CATALYTIC DECOMPOSITION OF HYDROGEN SULFIDE

By

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PREFACE

The feasibility of the decomposition of hydrogen sulfide using molybdenum as a catalyst was investigated and determined to be technically feasible. Some insight into the kinetics of the reaction was also obtained. The reaction was found to be a first order reaction.

I would like to express my sincere thanks to my major adviser, Dr. John H. Erbar, for his unfailing interest, excellent counseling, and continuous encouragement during the course of this study. My thanks too, to the School of Chemical Engineering, for giving me the opportunity to carry out this work.

I am very grateful to the Branns and the Rogers for their encouragement and making my stay at the University a truly pleasant and memorable one. I am also very grateful for all the help rendered me by the graduate students of the School of Chemical Engineering.

Last, but in no ways least, I am deeply indebted, and will always be, to my parents for giving me this opportunity to broaden my horizons.

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CHAPTER I

INTRODUCTION

The purpose of this investigation was to study the feasibility of the catalytic decomposition of hydrogen sulfide using molybdenum as a catalyst. This reaction was studied because of the increasing importance of air pollution control and the feasibility of a process to recover hydrogen and sulfur from the decomposition reaction. Current processes for hydrogen sulfide recover only sulfur. Thus "two birds are killed with one stone", that is, the waste disposal problem involving hydrogen sulfide is solved and hydrogen and sulfur, which are potentially useful products are recovered.

The disposal of hydrogen sulfide as a waste gas is under strict control by air pollution authorities. In metropolitan areas, for example, emission of hydrogen sulfide can be sufficient cause for shutting down a plant (9). The reasons for these strict controls are:

1. The gas is foul smelling.
2. The gas is toxic, the maximum allowable concentration of H_2S in the atmosphere is 15 mg/m^3 (22).

Another reason for this study was the useful nature of the products of the reaction. Hydrogen is a raw material for many process industries notably the ammonia fertilizer and the petrochemical industry. Liquid hydrogen has strategic importance as a rocket fuel. Sulfur, too, has well established uses: the manufacture of sulfuric acid, pharmaceuticals, tires, explosives, paper and many others.

CHAPTER II

THEORETICAL BACKGROUND

Literature Survey

The removal of hydrogen sulfide from sour natural gas and the subsequent conversion to sulfur, has been carried out for a number of years. Some of the important processes used for the removal of hydrogen sulfide from sour gas are the Amine Process, the Phosphate Process, and the Hot Carbonate Process (7). In these processes, the reagent and the hydrogen sulfide are regenerated. The hydrogen sulfide recovered is then converted into elemental sulfur.

The processes employed for the recovery of sulfur may be broadly classified as:

(1) semi-catalytic processes

(2) catalytic processes

The Partial-Combustion Process and the Split-Stream Process or the Claus Process are the most widely used semi-catalytic processes in the United States (7).

In the Partial-Combustion Process, the hydrogen sulfide is burned at a high temperature (1800 - 2400°F) in a combustion furnace with sufficient air to convert all the hydrogen sulfide to sulfur. The burning step is followed by catalytic oxidation at lower temperatures (400 - 700°F) to improve the yield. The preferred catalyst is bauxite or aluminum oxide (1). Other catalysts which have been tried are alkali sulfides on diatomaceous earth, clays, carbonaceous clays and titanium oxide.

In the Split-Stream Process (23), one-third of the hydrogen sulfide is burned completely to sulfur dioxide in a combustion process. This is then mixed with the other two-thirds of the hydrogen sulfide gas and burned over a catalyst to produce elemental sulfur and water. The mixed gases enter the first catalyst chamber at 400 - 700°F. In the catalyst converter, although the combustion reaction liberates 19,000 - 31,500 Btu per pound mole of hydrogen sulfide, the temperature rise is small. For the best recovery of sulfur, the converters are operated at a temperature slightly above the dew-point of the sulfur in the gases. Increasing the temperature, decreases the yield of sulfur. Bauxite is used as the catalyst.

Other processes which are known to be used for the recovery of sulfur from hydrogen sulfide are the Thylox Process (5), the Giammarco-Vetrocoke Process (7), the Townsend Process (18), and the Katasulf Process (12). In the Thylox Process, hydrogen sulfide is absorbed in a thioarsenate solution, which on regeneration produces elemental sulfur by oxidation. In the Giammarco-Vetrocoke Process, an alkaline solution of a mixture of arsenites and arsenates is used to absorb the hydrogen sulfide. This is then converted to monothioarsenate by lowering the pH of the solution. The monothioarsenate is then oxidized to produce elemental sulfur and simultaneously regenerate the arsenate solution which is recycled to absorb fresh hydrogen sulfide.

The Townsend Process used for the recovery of sulfur from natural gas has high efficiency in the recovery of sulfur. In this process, sulfur is first burned with air to sulfur dioxide (when processing acid gas from a scrubbing unit, one-third of the hydrogen sulfide is burned to form the required sulfur dioxide). The sulfur dioxide is then absorbed in a hygroscopic organic liquid such as di- or triethylene glycol, with no more than 10% of the water as the catalyst and reaction medium. The sulfur dioxide containing organic liquid is then passed countercurrent to the sour natural gas where the hydrogen sulfide reacts to form elemental sulfur and water. The resulting liquid stream is then processed to recover the elemental sulfur.

In the Katasulf Process, the hydrogen sulfide is first catalytically oxidized with air to form sulfur dioxide. The sulfur dioxide is then washed with a solution of ammonium sulfite-bisulfite. The ammonium sulfite-bisulfite solution is then acidified and heated under pressure at 290°F to form sulfur and ammonium sulfate.

In all these processes, the hydrogen sulfide is absorbed in some solution, usually an arsenate solution, and the resulting solution is oxidized to produce sulfur. An important limitation of all these processes is that the hydrogen is lost as water.

The catalytic processes for the decomposition of hydrogen sulfide have either been studied to investigate the kinetics of the reaction or for mass spectrometric studies of species or, they suffer the same major limitation as the semi-catalytic processes namely, the loss of hydrogen. Taylor and Pickett (21) studied the decomposition of hydrogen sulfide over a platinum filament in a

Pyrex glass chamber at 1740 - 2320°F. Their studies were carried out to elucidate the mechanism of gaseous reactions. The study indicated that the decomposition of hydrogen sulfide did occur and that it increased as the gas flow rate was increased. The actual mechanism was shown to consist of the primary adsorption of hydrogen sulfide oriented with the sulfur atom to the platinum; a splitting of the adsorbed molecule; liberation of hydrogen and the subsequent evaporation of sulfur from the heated filament.

Blanchard and LeGoff (4) studied the decomposition of H_2S, CS_2, SO_2, and CCl_4 over pure and carburized tungsten ribbon at 1800 - 3100°F, to obtain mass spectrometric data on the CS, SO and CCl_2 species produced in primary heterogeneous reactions. The vapor was passed at a pressure of 10^{-5} mm.Hg. at the rate of 5 liters per second through a reaction chamber in which the tungsten ribbon was mounted. They concluded that the decomposition proceeded at a faster rate on the carburized ribbon than on the tungsten ribbon at all temperatures. However, the amount of sulfur produced was greater with the tungsten ribbon. This was attributed to the decarburization of the ribbon to produce hydrogen and carbon disulfide.

