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## **DESIGN OPTIONS FOR A BUNSEN REACTOR**

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# DESIGN OPTIONS FOR A BUNSEN REACTOR

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## Abstract

This work is being performed for Matt Channon Consulting as part of the Sandia National Laboratories New Mexico Small Business Assistance Program (NMSBA). Matt Channon Consulting has requested Sandia's assistance in the design of a chemical Bunsen reactor for the reaction of  $\text{SO}_2$ ,  $\text{I}_2$  and  $\text{H}_2\text{O}$  to produce  $\text{H}_2\text{SO}_4$  and  $\text{HI}$  with a  $\text{SO}_2$  feed rate to the reactor of 50 kg/hour. Based on this value, an assumed reactor efficiency of 33%, and kinetic data from the literature, a plug flow reactor approximately 1" diameter and 12 inches long would be needed to meet the specification of the project. Because the Bunsen reaction is exothermic, heat in the amount of approximately 128,000 kJ/hr would need to be removed using a cooling jacket placed around the tubular reactor. The available literature information on Bunsen reactor design and operation, certain support equipment needed for process operation and a design that meet the specification of Matt Channon Consulting are presented.

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## EXECUTIVE SUMMARY

This work is being performed for Matt Channon Consulting as part of the Sandia National Laboratories New Mexico Small Business Assistance Program (NMSBA). Matt Channon Consulting has requested the design of a Bunsen reactor with a SO<sub>2</sub> feed rate of 50 kg/hour. The unit is to be used to produce HI.

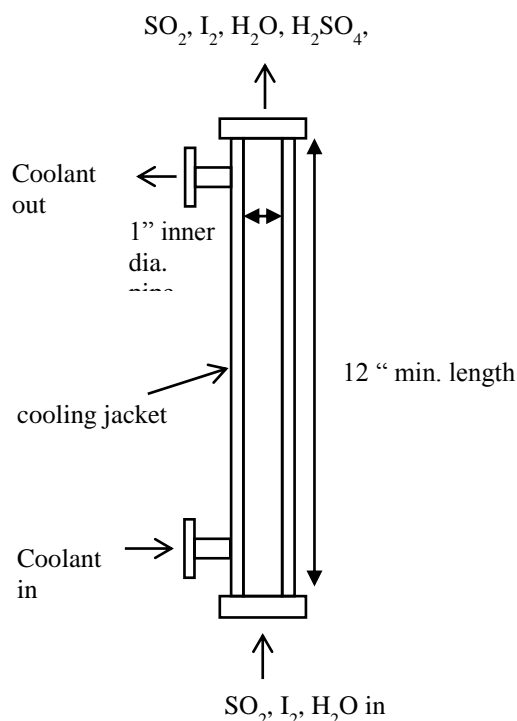
The Bunsen reaction utilizes SO<sub>2</sub>, I<sub>2</sub> and H<sub>2</sub>O to produce H<sub>2</sub>SO<sub>4</sub> and HI at a temperature of approximately 60 to 160°C. The reaction is given by:



The positive value of the Gibbs energy indicates the reaction is not thermodynamically favorable and therefore a large excess of I<sub>2</sub> and H<sub>2</sub>O is needed for the reaction to proceed as governed by Le Chatelier's Principle. Even with this large excess, 33% conversion is the highest value that can be achieved.

The Bunsen process can be performed in either a tubular or batch type reactor. Based on the low input rate of 50 kg/hr specified by Matt Channon Consulting it is recommended that a small tubular reactor would be the best option. This is based on the significantly lower cost of a small tubular reactor compared to a batch system, component availability and simplicity of process operation.

The reactor can be constructed using a section of glass lined steel pipe 1" in diameter and at least 12" in length. A cooling jacket can be installed around the pipe section to provide cooling. The design is given below.



# 1 INTRODUCTION

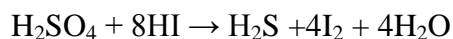
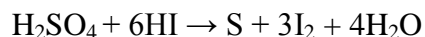
This project involves providing background information and design options for the construction of a Bunsen reactor. The work is being performed for Matt Channon Consulting as part of the Sandia National Laboratories New Mexico Small Business Assistance Program (NMSBA). Matt Channon Consulting has requested a Bunsen reactor with a SO<sub>2</sub> feed rate of 50 kg/hour. Literature data on Bunsen reaction kinetics, thermodynamics, design option for tubular and batch type Bunsen reactors are presented.

The Bunsen reaction uses SO<sub>2</sub>, I<sub>2</sub> and H<sub>2</sub>O to produce H<sub>2</sub>SO<sub>4</sub> and HI in an exothermic process at a temperature of approximately 60 to 160°C. The reaction is given by:



The reaction is not thermodynamically favorable and a large excess of I<sub>2</sub> and H<sub>2</sub>O is needed for the reaction to proceed as governed by Le Chatelier's Principle. Even with this large excess, 33% conversion is the highest value that can be achieved.

