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ENGINEERING AND BENCH-SCALE STUDIES OF THE SULFUR-IODINE CYCLE AT GENERAL ATOMIC

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ABSTRACT

The progress made on process engineering and bench-scale investigations of the General Atomic Company sulfur-iodine thermochemical water-splitting cycle is presented. A second generation process engineering flowsheet is under development. The results of this new flowsheeting effort not only represent a reiteration in the process engineering, but they also reflect the progress made in the laboratory; at the time of writing, approximately 60% of the flowsheet is complete. It is expected that the thermal efficiency of the process will be 45% or higher. In 1977, bench-scale investigations of the sulfur-iodine cycle have been initiated with the objective of verifying the practical chemical feasibility of the cycle. One of three subunits, which performs the main solution reaction, has been built and the first experimental run has been performed. The other subunits are planned to be ready for testing by 1979, and operation of the integrated unit is expected late in 1979.

A PROGRAM TO INVESTIGATE thermochemical water splitting using the High-Temperature Gas-Cooled Reactor (HTGR) as a heat source has been underway at General Atomic Company (GA) since October 1972. Current participants in this program are the Gas Research Institute (GRI) which carries on the sponsorship of the American Gas Association (AGA), the U.S. Department of Energy (DOE) and GA. Northeast Utilities Service Company and Southern California Edison Company have previously participated.

After an extensive computer aided search accompanied by laboratory scouting experiments, a cycle based on sulfur and iodine was selected in 1974 for process development.

The abbreviated chemical description of the sulfur-iodine cycle, which was first described by Russell (1)*, is:

$$2H_2O+SO_2+xI_2 \rightarrow H_2SO_4+2HI_x \quad 370K \qquad (1)$$

$$2HI_x \rightarrow xI_2+H_2 \quad 573K \qquad (2)$$

$$H_2SO_4 \rightarrow H_2O+SO_2+1/2O_2 \quad 1144K \qquad (3)$$

The HI_x represents the aqueous solution of several polyiodides formed in the main solution reaction (1). Separation of the H_2SO_4 and HI_x takes place under gravity, as the acids are almost completely immiscible. The upper phase contains almost all of the H_2SO_4, and the lower phase contains almost all of the HI_x. It takes, however, several process steps to perform the chemistry described by the reactions above. The following list of transformations, for example, is a more realistic description of the cycle.

$$H_2O(\ell)+SO_2(g)+xI_2(\ell) \rightarrow H_2SO_4(sol)+2HI_x(sol) \qquad (4)$$

$$H_2SO_4(sol) \rightarrow H_2SO_4(\ell) \qquad (5)$$

$$H_2SO_4(\ell) \rightarrow H_2SO_4(g) \qquad (6)$$

$$H_2SO_4(g) \rightarrow H_2O(g)+SO_3(g) \qquad (7)$$

$$SO_3(g) \rightarrow SO_2(g)+1/2O_2(g) \qquad (8)$$

$$2HI_x(sol) \xrightarrow{H_3PO_4} 2HI(g)+(x-1)I_2(\ell) \qquad (9)$$

$$2HI(g) \rightarrow H_2(g)+I_2(g) \qquad (10)$$

$$I_2(g) \rightarrow I_2(\ell) \qquad (11)$$

The interactions of these actual process steps, their effect on the heat balance and thus on the eventual economics of the process are the subject of process engineering studies. Results of these studies will also give guidance to further experimental investigations.

In 1975 the potentially attainable thermal efficiency of the process was estimated to be 52%** (2). Then, based on this encouraging result, the design of a complete process engineering flowsheet was started, which resulted in a 41% efficiency (3). For this process design, actual

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*Numbers in parentheses designate References at end of paper.

**Thermal efficiency defined as the ratio of the higher heating value of the H_2 produced to thermal output of the nuclear reactor.

data on reaction yields and physical separations were used, which by then had become available from laboratory experiments. Where data were still missing, realistic engineering judgments were made. Although this first complete flowsheet reflects a greater chemical engineering understanding than the initial evaluation, it is still an interim result. Moreover, continued efforts in the laboratory on the chemistry of the cycle (4)(5)(6) have developed improved yield data and alternate processing methods whose effects should be investigated. Therefore, in 1977 under DOE sponsorship, the design of a new flowsheet was initiated, which in a few areas will differ considerably from the previous one. At the time of writing, this work has not been completed and no conclusions can yet be drawn. It is expected, however, that this second generation flowsheet will show a thermal efficiency of approximately 45%.

