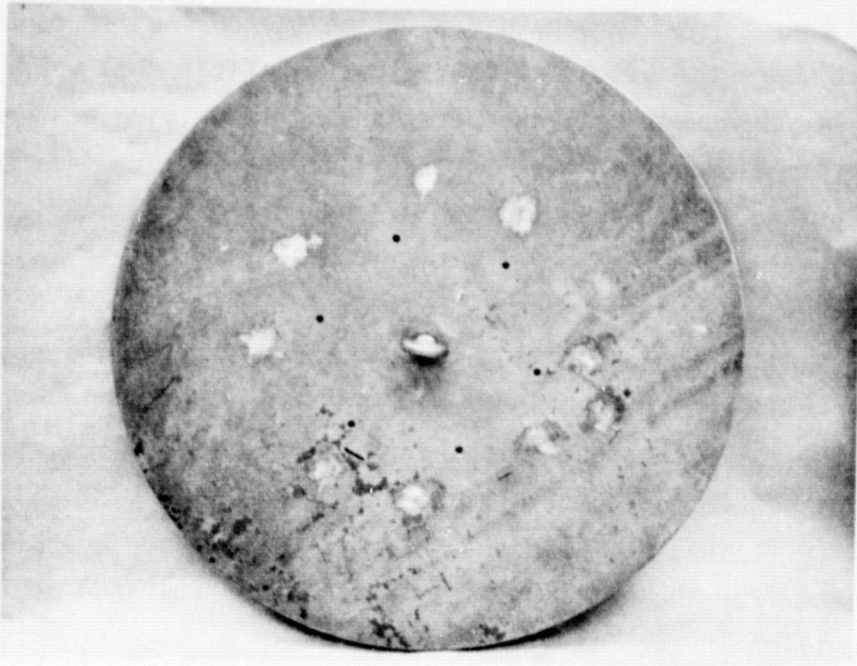
The final fabrication procedure developed and verified was to demonstrate the coiling of an alonized module to the desired configuration.

Alonizing is an intermetallic compound that is extremely brittle. Therefore, to prevent cracking, the bending process must be carried out at elevated temperatures.

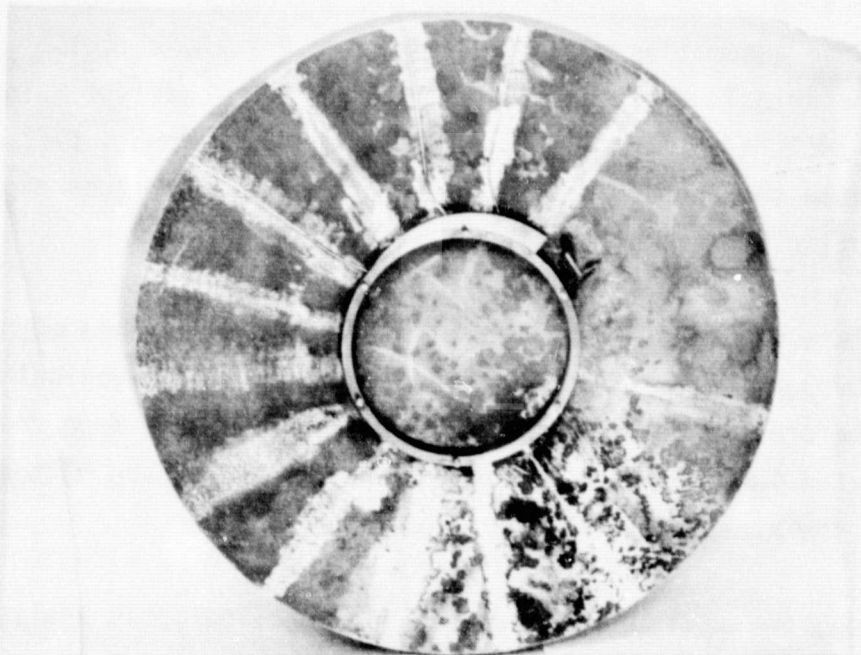
Alon, Inc. reported that the alonized tubes must be heated to greater than 1100°F (593°C) to prevent the tubes from cracking. Since there were no on-site facilities available to handle this type operation, an attempt was made to find a vendor with experience in bending alonized tubes. None were found. Therefore, a bending apparatus and the necessary techniques were developed at AESD.

A motorized welding table was located and adapted for use as a base for the bending apparatus. A variable speed controller was installed to control the table's rotational speed. A ten-inch diameter carbon steel pipe was used as the base on which the module was wrapped. Two circular pieces of carbon steel plate (~30-inch diameter) were used as top and bottom platons. The platons were spaced approximately one inch apart and used to maintain the module's flatness.

Figure 4-11 shows the top and bottom platons.



TOP PLATE



TAPERED
SHOE

HOLDER

BOTTOM PLATE

Figure 4-11. Photographs of Coiling Apparatus.

A bare 316 SS module was initially assembled and coiled. The coiling operation was carried out at room temperature. During the coiling procedure a kink occurred at the point where the second wrap overlaps the first coil wrap. The kink was eliminated in subsequent coiling operations by installing a shoe that tapered from the inside diameter of the first coil to the inside diameter of the second coil (see Figure 4-11). A photograph of the bare 316 SS module is presented in Figure 4-12.

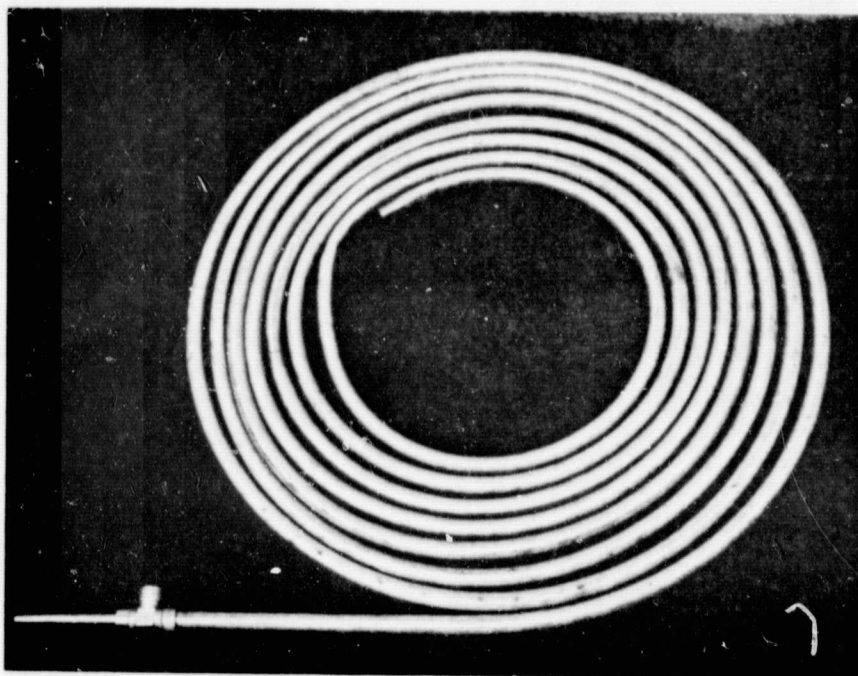
To coil the module, the end of the straight assembly was inserted into a holder (see Figure 4-11) and the welding table slowly and uniformly rotated. The tubing was fed as the table rotated. After the assembly was coiled, the top plate was removed. The module did not exhibit any stress in the vertical direction and maintained its flatness. The module did, however, produce a bit of tension as it was removed from the bottom plate. The module "unwound" approximately 90° from its position in the fixture.

The next step in this task was to bend an alonized module. Prior to bending, three hot bend tests were conducted to determine the minimum bend temperature at which cracking in the alonized layer did not occur. Several short (~1-1/2 ft.) concentric tube assemblies were built and used for these tests. The assemblies simulated the actual module except for the platinum catalyst. They were bent, over a five-inch radius, at 1100°, 1250°, and 1400°F (593°, 677°, and 760°C). The tests simulated the bending radius, temperature, and stresses experienced by the innermost coil of the module.

The temperature was measured at the inside of the inner tube with a thermocouple extending through the inner tube. The tubes were heated beginning approximately six inches before the bend begins up to the point of the bend. Two acetylene torches, located 180° apart, impinge on the outside surface of the outer tube and provide the necessary heat.

After bending, the samples were removed for metallographic analysis.

Figure 4-13 is an example of the type sample taken from each bend test. The outside radius of both the inner and outer tubes were examined for stress cracking.