Bigeleisen (3), et al., reported that hydrogen sulfide can be decomposed at 1292°F (700°C) on a uranium catalyst. The reactor used consisted of a flow system in which the uranium turnings were heated by an external furnace. They studied the reaction for the purpose of converting the hydrogen sulfide to hydrogen for analysis in a mass spectrometer.

Kingman (11) studied the decomposition of hydrogen sulfide on a molybdenum filament. The filament was placed in a Pyrex reaction vessel about 10 inches long and 1 inch in diameter. The gas was passed at a low pressure (0.088 mm.Hg.) and at 750 - 1260°F (400 - 685°C). Analysis of the results showed that the reaction could be accurately represented by a monomolecular equation and that the decomposition of hydrogen sulfide on a molybdenum filament resulted in the formation of hydrogen and free sulfur. The rate of the reaction was shown to be proportional to the pressure of the hydrogen sulfide.

Another process (16) has been described in which hydrogen sulfide was converted to hydrogen over a tungsten filament; however, this process was used to purify hydrogen gas contaminated by hydrogen sulfide. A process (6) has also been described in which hydrogen sulfide was converted to sulfur over a catalyst which consisted mainly of Al_2O_3 and $Al_2(SO_4)_3$. In this process, as in the other processes, the hydrogen was converted to water.

Thermodynamics

In the investigation of the reaction, a preliminary study was made of the thermodynamics of the reaction.

The thermodynamics of the reaction

$$2H_2S(g) \rightleftarrows 2H_2(g) + S_2(g)$$

indicate that the decomposition reaction is highly unfavorable and would be feasible only at very high temperatures or in the presence of a catalyst. The standard free energy change of the reaction was +35.1 kcals/mole and the standard heat of the reaction was +40.6 kcals/mole (see Appendix B for standard free energies and heats of

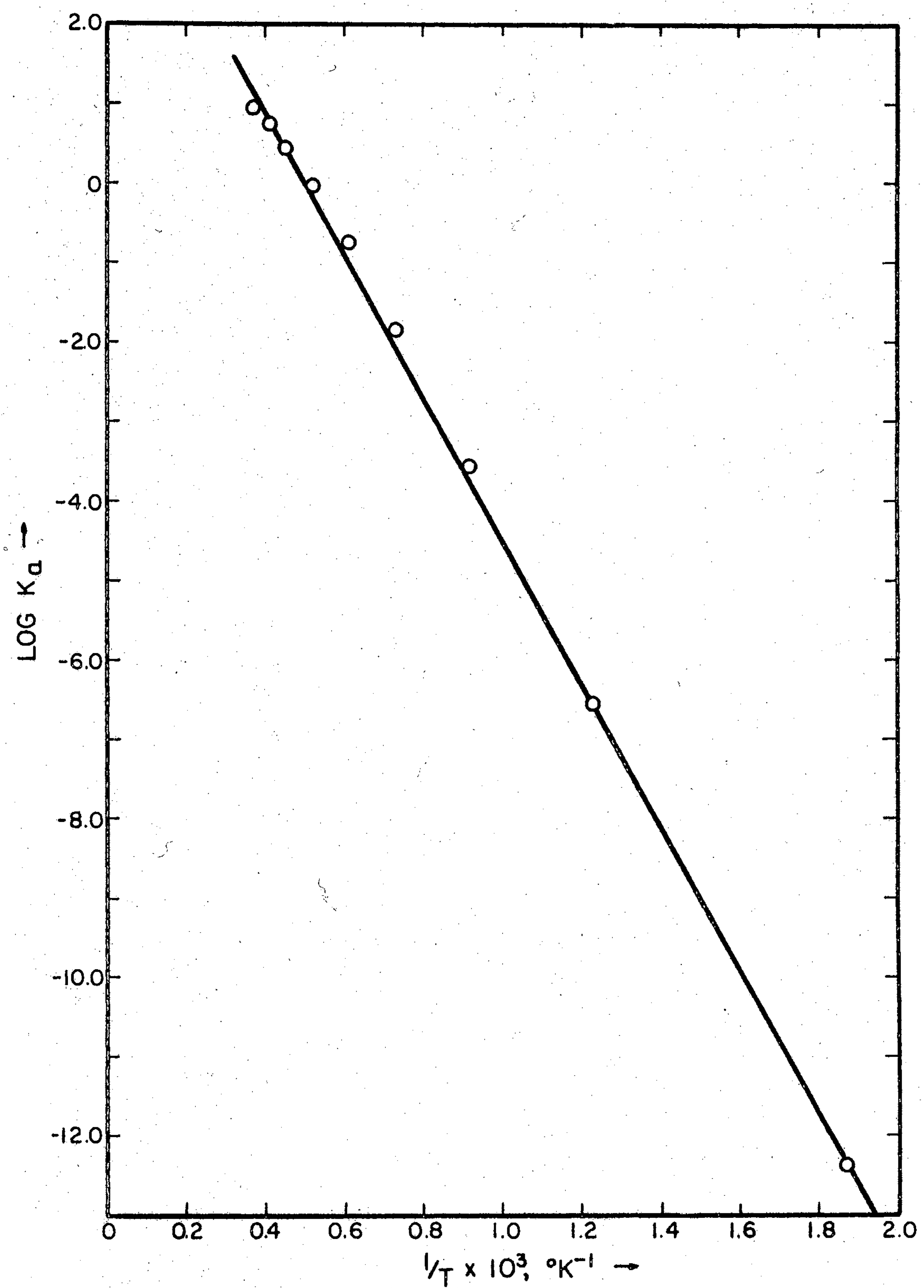

Figure 1. Effect of Temperature on the Equilibrium Constant for the Hydrogen Sulfide Decomposition

formation of reactants and products). Since the reaction is highly endothermic, heat has to be supplied to enable the reaction to occur. The equilibrium constant was calculated at various temperatures to determine the temperature at which the reaction would yield significant decomposition to products. Values of the equilibrium constant were plotted as a function of the reciprocal temperature. The resulting plot is shown in Figure 1. The temperature at which the hydrogen sulfide is essentially completely converted to products is over 3000°F.

The thermodynamics of the reaction indicates that the equilibrium conversion increases as the temperature is raised. In addition, using the Le Chatelier-Braun principle and the stoichiometry of the reaction leads to the conclusion that the equilibrium degree of conversion would be increased by a decrease in the pressure of the system.

Hence, from the study of the thermodynamics of the reaction, the following conclusions were reached:

a. The reaction had to be carried out at very high temperature or in the presence of a catalyst.

b. Heat had to be supplied continuously to the system.

c. The pressure of the system should be kept as low as possible.