At the same time, also under the sponsorship of DOE, bench-scale studies have been initiated to investigate the practical chemical feasibility of the cycle. These studies will give experimental information on the handling of fluids involved, on the operational behavior of key pieces of equipment, and on the effects of incomplete physical separations and possible side reactions.

This paper discusses the status of these latest engineering and bench-scale studies. Extensive reports on chemical and materials investigations relating to the GA sulfur-iodine cycle can be found elsewhere (4)(5)(6)(7).

PROCESS ENGINEERING

MAJOR OBJECTIVES AND CONSTRAINTS- In the process of screening thermochemical water splitting cycles, short-cut calculations (for example, on theoretical thermodynamic efficiencies) have their place in revealing potentially attractive cycles. But once screening has been done and a potential cycle is discovered, we believe that a complete process engineering flowsheet is the only basis for a good evaluation of that cycle. Such a flowsheet will reveal how the phase separations and the heat balances required to perform the chemistry of the cycle affect the actual performance of that cycle. The emphasis in our first flowsheeting was to show the relative importance of the various processing steps, and the main guideline in this was the overall thermal efficiency of the cycle. Our first results were, therefore, mainly used to guide laboratory efforts. As iterations in the chemical engineering continue with the new data, and as capital cost information gradually becomes available, process modifications will be guided more and more by cost considerations. The thermal efficiency, however, will still remain important, since it relates the cost of the chemical plant to the cost of the nuclear heat source. Several constraints and subobjectives guide this process engineering design effort:

1. The design reference heat source is a process heat HTGR, producing 1256K (1800°F) helium at the core outlet. The inlet temperature associated with this outlet temperature specification, for proper temperature distribution in the core, is 772K (930°F).
2. The heat content of the helium circulating through the core of the process heat HTGR is transferred to the water-splitting process through an intermediate helium heat-transfer loop. This assures isolation of the water-splitting process from radioactive contamination resulting from potential leaks in heat exchangers containing the reactor core primary helium.
3. The various transfer pump and gas compressor power demands of the process are supplied by turbomachinery in the intermediate helium heat-transfer loop, rather than under the more severe temperature service and more difficult radioactive contamination maintenance conditions of the primary helium.
4. Since a high thermodynamic efficiency is sought, the process heat demand vs. temperature function and the helium heat delivery vs. temperature function must be matched to avoid large thermodynamic irreversibilities associated with too-large values of heat transfer ΔT.
5. Heat available from within the water-splitting process must be reused whenever it is at a temperature greatly different from that of cooling water or cooling air; the tendency is to be more capital intensive with respect to energy reuse and efficient transfer of energy than has been justifiable, historically, in industrial chemical processes when energy was relatively cheap.
6. Many of the thermochemical data needed to optimize the design are being obtained but will not all be available at the time it is necessary to specify the base process flowsheet design. Best estimates will be used in portions of the design, necessitating flowsheet review and modification later as data become available.

From an engineering viewpoint, a major advantage of the GA thermochemical water-splitting process is that the system is virtually an all-fluid process. Potentially some solid iodine may be handled, but iodine melts at the relatively low temperature of 387K (236°F), and if its handling as a solid is inconvenient, it can be liquefied. We found the chemical complexity of the system to be moderate. Three primary chemical reactions permit a good flexibility in fitting the process to varying thermal matching requirements while not leading to overwhelmingly complex reaction and separation steps.

GENERAL PROCESS DESCRIPTION-In the

thermochemical process four distinct sections are identified. They perform the following operations:

Section I: The Main Solution Reaction Section. This section produces the acids H_2SO_4 and HI_x from H_2O, SO_2 and I_2. The SO_2 that is fed to this section contains all the O_2 generated by the decomposition of the H_2SO_4. The O_2 product is taken from this section.

Section II: The H_2SO_4 Section. This section concentrates and decomposes H_2SO_4 received from Section I.

Section III: HI Concentration Section. This section removes the very small amount of unreacted SO_2 from lower phase product ($HI_{x\ sol}$) and then separates it into HI, I_2, and H_2O using H_3PO_4.