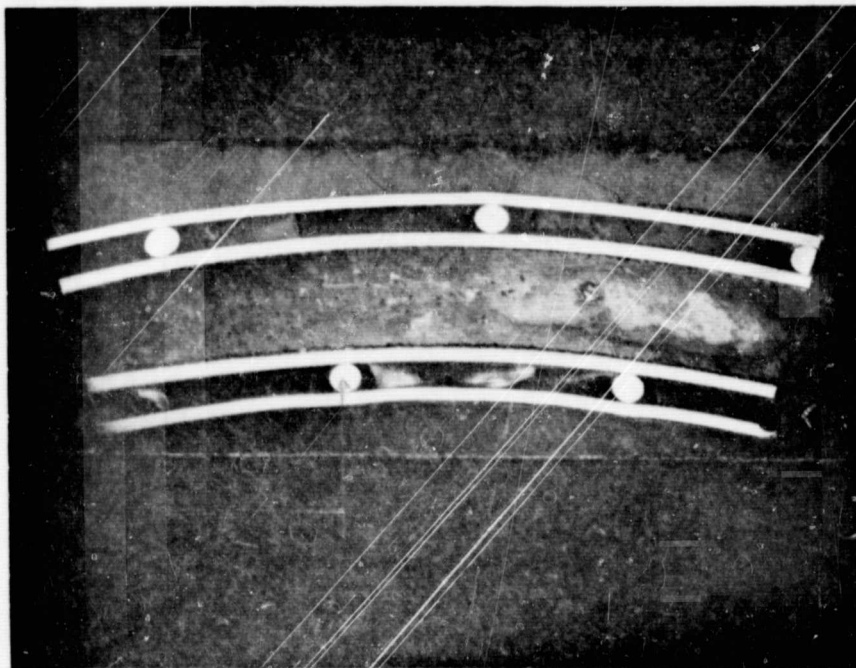


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Figure 4-12. Bare 316 SS Module.

OUTSIDE
RADIUS

SPIRAL
WRAP



INSIDE
RADIUS

Figure 4-13. Photograph of Bend Test Specimen.

Figures 4-14, 4-15, and 4-16 are photomicrographs of the bend tests at 1100°, 1250°, and 1400°F.

All of the bent tubes displayed some degree of cracking in the alonized outside surface of the inner tube. Higher magnification (than shown in Figures 4-14, 4-15, and 4-16) revealed that the cracks in the 1100°F sample propagate into the base metal. In comparison, the cracks in the 1250° and 1400°F samples terminate in the alonized region. The cracks that terminated in the alonized region were not considered to be a problem. Further analysis revealed that there were no cracks in the inside surface of the outer tube on any of the three samples.

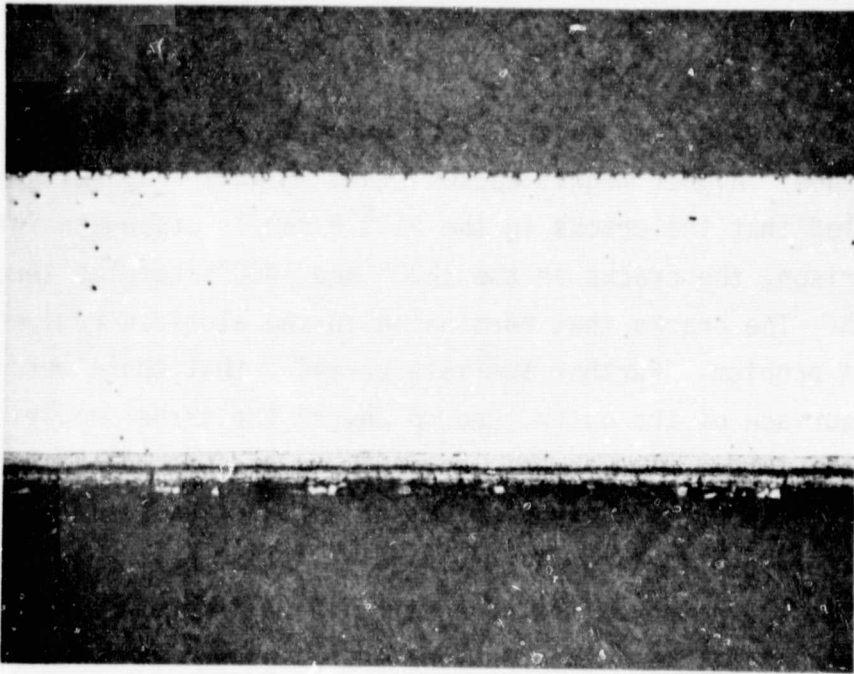
The inside surface of the inner tube experienced stress cracking in the 1100°F test but not at the 1250°F or 1400°F bend temperatures. The presence of base metal in the return tube may catalyze the reverse reaction, therefore 1100°F is not sufficient to safely bend alonized tubing. Since there was no difference in the 1250°F sample and the 1400°F sample, 1250°F was selected as the bending temperature.

A thirty-three foot alonized module was then coiled at a temperature >1250°F as measured at the inside of the inner tube. A check of the outside surface temperature of the outer tube showed it was ~200°F hotter than the inside temperature.

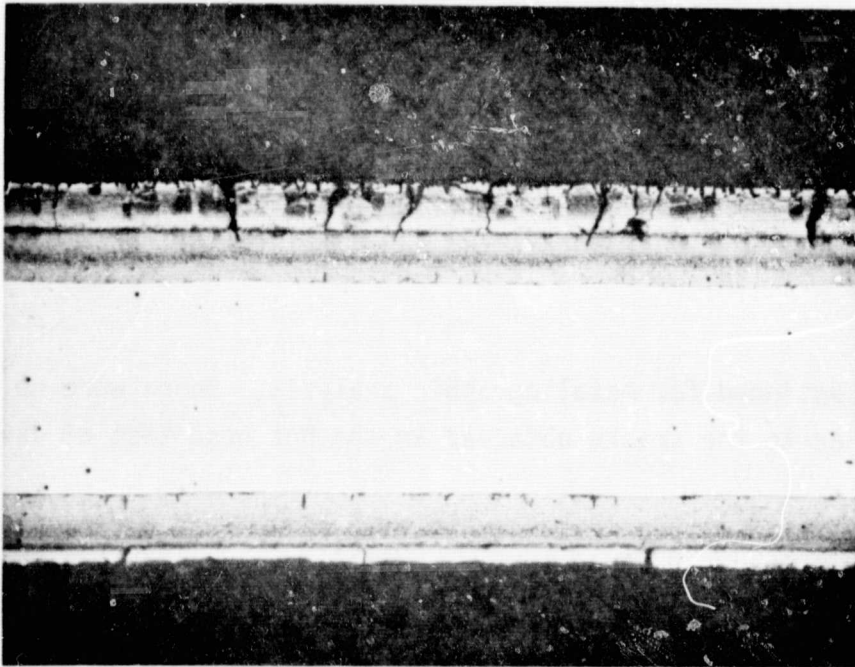
The coiling operation proceeded smoothly and no problems were encountered. After coiling X-rays of the module revealed that the concentricity of the two tubes was maintained.

The module was then sectioned for metallographic analysis. There were no cracks other than ones similar to the cracks observed in the hot bend test at 1250°F.

It was then decided to proceed and build a prototype Solar Chemical Receiver Module. All the techniques and procedures developed in the various fabrication verification tasks were integrated to produce a platinized-alonized module.