Determination of Controlling Resistances

The determination of the controlling resistance in the reaction is another important step in the study of the kinetics of the reaction, since this determines the controlling step of

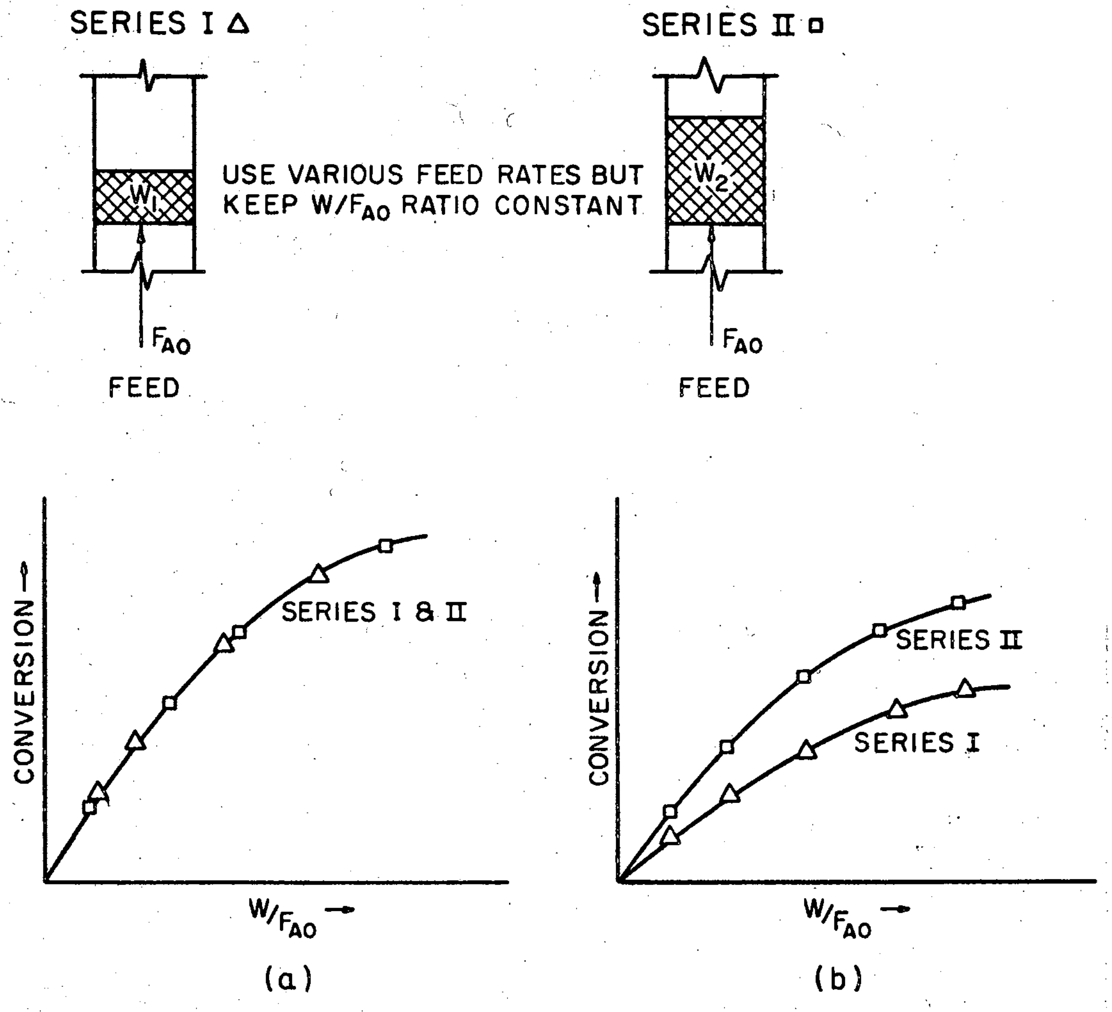

Figure 2. Summary of Procedure for Determining the Controlling Resistance

(a) Runs overlap, hence reaction rate controlling.

(b) Runs do not overlap, hence film diffusion rate controlling.

the reaction. Levenspiel (14) describes a procedure for determining the controlling resistance or step in the reaction. The procedure consists of carrying out series of experimental runs with different amounts of catalyst and feed rates, but at constant W/F_{Ao} values and determining the conversions obtained thereof. The conversions obtained are plotted versus W/F_{Ao}, for the various runs. If the runs overlap, reaction rate is the controlling step. However, if the runs do not overlap, film diffusion is the controlling step in the reaction. A summary of the procedure is given in Figure 2.

CHAPTER III

EXPERIMENTAL EQUIPMENT

Design Requirements

A reactor system was designed to carry out the study of the catalytic decomposition of hydrogen sulfide. The important factors to be considered in the design of the equipment were: the materials to be handled were corrosive, toxic and flammable; the reaction was to be carried out at high temperatures. Hydrogen sulfide, the raw material, is a highly toxic gas; its mixtures with air are flammable. Hydrogen sulfide also vigorously attacks most mild and stainless steels at high temperatures. Hydrogen, one of the reaction products, enters the interstices in steel and causes blistering or intergranular fissuring (8).

All these factors were taken into consideration in the designing of the equipment. A sketch of the reactor system is shown in Figure 3. The reactor was a 316 stainless steel tube which can withstand moderately high temperatures and corrosion. Stainless steel or tygon tubing was used for the connections as these materials were resistant to corrosion and readily available. Tygon tubing was used where the gas temperatures were less than 100°F. All thermocouples were sheathed. The pressure tubes in the pressure gauges were made of stainless steel. The fittings used to connect the various pieces were either of brass or steel. Brass fittings were used only in those places where little or no corrosion could occur and the chances of failure were small. Pyrex glass was used for the flasks in the H_2S absorber and the water-wash bottle.

Because of the flammability of the reactant and products, the room was well ventilated and a draft maintained when the experiments were conducted. The equipment set-up was thoroughly leak-tested to withstand gas pressures far in excess of the pressures encountered during the experiment.

Description of Individual Units

Rotameter 1

Rotameter 1 was used to meter the flow of hydrogen sulfide through the system. The rotameter was a Brooks Brothers "Sho-Rate" Type 2-1110 rotameter having 316 stainless steel inlet fittings and a built-in needle valve. The tube number was R-2-15-A and the flow was indicated by a sapphire ball-type float on a scale calibrated as a percentage of the maximum flow. The rotameter was calibrated for air with the wet test meter. The calibration curve is shown in Figure 7.

Rotameter 2

This rotameter was used to meter the flow of water into the system to quench the reaction. A Matheson Company, Inc. Type 603 rotameter with ball type floats of glass and stainless steel was used. The rotameter was calibrated for the stainless steel float using a stop watch and a calibrated beaker and noting the time it took to fill the calibrated beaker with water. The calibration data are shown in Figure 8.