Section IV: HI Decomposition Section. This section decomposes HI into H_2 and I_2 and separates products. The H_2 product is taken from this section.

Furthermore there is a special helium section which combines the helium related parts for the delivery of nuclear heat and the generation of process power. Most of this section will, therefore, be interwoven with the four chemical sections. These chemical sections are outlined in Figure 1.

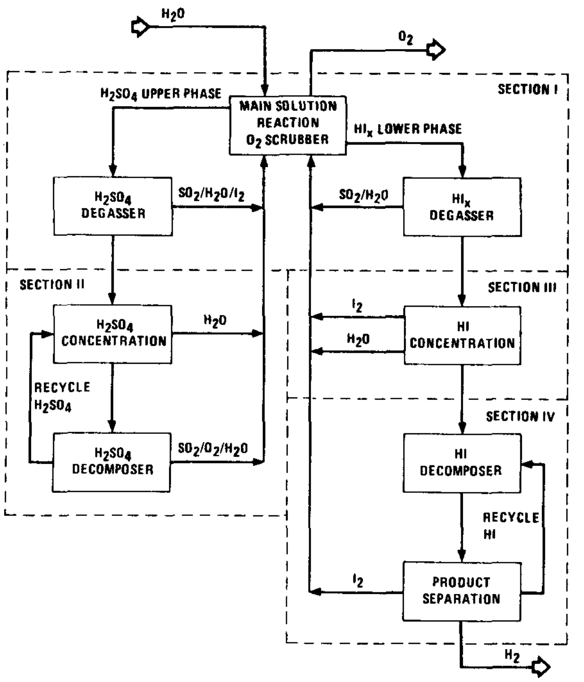

Fig. 1 - Sulfur-Iodine Cycle Process Model

PROCESS ENGINEERING DESIGN STATUS- Due to the complex nature of the overall process, several iterations of at least parts of the flowsheet are needed before a reasonable optimum result can be obtained, and, as the design, at this writing, has not yet been completed, the results obtained so far must be considered preliminary. In order to possibly expedite the engineering design, we have acquired the use of a process simulator computer code (DESIGN/2000, the ChemShare Corporation), and this code is now employed in certain areas of the design. Results obtained so far are given below.

Section I. In Section I, the main solution reaction (1) is performed, and the product H_2SO_4 and HI_x solutions are separated and degassed from SO_2. Initially, it was proposed to operate this reaction at a temperature below the ambient with a moderate excess of iodine. However, optimization studies performed in the laboratory, which used as a measure the heat required to recover unreacted reactants, have indicated that the reaction should be run at approximately 368K with an increased excess of iodine. As a result of this higher operating temperature, refrigeration is no longer required and the yield of the reaction is increased by approximately 20%.

By experiment, it has also been shown that O_2 in the main solution reaction (1) can pass as an inert substance. Therefore, the SO_2-O_2 product stream from the H_2SO_4 decomposition reaction (3) can be sent directly into the main solution reaction, which obviates the need for a separate SO_2-O_2 separation step. To obtain a clean O_2 off gas, countercurrent scrubbing towers are proposed for the main solution reaction. The gaseous SO_2-O_2 input from Section II becomes an O_2-I_2 gaseous output in which the I_2 residual is low, and it is relatively easier to remove from the final effluent O_2 product stream in an energy-efficient physical or chemical separation operation than is SO_2.

Figure 2, a diagram of the current conceptual flowsheet for Section I is shown. Iodine, water, and sulfur dioxide are collected from the other sections and are introduced such that the I_2 and most of the H_2O act as countercurrent scrub stages against the input SO_2-O_2 stream. The temperature of the SO_2 removal column is set somewhat higher than the main solution reaction column to ensure that $I_2(s)$ will not freeze out in the equipment. Most of the reaction and SO_2 removal is expected to occur in the main solution reaction column, with the SO_2 removal column performing a final polishing operation.

The H_2SO_4-HI_x liquid phases leave the bottom of the main solution reaction column and are partially concentrated by a throttle-flash from 180 kPa to ambient pressure. The gaseous portion of the flash mixture is separated from the liquid and is recycled directly to the main solution reaction column. The immiscible solutions are then separated by gravity.