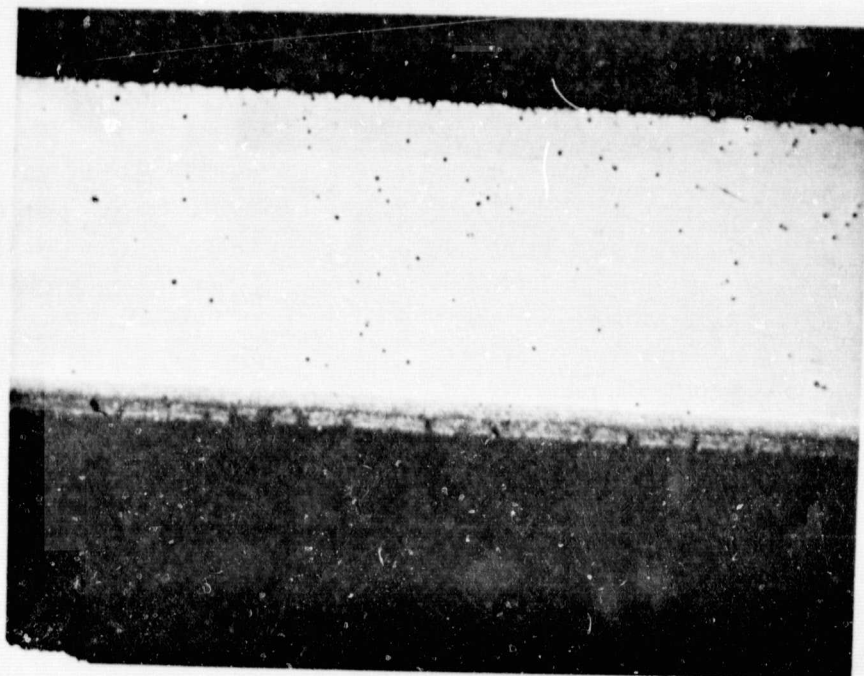


OUTER TUBE,
OUTSIDE
RADIUS

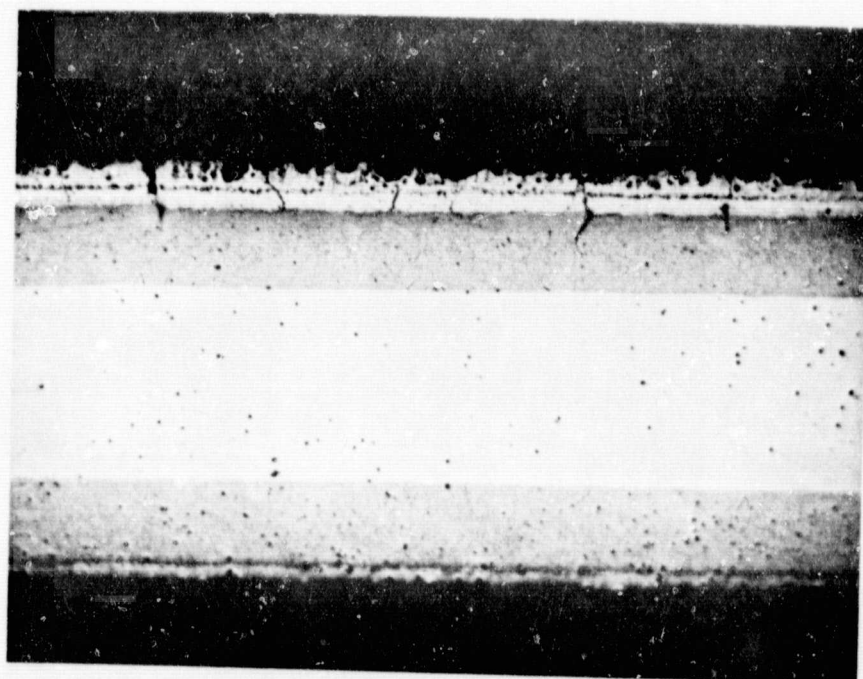


INNER TUBE,
OUTSIDE
RADIUS

Figure 4-14. Photomicrograph of Bend Test Sample, 1150°F. (50 X)

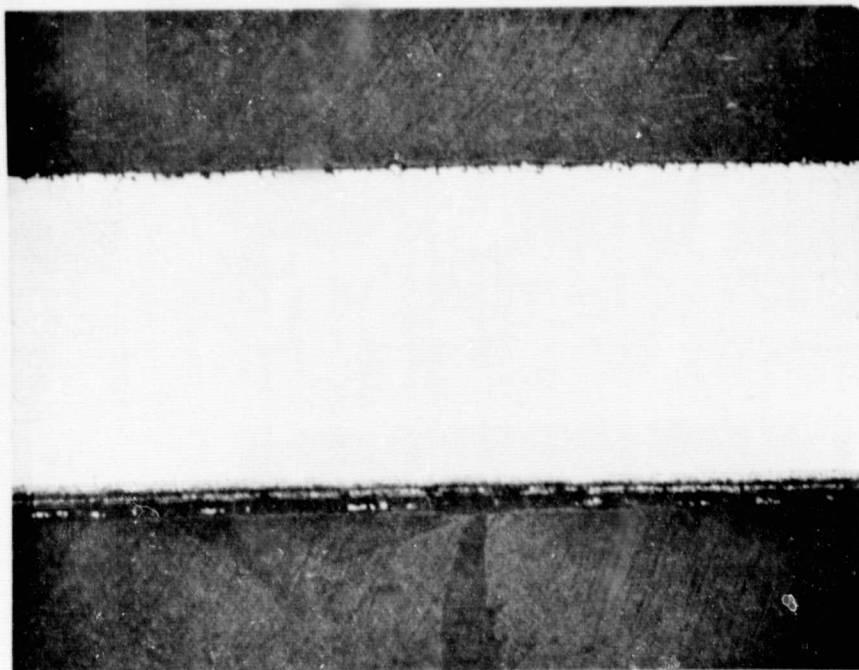


OUTER TUBE
OUTSIDE
RADIUS

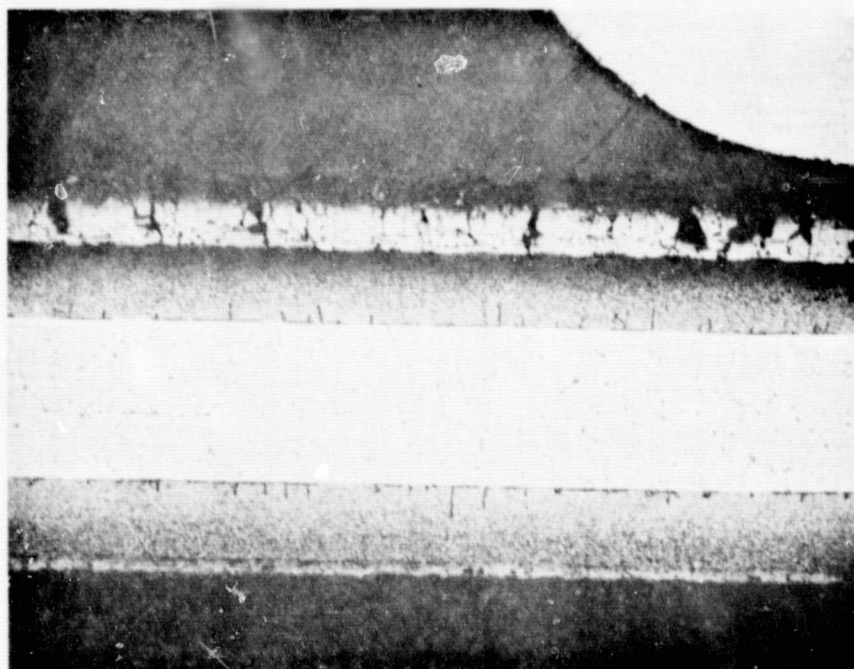


INNER TUBE
OUTSIDE
RADIUS

Figure 4-15. Photomicrograph of Bend Test Sample, 1250°F. (50 X)



OUTER TUBE
OUTSIDE
RADIUS



NOTE: SPIRAL
WRAP

INNER TUBE
OUTSIDE
RADIUS

Figure 4-16. Photomicrograph of Bend Test Sample, 1400°F.
(50 X)

The module consisted of two thirty-three foot concentric tubes. The outer tube (5/8-inch O.D. x 0.035-inch wall) was platinized and alonized on the last three feet of its inside surface. The inner tube (3/8-inch O.D. x 0.035-inch wall) was platinized and alonized on its outer surface's last three feet while its inner surface was alonized for the final ten feet.

No problems were encountered during the assembly.

The assembled module was then installed in the coiling apparatus and the coiling process initiated. Approximately halfway through the first wrap, the outer tube suffered catastrophic cracking. The cracks penetrated through the outer wall of the outer tube, exposing the spiral wrapped inner tube. The cracking is shown in Figure 4-17.

Metallographic analysis of the ruptured section revealed that the outside surface of the outer tube experienced sensitization.

Sensitization, which is a time-temperature dependent phenomena, occurs when stainless steel is slowly cooled down from high temperatures. In a temperature range of 538°-816°C, the carbon reacts with the chrome (both are present in stainless steel) to form chrome carbide. The chrome carbide forms in the grain boundary and reduces the ductility of the stainless steel. A large grain size was also observed indicating that the tubing had been held at elevated temperature for an extended time period during the alonizing process. Metallographic examination revealed that in some locations the wall thickness was composed of only two or three grains.