Pressure Gauges

Two pressure gauges were used to measure the pressure of the reactor. The gauges were manufactured by the Crosby Gage & Valve Company. Both gauges had stainless steel Bourdon tube pressure sensing elements, to guard against corrosion by hydrogen sulfide. These gauges were calibrated against a Budinberg dead weight tester which is accurate to 0.05%. The calibration data for the two gauges are given in Tables A-I and A-II.

Thermocouples

Four Con-o-clad type thermocouples were used to record the gas temperatures. The decision regarding the nature of the hot junction and its construction was based on:

1. The location of the thermocouple in the system.
2. Cost consideration.

All the thermocouples were grounded Iron-Constantan type thermocouples except thermocouple 3 which was ungrounded because at this point in the system, the temperatures of the exit gases and the reactor tube were different. A Chromel-Alumel thermocouple was used because the temperatures at this point in the reactor were expected to be beyond the range of an Iron-Constantan thermocouple.

The thermocouples were sheathed to protect against corrosion. The fittings were designed to ensure a leak-proof connection. The thermocouples were calibrated against a National Bureau of Standards thermometer (Range: 0 - 700°F) with an accuracy of ±1°F. The liquid used in the calibration was Dow Corning Silicone Oil (No: F-1-0173/100CS, Lot No. 43). An ice bath was used as a reference junction. The emf developed by each thermocouple was measured by a Leeds & Northrup Potentiometer (Catalog No: 8690) precise to ±0.01 mv. The calibration data of the thermocouples are shown in Figures 10 to 13.

Reactor Tube

The reactor tube was a 1/2-inch 316 stainless steel, Schedule 40 tube, 40 inches long. Stainless steel was chosen as the material of construction because of its ability to withstand high temperatures and its good corrosion resistance.

Catalyst

The catalyst used in the reactor was molybdenum metal. The catalyst was in the form of thin strips (thickness: 0.005 inch) about 2 inches long and 0.25 inches wide. This form was used because it gave maximum surface area for a given weight. A known weight of these strips was randomly placed in the reactor tube.

Furnaces

The four furnaces used as a heat source were Multiple Unit Electric Furnaces manufactured by the Hevi-Duty Equipment Company. The furnaces were Type 70 furnaces with a maximum rated temperature of 1950°F and a safe working temperature of 1850°F. They operated on 115/230 volts and had a power consumption of 750 watts.

Three of the furnaces were used in the reactor section and one was used in the preheating section. The other furnace used in the feed preheating section was a 220 volt furnace. This furnace was connected directly to the electric outlet (line voltage: 115 volts).

Powerstats

The powerstats used to regulate the power input to the heaters, and hence the reactor temperature, were manufactured by the Superior Electric Company. They were Type 116 powerstats with a rated primary voltage of 120 volts and a maximum current of 7.5 amperes. They had a maximum power supply of 1 KVA. Four powerstats were used, one for each Hevi-Duty furnace.

Analytic Section

The analytic section consisted primarily of the sulfur trap, H_2S absorber, water-wash flask and wet test meter. The sulfur trap and H_2S absorber consisted of Pyrex glass bottles with removable glass fittings to facilitate cleaning and refilling. The H_2S absorber consisted of three such bottles connected in series. The absorbent was a saturated solution of NaOH. Each flask or bottle was filled with a known weight of NaOH solution before each run. The water-wash bottle was made of glass and was filled with cold water. The water-wash bottle served two functions:

1. To saturate the gas with water before going to the wet test meter.

2. To remove any NaOH which might be escaping with the gas leaving the H_2S absorbers.

Wet Test Meter

The wet test meter used to determine the volume of hydrogen produced was a "Precision Wet Test Gas Meter" manufactured by the Precision Scientific Company, Chicago. The wet test meter had provision for determining the temperature and pressure of the exit gas stream.

Analytical Balance

An analytical balance was used to determine the weights of the flasks. The balance (University Inventory No: 020131) was manufactured by Voland & Sons, Inc. Weights could be determined precisely to ±0.005 grams.

The Process

The flow diagram of the process is given in Figure 3. The process consisted of passing the hydrogen sulfide over a fixed bed of metal catalyst at elevated temperatures. The temperature of operation depended on the nature of the catalyst. Catalysts suitable for this reaction were platinum (21), molybdenum (11), pure and carburized tungsten (4), and uranium (3). Molybdenum was selected as the catalyst to be investigated although literature studies indicated that platinum was a better catalyst.

The experimental procedure consisted of passing bottled H_2S (technical grade of 98.5% minimum purity) through a rotameter [2]