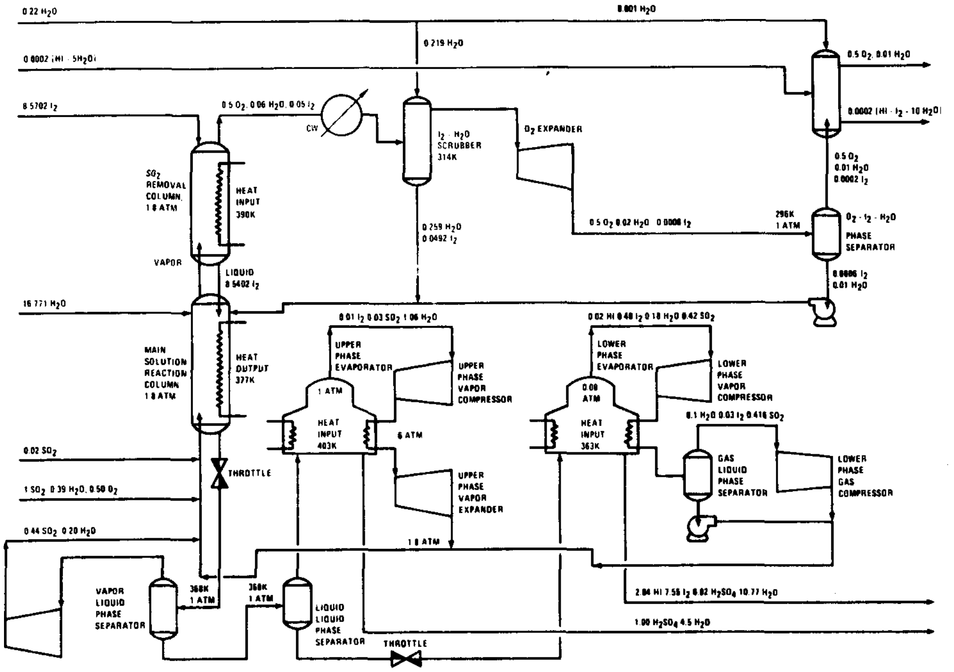

Fig. 2 - Section I, main solution reaction (H_2SO_4-HI production and separation, and O_2 removal)

The evaporation step for the H_2SO_4 light phase is to remove the corrosive residuals of HI and I_2 before the stream goes to Section II. The amount of water boilup associated with the back-reaction of HI and H_2SO_4 and the volatilization of elemental I_2 is probably conservative (i.e., the separation might be shown later, by laboratory data, to require less boilup). The evaporation step for the HI-I_2 heavy phase is to thoroughly remove the residual SO_2 under conditions sufficiently mild that the SO_2 and HI do not react.

The O_2 offgas from the SO_2 removal column is cooled first with water and then by adiabatic expansion to condense out most of the iodine. Virtually all remaining traces of I_2 are finally removed by scrubbing with a dilute solution of HI which forms an HI_3 complex in solution.

The system has been designed to be relatively work-intensive in order to avoid a high external heat demand. The heat requirements in Section I are among the lowest in the entire process. To meet these demands with direct supply from secondary helium would require extensive decompression/recompression of the secondary helium to make a thermally efficient matchup. Further, the option of using a low temperature bottoming power cycle on the large amount of reject heat at 377K from the main solution reaction column has not been foreclosed by designing for lower heat-higher power demand.

The net external work requirement is 33 kJ/Mol H_2, and the external heat requirement is 14 kJ/Mol H_2. The appropriateness of the particular flowsheet alternatives and assumptions made here will be reviewed after all other sections have been designed and integrated to make the secondary helium loop design. Table 1 shows net energy transfers in a partially summarized form.

