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DESIGN OF GA THERMOCHEMICAL WATER-SPLITTING PROCESS FOR THE MIRROR ADVANCED REACTOR SYSTEM

by
L. C. BROWN

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FOREWORD

GA interfaced the sulfur-iodine thermochemical water-splitting cycle to the Mirror Advanced Reactor System (MARS). The results of this effort follow as one section and part of a second section to be included in the MARS final report. Section 5 describes the process and its interface to the reactor. Partial Section 7 gives the capital and operating costs for the hydrogen plant.

SECTION 5.0
INTERFACING THE GA CYCLE

Contributor:
L. Brown

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5.0 INTERFACING THE GA CYCLE

A viable fusion-powered thermochemical synfuels economy will only become reality through close matching of the thermal output of the fusion reactor to load demands of the thermochemical process. Likewise, thermal energy sources within the chemical process must be matched to heat demands within the chemical process, and to heat requirements of the power production systems or power bottoming cycles.

We have proceeded from the integrated TMR driven synfuels plant developed two years ago (1) and shown in Figure 5.0-1 to develop an even deeper understanding of the factors governing the economic match of the fusion reactor to the synfuels plant. Last (32) year major modifications were made to the sulfuric acid processing sections, and economies were realized through applying energy conservation techniques to the HI purification section. Further optimization of the sulfuric acid section has resulted in this improved design. Details of the preliminary design of each process unit in the chemical plant are discussed. The design description included consideration of heat and mass flow, equipment sizes, and safety, and is based on chemical processes that have been demonstrated by laboratory experimentation.

5.1 Base Chemical Process

The GA water-splitting cycle(2,3) may be described by the four equations:

Figure 5.0-1 Overall Process Flow Diagram for the TMR/Synfuels Hydrogen Production Plant, 1981 Version

These equations represent three chemical reactions and one separations process. In these equations the species HI_x represents an aqueous complex of HI and I_2 formed by reaction 1. The H_2SO_4 product of reaction 1 is obtained as a moderately concentrated aqueous solution which is immiscible with HI_x .

The overall process is divided into five sections for design purposes. Sections I through IV roughly correspond to the four equations and section V represents the heat and energy transmission equipment required to match the fusion heat source to the chemical process. Descriptions of sections I through IV occupy the remainder of section 9 of this report. Section V is described in report section 6.

The developmental effort on the GA Sulfur-Iodine cycle has proceeded since 1974 along three parallel and mutually supportive lines:

- o Chemical investigations
- o Engineering flowsheet development
- o Process demonstration

Chemical investigations are the basis for the engineering flowsheet development and process demonstration. Synergistically, the flowsheet development and process demonstration efforts give impetus and direction to further chemical investigations. Likewise, each of these tasks raises new questions in its own area.

5.2 Recent Developments in Process Chemistry

The GA water-splitting process is not a static entity but is constantly being upgraded. Information has recently been made available on two chemical innovations to the process^(2,4).

These techniques have been developed to the preliminary flowsheet stage under separate funding. HI decomposition via homogeneous catalysis⁽²⁾ will eventually be incorporated into the fusion synfuels design, but since HI decomposition is relatively inexpensive there is little urgency. The Sulfur-

Sulfuric Acid Chemical Energy Storage system⁽⁴⁾, developed for solar applications, has the potential for long-term energy storage. Long-term storage could benefit a fusion synfuel plant by permitting the synfuel plant to maintain operation during scheduled and nonscheduled outing of the TMR. In addition, this system could provide on-site power for cold start-up of the TMR.

Neither of these process improvements have been included in this design. Discussion of this design resumes with section 5.3

5.2.1 HI Decomposition Via Homogeneous Catalysis

The present version of the sulfur-iodine water-splitting process employs HI decomposition in the liquid phase. Theoretical calculations indicate that high conversion levels are possible using such a scheme, and indeed high conversions were verified experimentally⁽¹⁰⁾. In addition, the same article reports that the magnitude of extrapolated rate data to high process temperatures appears sufficient to result in a viable process for HI decomposition. The above rate data were obtained by extrapolating measured rate data from lower temperatures and pressures in batch studies performed on supported platinum and ruthenium catalysts.

Recently, some data for a supported platinum catalyst at closer to actual engineering temperatures and pressures were obtained in a new flowing liquid HI bench-scale system. The rate value obtained gave credence to the use of the extrapolated lower temperature data and thus is verification of the present flowsheet and equipment sizing calculations.

Further in-depth studies, however, revealed a problem associated with the use of heterogeneous catalysis to decompose liquid HI. This is associated with the finding that at least platinum, whether supported or not, does dissolve in the liquid HI to a non-negligible degree. This requires the use of some kind of catalyst recovery and remanufacture scheme. Catalyst recovery and remanufacture are commonplace in the chemical industry but add to the

complexity and to the operational and capital costs of the overall process. It was due to the discovery of such an additional requirement of the process that some consideration was given to the use of homogeneous catalysis to decompose HI and to potential separation schemes inherent in the practical application of homogeneous catalysis.

A homogeneous catalyst concept has been devised⁽²⁾ in which there is an innate separation of the catalyst from the HI liquid, thereby allowing the catalyst to be totally recycled to the reactor. The concept is based on some unique findings surrounding the phase behavior of HI-I₂-H₂O mixtures. For certain compositions of these three chemicals, two liquid phases in equilibrium can exist. One phase is a very dry phase of HI and I₂; the other is an aqueous phase containing both I₂ and HI. It has been found that it is in this latter phase that certain homogeneous catalyst compounds tend to concentrate. This means that the aqueous phase acts essentially as the catalyst carrier and can be recycled to the HI decomposition reactor without having to recover the catalyst.

The potential advantages over a process employing heterogeneous catalysts with the need to recover and remanufacture 100% of the catalyst are clear. The proposed homogeneous process has been conceptually flowsheeted (but at too late a date for inclusion in this overall process design) and major areas requiring investigation have been identified. The needed information consists mainly in identifying the solubility and distribution of the catalyst in the fluids of the process train at engineering conditions, determining the best area of the HI-I₂-H₂O phase diagram in which to work, and determining the best schemes for catalyst recycle. The results of this work have supported this concept as a viable engineering process.

5.2.2 The Sulfur-Sulfuric Acid Chemical Energy Storage System

A sulfur-sulfuric acid energy storage system has been proposed for the GA sulfur-iodine water-splitting cycle for use in conjunction with a solar heat source⁽⁴⁾. This storage system has the potential for reducing the cost of fusion produced synfuels. During steady state operation of the TMR, a small

amount of sulfuric acid could be converted to sulfur. During outages of the TMR, the sulfur could then be burned in air providing both the feed stock and energy required to operate the hydrogen plant. Potentially, the system could provide sufficient energy for the cold start of the TMR after an outage.

Conversion of sulfuric acid to sulfur is a two step process. The first step is already included in the water-splitting process, Equation 5.2. Formation of sulfur from sulfur dioxide is then accomplished by the disproportionation reaction.

Kinetics of this reaction have been studied⁽¹¹⁾ at temperatures between 100° and 140°C. The observed reaction rates are compatible with expected holding times for the S and dilute H₂SO₄ products.

Thermodynamically, data from the JANAF Tables⁽¹²⁾ and Giauque⁽¹³⁾ suggest that the H₂SO₄ product can be made at up to 57% H₂SO₄, which is the same concentration as the sulfur-iodine cycle sulfuric acid product. This reaction, then, provides a method of upgrading an SO₂ product into a much more valuable S product at the expense of cycling two-thirds of the SO₂ back as H₂SO₄ solution. The stored energy in the S is recovered by combustion (in air) to SO₂, and the produced SO₂ is used in the SO₂-H₂O-I₂ reaction to make recycle H₂SO₄ and HI, which is the source of the H₂ in the cycle.

The SO₂ disproportionation makes a low-grade heat which is utilized in the cycle (for instance, for dehydrating H₂SO₄), while the burning of the S makes the very high-grade heat which drives the hydrogen producing plant both electrically and thermally to produce H₂SO₄ and H₂ from SO₂. This storage cycle has inefficiencies as do all energy storage schemes; however, it provides high energy storage density in low cost, easily stored chemicals and easily supplies large quantities of high-grade heat.

5.3 Plant Design Status

The present plant design is based on the most recent integrated flowsheet. The flowsheets for the main reaction (section I), HI purification (section III), and HI decomposition (section IV) are the same as for previous reports(1,32).

The sulfuric acid processing section (section I.) was revised to match the thermal energy available from the MARS blanket. The primary emphasis was upon increasing thermal energy recovery of the sulfuric acid concentration step while maintaining a good thermal match to the blanket heat source. At this level of preliminary design the size of the process equipment is determined sufficient to development equipment costs. The design calculations make use of standard chemical engineering correlations for the sizing calculations. When the required physical and transport properties have been unavailable they have been approximated.