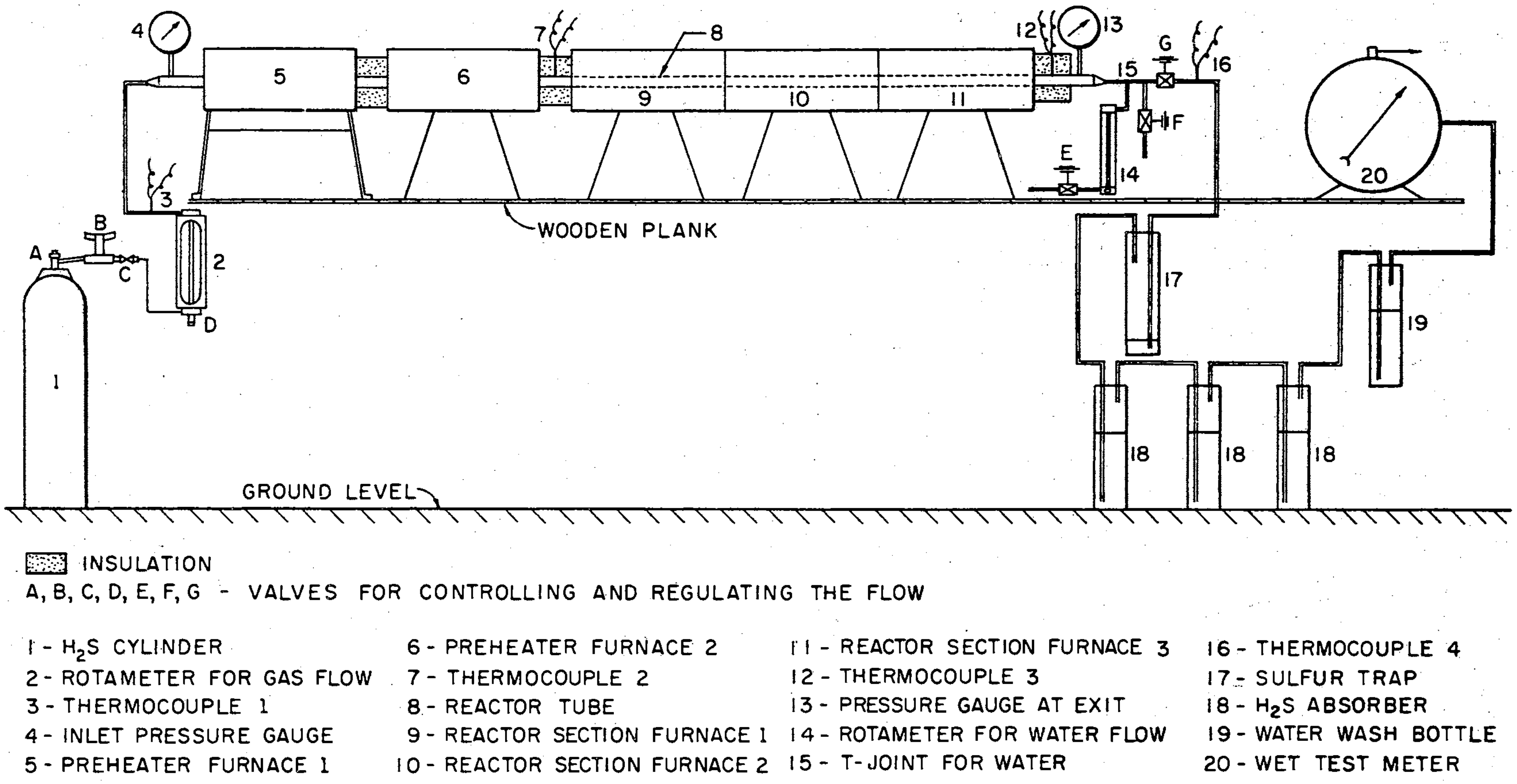

Figure 3. Equipment for the Study of the Catalytic Decomposition of Hydrogen Sulfide

where the flow rate was adjusted to the desired value. The inlet temperature of the gas was measured by a thermocouple [3] and the inlet pressure was determined by the pressure gauge [4]. The gas then passed through the preheating section consisting of two horizontal heaters, before entering the reaction chamber. The reaction chamber [8] consisted of an 1/2-inch diameter 316 stainless steel tube containing the molybdenum catalyst. The reactor was heated by three Hevi-Duty furnaces [9, 10, 11] where the decomposition reaction occured. The temperature of the exit gas stream was measured by the Chromel-Alumel thermocouple [12] and the exit pressure was determined by the pressure gauge [13].

A provision was made to introduce a stream of cold water [15] the rate of which would be determined by the rotameter [14]. The purpose of adding the water was to quench the reaction by decreasing the temperature of the effluent stream and by dilution. However, since the temperature of the exit gas stream was not high enough to warrant the quenching and since the back reaction was apparently small, the quench system was not used.

The product stream then passed to a sulfur trap [17] where the molten sulfur dropped out. The amount of sulfur collected was determined by gravimetric methods. The remaining product stream, consisting of the unconverted H_2S and H_2 then passed through a set of previously weighed flasks [18] containing a known weight of saturated NaOH solution. The unconverted H_2S was absorbed in these flasks. The exit gas stream (essentially all hydrogen), passed through a flask containing cold water [19] where any NaOH escaping with the gas stream was absorbed and the gas was saturated with water. The gas stream then goes to the wet test meter [20] where its volume was measured and was then discharged to the atmosphere. The volume of hydrogen produced was determined from the wet test meter readings. The gas leaving the wet test meter was qualitatively tested to determine the nature of its content.

Testing of the Experimental Set-up

The equipment was set up as shown in the flow diagram (Figure 3). The furnaces were adjusted, if necessary, to operate on 115 volts. The entire set-up was then tested for any gas leaks by passing air at around 40 psig and using a soap solution. Any detected leaks were removed by sealing the connection with a suitable sealant. The maximum pressure of 40 psig was chosen arbitrarily; however, it was a pressure that was several times the operating pressure.

CHAPTER IV

EXPERIMENTAL PROCEDURE

The experimental procedure consisted of passing hydrogen sulfide through the reactor tube and catalyst bed at different rates at known temperatures. The experimental runs were carried out at three different temperatures. Four different flow rates of H_2S were run at each temperature.

The experimental procedure for each run may be broadly classified as:

a. Auxiliary operations

b. Main experimental operations

The auxiliary operations consisted of the following:

1. Preparation of saturated solution of NaOH: The solution was prepared by dissolving about 300 grams of NaOH flakes in 0.5 liters of distilled water. The solution was continuously stirred and cooled to facilitate the solution of NaOH. The amount of NaOH flakes required to prepare a saturated solution was arrived at by trial and error.
2. Flushing the system: Before switching the furnaces on, the entire system was flushed with H_2S to remove any air present in the system. The H_2S was allowed to run through the system for about 30 minutes to ensure essentially complete removal of air, since the presence of air in the

system is detrimental to the reaction. This operation was carried out once at the start of the experimental runs and whenever the system was opened for repairs or changing the reactor tube.

3. Achieving steady state: After flushing the system with H_2S, the powerstats were switched on and adjusted to attain the desired temperature in the reactor section. Two to four hours were normally required for the reactor temperature to become constant at a given H_2S flow rate.
4. Standardizing the potentiometer: The reference junctions were packed into a vacuum flask containing crushed ice and the potentiometer circuit was standardized.

The analytical balance was checked and adjusted to read accurately. The flasks used in the H_2S absorber were cleaned, dried and weighed empty and with the solution. These weights were recorded. The flasks were then connected to the system. The connections were checked and any leaks present were removed.

The main operations for a run consisted of the following steps:

1. The atmospheric pressure was recorded from a Fortin's Barometer.
2. Referring to Figure 3, valve A on the H_2S cylinder was opened and valve B on the regulator was adjusted so that the discharge pressure was about 84 psi as recorded by the gauge on the regulator.
3. Valve C was opened and then needle valve D, on the gas rotameter, was opened to adjust the flow rate. A brief time delay was used to assure visual observation of the

float in the rotameter.

4. On opening valve D, the pressure gauges [4, 13] recorded an increase in pressure, which was released by opening valve F on the by-pass tube. The H_2S gas was allowed to flow into some spent NaOH solution, where it was absorbed. This operation was carried out to keep the H_2S concentration in the room at a safe level.
5. Valves C and D were then adjusted so that the flow of gas through the system was at the desired value. The gas was allowed to flow through the system at the set flow rate and the system was allowed to attain steady state, indicated by steady temperatures recorded by thermocouples 2 and 3.
6. When the system was judged to be at steady state, valve G was opened and valve F closed simultaneously, to force the gas to flow through the analytical apparatus.
7. The initial reading on the wet test meter was noted. The temperature and pressure of the exit stream from the wet test meter was also noted.
8. On opening valve G and closing valve F, a stop-watch was started and the gas was allowed to flow through the analytical section for about ten minutes.
9. During the run, all pressures, temperatures, and gas flow rates were recorded at various intervals, so as to obtain a mean value.
10. After about ten minutes, valve G was closed and the stop-watch stopped simultaneously.