Table 1 - Net Energy Transfers, Section I

Power	kJ/mol H_2SO_4
Gross Compressors Load	34.78
Expanders Power Recovery	-1.86
Net External Power Demand	32.92

Sensible Heat Transfers	kJ/mol H_2SO_4
Upper Phase SO_2 Strip Aux. Heat	-10.2
Lower Phase SO_2 Strip Aux. Heat	- 4.1
Main Reactor-O_2 Scrub Upper Sect.	1.6
Main Reactor-O_2 Scrub Lower Sect.	264.0
O_2 Cooler. HE-1	7.5
Net External Heat Output	258.8

Section II. In Section II the SO_2 and I_2 free aqueous H_2SO_4 (~55 wt.%) is concentrated and decomposed into H_2O, SO_2 and O_2. In Figure 3

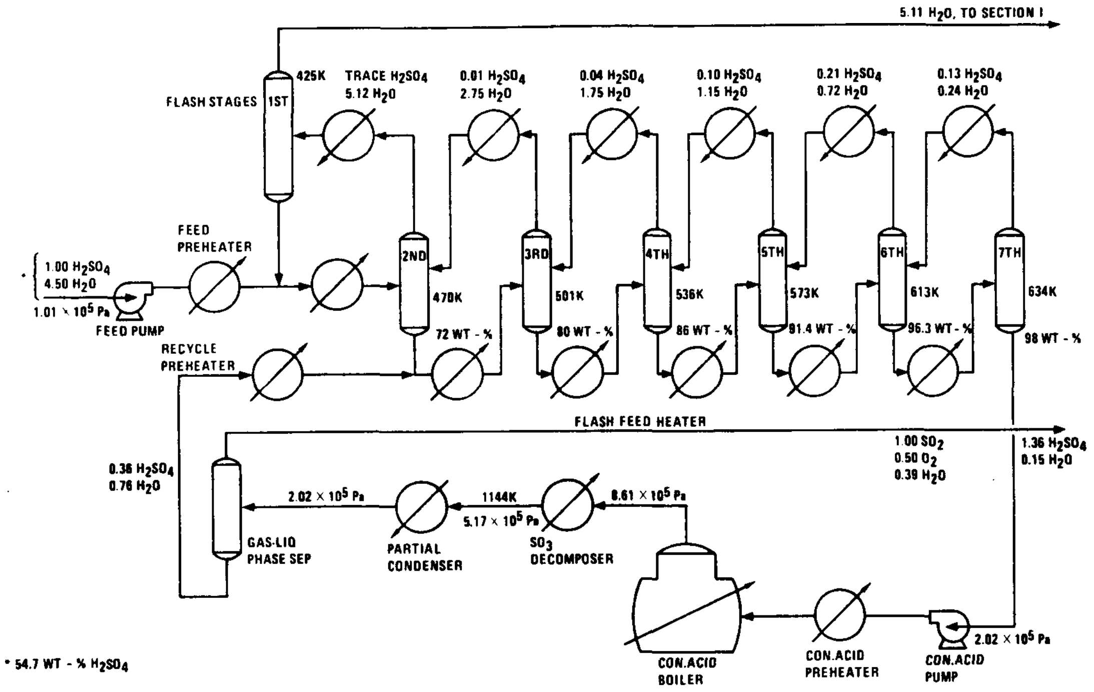

Fig. 3 - Section II, H_2SO_4 concentration and decomposition

a flow diagram of Section II is given. The H_2SO_4 feed received from Section I is concentrated at approximately 200 kPa to 98 wt.% in a series of six flash evaporation stages. The concentrated acid is then vaporized at 686K and decomposed throughout its heatup to 1144K, the maximum process temperature in the cycle. The last part of this heatup, from 800K, will be done in the presence of a catalyst to facilitate the decomposition of SO_3 into SO_2 and O_2. The reactant mixture is then disengaged from the catalyst and cooled back down. Eventually undecomposed SO_3 forms aqueous H_2SO_4 which at a concentration of 72 wt.% is separated from wet SO_2/O_2 mixture and recycled to the third concentration stage. The wet SO_2/O_2 product is sent back to Section I.

The thermal decomposition of H_2SO_4 will be performed at relatively high pressures, with a considerable allowance for pressure drop (850 to 200 kPa). This will have a negative effect on the thermal efficiency of the cycle since the yield per pass decreases with increasing pressure. It is, however, expected that the efficiency loss will be offset by a reduction in capital cost of the decomposer/heat exchanger. This component can be reduced in size because of increased heat transfer coefficients and decreased volume of the gas processed.

In Table 2 the heat transfers in Section II are shown. Since the overall net heat demand of the process will be dominated by this section, the internal heat matchup will be determined after all sections are finished.