Under certain conditions, such as operating the Bunsen reactor at a high temperature or with improper reactant concentrations, side reactions can result in undesirable byproducts. These are given by the equations:



Operating conditions to minimize the formation of byproducts are discussed.

The Bunsen process can be performed in either a tubular or batch type reactor. The products of the Bunsen reaction exist in two phases; a light and a heavy phase. These mixtures must pass through several separation processes to produce high purity HI that is needed by Matt Channon Consulting. The separation processes have typically been performed using distillation, however membrane separation process have been investigated.

Based on the low input rate of 50 kg/hr specified by Matt Channon Consulting it is recommended that a small tubular reactor would be the best option. This is based on the significantly lower cost of a small tubular reactor compared to a batch system, component availability and simplicity of process operation. Separation processes for achieving high purity HI are discussed.

## 2 BACKGROUND

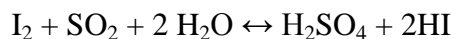
### 2.1 Bunsen Reactor Thermodynamic

Using the Gibbs energy for the Bunsen reaction, +82 kJ/mol at 400K the equilibrium constant can be calculated to be equal to  $1.96 \times 10^{-11}$ . The positive Gibbs energy and resulting very small equilibrium constant indicate the reaction is not thermodynamically favorable for the formation of the products. However, if excess reactants of H<sub>2</sub>O and I<sub>2</sub> are provided the reaction will proceed to the right as governed by Le Chatelier's Principle. The addition of excess I<sub>2</sub> and H<sub>2</sub>O also allows for the separation of the products into a light phase consisting mainly of H<sub>2</sub>SO<sub>4</sub> and a heavy phase containing I<sub>2</sub> and HI. (Hadj-Kali et al, 2009) Additionally, the use of excess I<sub>2</sub> can prevent the formation of undesirable byproducts.

Different values are reported for the enthalpy of reaction in the literature for the Bunsen reaction. Brown et al., (2003) reports a value of  $\Delta H = -216$  kJ/mole. Kubo et al., (2004) reports a value of -165 kJ/mole SO<sub>2</sub> consumed. A calculation performed with the HSC Chemistry software gives a value of -164 kJ/mole SO<sub>2</sub> consumed that is in agreement with the value reported by Kubo et al., (2004). This calculation assumes all reactants are at the reaction temperature of 120°C when introduced into the reactor.

### 2.2 Bunsen Reaction Kinetics and Stoichiometry

Kinetics of the Bunsen reaction are reported to be fast. Equilibrium can be established in 1 – 3 sec. The Bunsen reaction takes place at approximately 60-140°C and is exothermic. Therefore, heat must be continuously removed from the process. Brown (2012) states “Reaction kinetics appear to be a function of how fast heat can be removed from the system.” Therefore, Bunsen reactor design is very flexible and the process can be easily performed in a batch process or continuous flow process including a continuously stirred tank reactor (CSTR) or a tubular flow reactor. The reactants, SO<sub>2</sub>, H<sub>2</sub>O and I<sub>2</sub> are all preheated to the reaction temperature before introduction into the reactor. I<sub>2</sub> must be pumped into the reactor above its melting temperature of 113.7°C. SO<sub>2</sub> can be introduced as either a gas or liquid. Stoichiometrically the reaction is given by:



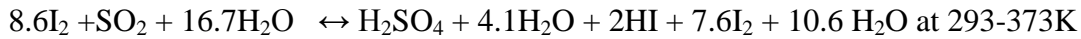
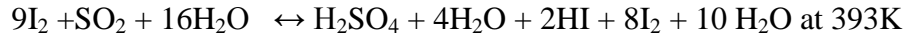
However, as previously stated, additional H<sub>2</sub>O and I<sub>2</sub> need to be added to the feed stream.

Lee et al., 2007 examined the optimal operating window for the Bunsen process. The study used existing experimental data from the literatures. Table 2-1 gives the results of the study. According to the authors the optimal conditions are consist of 4 moles of excess I<sub>2</sub> and 11 moles



of excess H<sub>2</sub>O over the stoichiometric requirements for a reaction temperature of 57°C and 4 - 6 moles excess I<sub>2</sub> and 11 - 13 excess moles H<sub>2</sub>O at 77°C. Even using these optimum values significant impurities were encountered in the light and heavy product phases.

The feed operating conditions proposed by Norman et al. (2004) are given by:



Lee recommends using the 4 moles excess of I<sub>2</sub>, 11 mole excess of H<sub>2</sub>O at 330K and 4-6 moles excess I<sub>2</sub> and 11 - 13 mole excess of H<sub>2</sub>O at 330 – 350K



**Table 2-1: Recommended Values for Feed to Bunsen Reactor**

Parameter	Reference					
	Norman et al., (1981)	Norman et al., (2004)	Kubo et al., (2005)	Giaconia et al., (2006)	Lee et al., (2008)	Lee et al., (2008)
Temperature (K)	293-373	393	343	353	330	330-350
x	7.6	8	4.4	3.57	4	4-6
m + n	14.7	14	24.6	11	11	11-13
m	4.1	4	8.9	-	5	5
n	10.6	10	15.7	-	6	6-8

## 2.3 Bunsen Reactor Design and Operation

Several design options and operational parameters are reported in the literature for Bunsen reactors. Cho et al. (2009) present a conceptual design for Bunsen reactor for Sulfur-Iodine (S-I) process for production of H<sub>2</sub>. The authors present the results from several different designs including the use of an electrodialysis cell to concentrate HI.