11. The by-pass valve F was then opened to relieve the system and the exit gas was allowed to flow into the spent NaOH solution.
12. Valves D and C were then closed in that order and then valve F was closed to prevent any solution, or air, from flowing back into the reactor.
13. The H_2S absorber flasks were then disconnected from the system and weighed. The weights of the flasks were recorded.
14. The final reading on the wet test meter was recorded.
15. During the course of the run, the wet test gas meter effluent was tested qualitatively. The exit gas was first tested for the presence of H_2S. A piece of white filter paper soaked in lead acetate solution was placed in the exit gas stream. If any H_2S was present in the gas stream, it would reduce the lead acetate to lead sulfide which would change the color of the filter paper to silver gray. Samples of the gas were collected over water and qualitatively tested for hydrogen. The test consisted of igniting the gas and noting the formation of water vapor on the wall of the tube. At higher reaction temperatures, where the conversions were significant, a flexible tube was connected to the exit nozzle of the wet test meter and the escaping gas was ignited. The flame was colorless and was placed on a cold, dry, polished metal surface. The formation of water as very fine droplets was noted, indicating the presence of hydrogen in the exit gas stream.

16. After determining the weights of the flasks in the H_2S absorber, they were again connected to the system, in a different order, so that the first flask in the chain contained a fresh solution of NaOH. This change was done in order to conserve NaOH. This operation was judged to be permissible since the first flask in the chain absorbed essentially all the undecomposed H_2S while the other flasks removed only traces.
17. The potentiometer was again standardized; the reading of the wet test meter was noted and the second run, at another flow rate was carried out in exactly the same manner as described above.

Experiments were first conducted with an empty tube at various temperatures with different flow rates, along the lines described, to determine the extent to which the material of the tube acted as a catalyst for the reaction. The reactor tube was then disconnected from the system and a known weight of the catalyst was randomly placed inside the tube.

The reaction was studied at three different temperatures, as recorded by thermocouple 3, and at each temperature four different gas flow rates were studied. The temperatures were controlled by means of powerstats connected to each furnace in the reactor section. The reaction was studied with the temperature increasing to the maximum possible value and then with the temperature decreasing to the minimum temperature studied, to act as a check.

Experiments were also carried out to determine whether the reaction rate or the film diffusion rate was the controlling step;

the reactor tube was disconnected and a different weight of catalyst was randomly packed in the tube. A series of runs were then carried out in a manner analogous to the one described.

At the termination of each series of runs, the analytical balance was checked and adjusted, if necessary. The flasks in the absorber chain were disconnected, cleaned, dried, and weighed empty and with fresh solution. The spend NaOH solution was collected and used as an absorbent at the end of the by-pass line.

CHAPTER V

RESULTS AND DISCUSSION

The prime object of this work was to determine the technical feasibility of the catalytic decomposition of hydrogen sulfide using molybdenum as a catalyst. Another objective was to obtain kinetic information about the reaction.

The technical feasibility of the reaction was amply demonstrated during the course of the study and the experimental results are presented in Tables I, II and III and Figures 4 and 5. The indicated temperatures are the temperatures of the effluent gas recorded by the thermocouple at the exit end of the reactor. Conversions around 85% were obtained at a gas temperature of 317.5°F.

The order of the reaction and the specific reaction rate constant for the reaction were determined according to the techniques presented by Levenspiel (15) and Smith (20). The calculations were based on the experimental results given in Table II. A sample calculation of the reaction order and the rate constant at 317.5°F is given in Appendix C. The reaction was determined to be a first order reaction and the values of the specific reaction rate constants for different temperatures are given in Table IV.

Having determined the reaction rate constants at the three temperatures by the above-mentioned technique, the rate constants were substituted into the equation for the first order homogeneous

TABLE I

EXPERIMENTAL RESULTS WITH NO CATALYST

Run	Temperature °F	Duration of Run Min.	Input Rate of H_2S at S.T.P. gm/min	Inlet Pressure mm.Hg. gauge	Rate of Gas Flow Thru WTM @ S.T.P. gm/min	Exit Pressure at WTM mm.Hg. abs.	Weight of Undecomposed H_2S gm/min	Amount of Error in Material Balance gm/min	% Conversion
1	170.0	10.00	0.562	124.12	0.016	750.00	0.533	0.013	2.76
2	240.0	10.033	0.980	51.71	0.052	750.00	0.864	0.064	5.33
3	340.0	14.083	1.539	142.22	0.596	750.00	0.685	0.258	38.60

TABLE II

EXPERIMENTAL RESULTS WITH 75.49 GRAMS OF CATALYST

Run	Duration of Run Min.	Input Rate of H_2S at S.T.P. gm/min	Inlet Pressure mm.Hg. gauge	Rate of Gas Flow Thru WTM @ S.T.P. gm/min	Exit Pressure at WTM mm.Hg. abs.	Weight of Undecomposed H_2S gm/min	Amount of Error in Material Balance gm/min	Residence Time $\frac{\text{g-cat}}{\text{g-}H_2S\text{/min}} \times 10^{-2}$	% Conversion
Temperature = 190.0°F		△							
4	16.195	0.540	134.46	0.005	750.0	0.512	0.023	1.400	0.885
5	10.409	0.980	134.46	0.007	750.0	0.914	0.059	0.769	0.760
6	10.588	1.439	134.46	0.004	750.0	1.218	0.217	0.525	0.255
7	7.383	1.761	103.43	0.005	750.0	1.624	0.132	0.430	0.306
Temperature = 259.5°F		⊡							
8	12.295	0.555	134.46	0.211	750.0	0.314	0.030	1.360	38.10
9	11.700	0.989	134.46	0.229	750.0	0.751	0.009	0.760	23.10
10	10.266	1.439	134.46	0.181	750.0	1.093	0.165	0.516	12.60
11	8.925	1.766	134.46	0.170	750.0	1.384	0.212	0.419	0.981
Temperature = 317.5°F		⬡							
12	10.766	0.547	129.29	0.468	750.0	0.037	0.042	1.380	85.6
13	10.058	0.980	129.29	0.806	750.0	0.030	0.144	0.760	82.2
14	10.275	1.459	134.46	0.724	750.0	0.553	0.182	0.516	49.5
15	10.110	1.796	155.14	0.575	750.0	0.942	0.279	0.419	31.8
Temperature = 282.5°F		◇							
16	10.450	0.562	124.12	0.231	750.0	0.330	0.001	1.340	41.0
17	10.155	0.989	129.29	0.281	750.0	0.703	0.005	0.760	28.4
18	10.241	1.463	160.32	0.117	750.0	1.182	0.164	0.516	8.0
19	10.050	1.796	206.86	0.126	750.0	1.570	0.100	0.419	7.0
Temperature = 190.5°F		▽							
20	10.766	0.562	129.29	0.015	750.0	0.532	0.015	1.340	2.72
21	10.166	0.915	144.80	0.008	750.0	0.903	0.004	0.840	0.885
22	10.991	1.446	170.66	0.004	750.0	1.259	0.183	0.521	0.260
23	10.110	1.796	181.00	0.007	750.0	1.662	0.127	0.421	0.377