Table 2 (Part 1) - Heat Loads, Cold Sides, Section II

	Duty, kJ/mol H_2	Temperature, K	
		In	Out
Feed Preheater	11.013	403	425
Flash Feed Heater	123.457	425	470
Flash Feed Heater	98.819	470	502
Flash Feed Heater	49.508	502	536
Flash Feed Heater	49.215	536	573
Flash Feed Heater	59.944	573	613
Flash Feed Heater	34.334	613	634
Recycle Preheater	6.103	418	470
Concentrated Acid Preheater	13.558	635	686
Concentrated Acid Boiler	132.663	686	686
SO_3 Decomposer	254.810	686	1144

Table 2 (Part 2) - Heat Loads, Hot Sides, Section II

	Duty, kJ/mol H_2	Temperature, K	
		In	Out
Flash Feed Condensors	8.690	470	425
Flash Feed Condensors	4.525	502	470
Flash Feed Condensors	6.999	536	502
Flash Feed Condensors	11.997	573	536
Flash Feed Condensors	24.199	613	573
Flash Feed Condensors	16.355	634	613
Decomposer Partial Condensor	123.818	1144	579
Decomposer Partial Condensor	93.515	579	418

The sulfuric acid vaporization is the only major isothermal heat load in the cycle; the reboiler of the distillation tower from the previous flowsheet, which had a comparable heat load, is omitted here as a result of the multiple flash evaporation method of concentration. This should improve the thermal matchup with the non-isothermal, secondary helium heat source as compared with the previous flowsheet.

Section III. In Section III the HI_x(sol) received from Section I is separated into anhydrous HI, I_2 and H_2O. For this separation H_3PO_4 appears to be more attractive than the LiI which was considered previously. I_2 can be separated as a liquid, and the actual boiling of the H_2O can occur at relatively low temperatures from H_3PO_4. As compared to the previous flowsheet, this should improve the heat economy, and the materials cost associated with this evaporation step should be less as well. At this writing, however, the design has not yet been started.

Section IV. Two conceptual process schemes were developed for Section IV, the decomposition of HI. Each scheme has one HI decomposer and each depends on both condensation and distillation to recover H_2 and separate HI and I_2 for recycling. Each scheme also has a water wash step to remove a small amount of HI from the recovered H_2. The schemes differ as follows:

- Scheme A - The effluent from the decomposer is fed to a distillation unit which separates it into an I_2 fraction and an H_2-HI fraction. The latter is cooled to condense out the HI.
- Scheme B - The effluent from the decomposer is cooled to condense out the I_2 and a large fraction of the HI. The HI-I_2 liquid is distilled to separate the HI and I_2. The H_2-HI gas phase is cooled further to condense out the HI.

More than one temperature level for the decomposer had to be considered because the conversion per pass increases with an increase in temperature. Laboratory work has shown that, by using a catalyst, decomposition of HI can proceed essentially to equilibrium conditions in a very short time (seconds) at temperatures as low as 473K. However, at some higher temperature level, equilibrium at a higher HI conversion can also be reached in comparably short times even without a catalyst. The higher the temperature level the faster the decomposer effluent must be cooled to prevent any significant back reaction; at very high temperatures the back reaction must be prevented by a quench which is thermodynamically inefficient.

After preliminary studies, Scheme B was chosen on the basis of its more efficient product separation. Figure 4 shows the flow diagram of this section. With the minor exception of the water-wash tower, this entire section has been modeled with DESIGN/2000. It is now possible to quickly recalculate this section, for example, for a different temperature in the HI decomposer reactor such that a better heat matchup in the overall process can be obtained.