The outer tube experienced several excursions through the sensitization temperature range:

- 1) during the high temperature alonizing process;
- 2) twice during the high temperature catalyst placement procedure; and
- 3) once in the coiling operation.

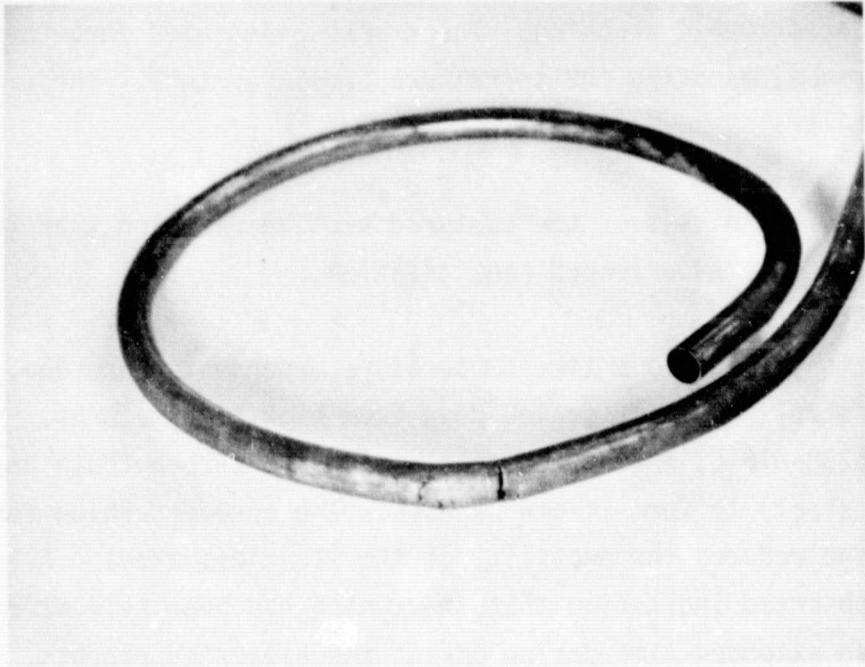


Figure 4-17. Alonized-Platinized Module (1000°C).

Since the alonizing temperatures and bending temperatures are unavoidable, the catalyst placement procedure was examined to determine if the temperatures and/or exposure time could be reduced.

In the platinizing procedure, the tubes were gradually heated to prevent blistering due to rapid decomposition of the organic binder and then allowed to slow cool in the furnace. The tubes were exposed in the sensitization temperature range for an extended time period.

In order to determine if minimizing the sensitization exposure would be beneficial, a second outer tube was painted with the platinum ink and heated as before to $>1000^{\circ}\text{C}$. After reaching peak temperature, the tube was air quenched by removing it from the furnace and allowing it to cool rapidly. This procedure significantly reduced exposure time in the sensitization temperature range from hours to minutes.

The tube was assembled and coiled using the previously developed hot bending procedure. The outer tube again failed in the same manner; i.e., intergranular cracking.

It appears that the high temperature required to decompose the platinum ink aggravated the sensitization problem. Reexamination of the successfully coiled alonized but nonplatinized module revealed minute cracks in the outer surface of the outer tube. Since the heat treatment associated with the platinizing process appears to worsen the effects of sensitization, the possibility of modifying the platinizing process was explored. The platinum ink supplier was contacted to determine if the platinum could be deposited at a lower temperature. According to Englehard Industries, the organic binder in the ink decomposes at $300\text{-}350^{\circ}\text{C}$. However, to insure a good bond between the alonized substrate and the platinum, higher temperatures are required.

Two samples of alonized 316 SS were painted with the platinum ink and fired at 350° and 550°C .

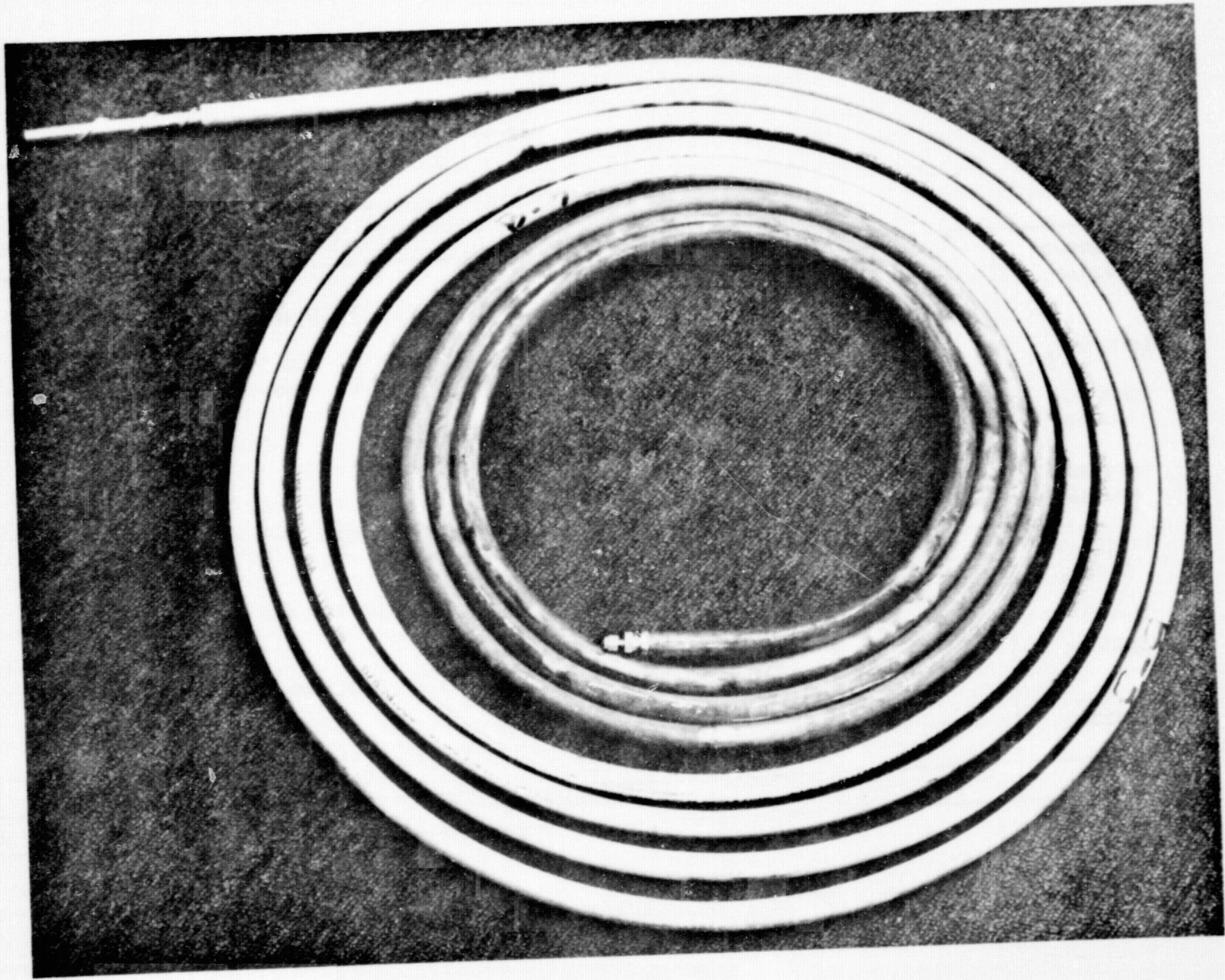
The platinum did not bond to the substrate at 350°C as it was easily removed during normal handling operations. The 550°C sample had a slightly better adhesion although it, too, could be removed with little effort. In addition, the alonized tube platinized at 550°C also cracked during the bending operation.

The sensitization problem produced by the alonizing process can be averted by use of stabilized stainless steel such as 318 or 347 or a low carbon stainless steel such as LC 304 or LC 316. Use of these materials should be explored during future investigations.

In an effort to manufacture a complete Solar Receiver Module and circumvent the cracking problems, a modified module was constructed. This back up module consisted of a bare 316 SS outer tube and an alonized, platinized inner tube. The module shown in Figure 4-18, was successfully coiled. There were no visible cracks in the outside surface of the outer tube.

The module was built as an alternate to the base module to demonstrate the concept of the SCR. It contained approximately 30-40% of the catalyst present in the base module. However, the reduced catalyst loading may or may not have an adverse effect on the receiver's performance. The actual performance was not measured, as the testing was terminated due to operating problems as discussed in the following section.

Table 4-1 presents a listing of all the modules built and coiled and their degree of success.