5.4 Main Solution Reaction Step (Section I)

5.4.1 Design Considerations

The main solution reaction step, Section I of the process, comprises all equipment associated with the chemical reaction:

where gaseous sulfur dioxide reacts with an excess of molten iodine and water to produce two immiscible liquid phases. The designation L_1 indicates that water is the major component in the H_2SO_4 product phase, and L_2 that iodine is the major component in the HI product phase. The H_2SO_4 phase which is the lighter (lower density) phase, also contains a trace of iodine. The heavy phase, referred to as HI_x , because it contains excess iodine in a complexed form, also contains water. The main solution reaction is exothermic and Section I requires no heat input from the fusion reactor but exports a

significant quantity of low-grade heat to a power bottoming cycle. The Section I process is shown schematically in Figure 5.4-1.

Even though Section I requires no heat input from the fusion reactor, the design of the main solution reaction step has a major influence on the overall process efficiency and thus upon the reactor size and the hydrogen production cost. Although decisions on heat and power recovery within Section I have an effect on efficiency, the composition and temperature of the light phase flowing to process Section II (the H_2SO_4 processing step) and of the heavy phase flowing to Section III (the HI concentration step) strongly influence the overall efficiency of the process. In the interest of efficiency, both streams should leave Section I at as high a temperature as possible and at the highest acid concentration possible. Since the equilibrium of an exothermic reaction shifts towards the reactants as temperature is raised, a compromise must be made between high temperature and high acid concentrations in the product. In deciding the operating conditions for Section I some economic trade-off calculations were made but a full-scale process optimization was not in the scope of this work. The basis for the resulting decisions, as well as those for the rest of this study, may best be termed "engineering judgment." When trade-off calculations were not definitive, the decision was usually made on the side of "high efficiency" not "low capital cost."

Consideration of two factors dominates the design of Section I: (1) the large quantity of heat produced by the main solution reaction must be removed, and (2) the combination of hydrogen iodide and iodine in the heavy aqueous phase is extremely corrosive to materials normally employed for heat transfer surfaces. The only metals known to resist HI_x are refractory metals such as niobium, tantalum, and molybdenum. The present design employs niobium for heat transfer surfaces contacting HI_x , which permits the reaction energy to be used in the power bottoming cycle.

Niobium is relatively expensive: therefore, to reduce the capital cost of Section I, a number of design techniques have been employed which improve heat transfer and decrease the amount of Nb required. Some of these are:

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Figure 5.4-1 Schematic of Section I, Reaction of SO_2 with Iodine and Water to give H_2SO_4 and HI_x as Immiscible Liquid Products

1. Cool feed streams to Section I. Prior to carrying out the main solution reaction, we transfer the heat from the Section I feed streams to the power bottoming cycle. This involves less expensive heat transfer materials than Nb and reduces the amount of heat transferred from the reaction products of the main solution reaction.
2. Operate adiabatically. Instead of cooling the reaction products from 393 to 368 K (to shift the equilibrium towards the products) and then reheating the separated products, we operate adiabatically, and maintain the desired production rate of the main solution reaction by increasing the iodine concentration to provide the needed shift in the equilibrium.
3. Use direct-contact heat exchange. Where possible, we employ direct contact heat exchange between immiscible liquid phases or gas and liquid phases.
4. Apply enhanced heat transfer techniques. Utilizing two-phase gas-liquid flow through the heat exchanger gives higher than normal convective heat transfer coefficients. Spiral-fluted tubing provides enhanced heat transfer and, due to the wall stiffening effect, permits thinner tube walls.

5.4.2 Heat Exchanger Reactor

Almost 52% of the chemical reaction forming HI and H_2SO_4 takes place in the Heat Exchanger Reactor (R-101 in the flow diagram of Figure 5.4-1) and in the piping immediately preceeding the reactor. The reactor selected is a shell and tube heat exchanger with fixed tube sheets. The vessel, tube sheets, and heads are fabricated from mild steel. The heads are lined with fluorocarbon and the spiral-fluted niobium tubes are welded to a niobium tube sheet liner. The process fluid is on the tube side of the heat exchanger and the shell side is part of the power bottoming cycle.

Upstream of the heat exchanger, the SO_2/O_2 from Section II (sulfuric acid processing) is mixed with the predominantly iodine stream from the Boost Reactor (C-103) and aqueous streams from the scrubbing reactors (C-101 and C-104). The combined streams react exothermically as they flow through a fluorocarbon-lined pipe and into the heat exchanging reactor. The pressure drop through the heat exchanger reduces the system pressure from an initial 0.50 MPa to 0.44 MPa at which point the temperature reaches 393 K. At the exit of the heat exchanger the O_2 is removed from the stream in the first of two separators (S-101). In the second separator (S-102), operating at 0.11 MPa, three phases separate: the heavy liquid phase (HI_x), the light liquid phase ($\text{H}_2\text{O}/\text{H}_2\text{SO}_4$), and a gas phase consisting of SO_2 and steam. The vaporization of water and SO_2 results in the temperature decreasing from 393 K to 385 K.

5.4.3 Lower Phase SO_2 Stripper

The lower phase solution (HI_x) is saturated with SO_2 which when allowed to remain forms sulfur and H_2S via tramp reactions. An oxygen recycle stream strips most of the SO_2 in a packed bed stripper (C-102) thus minimizing the tramp reactions. A minimum amount of oxygen is employed for this operation as the evaporation of water into the oxygen cools the HI_x requiring more heat input in Section III (HI purification). With a 10% O_2 recycle the HI_x is cooled from 385 K to 381 K.

The stripper, sized for operation at 70% of flooding, is a standard packed column design. The fluorocarbon-lined mild steel vessel is packed with 50 mm ceramic Raschig rings. Since the operating pressure is near atmospheric, a glass-lined steel vessel is a viable option but would require fluorocarbon packing to avoid liner damage.

5.4.4 Boost Reactor

In the boost reaction, the sulfuric acid concentration of the light phase is increased from 50% to 57% by contacting with molten iodine in the presence of sulfur dioxide. The increased H_2SO_4 concentration is realized through the action of the main solution reaction. Water is used up by reaction with SO_2

and I_2 to form H_2SO_4 and HI . Over 7% of the total chemical reaction of Section I occurs in the Boost Reactor. Since the contact is performed in a counter-current manner, the reactor also acts as a direct contact heat exchanger, raising the temperature of the sulfuric acid stream from 383 K to 393 K.

Although the mechanical design of the Boost Reactor is straightforward, i.e., a fluorocarbon-lined mild steel vessel packed with 50 mm ceramic Raschig rings, the sizing calculations are not. Common packed columns operate with either a gas rising through a descending liquid phase, or a light liquid phase rising through a descending heavy liquid phase. The Boost Reactor has both a gas phase (SO_2 in O_2) and a light liquid phase (H_2SO_4 and water) rising through the descending heavy liquid phase (I_2). The present design is based on adding the cross-sectional areas required if the gas and light liquid separately contacted the heavy liquid. This is a very conservative approach. We believe that a design based upon information gained in a pilot plant would result in a smaller Boost Reactor vessel.

5.4.5 Scrubbing Reactors

The oxygen is purified before discharge to the atmosphere in the Scrubbing Reactors. The packed column reactors operate in a titration mode in which sufficient iodine is added to the scrub water in the lower part of the column to react stoichiometrically with the sulfur dioxide present in the oxygen. In the upper part of the column the oxygen is washed with pure water. The primary Scrubbing Reactor (C-101) operates at 0.44 MPa purifying the gaseous product of the Heat Exchanger Reactor (R-101). Over 19% of the Section I reaction takes place in the primary Scrubbing Reactor. Almost 22% of the total reaction takes place in the secondary Scrubbing Reactor at 0.10 MPa. The secondary scrubber cleans up the oxygen stream exiting the Boost Reactor as well as the steam/ SO_2 stream produced during depressurization of the liquid reaction product of the Heat Exchanger Reactor.

Both scrubbing reactors perform a second function as direct contact heat exchangers. Oxygen leaving the process is cooled to near ambient conditions while preheating the water entering the process. The scrubber vessels are of

fluorocarbon-lined mild steel and the packing is 50 mm ceramic Raschig rings. Pilot plant tests may demonstrate reduced vessel costs. The upper portions of the scrubber contain only water and oxygen so no lining should be required in this area. Depending upon the temperatures reached in the lower portions of the scrubber, less expensive linings may be possible.

5.4.6 Heat and Power Recovery

The heat transferred to the power bottoming cycle from Section I totals 802 MW_t. The majority of this is transferred via the Heat Exchanger Reactor but significant quantities of heat are also transferred from the hot water products of Sections II and III and from the SO₂/O₂ product of Section II. Since the heat transfer materials used for water and SO₂ are much less expensive than the niobium used in the Heat Exchanger Reactor, there is the potential for further cost reduction by performing more of the cooling on water and SO₂ streams. Ultimately an economic optimization must determine the split in heat transfer duties on the basis of minimum hydrogen production cost.

A total of 5.5 MW is recovered in Section I using turbines for pressure reduction. Preliminary indications are that the turbines are economic but a final determination must await an analysis based on the hydrogen production cost resulting from this study.

5.5 H₂SO₄ Processing Step (Section II)

5.5.1 Design Considerations

The sulfuric acid processing step, Section II of the process, takes 57% H₂SO₄ from Section I, decomposes the H₂SO₄ and returns the resulting SO₂, O₂, and H₂O back to Section I. The process flow diagrams is given in Figure 5.5-1.