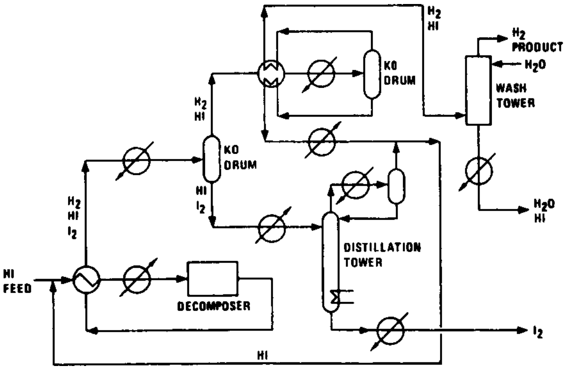

Fig. 4 - Section IV, HI decomposition

BENCH-SCALE TESTING

In 1977, under a DOE contract, GA started the design and construction of a bench-scale unit that would perform all the steps in the sulfur-iodine thermochemical water-splitting cycle as one continuous operation. Thus, with the exception of water being fed in and hydrogen and oxygen taken out as products, all the other materials will be internally recycled. The objective of this effort is to test the practical chemical feasibility of the cycle. The only side reactions of importance in the sulfur-iodine cycle are those which reduce the SO_2 to S or H_2S. The new conditions for the main solution reaction, higher temperature and higher iodine content, have reduced the rates of these side-reactions to a very low level. Only continuous operation of the process with recycled materials over extended periods of time can reveal whether these side-reactions will still be significant and, if so, how they can be remedied.

In the bench-scale testing, there is no attempt to prove the energy efficiency of the cycle; that will be done in the flowsheeting effort. Components can and should, however, be selected which can potentially perform efficiently on a large scale. For instance, a flow reactor may be selected over a stirred vessel. It is also not the object of the bench-scale work to prove materials of construction; wherever possible, materials with known corrosion resistance, such as inert plastics and glass, will be used.

For these investigations, the process is divided into three subunits, which, after they have been proven to operate satisfactorily alone, can then be linked together. The design goal of the bench-scale system is an H_2 production rate of approximately 17×10^{-6} m^3/s (at normal temperature and pressure). The three subunits are described below.

THE MAIN SOLUTION REACTION SUBUNIT-In this unit, the main solution reaction

$$2H_2O+SO_2+xI_2 \quad H_2SO_4+2HI_x \tag{1}$$

is performed, the two acids H_2SO_4 and HI_x are separated, and unreacted SO_2 is removed from the HI_x phase. Currently, design and construction are complete except for the HI_x degassing and SO_2 recycle systems. Figure 5 presents a flowdiagram of the subunit.

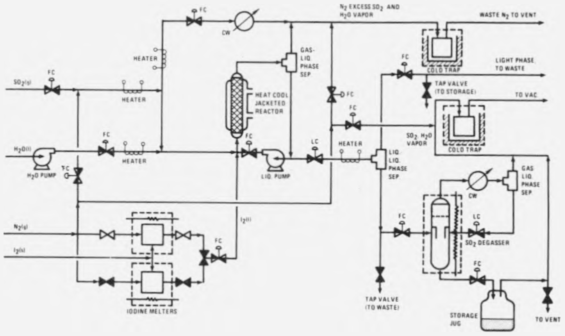

Fig. 5 - Subunit I, main solution reaction (simplified bench-scale flowsheet)

The iodine is delivered as a liquid from either of two 4-liter iodine supply vessels. Its delivery rate is measured with a manometer-orifice device, and it enters the reactor downstream of metered supplies of H_2O and SO_2. The combined feeds move upward into the entrance of the reactor, where the HI_x and H_2SO_4 are created during the residence of the fluids.* The temperature is stabilized by a cooling jacket around the mid-section of the reactor. The reaction effluent passes into a gas separation chamber where excess SO_2, which drives the fluids through the reactor, is separated from the liquid stream and bled to a recycle system (not yet installed). The two liquid phases are taken from the bottom of the gas-liquid separator and fed into a central point in a liquid-liquid separator. The lighter H_2SO_4 phase is drawn off the top of this separator while the heavier HI_x phase is drawn off the bottom. Figure 6 shows the reactor and the phase separators during a cold testing phase with simulating fluids.

Construction of the unit shown in Figure 6 has been extended since the picture was taken.

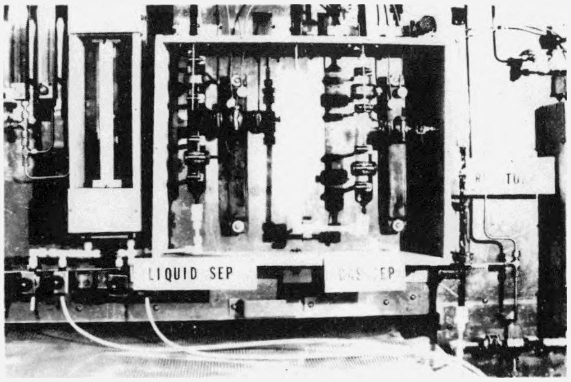

Fig. 6 - A closeup of the reactor and separator sections of bench-scale subunit No. 1

The iodine supply vessel, the inlet iodine lines, and the exit lower phase lines are currently contained in ovens with appropriate windows for viewing. Heating and control systems were installed and the unit was tested with water for temperature control. At the time of writing, the unit has been run with I_2, and H_2SO_4 and HI_x products have been made. This has qualitatively proven the operating concept of the main solution reaction. Experimental results still need to be evaluated, however, before quantitative conclusions can be drawn.