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Figure 4-18. Photograph of Alternate Solar Chemical Receiver Module.

TABLE 4-1: SCR MODULE FABRICATION

TEST	DESCRIPTION	RESULT
1	Bare 316 SS	Successful
2	Alonized as per base design*	Successful
3	Alonized, platinized (1000°C) as per base design, furnace cooled	Severe cracking
4	Alonized, platinized (1000°C) as per base design, air cooled	Severe cracking
5	Alonized, platinized (1000°C) as per base design, air cooled, new furnace	Severe cracking
6	Bare outer tube, inner tube alonized, platinized (1000°C) as per base design, air cooled	Successful
7	Alonized, platinized (530°C) as per base design, air cooled	Severe cracking

Base Design: Outer Tube: O.D. Bare 316 SS
I.D. Alonized, platinized 3 ft

Inner Tube: O.D. Alonized, Platinized 3 ft
I.D. Alonized 10 ft

5.0 BENCH TESTS

Two primary and two supplemental bench tests were planned. The SCR Bench Test Outline is presented in Table 5-1.

Bench Test 1, the baseline design, was to characterize the module performance while Bench Test 2 was to verify the results of Test 1 and to measure the extent of the back reaction. Test 1 consists of a ten-foot straight concentric tube module with all three wall surfaces in contact with the gas stream (i.e., outer tube I.D., inner tube O.D. and I.D.) alonized. The outer tube I.D. and inner tube O.D. were also platinum coated. Test 2 was to be identical to Test 1 except that the inner tube I.D. was bare 316 SS. Comparisons between these two tests would indicate the extent of the back reaction without alonizing and would also indicate the consistency of performance between individual modules.

However, due to unexpected auxiliary equipment failure, neither of the tests were completed. Test 1 was carried out using 96 w/o H_2SO_4 rather than SO_3 and had to be terminated due to a system malfunction before steady state conditions were achieved.

Two supplemental bench tests were also planned (see Table 5-1). These tests were considered subordinate to the primary tests. Test S1 was completed within the overall schedule while Test S2 was eliminated. Test S1 was the direct measurement of SO_2 oxidation in a bare 316 SS tube.

In addition, the test plan called for a functional performance test of the prototype module. This test was to be conducted under design requirements. Due to the test system failure, this test had to also be cancelled.

In the following sections, a description of the Bench Test Facility (Section 5.1) and a discussion of the Bench Test Results (Section 5.2) are presented.

TABLE 5-1: SOLAR CHEMICAL RECEIVER BENCH TEST OUTLINE

TEST NUMBER	PURPOSE	MODULE DESCRIPTION	FEED COMPOSITION	FLOW RATE (lb/hr)	POWER (watts)
1	Characterize Module Performance	10' Straight Concentric Module, All Alonized	SO ₃ , SO ₂ , O ₂ (Equil. at 500° C)	2.5, 5.0, 10.0	200, 400
2	Verify Test 1 Performance Measure Back Reaction	10' Straight Concentric Module, Return Tube Bare	SO ₃ , SO ₂ , O ₂ (Equil. at 500° C)	2.5, 5.0, 10.0	200, 400
SUPPLEMENTAL TESTS					
S1	Measure Bare Tube Back Reaction Rate	3/8" OD, 316 SS Tube, 3' Long	SO ₂ - 67% Vol. O ₂ - 33% Vol.	5.0	T=500 to 900° C
S2	Determine Reaction Rate Constant	3/8" OD, 316 SS Tube, Inside Surface Alonized With Pt	SO ₃ , SO ₂ , O ₂ (Variable)	2-10	T=700 to 1000° C

5.1 BENCH TEST APPARATUS DESCRIPTION

The Solar Chemical Receiver Bench Test Facility was designed to accommodate a ten foot straight, bench test module and, with slight modification, the thirty-three foot coiled module.

A schematic diagram of the Straight Tube Bench Test Facility is presented in Figure 5-1 and the Coiled Bench Test Facility in Figure 5-2. A photograph of the Bench Test Facility, with a ten-foot module installed, is presented in Figure 5-3.

The hazards associated with anhydrous SO_3 required the installation of a high velocity ventilation hood and glove box (see Figure 5-3).

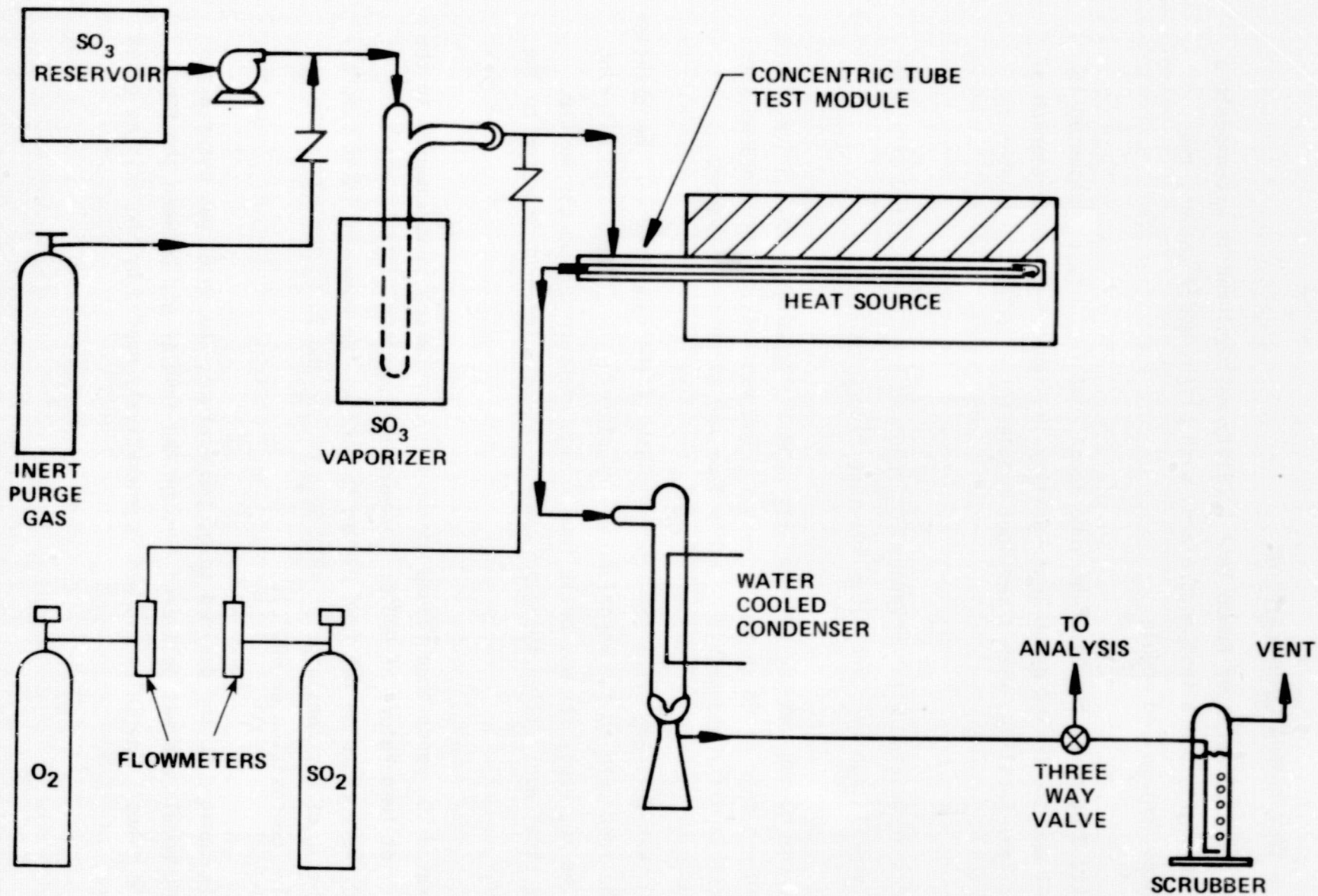
The glove box housed the SO_3 storage flask and SO_3 transfer pump. A dry, inert atmosphere was maintained in the glove box. The ventilation hood covered the entire test facility and provided safety from any escaping SO_3 vapors.

Liquid SO_3 was delivered from the storage flask to the vaporizer via the transfer pump. The feed rate to the vaporizer was regulated with a calibrated metering valve. The SO_3 vaporizer was built from a high pressure 304 SS gas bottle. Stainless steel wool filled the vaporizer to increase the heat transfer to the liquid SO_3 .