Major unit operations of Section II are sulfuric acid concentration, vaporization, and decomposition. Equipment layout and designs are based on previous work, but the section has been completely reflowsheeted to match the MARS blanket. Newly available thermodynamic data was used in the flowsheeting.

Figure 5.5-1 Schematic of Section II, Concentration and Decomposition of Sulfuric Acid

5.5.2 Acid Concentration

Early version of the sulfur-iodine water-splitting process relied on vapor recompression to achieve high thermodynamic efficiency, though at high capital cost.

Several techniques were tried to accomplish the goal of heat recovery without vapor recompression. These included multi-effect evaporators, pressure staged flash evaporators (adiabatic and non-adiabatic) and column evaporators. It became evident that no single technique could achieve the desired goal. A combination of four different concentration techniques was used in the final flowsheet.

Staged isobaric boiling at 7.5 MPa is used to achieve the goal of water remove at useful steam temperatures. Low pressure distillation is used for final concentration of the acid to the azeotropic composition. Two adiabatic flash stages at 2.0 MPa and 0.5 MPa are used to match the isobaric staged boiling to the low pressure distillation column. Finally, staged partial condensation is used to remove H_2SO_4 from the decomposer product along with a minimum of water.

The isobaric staged boiling is accomplished in what can be best described as a horizontal distillation column. The design is patterned after the H_2SO_4 vaporizer described in previous work and shown in Figure 5.5-2. The horizontal vessel is fluorocarbon lined to protect the mild steel from the sulfuric acid and brick lined to protect the fluorocarbon lining from heat. Siliconized silicon carbon tubes provide the heat of vaporization for the water. The vessel is partitioned into multiple stages by weirs made from acid brick. Above the weirs the vapor space is interconnected, with the vapor outlet at the low acid concentration end of the unit. Mixing of the vapors from many stages results in a vapor composition typical of the mass average temperature. The vapors are cooled slightly to condense the trace of acid present. The separated acid is added to the acid product of the concentrator. After removal

Figure 5.5-2 Design for the H_2SO_4 Azeotrope Vaporizer

of the acid, the steam passes through a total condenser transferring the heat of condensation to the evaporators of section III.

The acid, concentrated to 79%, at 7.5 MPa is flashed first to 2.0 MPa and then to 0.5 MPa and again to 0.02 MPa. The vapors from the first two flashes are treated by partial condensation to remove H_2SO_4 and recycled to Section I. The combined liquid/vapor stream from the third flash is fed to the low pressure distillation column.

An eight-stage distillation column completes the concentration of the acid up to 98%. Since the column must accommodate a large variation in liquid rates over its length, a tray type column is preferred over a packed column. Ceramic internals are used in the form of trough type trays. The bottom reboiler and the intermediate reboilers are again constructed with siliconized silicon carbide U-tubes. Each tray, except for the top, has its own intermediate reboiler.

The product gases from the decomposer are subjected to five stages of partial condensation so as to remove the undecomposed acid with a minimum of water. The liquid products from the partial condensation stages are returned to the atmospheric distillation column, the liquid from the final stage providing column reflux.

5.5.3 H_2SO_4 Vaporizer

The H_2SO_4 Vaporizer is a particularly challenging design problem since either ceramic materials or high-silicon metallic alloys need to be employed to simultaneously withstand the H_2SO_4 corrosion and the 3.0 MPa pressure from the helium heat transfer fluid.

Our best choice is to use siliconized silicon carbide U-tubes for the heat exchangers in the H_2SO_4 Boiler. This approach has been used on a developmental basis by the Norton Company⁽¹⁵⁾ in conjunction with AiResearch, Inc. for high pressure (5 MPa) helium "heat exchanger applications." We developed a special

geometric configuration that established a rough optimization between the heat transfer and pumping losses in the helium and the boiling instabilities that could occur in the liquid H_2SO_4 . This configuration was shown in Figure 5.5-2.

5.5.4 SO_3 Decomposer

The decomposer consists of staged catalytic fluid bed reactors operating up to 1100 K. Each unit contains internal heat exchanger tubes to provide the heat required to drive the highly endothermic SO_3 decomposition reaction. Final conversion of 53% is achieved using both Pt on Zr_2O_3 and Fe_2O_3 catalysts. The less expensive Fe_2O_3 catalyst is employed at 1100 K, but it loses its catalytic activity at significantly lower temperatures due to reversible sulfation.⁽⁵⁾ Noble metal catalysts on non-sulfating supports may be used over the whole temperature range. Details of a single stage of the Fluid Bed Decomposition are given elsewhere⁽¹⁹⁾ so only a brief overview is provided here.

Figure 5.5-3 shows some of the details of the fluidized bed design, using 0.5 mm diameter catalyst spheres. The fluidized bed is on the shell side of the unit with the helium on the tube side. The T_s across the helium film within the tube are about 20 K, and the T_s on the outside of the tubes in the fluidized bed region are expected to be around 14 K. The helium T presents a difficult design problem and the 20 K is a compromise between higher helium source temperatures and helium pressure.

The Fluidized Bed Decomposer consists of eight two-stage decomposers in parallel to handle needed production rate. A portion of the heat for the low temperature bed of each bed is supplied while cooling the high temperature product gases. A separate tube bundle is used for recuperation.

5.5.5 Decomposer Recuperator

The Decomposer Recuperator is a heat exchanger unit that simultaneously preheats the feed to the decomposer using heat from the hot SO_2 , O_2 , H_2O , and SO_3 that leave the decomposer. This unit is important, since without it, a larger quantity of sensible heat in the temperature range of 680 K to 1000 K

Figure 5.5-3 Design of the Catalytic Fluidized Bed SO_3 Decomposer for the Two-Zone Blanket

would have to be supplied from the blanket. In addition to the sensible heat requirement, there is, of course, additional heat required to supply the endothermic heat for decomposing SO_3 into SO_2 and O_2 . Recuperation is insufficient to provide the total sensible heat required to treat the sulfuric acid vapors from the boiling point to the decomposer inlet temperature because the heat of decomposition of H_2SO_4 to H_2O and SO_3 must be provided over this temperature range. The Recuperator thus includes a "boost" loop of TMR-supplied heat. This Decomposer Recuperator is a difficult design to accomplish since it is a gas-to-gas heat exchanger, and these types of units characteristically have very low heat transfer coefficients. The result is a high pressure drop, high pumping power, large and expensive heat transfer areas, and high alloy temperatures. The design we evolved was again a techno-economic compromise. We used Incoloy-800H in a standard shell and tube configuration as shown in Figure 5.5-4.

5.5.6 Decomposer Coolers

The Decomposer Cooler continues cooling the decomposer product after recuperation is no longer possible. Heat removed in the decomposer coolers is supplied to the low pressure still and the multistage isobaric flash preheater. Sulfuric acid condenses out as the decomposer products are cooled. This sulfuric acid is removed in several stages. The liquid phase from the final cooler is predominantly water; therefore, it is returned to Section I along with the gases.

Silicon carbide is required where sulfuric acid is condensing. At high temperatures, before the onset of condensation, Incoloy 800H would suffice except that silicon carbide is required for boiling sulfuric acid duty on the cold side of the tubes. After the majority of the sulfuric acid has been condensed and decanted, more conventional materials of construction are permitted. In the low temperature region the requirements of heating 57% sulfuric acid dominates the material selection.

2 units, 4366 tubes per unit
 Total heat transfer area per unit = 7407 m²

Figure 5.5-4 Design for the SO₃ Decomposer Recuperator

5.6 HI Concentration Step (Section III)

5.6.1 Design Considerations

The hydrogen iodide concentration step, Section III of the process, entails separation of a mixture of hydrogen iodide, iodine, and water (HI_x) into its component parts. A simplified flow diagram of Section III is presented in Figure 5.6-1. The iodine and water are separately returned to Section I, the main solution reaction step, and the hydrogen iodide is decomposed in Section IV of the process. The use of phosphoric acid as an extractive distillation agent, first proposed in 1975⁽²⁰⁾, remains as the reference process for HI purification. The alternative of liquid HBr extraction, although attractive from a theoretical standpoint, is not yet able to compete with the H_3PO_4 process on an efficiency basis.

The system $\text{HI-H}_2\text{O}$ forms a maximum boiling azeotrope at a composition of 57% HI which is approximately the composition of HI_x on an iodine free basis. Iodine is held in the $\text{HI-H}_2\text{O}$ solution through the formation of polyiodides such as HI_3 , HI_5 , HI_7 , etc., which are formed only in the presence of water. Addition of H_3PO_4 lowers the activity of water which performs two functions; first, it destabilizes the polyiodide complexes permitting iodine to form a separate liquid phase; secondly, it breaks the azeotrope which allows the HI to be distilled from the mixture. The HI distillation is performed under pressure so that liquid HI is available for decomposition in Section IV. Water is removed from the phosphoric acid by evaporation, and the phosphoric acid is recycled back into the process.

5.6.2 Iodine Separation

Liquid iodine is separated from the HI_x in two steps. Both operational steps are performed in fluorocarbon-lined mild steel vessels packed with 20-mm ceramic saddles. In the iodine knock-out column (C-302), the HI_x is contacted counter-currently with 96% H_3PO_4 . The HI and H_2O are extracted into the H_3PO_4 .