THE H_2SO_4 SUBUNIT-This subunit will concentrate H_2SO_4, decompose it at high temperatures (up to 1144K) and separate undecomposed H_2SO_4 and the reaction products H_2O, SO_2 and O_2. Figure 7 shows a flowsheet of the subunit. H_2SO_4 feed received from the main solution reaction subunit, or prepared in a special make-up section, is

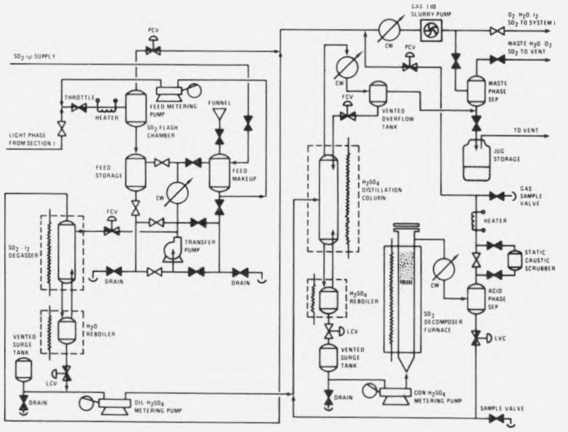

Fig. 7 - Subunit II H_2SO_4-H_2O separation and decomposition (simplified bench-scale flowsheet)

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*The required mixing is accomplished by alternating spiral glass vanes.

sent to an SO_2-I_2 degasser which removes SO_2, I_2, HI and some H_2O. The more concentrated acid is then fed into a distillation column where azeotropic H_2SO_4 is produced. In the SO_3 decomposer, the concentrated H_2SO_4 is boiled and heated to approximately 1144K. In the last part of the decomposer, a catalyst can be placed to facilitate the SO_3 decomposition. Products then are cooled with water condensing the undecomposed H_2SO_4 which is recycled to the still. Wet SO_2-O_2 product may then either be crudely analyzed by passing it through a caustic scrub prior to metering the residual gas to waste, or the total gas may be recycled to subunit I without removal of SO_2.

This subunit has been designed and construction and testing of it is expected in 1978.

THE HI SUBUNIT-This subunit will separate HI out of the lower phase product of the main solution reaction and thermally decompose it into H_2 and I_2. The conditions for these steps have not yet been fixed; however, H_3PO_4 will be used for the HI separation step and the HI decomposition will be done catalytically at moderate temperatures. This subunit will be designed in 1978.

It is projected that the first operation of the subunits as an integral benchunit will take place in 1979. After that, various experiments are planned which will explore the operational conditions of the main solution reaction, will test catalysts for decomposition of HI and H_2SO_4, will test oxygen and hydrogen cleanup systems, and will test possible novel concepts which by then may have emerged from continuing parallel laboratory experiments. Although not an initial objective of the bench-scale operation, it is planned to eventually perform corrosion experiments with candidate construction materials obtained from screening tests.

CONCLUSIONS

The sulfur-iodine thermochemical water-splitting cycle appears to be a promising approach to producing hydrogen from nonfossil sources. It has heat requirements that match very well the thermal output capability of the HTGR, and it can be conducted as an all-fluid process, which is a characteristic that should give it considerable engineering advantage over a cycle requiring solids handling.

A new process engineering flowsheet is being developed which will incorporate recently obtained chemical data and revised processing methods. It is expected that this flowsheet will result in an overall thermal efficiency for the process of 45% or greater.

A continuous-flow bench-scale unit is under construction and operation is expected in 1979. The engineering and bench-scale investigations will give a better insight into the technical feasibility of the sulfur-iodine cycle and will give valuable guidance to future chemical and materials investigations.

ACKNOWLEDGEMENT

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