Hadj-Kali et al. (2009) present a thermodynamic model for the Bunsen section of an S-I cycle. The model is based on using the UNIQUAC activity coefficient model with Engel's solvation model. The complete model is discussed and utilizes nearly 100 parameters. A 15 parameters model is proposed after defining new apparent chemical species.

Parisi et al. (2011) performed experiments to determine the effect of temperature and reagent concentration on the Bunsen reaction. Experiments were performed using a semi-batch procedure by feeding gaseous  $\text{SO}_2$  in to an iodine/hydriodic acid/water solution.

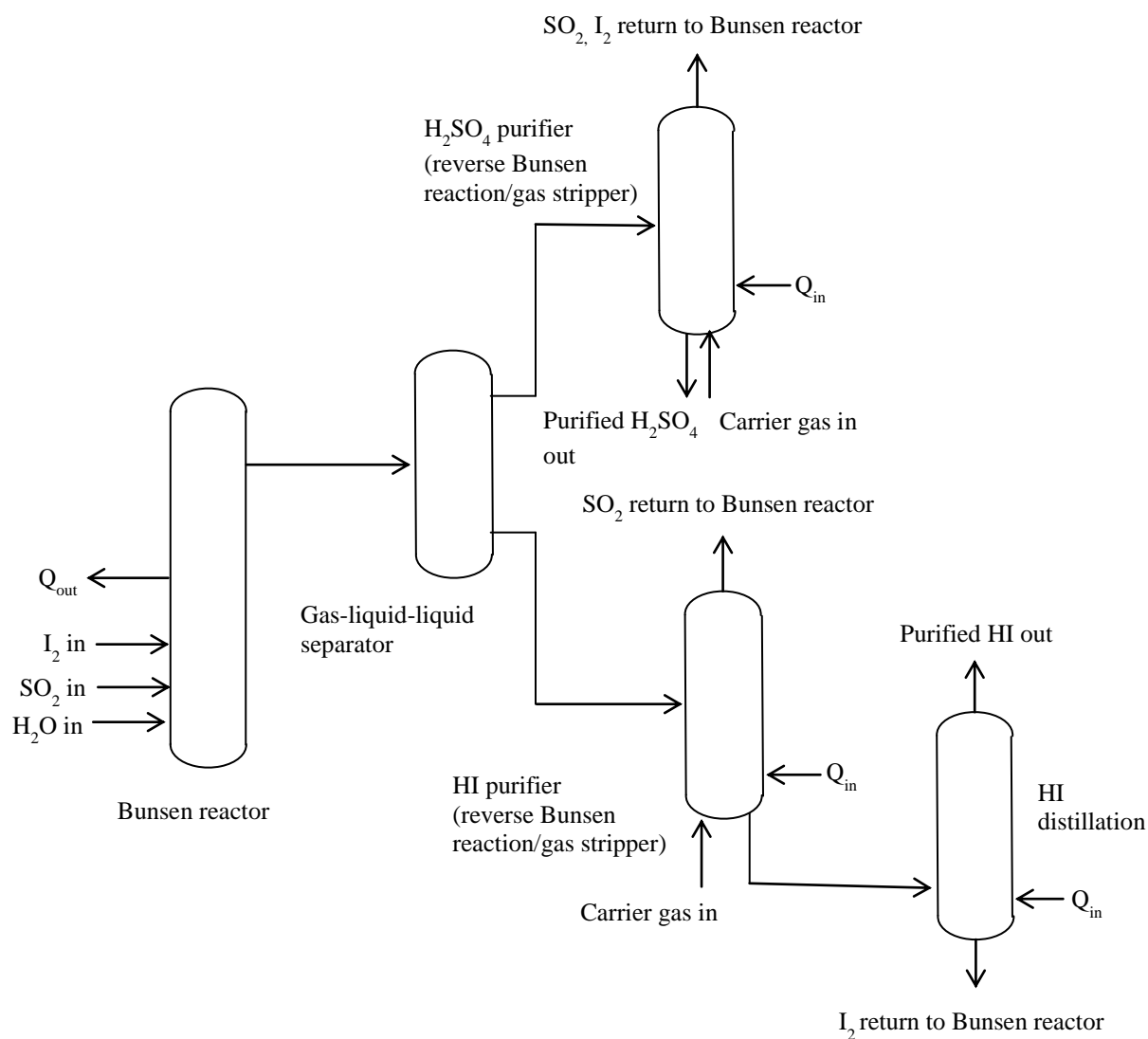
A survey of alternative routes for performing the Bunsen reaction other than the use of excess  $\text{H}_2\text{O}$  and  $\text{I}_2$  in the process is given by Giaconia et al. (2009). The alternatives include 1. Use of a solvent other than water, 2. The addition of a precipitating agent to separate iodine from sulfate and 3. performing the Bunsen reaction in an electrochemical cell. The authors performed experiments to evaluate the use of these alternative methods. Unfortunately, there is not enough information to date on these alternative methods for practical applications.