The saturated SO_3 vapor then flowed to the preheater where it was heated to the desired inlet temperature of 500°C . Immediately upstream of the preheater, SO_2 and O_2 were added to the feed stream to make up the required inlet composition. The inlet gas consisted of $\sim 91\%$ SO_3 , 6% SO_2 and 3% O_2 (which is the equilibrium composition at 500°C).

After being heated to the desired inlet temperature, the inlet gas entered the bench test module and was delivered through the annular gap between the two concentric tubes. The inlet gas was regeneratively heated by the exiting gases to $\sim 725^\circ\text{C}$.

5-4



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Figure 5-1. Solar Chemical Receiver Straight Tube Bench Test Facility.

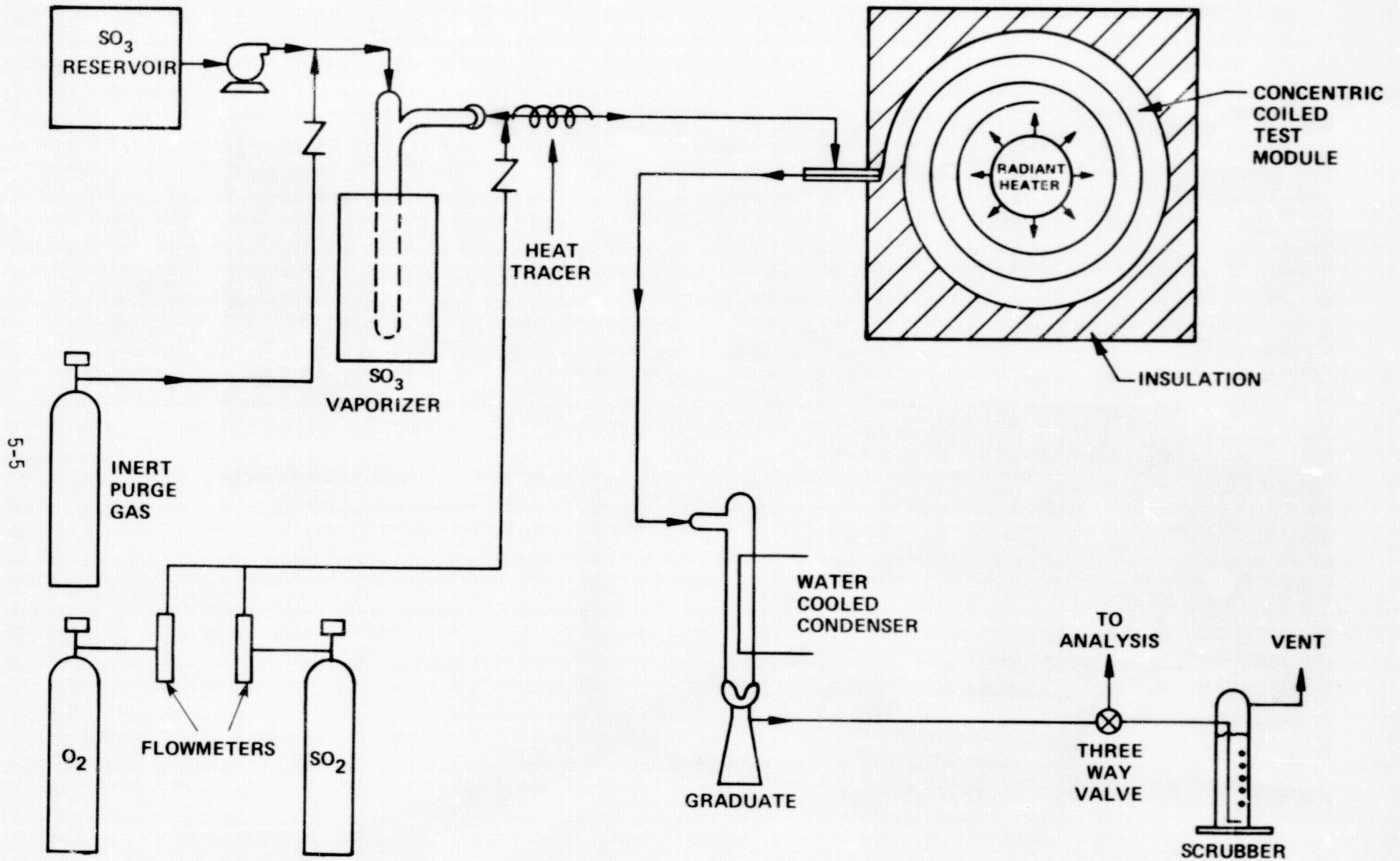


Figure 5-2. Solar Chemical Receiver Coiled Module Test Facility.

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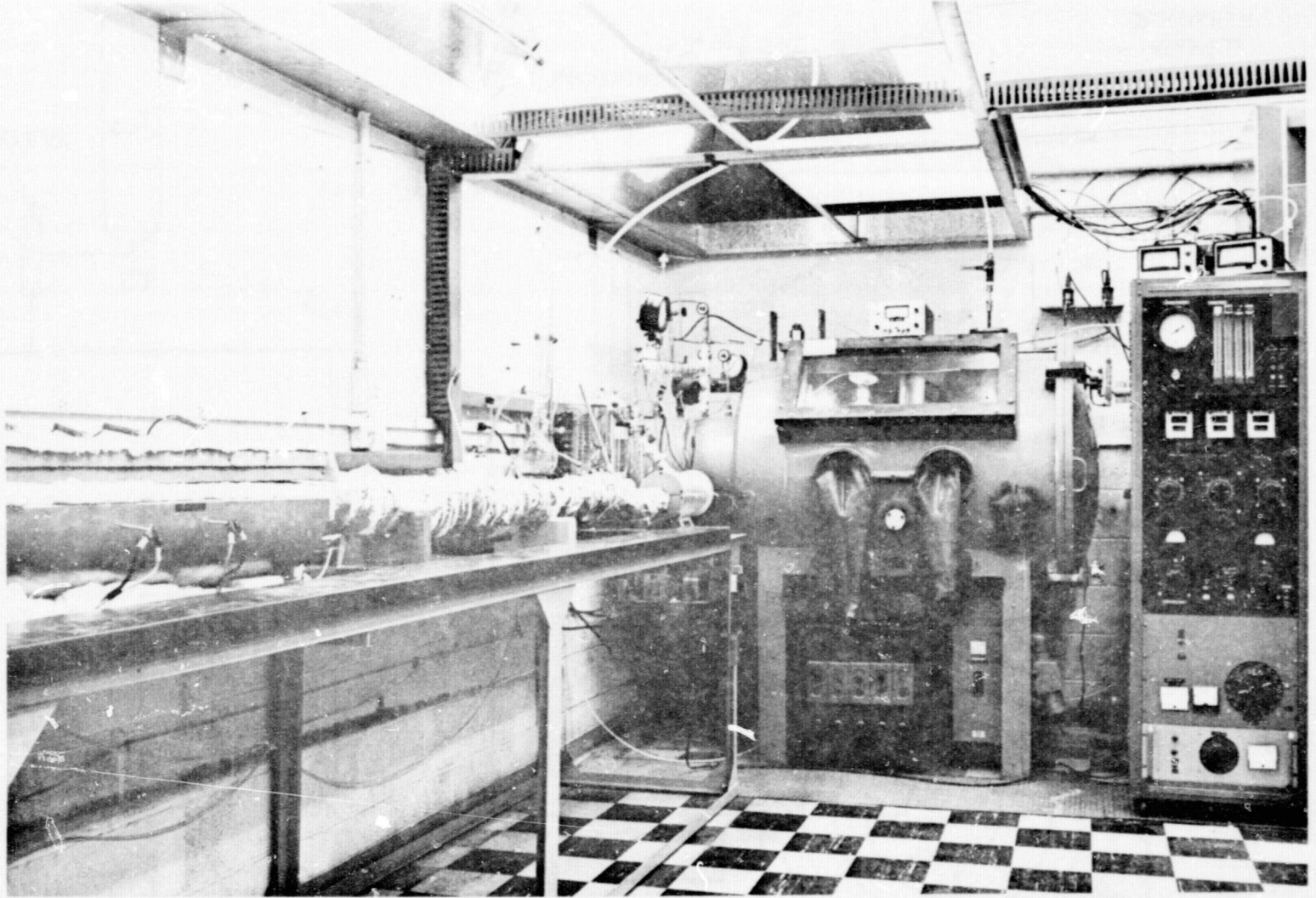


Figure 5-3. Photograph of the SCR Bench Test Facility.