Figure 5.6-1 Schematic Flow Diagram of Section III, Separation of Aqueous HI_x into HI , I_2 , and H_2O

leaving molten iodine saturated with H_3PO_4 . H_3PO_4 is washed away from the iodine with water in the iodine wash column (C-301). The wash column is operated at 0.3 MPa and 393 K so as to maintain both iodine and water in the liquid state.

5.6.3 Hydrogen Iodide Distillation Column

HI is distilled from the H_3PO_4 solution in a plate column operating at 0.9 MPa. Operation at this pressure requires a higher temperature in the bottoms than would be required at low pressure, but an expensive HI liquefaction step is eliminated. Efficient use of the thermal energy, required for operation of the HI Distillation Column, is made possible by producing the HI product as a liquid and thus eliminating the thermodynamic inefficiency associated with a liquefaction compressor and that associated with production of power to operate it. Additionally, an intermediate condenser allows part of the condensing heat to be withdrawn from the column at a useful temperature. Although a majority of the heat required to preheat the feed and to operate the column is obtained by heat recovery within Section III, 355 MW is required from Section II at 523 K.

Design of the HI Distillation Column entailed a trade-off between capital and operating costs. Use of an intermediate condenser and operation at a relatively low reflux ratio reduce thermal energy requirements, but require more trays in the column for adequate chemical separation. The final design included 50 Hastelloy-C trays in a Hastelloy-C clad mild steel tower. The trays selected were the trough type, being a reasonable compromise between the higher efficiency, higher cost of bubble cap trays and the lower efficiency, lower cost of sieve trays.

An additional small design complication arises because the feed to the HI Distillation Column is saturated with iodine. Liquid iodine must be removed continuously from the middle of the column to avoid buildup. A stream, containing two liquid phases consisting of phosphoric acid and liquid iodine, is withdrawn from the iodine buildup region. The phosphoric acid returns to the column from the top of a liquid-liquid separator and the bottom iodine phase

passes to the iodine wash column for phosphoric acid removal before returning to Section I.

The small quantities of H_2SO_4 and SO_2 , remaining in the lower phase product from Section I, react chemically with HI in the feed preheater for the HI Distillation Column. The product of the reactions are H_2O , I_2 , H_2S , and S. The quantities involved are so small that the relative amounts of S and H_2S have not been determined. For flowsheeting and cost estimating purposes, it has been assumed that half the sulfur in H_2SO_4 and SO_2 ends up as H_2S and half as S. The H_2S will leave the column in the overhead product and the S is assumed to exit the column with the H_3PO_4 . The liquid S is separated from the H_3PO_4 and oxidized back to SO_2 in Section II.

5.6.4 Phosphoric Acid Concentration

Removal of water from the phosphoric acid is accomplished in three stages of vapor recompression-driven flash evaporation. The operating condition of each stage has been modified so as to reduce capital cost for heat exchange. Each stage now operates at a different pressure with the pressure decreasing as the acid concentration increases. The major cost saving came through elimination of the heat exchanger which conditioned the evaporator feed. Only the first and last stages require any heat input from the fusion reactor, but all three stages require significant quantities of power for vapor recompression. A total of 641 MW of shaft power is required to operate the compressors whereas only an additional 104 MW of thermal power at 484 K is required from Section V to heat the high temperature evaporator. Heat is recovered within each evaporation stage from interstage cooling of the six-stage compressor, from the condensation of the compressed steam and from the concentrated phosphoric acid product.

The phosphoric acid concentration step is simple in concept but capital costs of the turbine compressors and heat exchangers are significant in the overall hydrogen production cost. Even though a considerable reduction in the cost of H_3PO_4 concentration has been made, alternative chemical systems are *under continuing investigation with the goal of eliminating H_3PO_4 from the process.*

5.7 HI Decomposition Step (Section IV)

5.7.1 Design Considerations

The HI decomposition step (Section IV of the process) requires the following operations:

- o Decompose HI(l) to $\text{H}_2\text{(g)}$ and $\text{I}_2\text{(l)}$
- o Separate HI from the I_2 and recycle to the decomposer
- o Separate HI from the H_2 and recycle to the decomposer
- o Scrub the H_2 product in preparation for distribution

A simplified schematic flow diagram of Section IV is presented in Figure 5.7-1.

Three process variables dominate the design of Section IV and have a direct impact on the fusion reactor interface. The temperature and pressure of the HI Decomposition Reactor govern the equipment size and amount of recycle through the reactor, whereas the HI- I_2 Distillation Column pressure determines the maximum process temperature required from the fusion reactor. Variables governing the cleanup of the hydrogen product have a smaller, but still significant, impact on the hydrogen production cost.

5.7.2 HI Decomposition

Hydrogen iodide decomposition is accomplished in the HI Decomposition Reactor (R-401). The extent of the decomposition reaction is limited by thermodynamic equilibrium. Therefore, to limit the amount of recycle of HI back through the reactor, process conditions must be chosen so as to give a high conversion per pass through the reactor. Previous studies at the General Atomic Company⁽¹⁰⁾ have demonstrated that when decomposition is carried out under high pressures so that HI and I_2 are present as liquids, a much higher decomposition yield is obtained than with the analogous gas phase decomposition. Selection of the temperature and pressure involve a number of tradeoffs. The critical temperature of HI places an upper limit upon the

Figure 5.7-1 Schematic Flow Diagram of Section IV, the HI Decomposition Step

initial reaction temperature of an adiabatic flow reactor, while the required hydrogen delivery pressure places a lower bound upon the system pressure.

For this system design we still rely upon an adiabatic flow reactor using an activated charcoal catalyst. The recently developed homogeneous catalysis concept⁽²⁾ became available after this year's design base was fixed.

The reactor was sized for a 4-minute residence time. The mild steel vessel is lined with fluorocarbon to protect it from the HI and iodine. The reactor operates at 8.3 MPa. the reaction temperature increases from 415 K at the inlet to 424 K at the outlet due to the slightly exothermic nature of the reaction under these conditions. The majority of the energy required to bring the HI to reaction temperature is supplied by heat reuse within Section IV. Only 6% of the energy (10.9 MW_t) must be supplied from the fusion reactor.

A design based upon a continuous stirred tank reactor deserves future consideration. It would require a considerably longer residence time than the adiabatic flow reactor, but the system pressure could be lowered to 5 MPa, which may lead to reduced capital costs.

5.7.3 HI-I₂ Distillation

The liquid product from the HI Decomposition Reactor passes through a pressure reducing turbine to the HI-I₂ distillation column (C-401). The pressure of the still (5.1 MPa) sets the temperature of the still bottoms at 71° K. The still pressure and temperature may be decreased further, thus decreasing the high temperature heat load on the fusion reactor but at the expense of increased low temperature heat requirements to reheat the still overhead product for recycle to the HI cracker. In addition, the heat now supplied from the overhead condenser to the evaporative refrigerator and from the bottoms product to the liquid HI heat exchangers, would instead have to be supplied from other sources.

In addition to the 41 MW required from the fusion reactor helium stream, an additional 36 MW is required at 522 K. This additional heat may be used at a lower temperature because the distillation column is designed with intermediate reboilers. The use of intermediate reboilers increases the capital cost due to the cost of both the boilers and additional trays in the column, but the overall thermal efficiency is improved significantly.

The distillation column, constructed from Hastelloy-B clad mild steel, is 2.7 m in diameter for the bottom 12 m and expands to 4.7 m in diameter for the upper 3 m. There are 25 Hastelloy-B trough type trays in the bottom section and 6 in the top. The expanded top section is required by both the higher flow rates and lower density encountered in this section of the column.

5.7.4 Hydrogen Cleanup

The hydrogen from the decomposer is cleaned in three operations. First, the bulk of the hydrogen iodide is removed by condensation. Second, remaining HI is removed with a water wash. Finally, the trace of H_2S is removed by a combination of chemical reaction and water wash.

The gaseous product of the HI Decomposition Reactor is cooled in three stages. A heat exchanger removes the high temperature portion of the heat to the power bottoming cycle and dumps the low temperature heat to cooling water. The stream is further cooled from 303 K to 291 K via an absorption refrigeration system. The LiBr based refrigeration⁽²¹⁾ is driven by waste heat from the condenser of the HI-I₂ Distillation Column. After condensate separation, only a small quantity of HI and possibly H_2S remain in the hydrogen.

The remaining HI is easily removed via a water wash (C-402). A minimum amount of water is employed because any water added at this point must be removed by distillation in Section III. In the absence of iodine, HI is not corrosive to nonmetallics; therefore, hydrocarbon-lined mild steel is sufficient for this application. Since traces of iodine could enter the HI

scrub column due to process upsets in the HI condensation system, the lower portions of the column are lined with fluorocarbon. The column is packed with 25-mm ceramic Raschig rings.