Giaconia et al., (2007) studied the phase separation process in the Bunsen process. The authors performed experiments in 50 mL jacketed quartz vessel at temperatures of 80, 95 and 120°C.  $\text{HI}$ ,  $\text{I}_2$ ,  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{O}$  were mixed in the vessel and maintained at the experimental temperature for 1 hour for the gravimetric separation process to proceed to completion. Both phases were then analyzed. The authors concluded that temperature and  $\text{I}_2$  content have little effect on the sulfuric acid and  $\text{HI}$  concentrations in the two phases. Increasing water content leads to dilution of the acid whereas  $\text{HI}$  concentration in the heavy phase is always well over the azeotropic point provided the distillation is performed at pressure of 22 bar or greater. The purity of both phases was not significantly affected by iodine content and temperature.  $\text{I}_2$  and  $\text{HI}$  in the sulfuric acid phase was always 1-2% of the total, while sulfates in the hydriodic acid phase are always more than 15% of the total. The authors did not observe the formation of byproducts provided that the reagents were initially mixed at 80°C. The authors state the smallest  $\text{I}_2$  content should be 3.9 mole  $\text{I}_2$ /mole  $\text{H}_2\text{SO}_4$  to prevent byproduct formation. At a temperature of 80°C, recommended by the authors, the initial molar fractions of  $\text{HI}/\text{H}_2\text{O}/\text{I}_2/\text{H}_2\text{SO}_4 = 0.11/0.61/0.22/0.06$ .

## 2.4 Processing Operations and Options

Products from the Bunsen reactor require multiple processing operations to obtain high purity  $\text{HI}$ . A simplified schematic diagram of the Bunsen reaction and subsequent separation and purification operations is given in Figure 2-1.  $\text{I}_2$ ,  $\text{SO}_2$  and  $\text{H}_2\text{O}$  are fed into the Bunsen reactor operating at a temperature between 60 to 160°C. Kinetically the reaction is fast and two liquid phases are rapidly formed. Both the light and heavy liquid phases,  $\text{H}_2\text{SO}_4$  and  $\text{HI}$  rich, respectively, are transferred to a liquid-liquid separator. The light and heavy phases are then transferred to purification processes to remove any unreacted reagents. For both light and heavy phases this is accomplished by employing the reverse Bunsen reaction and use of a carrier gas to separate out the impurities. For the heavy or  $\text{HI}$  rich phase, an additional purification process, typically distillation, is required to produce a high purity  $\text{HI}$ . Although the Bunsen process itself is more than 150 years old, the construction and optimization of the Bunsen process and

subsequent separation/purification processes are still under investigation.



**Figure 2-1 Simplified Schematic Diagram of the Bunsen reaction Including Separations Processes**

## 2.5 Materials of Construction

$H_2SO_4$ ,  $SO_2$  in the presence of water, HI, and  $I_2$  are all extremely corrosive to most metals. This is particularly true when these chemicals are at elevated temperature such as those in the Bunsen process. Experience with these compounds in the laboratory-scale Sulfur-Iodine process performed at the General Atomics facility in San Diego, CA indicated the most corrosion