The heat exchanger portion (the first seven feet of the bench test module) was well insulated and the outside surface temperature measured every twelve inches.

The reaction zone region (the final three feet) was circumferentially heated on its bottom half. This was done using one-half of a split tube furnace.

Top and bottom tube surface temperatures were measured every six inches in this region. One thermocouple was located on the bottom (furnace) half of the tube and the other 180° opposite on the top (insulation) half. Additionally, an internal thermocouple positioned near the turn-around point measured the gas temperature.

The reaction zone furnace consisted of three separately controlled heating zones. This insured that each zone operated at the same power level or temperature.

The product gas, SO_2 , O_2 , and unreacted SO_3 , returned through the inside of the inner tube. This stream gave up its sensible heat to the cooler incoming feed gas. The product gas exited the module and was cooled, condensing out the unreacted SO_3 . The remaining gases, SO_2 and O_2 , were either analyzed or scrubbed and vented. Analysis was done using a DuPont 400 Photometric SO_2 Analyzer.

Alternately, the product stream could also be analyzed at the turn-around point (the end of the reaction zone).

The entire test apparatus was thoroughly instrumented with thermocouples, temperature controllers and alarms, power controllers, etc. Internal temperatures (i.e., gas temperatures) were measured inside the SO_3 vaporizer, before and after the preheater, at the end of the reaction zone, and at the end of the module. The surface temperatures were measured at numerous points throughout the system. A data acquisition system provided a means to rapidly and continuously record all pertinent information. System pressure was monitored, controlled, and recorded manually.

The entire delivery system was constructed of 316 SS tubing and heat traced to prevent the saturated SO_3 vapor from condensing. All valves, fittings, etc. were 316 SS. The analytical train was built of Teflon and quartz to prevent the back reaction.

After an initial system debugging, the SO_3 metering valve was calibrated. The initially installed 316 SS valve failed to produce consistent results. Therefore, this valve was removed and a Teflon metering valve installed. Calibration was carried out using 96 w/o H_2SO_4 rather than SO_3 due to safety considerations. The viscosity and density of SO_3 and H_2SO_4 are relatively equal and a correction factor was developed to adjust the obtained values for SO_3 flow rate. The calibration measured flow rate as a function of pressure drop across the valve at various valve settings. This provided an accurate means of measuring the flow of the feed stream.

The next step in evaluating the test facility was to conduct a thermal performance checkout. The reaction zone furnace, vaporizer, preheater, etc. were all operated under design requirements. Argon was used as the working fluid for all the checkout runs.

During the checkout runs, the gas temperature at the turn-around point was between 400-500°C, well below the ~800°C designed operating level. Additional

gas, contrary to the design requirements. The inlet gas, entering the module at 500°C, was losing a significant amount of heat in the heat exchanger portion of the module. A temperature profile of the outside surface of the heat exchanger revealed that there was substantial heat loss. As a result, the inlet gas entering the reaction zone furnace was much cooler than desired. This reaction zone furnace then reheated the gas back to ~400-500°C.

To reduce the heat loss from the gas to the outside surface, the heat exchanger section was heat traced and reinsulated.

In the next series of thermal performance checks the heat tapes were controlled at ~500°C.

This modification increased the turn-around gas temperature to $\sim 740^{\circ}\text{C}$. Since the system, for this particular run, had not reached thermal equilibrium and the temperature was still increasing, the facility's thermal performance was determined to be satisfactory.

The thermal performance evaluation tests also served to provide the operators with hands-on experience in running the system. All the necessary procedures for startup, operating, and shutdown were established and reviewed and approved by a division Safety Committee representative. The procedures were implemented and verified during the thermal performance tests. Also reviewed and satisfactorily conducted were exercises in shutting down the system in a worst-case situation (a ruptured SO_3 delivery line).

The final step in checking out the bench test facility was to do a two-point calibration of the metering valve with SO_3 . The valve was initially calibrated with 96 w/o H_2SO_4 and a correction factor developed to compensate for the slight difference in viscosity. This two-point analysis was carried out to verify the correction factor.

After one point was verified (two actual tests) the SO_3 transfer pump failed. An in-house backup pump was installed, and it also failed. Since the delivery time for a new pump was 10-12 weeks, an alternate method of delivering the SO_3 had to be found. An attempt was made to pressurize the SO_3 storage flask. This attempt failed as the storage flask leaked upon pressurizing.

It was then decided to substitute 96 w/o H_2SO_4 for SO_3 in the bench tests. Pumps compatible with H_2SO_4 were on hand and ready for immediate use.

A discussion of the bench test results is given in the following section.

To convert the test facility from straight tube testing to coiled module testing, the reaction zone furnace had to be removed.

An insulated container, holding the coiled module, would then be installed. The inlet adapter, etc., is identical to the straight tube module; therefore, the

delivery system and analysis train remain unchanged. Heat is supplied to the coiled module with a radiant heater. The test apparatus modification for prototype module testing was not made due to the termination of the test program.

5.2 BENCH TEST RESULTS

5.2.1 SUPPLEMENTAL BENCH TEST 1

The objective of Test S1 was to determine the catalytic activity of 316 SS on the oxidation of SO_2 (the "back reaction");



This test was conducted in a simple apparatus consisting of a 3/8-inch O.D. 316 SS tube centered in a split tube furnace. SO_2 and O_2 were delivered to the tube and the gas composition measured before entering and after exiting the split tube furnace. The analysis equipment and delivery system were the systems used (and described) in the Bench Test Facility.

By comparing the % SO_2 and the gas flow rate of the inlet and outlet streams, a direct measurement of SO_2 oxidation was made. This, in turn, reflected the catalytic activity of the bare 316 SS tube.

The inlet stream consisted of 67% SO_2 and 33% O_2 . The mass flow rate was 0.22 lb/hr, corresponding to a volumetric flow of ~500 scc/min SO_2 and 250 scc/min O_2 . Outside surface temperatures were measured every three inches in the 12-inch split tube furnace. The furnace control thermocouple was centered in the furnace.

For this particular test, the temperature profile was:

TC #1 - 813°C - inlet surface temperature

TC #2 - 889°C

TC #3 - 904°C - control thermocouple

TC #4 - 866°C

TC #5 - 654°C - outlet surface temperature

The measured inlet gas was ~458 scc/min SO₂ (67% total) and ~225 scc/min O₂ (33% of total). The outlet composition flow rate was 638 cc/min with a 67% SO₂ content. To calculate the conversion of SO₂ to SO₃, the following equation was used:

$$\% \text{ conversion} = \left(\frac{\text{SO}_2 \text{ flow in} - \text{SO}_2 \text{ flow out}}{\text{SO}_2 \text{ flow in}} \right) \times 100 \quad (5-2)$$

Since the analysis is done at room temperature and pressure for both inlet and outlet streams, no temperature or pressure corrections were necessary.

Therefore, using the above data and Eq. 5-2, the calculated conversion was:

$$\begin{aligned} \% \text{ conversion} &= \left(\frac{458 - 428}{458} \right) \times 100 \\ &= 6.7\% \end{aligned}$$

A second test, conducted several hours after the first, produced a conversion rate of ~4.5%.

In both tests, the generated SO₃ was not completely condensed out of the outlet gas sample. The characteristic white cloud associated with SO₃ vapor was observed in the outlet sample.

The averaged conversion rate of ~5.6% must be considered in the context of the experiment. These tests were designed to be qualitative and to indicate the tendency for back reaction. The operating parameters (temperature, pressure, and flow rates) and the residence time were not identical to those experienced in the prototype SCR module. With these points considered, it appeared that the alonizing on the inside surface of the inner tube may not be necessary, since the actual extent of back reaction can be expected to be considerably less than that achieved during these tests. However, before a final conclusion is made additional testing should be conducted. It had been planned that these additional tests would be part of the bench tests (by comparing Bench Test #1 and Bench Test #2).

The purpose of Test S2 was to measure the SO₃ reaction rate constant for wall-deposited platinum. This is more theoretical in nature, and actually was intended to improve the proposed system mathematical model.

Since the major emphasis of the project was directed to fabricating a coiled module, Test S2 was eliminated because of insufficient time.