Due to the high acidity in the lower portions of the column, H_2S cannot be absorbed. H_2S builds up slightly in the upper section of the column until at steady state, H_2S passes on to the H_2S removal column (C-403) at the same rate it enters the HI scrub column. Since H_2S has a relatively low solubility in water, a simple water scrub would require that an excessive amount of water be processed through Section II to reconvert the H_2S to SO_2 . The problem is solved by adding a small amount of SO_2 to the H_2S absorber. H_2S reacts with SO_2 in the presence of water to produce a sulfur slurry. The slurry is concentrated via a filter with back-flush so only a small amount of water accompanies the S back to Section II where it is converted to SO_2 via reaction with SO_3 . The mild steel column, packed with 25-mm ceramic Raschig rings, is hydrocarbon lined to prevent corrosion.

The H_2 pressure is dropped to the distribution pressure of 5.1 MPa via a power recovery turbine and thus exits the process.

5.8 Materials Selection for the Thermochemical Plant

The corrosive nature of the chemicals involved in the GA cycle has necessitated an extensive materials testing program be carried out during the process development effort. Since sulfuric acid is common to other thermochemical cycles and is industrially significant, other workers have extensively investigated materials for the H_2SO_4 - H_2O system.

5.8.1 Materials Selection for Section I

The extensive material testing program for the GA cycle has produced a great deal of data on corrosion in iodine systems⁽²²⁾. Table 5.8-1 presents a summary of the test results. Early results indicated that niobium is impervious to attack by the HI- I_2 - H_2O solutions typical of the main solution reactor, but the most recent work regarding the effect of the H_2SO_4 upon

PRINCIPAL UNIT-OPERATIONS	FLUIDS	TEMPERATURE RANGE		MATERIAL CANDIDATES FOR: HEAT EXCHANGERS, VESSELS, PUMPS AND HARDWARE
		°C	K	
MAIN SOLUTION REACTION	$\text{SO}_2 + \text{I}_2 + \text{H}_2\text{O} \longrightarrow$ $\text{HI}_x + \text{H}_2\text{SO}_4$ (55 wt % ACID)	125	398	GLASS-LINED STEEL; FLUOROCARBON PLASTICS & ELASTOMERS; CERAMICS — SiC , Al_2O_3 , CARBON IMPERVIOUS GRAPHITE TANTALUM
I_2 AND H_2O SEPARATION	$\text{HI}_x + \text{H}_3\text{PO}_4$	120–158	393–431	GLASS-LINED STEEL; HASTELLOY B-2; TANTALUM-LINED STEEL; FLUOROCARBON PLASTICS
I_2 RECYCLE	I_2	120	393	HASTELLOYS B-2, G, C-276; GLASS
DECOMPOSITION OF LIQUID HI_x	HI_x (LOW H_2O); I_2 ; H_2	120	393	HASTELLOY B-2; GLASS-LINED STEEL; FLUOROCARBON PLASTICS;

Table 5.8-1 Material candidates for handling process fluids containing HI_x and I_2

H₂SO₄ upon the system questions the use of niobium in this portion of the process. Tantalum can be substituted for the niobium, at higher cost, but this has not been done in the present equipment design.

Although glass-lined steel is an ideal material for use with the HI-I₂-H₂O system, it is unavailable in the equipment sizes required for the TMR powered plant. Fluorocarbon-lined steel performs the same function and is available in the desired equipment sizes.

5.8.2 Materials Selections for Section II

Materials selections need to be made for the following process units for Section II:

- o Isobaric Concentrator
- o Low Pressure Distillation Column
- o H₂SO₄ Boiler
- o Fluidized Bed Decomposer
- o Decomposer Recuperator
- o Decomposer Cooler

Table 5.8-2 presents a summary of the material investigations by GA⁽²²⁾ for the sulfuric acid sections of the process.

H₂SO₄ Boiler

Materials problems are similar for the Isobaric Concentrator, the Low Pressure Distillation Column, and the H₂SO₄ Boiler. The main problem is to provide a heat exchanger that is tolerant of hot concentrated sulfuric acid. All known metallic heat exchanger materials (except for expensive noble metals such as Au and Pt), are severely corroded by these H₂SO₄ solutions above temperatures of 500 K. A number of ceramic and intermetallic materials have been tested in hot concentrated H₂SO₄ at 633-693 K in recent years at LLNL^(23,24) and at Westinghouse⁽²⁵⁾. The top candidate materials from both corrosion resistance and heat exchanger design standpoints are the following:

PRINCIPAL UNIT OPERATION	FLUIDS	APPROXIMATE FLUID TEMPERATURES		MATERIAL CANDIDATES FOR: HEAT EXCHANGERS, VESSELS, PUMPS AND OTHER HARDWARE	
		°C	°K		
MAIN SOLUTION REACTION	$\text{SO}_2 + \text{I}_2 + \text{H}_2\text{O} \longrightarrow$ $\text{HI}_x + \text{H}_2\text{SO}_4$ (55 wt %)	125	398	GLASS-LINED STEEL; FLUOROCARBON PLASTICS & ELASTOMERS;	CERAMICS—SiC, Al_2O_3 , CARBON; IMPERVIOUS GRAPHITE TANTALUM
CONCENTRATION	H_2SO_4 55–65 wt %	95–150	368–423	HASTELLOYS B–2 OR C–276 IMPERVIOUS GRAPHITE	GLASS OR BRICK-LINED STEEL
CONCENTRATION	H_2SO_4 65–75 wt %	150–180	423–453	HASTELLOYS B–2 OR C–276 IMPERVIOUS GRAPHITE	GLASS OR BRICK-LINED STEEL
CONCENTRATION	H_2SO_4 75–98 wt %	180–420	453–693	BRICK-LINED STEEL; CAST Fe–14 wt % Si	
VAPOR FORMATION AND DECOMPOSITION	$\text{H}_2\text{SO}_4 \longrightarrow \text{H}_2\text{O} + \text{SO}_3$	330–600	603–873	BRICK-LINED STEEL; CAST Fe–14 wt % Si SILICIDE COATINGS ON STEEL; HASTELLOY G	
VAPOR DECOMPOSITION	$\text{SO}_3 + \text{H}_2\text{O} \longrightarrow$ $\text{H}_2\text{O} + \text{SO}_2 + 1/2 \text{O}_2$	500–850	873–1123	INCOLOY 800H WITH ALUMINIDE COATING	

Table 5.8-2 Candidates construction materials for sulfuric acid

- o Siliconized SiC
- o "CrSi₂" coated Incoloy-800
- o Durichlor-51 (Fe-14% Si-4% Cr)

All of these materials depend upon the development of a corrosion resistant SiO₂ scale on the surface to provide protection against corrosion by H₂SO₄. Similarly, it is well known that pure silica glass (SiO₂), such as in the form of silica brick, is inert to corrosion by H₂SO₄.

SiC currently presents the best prospect as a heat exchanger material. Siliconized SiC (a two-phase composition consisting of a mixture of SiC and Si) is produced at the Norton⁽²⁶⁾ and Carborundum⁽²⁷⁾ companies and is especially suited for this type of application. This type of material, which contains about a 10-15% excess of silicon metal, is impervious to gases, has a high thermal conductivity, high strength, good thermal shock resistance, and can be fabricated in complex shapes and bonded together to form heat exchanger assemblies⁽²⁸⁾. Corrosion testing of SiC for 1121 h and Si for 592 h in 97% H₂SO₄ at 673 K at the Lawrence Livermore National Laboratory showed no evidence of corrosion⁽²³⁾, thus confirming the corrosion resistance of both SiC and Si for this application.

Specimens of Incoloy-800 coated with chromium silicide of nominal composition CrSi₂ were tested for corrosion for 240 h at 673 K in 97% H₂SO₄ at the Lawrence Livermore National Laboratory⁽²⁴⁾ and found to show a very low rate of corrosion (extrapolated weight loss rate of 15 mg/cm²-y). The coatings were about 120 m thick, uniform in thickness, well-bonded to the substrate, and without evidence of fractures. The coatings were prepared by Dr. Charles M. Packer of Lockheed Missiles and Space Company of Palo Alto, California, using a slurry coat and rapid melt technique. Although the above results are preliminary in nature, they are encouraging in that a conventional heat exchanger material such as Incoloy-800 can be protected against corrosion by a coating process.

Durichlor-51 is a commercial high silicon cast iron material that is yet another candidate as a heat exchanger or container material. It is basically an intermetallic compound with a composition of approximately Fe_3Si and, as such, presents the disadvantages common to most intermetallics of poor ductility, low tensile strength, and poor machining and fabrication characteristics. Also, being a cast material, the control of internal porosities and non-uniformities present production problems. Nonetheless, with proper quality control and with good engineering design and practices, these problems can be controlled. From a corrosion standpoint, tests of up to 524 h on Durichlor-51⁽²³⁾ have shown only a moderate amount of attack (5 micron surface penetration); but very importantly, corrosion appears to proceed very uniformly over the surface of this material. It therefore seems reasonable to extrapolate to a lifetime of the order of 5 y for this application. In contrast to Durichlor-51, duriron is another high silicon cast iron alloy that shows a rather irregular corrosion behavior with grain pullouts, corrosion pits, and spallation in similar H_2SO_4 corrosion tests at 673 K⁽²³⁾.