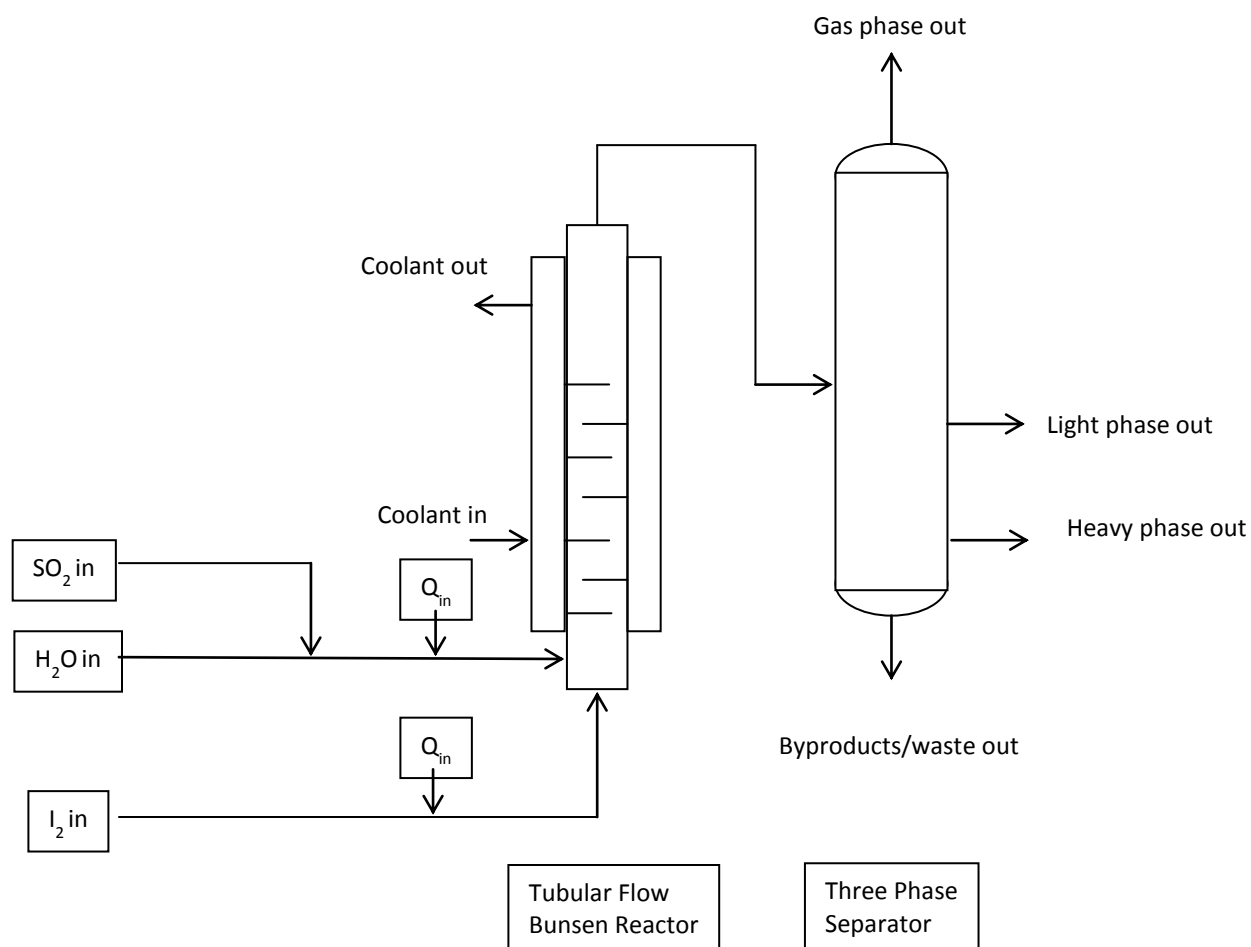
resistant materials for  $\text{H}_2\text{SO}_4$  and wet  $\text{SO}_2$  were Teflon, silicon carbide and glass. All metal alloys tested with the exception of tantalum including steels and nickel based materials suffered severe corrosion. It was determined in the operation of a demonstration scale Sulfur-Iodine process for  $\text{H}_2$  production that glass preforms significantly better than Teflon linings for handling  $\text{I}_2$  and  $\text{SO}_2$ . Teflon is permeable to both of these compounds. (Moore et al., 2012; Russ et al., 2012) Teflon and glass lined piping and components are commercially available.