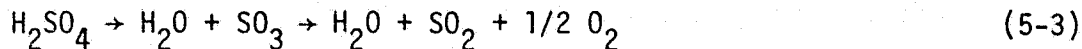
5.2.2 BENCH TEST RESULTS

The bench test outline shown in Table 5-1 called for two primary bench tests, each consisting of a number of runs at varying mass flow rates and thermal inputs. Bench test #1 evaluated a straight 10-foot baseline design module (platinized and alonized on 3 ft of the inner tube O.D. and outer tube I.D. and an alonized inner tube I.D.) while bench test #2 examined a similar module except that the inner tube I.D. was bare 316 SS. A comparison of these two tests would give an indication of the extent of the back reaction and verify individual module performance.

Bench test module #1 was installed in the bench test facility and used in the system checkout as previously described.

During the final calibration of the system, a critical component failed (see Section 5.1) and prevented the SO₃ from being delivered to the module. This impacted on the type of bench test that could be safely carried out. Consequently, it was decided to conduct the bench tests substituting concentrated sulfuric acid for anhydrous SO₃.

For the two reactions:



the equilibrium conversion for Eq. 5-3 is higher than that of Eq. 5-4. Therefore, slightly higher conversions to SO₂ were expected when using H₂SO₄ in place of SO₃. Although the absolute conversion rate would be higher with

H_2SO_4 , it was expected that the percent of equilibrium achieved would be relatively equal. Concentrated H_2SO_4 is ~45 mole percent compared to 100 mole percent SO_3 . The equilibrium conversion for H_2SO_4 is higher therefore due to the presence of inerts (water).

The design requirements specified a feed composed of the equilibrium composition at 500°C; 91.5% SO_3 , 5.6% SO_2 , and 2.8% O_2 (all mole percent). The concentrated H_2SO_4 provided a feed of ~45 mole percent SO_2 or approximately half the specified concentration.

Although the molar flow rate of SO_3 could not be equaled, the total mass flow rate of 5 lbs/hr was. This translated to a volumetric flow of ~20 cc/min liquid sulfuric acid.

The bench test was started by setting the module furnace to the specified thermal inputs and the vaporizer, etc., to their required temperatures. An argon flow was maintained during the primary heatup. After the furnaces reached thermal equilibrium, the argon flow was terminated and the sulfuric acid feed initiated at ~20 cc/min.

At this point in the run the module furnace was cycling at ~900°C (as measured on the outside surface on the furnace half of the module), the turn-around gas temperature (argon) was ~630°C and the system pressure ~30 psig.

Upon adding the sulfuric acid, the turn-around gas temperature rapidly increased to ~665°C.

The sulfuric entered the module at ~507°C and exited at ~520°C. Acid vapor exited the module and entered the water-cooled condenser. The condenser proved to be inadequate as acid vapor was evident in the lines to the SO_2 analyzer. Some unreacted liquid sulfuric was condensed out, however,

After running approximately 15 minutes, the turn-around gas temperature increased to 670°C, inlet temperature to ~500°C and the outlet temperature ~497°C.

The first sign of trouble appeared when the system pressure began to decrease rapidly. The acid feed was checked and flowing normally. In an attempt to increase the pressure, the outlet throttling valve was slightly closed. (This valve was incorporated into the design to maintain the system pressure). This did not increase the system pressure. Additionally, there were no visual signs (i.e., acid vapor) of any leaks in the system.

Approximately 21 minutes into the test, the system pressure gauge ruptured, releasing acid vapor into the laboratory. Emergency shut down procedures were invoked, terminating the run.

At shut down, the gas turn-around temperature was $\sim 673^{\circ}\text{C}$ and steadily increasing. Inlet gas temperature was 515°C , outlet 498°C . The module furnace was cycling at $\sim 890^{\circ}\text{C}$.

An analysis of the collected unreacted sulfuric acid produced a conversion rate of $\sim 34\%$. This conversion rate was calculated by titrating (with 1N NaOH) the feed acid and the condensed unreacted sulfuric acid. The difference in concentrations was used to calculate the conversion rate by:

$$\% \text{ conversion} = \frac{[\text{conc. of feed}] - [\text{conc. of condensate}]}{[\text{conc. of feed}]} \quad (5-5)$$

This calculation was an approximate conversion rate averaged for the entire 20 minute run. The unreacted acid that was not condensed would have most likely decreased the condensate concentration, resulting in an increase in the conversion rate.

The equilibrium conversion rate was difficult to determine since the system was in a transient state. If an average temperature of 670°C is assumed, the equilibrium conversion (at 3 atm) is $\sim 40\%$. However, due to the malfunctioning pressure gauge, the actual pressure may have been higher. However, it did not exceed 55 psig as a safety relief valve designed to open at this pressure did not activate. The valve was checked after the test and found to be operating normally.

Assuming that the equilibrium conversion was ~40%, the observed conversion rate of 34% represents an efficiency of ~85%.

An attempt was made to calculate a mass balance on the system, however, due to the vapor that was released to the atmosphere, this was impossible.

After the system was returned to a safe condition, the ruptured pressure gauge was removed and inspected. The gauge was found to be mislabeled. According to the information received, the entire gauge was 304 SS, however, upon inspection it was found that the bourden tube was brass. The solder joint between the bourden tube and the socket, not compatible with the acid environment, corroded and ruptured. The earlier observed decrease in system pressure was an indication that the gauge had failed and therefore the system pressure was suspect.

The ruptured gauge was an auxiliary piece of equipment, separate from the bench test module. A visual inspection of the module revealed that it was not damaged.

As a result of gauge failure, the second bench test and the functional performance tests were cancelled.

6.0 CONCLUSIONS

Based on the design analysis, it is concluded that a solar chemical receiver utilizing the endothermic decomposition of SO_3 can meet or exceed all of the functional requirements set forth by JPL. It is further concluded that the SCR should utilize a protective Alonized coating in the reaction zone. Wall-deposited platinum is considered the preferred catalyst.

Special fabrication procedures are required to manufacture and assembly the SCR. Several of these procedures have been developed and are discussed in this report. Coiling of an alonized-platinized module is the critical manufacturing task. Existing bending methods were unsuccessful when the alonized 316 SS was subjected to further thermal cycling during catalyst placement. A suggestion of alternate tubing materials to prevent "sensitization" has been made.

Experimental results with a bench-scale SCR were inconclusive. Because of a transfer pump failure, no tests were conducted with anhydrous SO_3 as had been planned. An abbreviated 20 minute test with 96% sulfuric acid was unable to reach steady-state, but the limited results were encouraging. The wall-deposited platinum catalyst appears to be an effective catalyst for SO_3 decomposition under the proposed operating conditions.

7.0 RECOMMENDATIONS

Although the experimental portion of this work was terminated before all of the proposed tests could be conducted, the theoretical analysis suggests that all of the desired functional performance goals can be met. It is therefore recommended that further experimental verification of the actual SCR performance be conducted. It is also recommended that an evaluation of alternate reactor materials be made in order to prevent sensitization during Alonizing and catalyst placement.

Upon successful demonstration of SCR performance and physical integrity (i.e., no cracking), a complete solar chemical receiver, with several manifolded SCR modules, should be designed and fabricated.

8.0 NEW TECHNOLOGY

No reportable "New Technology" items have been identified during the course of this contract.

9.0 REFERENCES

1. J. R. Wertz, C. E. Wicks, R. E. Wilson, Fundamentals of Momentum, Heat and Mass Transfer, John Wiley and Sons, Inc., New York, N.Y., 1969.
2. Westinghouse Electric Corporation, "Solar Thermal Hydrogen Production Process," Annual Technical Progress Report, DOE Contract ET-78-C-02-47051 December, 1978.
3. Octave Levenspiel, Chemical Reactor Engineering, John Wiley and Sons, Inc., New York, N.Y., 1972.
4. W. H. McAdams, Heat Transmission, McGraw-Hill Book Co., New York, 1954.
5. R. A. Seban and A. Hunsbedt, "Friction and Heat Transfer in the Swirl Flow of Water in an Annular," Int. Journal of Heat Transfer, Vol. 16, 1973.
6. Y. Mori and W. Nakayama, "Study on Forced Convective Heat Transfer in Curved Pipes," (1st Report, Laminar Region), Int. Journal of Heat Transfer, Vol. 8, 1965.
7. Y. Mori and W. Nakayama, "Study on Forced Convective Heat Transfer in Curved Pipes," (2nd Report-Turbulent Region), Int. Journal of Heat Transfer, Vol. 10, 1967.
8. A. L. Edwards, "TRUMP: A Complete Code for Transient and Steady-State Temperature Distributions in Multidimensional Systems," Lawrence Livermore Laboratory, September, 1972.

END

DATE

JUL. 10, 1981