In view of the developments thus far, our choice of material is siliconized SiC for the H_2SO_4 heat exchangers. We select silica brick as a liner material for the vessels, both because of the inertness of silica to corrosive attack by sulfuric acid, and to provide thermal insulation so that more conventional materials (e.g., Teflon-lined steel) can be used for the vessel wall. We select Durichlor-51 for the bodies of the liquid H_2SO_4 pumps.

Fluidized Bed Decomposer

The main materials problem in the Fluidized Bed Decomposer is selection of a heat exchanger material to meet corrosion resistance and creep strength requirements.

Corrosion tests on a number of heat exchanger alloys for H_2SO_4 vapor decomposers have been carried out at JRC Ispra and at the General Atomic company at temperatures in the 773-1173 K range. Their results⁽²⁹⁾ indicate that as bare uncoated alloys, Incoloy-800H and Inconel-625 provide the best

materials with anticipated lifetimes of a few years. A substantial gain in lifetime can be achieved by using an aluminide-coated Incoloy-800H on the surface exposed to the decomposing SO_3 gas. This gives an unusually stable coating-substrate combination that stabilizes in coating thickness and weight change after an exposure of 200-300 hours to the gaseous H_2SO_4 products⁽²⁵⁾. Corrosion protection is apparently afforded by an Al_2O_3 film, and imperfections in the coating are self-healing. The useful life of aluminide-coated Incoloy-800H for this application is believed to be of the order of 20 years. On the high pressure helium side, corrosion is not a problem.

From a consideration of creep strength, cost, and fabrication issues, Inconel-617, Inconel-625, and Incoloy-800 (or Incoloy-800H) appear to provide the best alternatives. Stress rupture and creep properties for Inconel-625 and Incoloy-800 are comparable⁽³⁰⁾. Although creep strength data are not available for Inconel-617, it shows 1000-hour stress rupture strengths that are three times higher⁽³⁰⁾ than for the other two alloys. Thus, extrapolations of the available data for Inconel-625 and Incoloy-800 suggest a tensile creep strength for 1% creep in 100,000 hours of $2000 \pm 50\%$ psi (1.4×10^7 Pa) at 1100 K and $1200 \pm 30\%$ psi (8.2×10^6 Pa) at 1150 K. We estimate the corresponding values for Inconel-617 to be $6000 \pm 50\%$ psi (4.1×10^7 Pa) at 1100 K and $3600 \pm 30\%$ psi (2.4×10^7 Pa) at 1150 K based on the argument that the creep strengths scale as a fraction of the long-term creep rupture data similar to Inconel-625 and Incoloy-800. Although no creep data are available for Incoloy-800H, it is a material that has been designed for higher strength than Incoloy-800 and should give a better performance.

From a consideration of both corrosion and creep strength we select Incoloy-800H as the heat exchanger material for the Fluidized Bed Decomposer. A minimum wall thickness 3 mm is required to maintain material integrity after allowances for 0.5 mm corrosion penetration. An aluminide protective coating is recommended on the SO_3 side of the heat exchanger to obtain a longer equipment life.

Incoloy-800H is also the material of choice for constructing the vessel and gas distributor for the Fluidized Bed Decomposer. Alternatively, the walls

could be constructed of silica brick backed with 316 stainless steel. For stainless steel to be successfully employed it must be maintained above the condensation temperature of sulfuric acid (680K).

Decomposer Recuperator

The main materials problem for the Decomposer Recuperator is for the Heat Exchanger, and as was the case for the Fluidized Bed Decomposer, corrosion and long-term creep strength are the main considerations. Temperatures are sufficiently lower here (1100 K) that the problems are less severe. Therefore, an aluminide coating should not be needed. We select Incoloy-800H at a wall thickness of 3 mm as the preferred material with Inconel-617 as a backup for the Decomposer Recuperator heat exchanger. Selections for the vessel walls are either silica brick-lined 316 stainless steel or Incoloy-800H.

Decomposer Cooler

Transport piping from the Decomposer Recuperator to the Decomposer Cooler would be made from Incoloy 800H. The Decomposer Cooler heat exchanger is an integral part of the acid concentration system and the heat exchanger tubes would be made from siliconized SiC.

Summary of Materials Selections for Section II

The selection of materials for Section II are summarized below in Table 5.8-3.

Table 5.8-3 Summary of Materials Selections for Heat Exchanger and Vessel
Materials for Section II of the GA Thermochemical Cycle

PROCESS UNIT	Hx MATERIALS	VESSEL MATERIALS
Concentrator	Siliconized SiC tubing, Durichlor-51 pumps	Silica brick liner on Teflon-lined mild steel
H ₂ SO ₄ Vaporizer	Siliconized SiC tubing, Durichlor-51 pumps	Silica brick liner on Teflon-lined mild steel
Fluidized Bed Decomposer	Aluminide-coated Incoloy-800H	Incoloy-800H, or silica brick liner on 316 S.S.
Decomposer Recuperator	Incoloy-800H	Incoloy-800H, or silica brick liner on 316 S.S.
Decomposer Cooler	Siliconized SiC tubing	Vessel is integral part of concentrator.

5.8.3 Material Selection for Sections III and IV

Material selection for Sections III and IV is similar to that selected for Sections I and II. The information in Table 5.8-1 is applicable to the iodine-HI regions of Sections III and IV. Phosphoric acid adds no new complexity except in the high temperature portions of the process, acid brick is necessary as a thermal protection for teflon liners. In addition, Hastelloy C has been recommended⁽³¹⁾ for use with boiling concentrated phosphoric acid.

5.9 Safety Considerations

The main safety issues involve the control, handling, and containment of tritium, liquid lithium lead, and sulfuric acid. The safety issues regarding tritium and liquid lithium were reviewed and discussed in some detail in the 1980 report⁽¹⁹⁾, and it was concluded that adequate controls were designed into the system to maintain proper safety. We have applied similar considerations in the current designs, including particular attention to both residual and accidental leakages of tritium into the environment or product, tritium inventories in the system, and potential problems of liquid lithium fires. We feel that the safety issues have been well met, and many of the details have been covered in this report. We refer the reader to previous reports for further details.

In regard to safety of sulfuric acid, it is important that conservative designs be maintained on the vessels and piping containing hot liquid and gaseous sulfuric acid and its decomposition products, since sulfuric acid introduced into the environment is a serious health hazard. We feel that we have taken adequate measures in our design studies, and recognize further that the sulfuric acid industry has been a large and well-established industry and one that we can draw on for future design information as needed for assuring a safe plant.

5.10 Plant Layout and Plot Plan

5.10.1 Plant Layout

A complete plant process layout, assembling all of the sections, was prepared for a previous version of a TMR driven water-splitting plant⁽¹⁾ and is given earlier in the report in Figure 5.0-1, complete with important process parameters and labels on key process sections and on process units. The legend describes the symbolic representation used. Generally we have attempted to arrange the highest temperature processes at the top of the figure and the lower temperature processes at the bottom. At the immediate left is the Tandem Mirror Fusion driver and the associated helium coolant streams operating turbo-generators and process heat exchangers as a topping cycle and bottoming into a steam-Rankine cycle for additional electricity generation.

A larger version of this complete process flow diagram and flowsheet are available upon request. However, this reduced-size figure has been included to help one grasp the overall picture of the Fusion/Synfuels Process concept.

The MARS version of the flowsheet has not been integrated into a similar one full layout but simplified flowsheets for each section have been presented in the text of this section. The composite flowsheet still serves the purpose of presenting the scale of the total thermochemical process.

5.10.2 Plot Plan

An artist's conceptual drawing of the plot plan as given previously⁽¹⁾ report is shown in Figure 5.10-1. Changes in this year's design did not materially affect space requirements so this plot plan still gives a rough idea of the approximate land area and relative sizes of the TMR nuclear island, turbine-generator, steam generator and process heat exchanger building, as well as the rest of the chemical plant. We conclude from the plot plan that this plant is quite compact, and raises no new issues regarding heat transport distances, safety, etc.

Figure 5.10-1 TMR-Synfuels Thermochemical Hydrogen Production Plant Plot Plan, 1981 Version

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SECTION 7.0

PRELIMINARY COST ESTIMATES

Contributor:
L. Brown

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SECTION 7.0

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7.0 PRELIMINARY COST ESTIMATES

Installed equipment costs and direct operating costs were estimated for the chemical plant as matched to the MARS blanket. The plant is based on a TMR with a two temperature zoned blanket and a Fluidized Bed Decomposer (FBD).

7.1 The Economic Basis

We present costs based on constant, July 1980 dollars, and use currently available chemical plant technology. Since the chemical portion of the plant will be manpower intensive and cannot be operated with as few people or as little supplies or maintenance as in an electric power plant, we have used chemical plant experience⁽¹⁾ in estimating all labor costs.

7.2 Costing Procedures

The chemical industry has developed considerable expertise in estimating the capital cost as well as the projected operating costs of their respective plants.

Costs for the Chemical Plant and the power systems interface are obtained by standard chemical engineering costing techniques. These costing techniques are based upon using actual construction experience from many chemical plants to predict capital and operating costs from FOB equipment costs. The new reference work by Peters and Timmerhaus⁽¹⁾ was used to augment the older Guthrie methods^(5,6) both for estimating the FOB costs of equipment, as well as deriving from the FOB cost estimates of the installed direct capital cost, the total plant investment and the operating cost. Where standard works could not provide capital costs, as in most of Section II and the power recovery systems, vendor estimates or other special sources of FOB cost data were used. The industry standard, Marshall and Swift (M&S)⁽⁷⁾, equipment cost index was used to reduce all costs to the same basis, July 1980.