### **3 DESING OPTIONS**

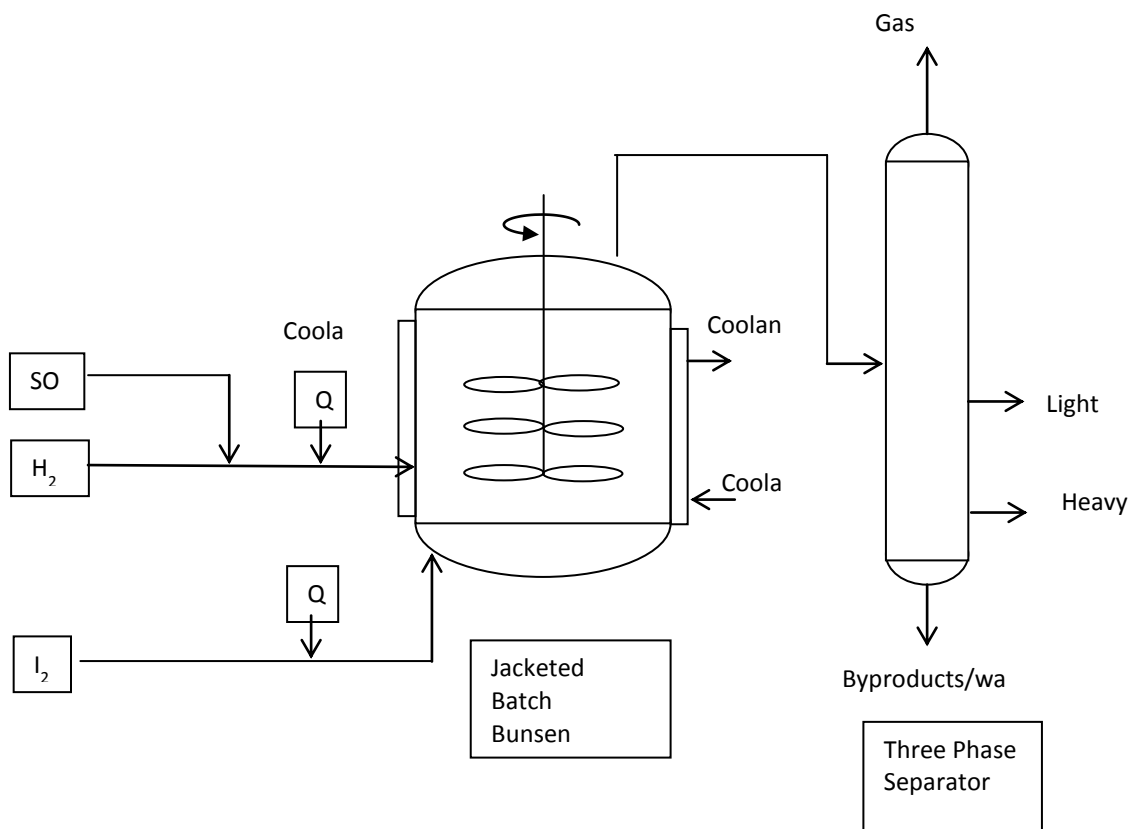
#### **3.1 Bunsen Reactors**

The two options for a Bunsen reactor are given in Figures 3-1 and 3-3. Figure 3-2 is a schematic drawing of a tubular reactor. These units can be easily constructed using pipe sections with a jacket for cooling or heating. Figure 3-3 is a schematic drawing of a batch type chemical reactor or continuously stirred tank reactor (CRTS) for use as a Bunsen reactor. These units are commercially available in different sizes including small volume units for pilot plant process.

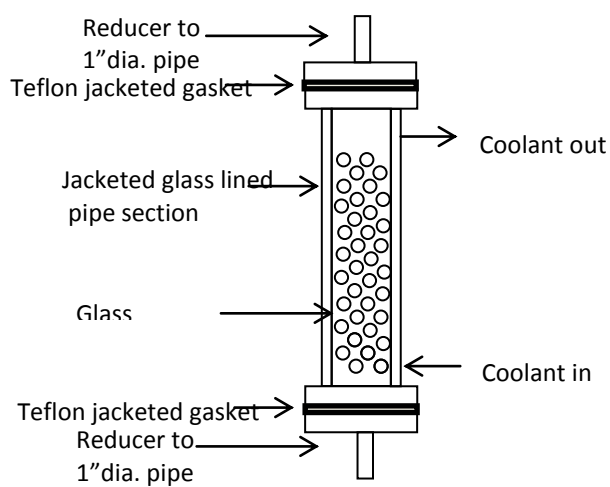
There are advantages and drawbacks to each approach for the Bunsen reaction. A tubular reactor can be constructed using a glass lined steel pipe section with a cooling jacket. Since the reaction temperature is above the boiling point of water, an oil would be a better selection for a cooling fluid. The reactor does not have to be equipped with fins to enhance mixing as shown in Figure 3-2. A packing material such as glass rings can be used instead to provide mixing. An example is given in Figure 3-4. Such a unit would be easy to construct using off the shelf components and relatively inexpensive.



**Figure 3-1: Tubular Flow Bunsen Reactor and Three Phase Separator**



**Figure 3-2: Bunsen CSTR with Three Phase Separator**



**Figure 3-3: Bunsen Reactor Constructed from a Jacketed Glass Lined Pipe Section.**

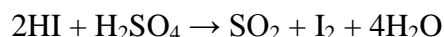
### 3.2 Liquid-Liquid Separator

Separation of the two liquid phases and the gas phase from the Bunsen reaction can be accomplished with a simple glass lined gas-liquid-liquid separator. These unit are no more than a glass lined tank with a vent to purge any gas that may remain in the system. These units are commercially available.

### 3.3 Purification Units

#### 3.3.1 Reverse Bunsen Reaction and Gas Stripping

To remove impurities, the reverse Bunsen reaction is use to convert impurities in each phase back to reactants for the Bunsen reaction and these compounds are stripped from the solution using a carrier or sweeping gas. The reverse Bunsen reaction is given by:



Gao et al., (2007) investigated the purification processes for the light and heavy phases formed by in the Bunsen reaction. Experiments were performed in a packed column 34 cm in length by 4 cm diameters. The packing was small glass-ring fillings, 0.3cm x 2 cm. The mixtures were fed into the top of the column and purified product exited at the bottom. The nitrogen sweeping gas was fed at the bottom of the column. The results indicate for the light or  $\text{H}_2\text{SO}_4$  phase with a feed rate of 1.6 ml/min (96 mL/hr) the optimum gas flowrate and temperature for removing the impurities were 50 mL/min and 140°C, respectively. For the heavy or HI phase with the feed rate of 0.5 to 1.0 mL/min. the best results were obtained at a temperature of 160°C and a gas flowrate of 200 ml/min. In summary, for the light phase a relatively low flow rate of sweeping gas and a temperature of 140°C are desirable whereas for the heavy phase a higher temperature of 140-160°C and significantly high gas flowrate are required.