TABLE III

EXPERIMENTAL RESULTS WITH 26.48 GRAMS OF CATALYST

Run	Duration of Run Min.	Input Rate of H_2S at S.T.P. gm/min	Inlet Pressure mm.Hg. gauge	Rate of Gas Flow Thru WTM @ S.T.P. gm/min	Exit Pressure at WTM mm.Hg. abs.	Weight of Undecomposed H_2S gm/min	Amount of Error in Material Balance gm/min	Residence Time $\frac{\text{g-cat}}{\text{g-}H_2S\text{/min}} \times 10^{-2}$	% Conversion
Temperature = 343.0°F									
24	10.316	0.157	124.12	0.129	740.0	0.024	0.004	1.690	82.16
25	10.095	0.316	124.12	0.135	740.0	0.156	0.025	0.838	42.72
26	10.141	0.517	118.94	0.200	740.0	0.258	0.059	0.512	38.68
27	10.355	0.805	124.12	0.282	740.0	0.490	0.033	0.329	35.03
Temperature = 281.0°F									
28	10.175	0.151	129.29	0.060	740.0	0.095	-0.004	1.755	39.74
29	6.853	0.342	118.94	0.104	740.0	0.229	0.009	0.775	30.41
30	9.755	0.532	103.43	0.028	740.0	0.475	0.029	0.498	5.26
31	10.075	0.817	129.29	0.021	740.0	0.762	0.034	0.324	2.57