It is recognized that some of the best costing techniques are maintained as proprietary by A&E, chemical and oil companies. A proprietary costing method, available at GA was used to spot check costs for a representative number of items. No overall bias could be observed although variations

between different types of equipment were noted.

We believe that our cost estimates are well within our goal of + 30% accuracy.

7.3 Preliminary Capital Cost

7.3.1 Main Solution Reaction Step (Section I)

The simplified schematic of Section I was presented in Figure 5.3-1. Table 7.3-1 presents a detailed list of the equipment required for performing the main solution reaction step, together with size and cost data. With the exception of the Heat Exchanger Reactor (R-101), all items have been costed by the Guthrie method.^(5,6)

The structural material used throughout Section I is mild steel. If other than dry SO_2 , oxygen and or water are present, the steel must be protected by an appropriate coating or lining. Although spray on hydrocarbon based coatings are adequate when moist SO_2 is present, liners of bulk fluorocarbon are specified when H_2SO_4 , HI or I_2 are present. The installed cost of fluorocarbon linings is estimated to be \$1100/m².

The Heat Exchanger Reactor is unique because of the material involved, niobium. The \$220/kg cost of the niobium tubing dominates the reactor cost. Added to the \$15.8M cost of the tubing is an appropriate amount for installation of the tubing (\$3.2M), plus the FOB cost of the equivalent mild steel heat exchangers (\$1.2M), giving an FOB cost of \$20.2M. Installation cost was estimated from the cost of the equivalent mild steel heat exchanger since niobium is required only for the heat transfer surfaces.

The Guthrie method allows specification of the piping materials separate from the specification of the materials of construction of the equipment. Either mild steel or standard fluorocarbon lined mild steel piping was specified as appropriate. Pumps are either cast iron for water, or cast iron with a molded fluorocarbon liner for corrosive solutions.

Since Section I operates essentially as six systems in parallel, 83% of production capacity may be maintained if any one system is down for repairs.

Table 7.3-1 - Preliminary Capital Costs for Section I^a - ~~M~~ July 1980

Item No.		Parallel Units	Diameter Meters	Length Meters	Equivalent Mild Steel FOB Cost	Actual FOB Cost Plus Adders	Installed Direct Capital Cost	Total Plant Investment Basis
C101	Primary scrubbing reactor	6	3.8	9.0	0.334	1.388 ^b	2.267	3.009
C102	Lower phase SO ₂ scrubber	6	5.1	19.5	0.869	3.967 ^b	6.252	8.232
C103	Boost reactor	6	5.1	19.5	0.869	3.967 ^b	6.252	8.232
C104	Secondary scrubbing reactor	6	4.5	8.6	0.391	1.693 ^b	2.721	3.597
S101	High pressure flash drum	6	3.6	13.5	0.501	1.787	3.004	4.057
S102	Low pressure flash drum	6	4.2	12.0	0.464	1.735	2.948	3.947
S104	Primary water knockout drum	6	3.0	3.8	0.116	0.116	0.346	0.518
S105	Secondary water knockout drum	1	3.0	3.9	0.019	0.019	0.057	0.086
R101	Heat exchanger reactor	6	1.8	7.5	1.205	20.686	22.772	27.194
E101	SO ₂ heat exchanger	6	1.7	12.0	0.731	1.112	2.075	3.020
E102	Sec. III water heat exchanger	2	1.1	12.0	0.178	0.178	0.413	0.631
E103	Sec. II water heat exchanger	3	1.2	12.0	0.282	0.282	0.654	0.997
P101	Water feed pump	6+1	-	-	0.002	0.002	0.008	0.012
P103	Reactor feed pump from C101	6+1	-	-	0.009	0.017	0.032	0.043
P104	Iodine feed pump	6+1	-	-	0.018	0.037	0.067	0.092
P105	Reactor feed pump from C103	6+1	-	-	0.034	0.068	0.121	0.167
P106	Reactor feed pump from C104	6+1	-	-	0.047	0.094	0.173	0.239
TE101	O ₂ power recovery turbine	1	-	-	0.996	0.996	2.148	3.238
TE103	Iodine power recovery turbine	1	-	-	0.058	0.169	0.250	0.329
Total Capital Cost					7.123	38.313	52.560	67.640

^a Based on a hydrogen plant producing 5025 moles per second.
^b Adders include the field installation of liners.

Pumps are cross-connected such that one installed spare backs up the active six. The power recovery turbines are not spared except by a by-pass valve.

The column labeled "Total Plant Investment Basis" is Table 7.3-1 indicates that the major unit costs of Section I are associated with the Heat Exchanger Reactor (R-101). The cost of R-101 may be decreased if more of the heat load is shifted to water-based heat exchangers. If the power bottoming cycle were eliminated the cost of R-101 would be significantly decreased due to the much larger differential temperatures across the exchanger.

7.3.2 H₂SO₄ Processing Step (Section II)

The simplified schematic for the Sulfuric Acid Processing step was presented in Figures 5.4-1. Detailed lists of the equipment required for H₂SO₄ concentration and decomposition together with cost data are presented in Tables 7.3-2. All cost estimates were made using Guthrie's techniques.^(5,6)

The major costs in Section II are associated with heat transfer equipment. Silicon carbide is the material of choice for heat transfer surfaces where liquid-gas interfaces occur. Silicon carbide is used for all heat transfer involving concentrated sulfuric acid except in the Recuperator/Decomposer Preheater. Incoloy-800H is used in the Recuperator/Decomposer Preheater as only gases exist at the temperatures encountered.

Vessels are fabricated from Fluorocarbon-lined mild steel, with this lining thermally insulated from the process via acid brick linings.

Costs of silicon carbide and vessel liners are treated as adders to the base carbon steel equipment costs. Silicon carbide U-tube costs are estimated to be \$32.7/M⁽⁸⁾ for the 5 cm diameter tubes specified for the H₂SO₄ vaporizer, and this cost was used in estimating the rest of the silicon carbon heat exchangers.

7.3.3 HI Concentration Step (Section III)

Capital costs for Section III are presented in Table 7.3-3. The simplified flow diagram for this section was presented in Figure 5.7-1.

Table 7.3-2 Preliminary Capital Costs for Section IIA^a

Item No.		Parallel Units	Diameter Meters	Length Meters	Equivalent Mild Steel FOB Cost	Actual FOB Cost Plus Adders ^b	Installed Direct Capital Cost	Total Plant Investment Basis
C201	Isobaric Concentrator Vessel	2	5.0	30.0	0.615	2.184	3.634	4.658
C202	Low Pressure Distillation Column	2	4.5	12.0	0.162	0.953	1.572	1.980
R201	Decomposer Vessel	8	6.0	12.0	0.900	15.829	18.659	22.159
S201	Condensate Separator on C201	2	1.4	2.4	0.035	0.073	0.156	0.208
S202	Condensate Separator on C202	2	1.4	2.4	0.035	0.073	0.156	0.208
S203	Intermediate Flash Separator	2x2	3.6	4.8	0.155	0.650	1.016	1.290
S204	Secondary Condensate Separator	2x2	1.4	2.4	0.070	0.146	0.312	0.416
S205	Acid Knock-Out Drum	2x5	3.6	3.0	0.211	0.854	1.352	1.719
E201	Concentrator Heat Exchangers	--	--	--	1.394	5.808	9.449	11.995
E202	Distillation Column Heat Exchangers	--	--	--	0.745	1.804	3.750	4.916
E203	Partial Condenser on C201	2	1.0	6.0	0.103	0.103	0.236	0.351
E204	Partial Condenser on C202	2	1.2	6.0	0.116	0.116	0.264	0.398
E206	Vaporizer Preheater	4	3.0	7.2	0.478	1.955	3.173	4.034
E207	Vaporizer	16	3.0	10.0	2.361	10.670	16.736	21.147
E208	Decomposer Preheater	32	3.0	9.0	2.622	25.969	39.696	47.298
E209	Decomposer Heat Exchanger Tubes	--	--	--	2.683	16.095	29.480	31.475
E210	Feed Preheater to C201	6	3.0	7.2	0.732	3.995	3.995	7.348
E211	Partial Condenser on Inter. Flash	2x2	1.2	6.0	0.238	0.238	0.543	0.817
E212	Total Condenser on Inter. Flash	2x2	1.5	6.0	0.293	0.293	0.669	1.006
E213	Total Condenser on C201	8	2.0	6.0	0.857	0.857	1.794	2.757
E214	Total Condenser on C202	4	2.0	6.0	0.457	0.457	1.043	1.569
P201	Dilute Acid Feed Pump	6+1	--	--	0.434	1.306	2.415	3.140
P202	Vaporizer Feed Pump	6+1	--	--	0.145	0.435	0.805	1.047
P203	Distillation Column Overhead Pump	6+1	--	--	0.027	0.027	0.063	0.096
TE201	Liquid Expander From H.P. Conc.	1	--	--	0.180	0.180	0.427	0.642
TE202	Liquid Expander From Inter. Flash	1	--	--	0.053	0.053	0.125	0.189
Total Capital Cost					16.066	91.050	137.364	172.655

^a Based on a hydrogen plant producing 5025 moles per second.^b Adders include installation of silicon carbide tubing, acid brick, and fluorocarbon lining.