The specific units for performing the reverse Bunsen reaction and gas stripping can be constructed of commercially available components. The tank is a glass lined steel tank with a jacket for providing the heat transfer fluid to maintain the process temperature at the optimum value. The column is packed with glass or ceramic Rasching rings to provide good contact between the liquid and stripping gas. The stripping gas can be  $\text{N}_2$  or Ar. The stripping gas and feed should be preheated before entering the separation tank. A simple glass lined heat exchanger can be used to preheat the feed.

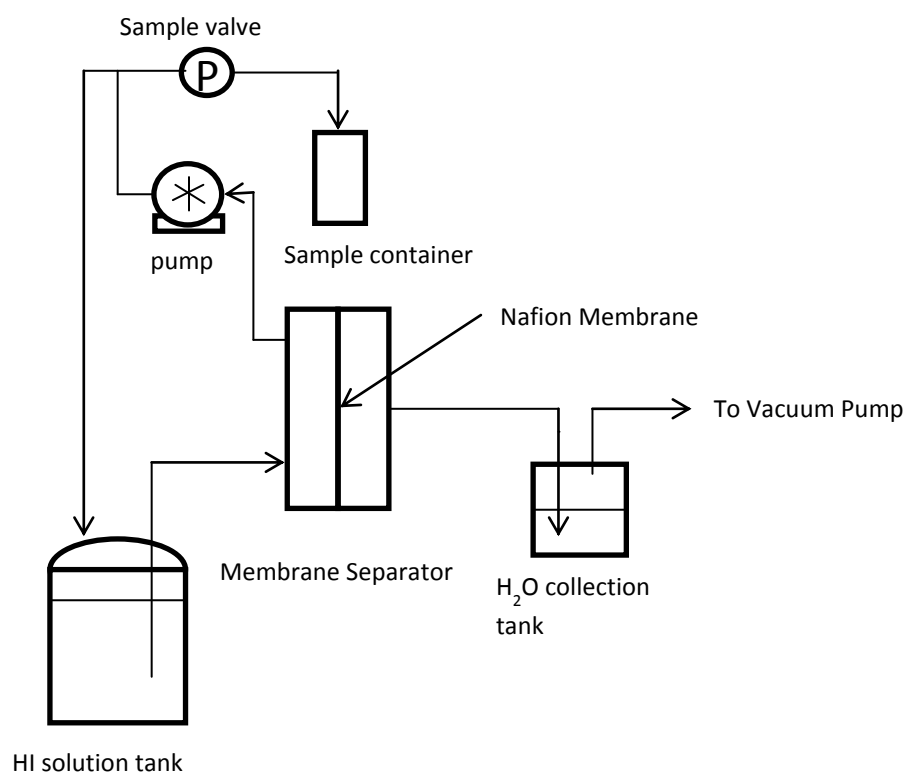
### 3.4 Membrane Separation of HI phase

H<sub>2</sub>O in the HI must be removed before further processing can be performed. This has been performed in the past by distillation. Because HI and H<sub>2</sub>O form an azeotrope at 57wt/o HI, a chemical must be added to break the azeotrope for complete removal of the H<sub>2</sub>O from the HI phase. This is accomplished by adding concentrated phosphoric acid to the HI phase before distillation. This process is highly energy intensive and involves the use of another highly corrosive chemical.

An alternative to distillation with phosphoric acid is the use of membrane separation. Membrane separation is a relatively new process for removing H<sub>2</sub>O from the HI phase. Elder et al. (2009) give the results from a simulation using a membrane separation step for H<sub>2</sub>O removal. The authors report an efficiency as much as 50% could be achieved however, no experiments were performed in the study to validate the model. This is significantly lower than what is required in the present project for a target value of >99% pure HI.

Elder et al. (2009) performed batch and continuous flow experiments for removing H<sub>2</sub>O from HI using Nafion 117 and Nafion 212 membranes. A schematic drawing of the equipment is given in Figure 3-4. Experiments were performed from 50°C to 120°C. In both batch and continuous experiments the flux of water through the membranes was high and separation factors as high as 20,000 were measured. The permeate was analyzed and determined to be almost pure H<sub>2</sub>O. Separation factors were significantly higher at 100°C than at 50°C. The authors report operating at 120°C with an azeotropic feed composition gives almost pure water with a flux rate of 0.7 kg/m<sup>2</sup>h using Nafion 117 and 0.45 kg/m<sup>2</sup>h. Using Nafion 212 the flux increases to 1.35 kg/m<sup>2</sup>h.

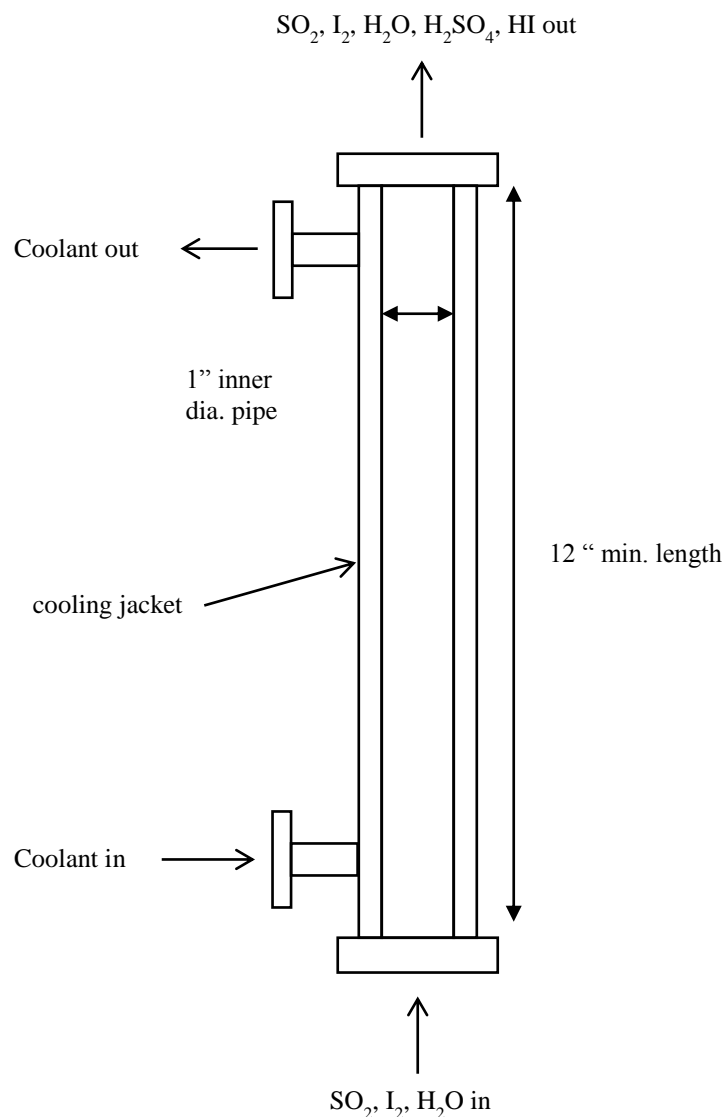




**Figure 3-4: Experimental Set Up For Membrane Separation of H<sub>2</sub>O from HI Solution (Elder et al. (2009))**

## 4 PROCESS RECCOMENDATIONS

Based on the low flowrate of  $\text{SO}_2$  into the Bunsen reactor specified by Matt Channon Consulting a tubular flow reactor is likely a better choice over a CSTR. A unit to meet the specifications of Matt Channon Consulting is given in Figure 4-1



**Figure 4-1: Tubular Bunsen reactor with cooling jacket.**

The unit is constructed using a glass lined pipe section equipped with a cooling jacket. Based on the design specification of 50 kg/hr SO<sub>2</sub> the unit needs to be 1” in diameter and a minimum of 12” in length. The materials of construction for the cooling jacket will depend on the coolant selected.

Using a SO<sub>2</sub> feed rate of 50 kg/hr to the Bunsen reactor and the optimized values reported by Lee et al (2008) for Bunsen reactor operation at 330K, the feed composition, output composition, recycle stream and fresh feed required can be calculated by mass balance. The values are given in Table 4-1.

**Table 4-1: Feed Rate and Output to and from Bunsen Reactor Based on 50 kg/hr SO<sub>2</sub> Feed Rate and Values Recommended by Lee et al., (2008) for Operation at 330K (57°C) with Excess I<sub>2</sub> and H<sub>2</sub>O.**

Species	Feed in to reactor (kg/hr)	Output from reactor (kg/hr)	Recycle From reactor (kg/hr)	Fresh Feed to reactor (kg/hr)
I <sub>2</sub>	991.4	925.9	925.9	65.5
SO <sub>2</sub>	50	33.5	33.5	15.5
H <sub>2</sub> SO <sub>4</sub>	-	25.26	-	-
HI	-	65.9	-	-
H <sub>2</sub> O	182.8	173.5	173.5	9.3

These values can be used to calculate the heat requirements for the Bunsen reactor and design of the separation processes.

## 5 CONCLUSIONS

This work is being performed for Matt Channon Consulting as part of the Sandia National Laboratories New Mexico Small Business Assistance Program (NMSBA). Matt Channon Consulting has requested Sandia's assistance in the design of a chemical Bunsen reactor for reaction of  $\text{SO}_2$ ,  $\text{I}_2$  and  $\text{H}_2\text{O}$  to produce  $\text{H}_2\text{SO}_4$  and  $\text{HI}$ . Matt Channon Consulting requested a Bunsen reactor with a  $\text{SO}_2$  feed rate of 50 kg/hour. Based on this value, an assumed reactor efficiency of 33%, and kinetic data from the literature conversion, a plug flow reactor approximately 1" diameter and 12 inches long would be needed. Because the process is exothermic heat in the amount of 128,084 kJ/hr would need to be removed using a cooling jacket placed about the tubular reactor.

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