Table 7.3-3 Preliminary Capital Costs for Section III^a - M\$, July 1980

Item No.	Parallel Units	Diameter Meters	Length Meters	Equivalent Mild Steel FOB Cost	Actual FOB Cost Plus Adders	Installed Direct Capital Cost	Total Plant Investment Basis
C301 Iodine wash column	3	4.5	18.0	0.343	2.769 ^{bc}	3.346	4.139
C302 Iodine knockout column	10	7.7	24.0	3.137	29.265 ^{bc}	34.476	42.272
C303 HI distillation	3	6.9	21.6	0.912	22.194 ^b	25.016	29.478
S301 Surge drum - C303 reflux	3	2.7	10.2	0.128	0.424 ^b	0.756	0.969
S303 Flash drum - 1st H ₃ PO ₄ stage	6	6.0	9.0	0.499	1.660 ^c	2.949	3.780
S304 Flash drum - 2nd H ₃ PO ₄ stage	5	3.6	16.2	0.357	1.192 ^c	2.114	2.708
S305 Flash drum - 3rd H ₃ PO ₄ stage	4	6.6	10.7	0.463	3.237	5.797	7.027
S306 S-H ₃ PO ₄ separator	3	5.9	22.2	0.558	3.907	6.789	8.242
E302 Intermediate condenser on C303	3	1.6	12.0	0.148	1.496	2.081	2.513
E303 Reboiler on C303	6	1.1	12.0	0.903	3.951	5.110	6.608
E304 Condenser on C303	6	1.6	12.0	0.820	4.063	5.480	6.967
E305 Iodine cooler	1	1.4	12.0	0.102	1.023	1.423	1.719
E306 Heater - 1st H ₃ PO ₄ stage	65	1.5	12.0	3.229	25.696	34.975	42.920
E307 Heater - 2nd H ₃ PO ₄ stage	50	1.5	12.0	2.639	21.417	29.262	35.789
E308 Heater - 3rd H ₃ PO ₄ stage	49	1.5	12.0	1.774	13.083	17.720	21.812
E309 Concentrated H ₃ PO ₄ cooler	6	1.8	12.0	2.148	21.701	30.168	36.434
E310 Water cooler - 1st H ₃ PO ₄ stage	1	1.7	12.0	0.239	1.703	2.386	2.938
E311 Water cooler - 2nd H ₃ PO ₄ stage	1	1.8	12.0	0.224	1.647	2.229	2.643
E312 Water cooler - 3rd H ₃ PO ₄ stage	1	1.7	12.0	0.160	1.181	1.600	1.970
P301 Lower phase feed pump	10+1	-	-	0.212	0.425	0.814	1.112
P302 Iodine wash water pump	3+1	-	-	0.006	0.006	0.013	0.021
P304 Feed pump - C303	10+1	-	-	0.742	1.486	2.721	3.749
P305 Concentrated H ₃ PO ₄ pump	10+1	-	-	0.478	0.956	1.750	2.406
TE302 1st H ₃ PO ₄ stage PR turbine	1	-	-	0.398	0.398	0.741	1.092
TE303 2nd H ₃ PO ₄ stage PR turbine	1	-	-	0.257	0.257	0.562	0.892
TE304 3rd H ₃ PO ₄ stage PR turbine	1	-	-	0.240	0.240	0.516	0.757
TE305 Iodine power recovery turbine	1	-	-	0.087	0.171	0.291	0.396
TC301 1st H ₃ PO ₄ stage steam comp.	6	-	-	17.321	17.321	37.378	61.467
TC302 2nd H ₃ PO ₄ stage steam comp.	5	-	-	14.434	19.562	31.149	46.949
TC303 3rd H ₃ PO ₄ stage steam comp.	4	-	-	11.547	11.547	24.919	37.559
Total Capital Cost				64.610	214.038	314.531	417.328

^a Based on a hydrogen plant producing 5025 moles per second.^b Adder includes field installation of packing.^c Adder includes field installation of liner.

Materials of construction and costing techniques are similar to those of Section I. Where, because of thermal or mechanical limitations, fluorocarbon linings are not acceptable, we have used Hastelloy-C.

The hydrogen iodide concentration step is the most capital intensive portion of the chemical plant. Although significant costs are associated with iodine knockout and hydrogen iodide distillation, the largest costs are associated with phosphoric acid concentration.

The high cost components of the phosphoric acid concentration system are steam compressors and heat exchangers. The compressor costs are based on vendor estimates⁽⁹⁾ and reduced to the 1980 base using the M&S⁽⁷⁾ cost index. These compressor costs appear to be fixed unless future developments bring down the relative cost of turbine compressors. Flowsheet modifications have the potential for reducing the heat transfer costs, particularly if direct contact heat transfer is employed between immiscible streams in Sections I and III and between Sections III and IV. The large amount of rotating machinery in the phosphoric acid concentration system makes this part of the process a potential source of downtime. The compressors in the third evaporation stage were deliberately oversized to make them identical to the first and second stage units. If any one of the 15 units is down, intermediate pressures may be shifted to permit operation at 93% of capacity with only a slight overall efficiency loss.

7.3.4 HI Decomposition Step (Section IV)

The HI decomposition step has the lowest flow rates and lowest costs of the four chemical process steps. The costs are as high as given in Table 7.3-4 only because of the high pressures involved. The simplified flowsheet for Section IV was given in Figure 5.8-1.

7.4 Chemical Plant Operating Costs

Preliminary estimates of the operating and maintenance costs of chemical plants may be calculated from the F.O.B. costs of the capital equipment. According to Guthrie,⁽⁶⁾ the yearly operating and maintenance cost is 20% of the direct capital cost, assuming mild steel construction.

Table 7.3-4 Preliminary Capital Costs for Section IVA - M³, July 1980

Item No.	Parallel Units	Diameter Meters	Length Meters	Equivalent Mild Steel FOB Cost	Actual FOB Cost Plus Adders	Installed Direct Capital Cost	Total Plant Investment Basis
C401 HI-12 Distillation column	1	4.7	15.0	0.174	1.794b	2.198	2.663
C402 HI Absorber	6	4.1	9.6	1.077	1.342bc	4.411	5.850
C403 H ₂ S Scrubber	2	3.8	27.3	0.718	1.049bc	2.900	4.083
S401 Reactor effluent V-L separator	1	2.9	13.2	0.166	0.300c	0.728	1.009
S402 H ₂ -HI vapor liquid separator	1	2.4	9.6	0.124	0.206c	0.527	0.735
D401 Reflux surge drum - C401	1	3.3	15.0	0.077	0.254c	0.373	0.510
R401 HI decomposition reactor	4	4.5	27.0	1.795	3.782bc	8.411	11.532
E400 Misc. integrated heat exch.	13	-	-	1.333	6.526	9.457	12.100
E4XXd Absorption refrigeration unit	1	-	-	1.732	1.732	2.891	4.678
P401 Reactor feed pump	4+1	-	-	0.654	1.892	2.980	3.975
P402 HI recycle feed pump	1+1	-	-	0.294	1.442	2.272	3.029
P403 Reflux pump - C401	1+1	-	-	0.013	0.038	0.059	0.077
P404 Make-up water feed pump	6+1	-	-	0.021	0.049	0.074	0.074
P405 Recycle pump - C402	6+1	-	-	0.025	0.071	0.112	0.149
P406 S02-H ₂ O pump to C403	6+1	-	-	0.018	0.018	0.043	0.065
P407 Recycle pump -C403	2+1	-	-	0.015	0.015	0.036	0.053
TE401 HI-12 power recovery turbine	1	-	-	0.357	1.033	1.521	2.048
TE402 Hydrogen power recovery turbine	1	-	-	1.666	1.666	3.596	5.421
Total Capital				10.259	23.281	42.564	58.051

a Based on a hydrogen plant producing 5025 moles per second.

b Adder includes field installation of packing.

c Adder includes field installation of liners.

d The absorption refrigerator includes heat exchangers E408, E410, and E411.

Assuming the cost of the mild steel plant is 243.2% of the F.O.B. cost of the equivalent mild steel equipment,⁽⁵⁾ the yearly operating cost of the chemical plant is \$48.6 M as indicated in Table 7.4-1.

TABLE 7.4-1
Estimated Operating Costs of Chemical Plant - M\$ July 1980

<u>Section</u>	<u>Equivalent Mild Steel F.O.B. Cost</u>	<u>Total Plant^a Investment Based on Mild Steel^a</u>	<u>Yearly Operating Cost^b</u>
I	7.123	17.679	3.536
II	16.066	39.727	7.945
III	64.610	160.362	32.072
IV	<u>10.259</u>	<u>25.463</u>	<u>5.093</u>
	98.058	243.231	48.646

a 248.2% of F.O.B. equipment cost.

b 20% of Total Plant Investment.

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