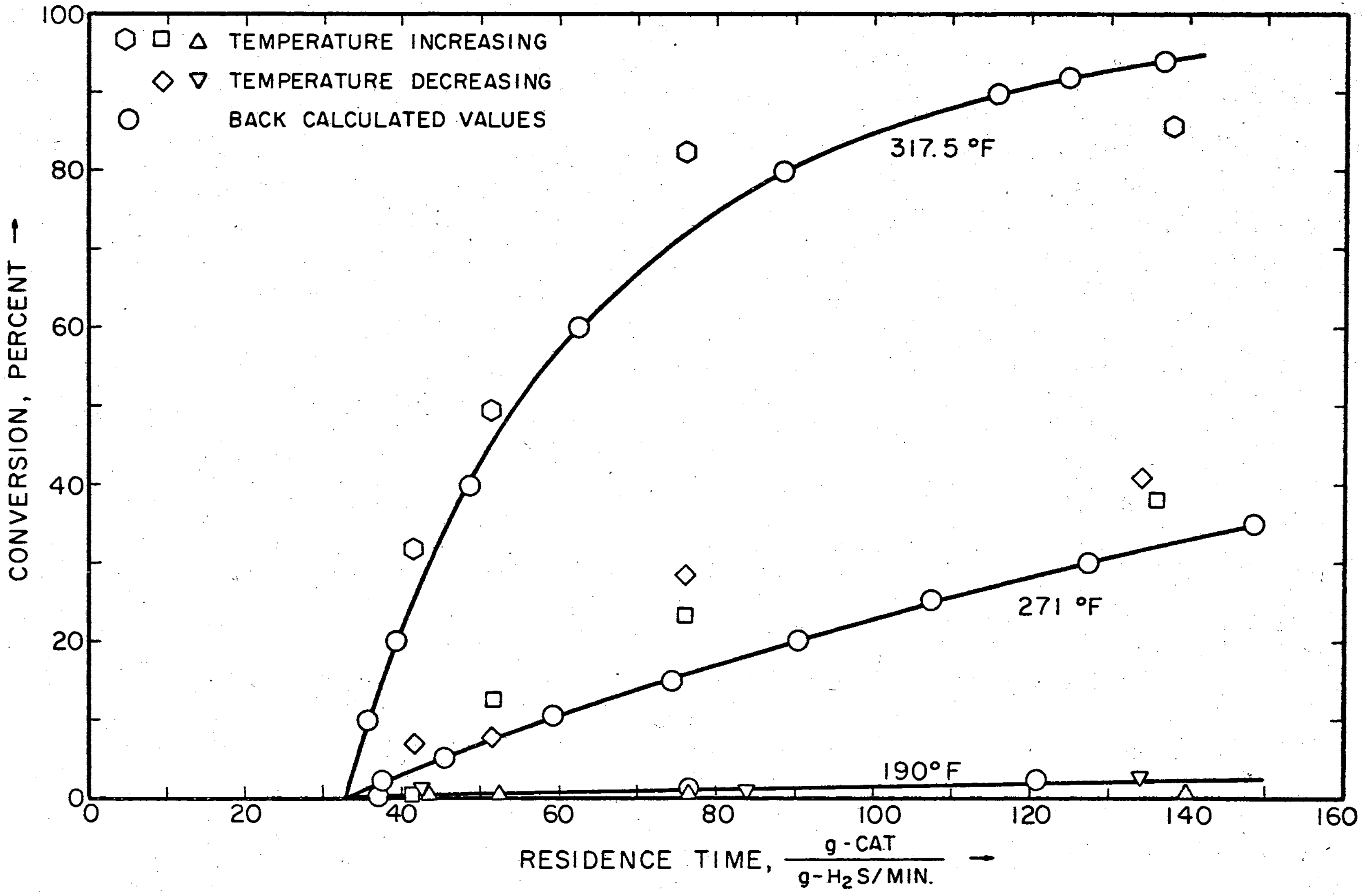

Figure 4. Residence Time - Conversion Plots for H_2S Decomposition Reaction

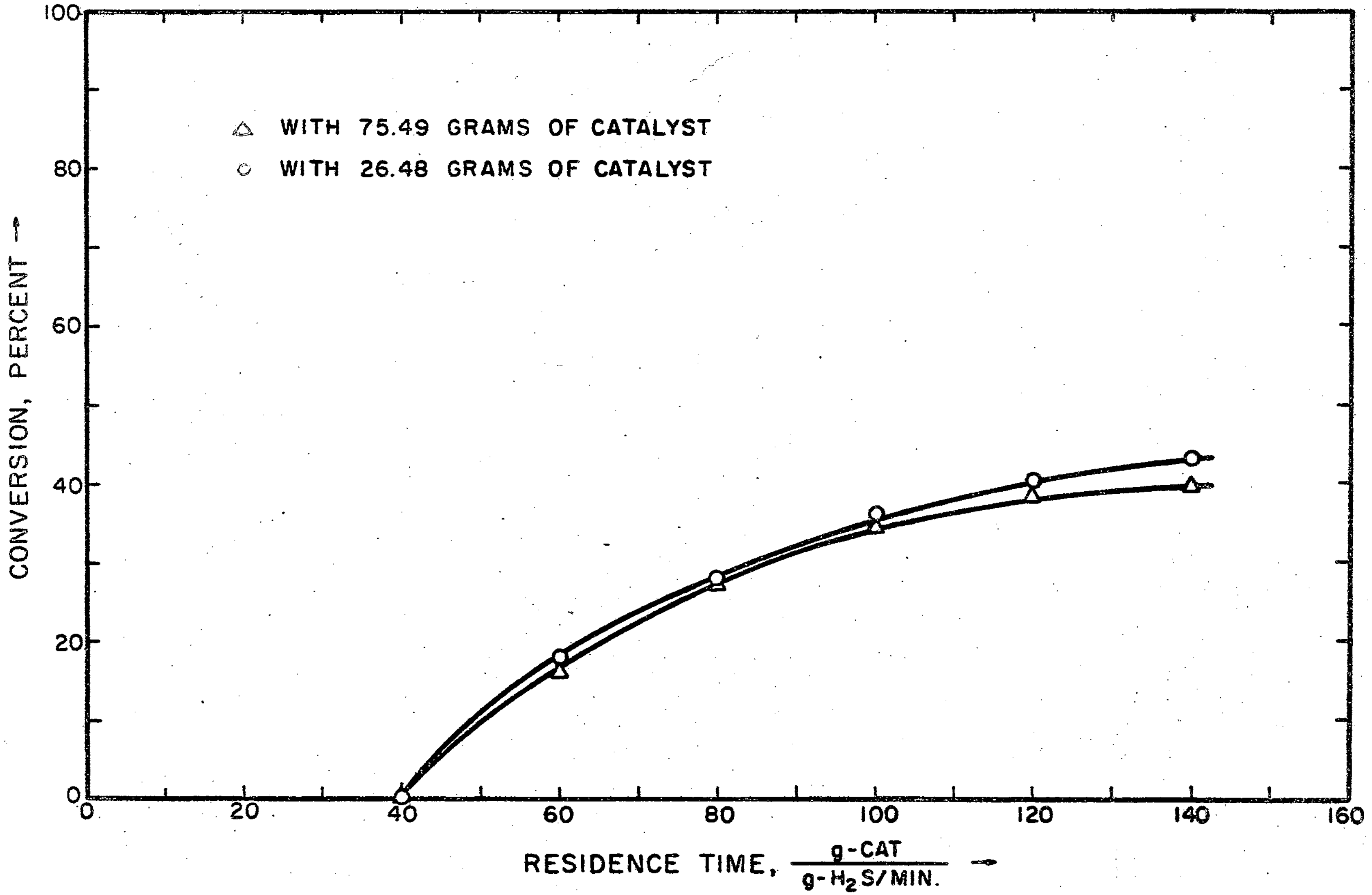

Figure 5. Determination of Controlling Step of Reaction

TABLE IV

SPECIFIC REACTION RATE CONSTANTS AT VARIOUS TEMPERATURES

Temperature t °F	Specific Reaction Rate Constant k liter/(g-cat)(min)
190.0	0.000174
271.0	0.00347
317.5	0.0326

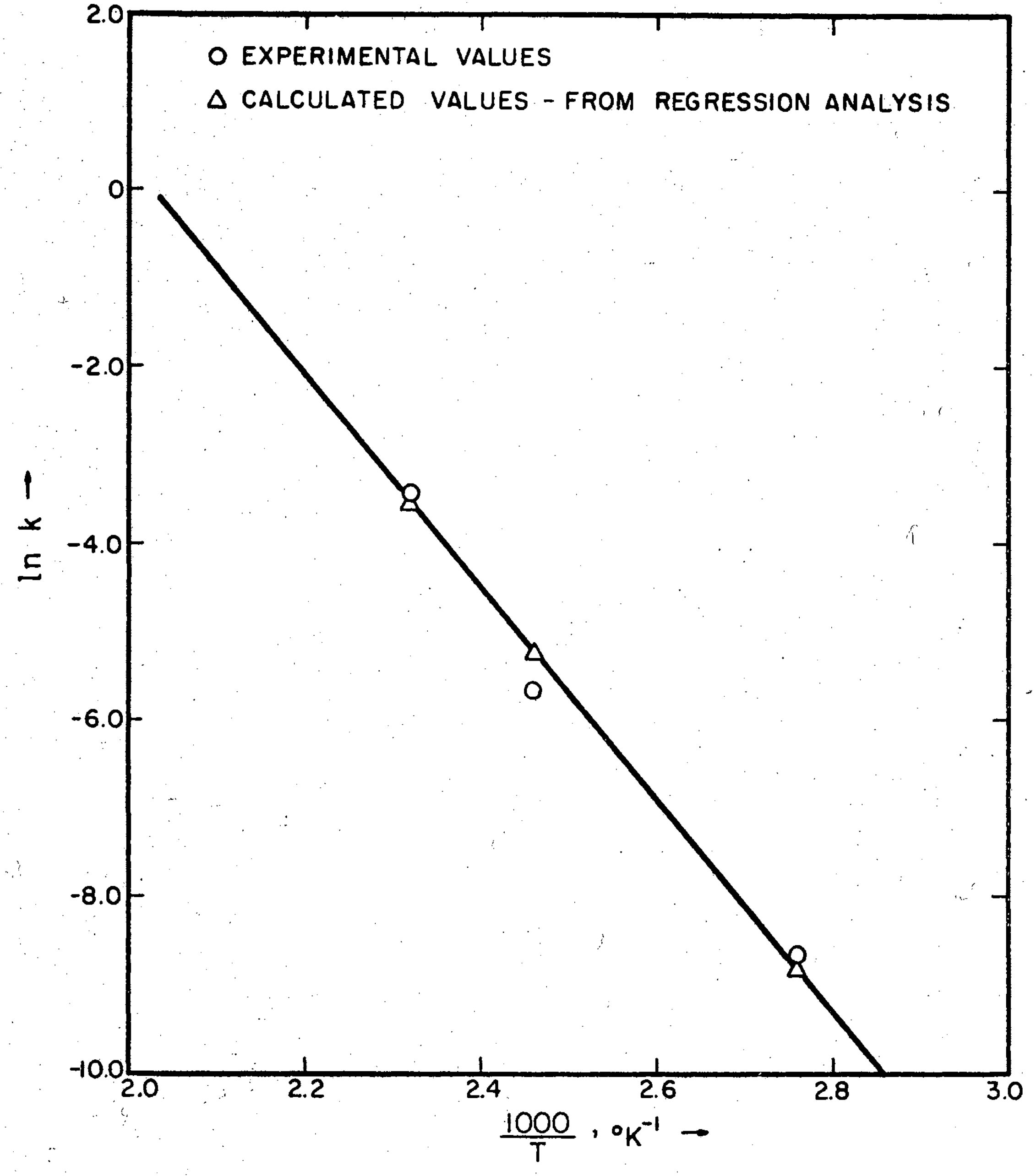

Figure 6. Temperature Dependence of Reaction Rate Constant

reaction in a plug flow reactor (13) viz:

$$kC_{Ao}\tau = (1 + \varepsilon_A)\ln \frac{1}{1 - X_A} - \varepsilon_A X_A \tag{1}$$

and values of τ were calculated for various conversions, X_A. These values are plotted in Figure 4 for the temperatures studied. This figure shows that the back-calculated values generally agree with the experimental values; however, the calculated values tend to lie below the experimental values. The differences may be attributed to experimental error and the use of the homogeneous plug flow equation to describe the behavior in a heterogeneous plug flow reactor.

By making a series of runs at a given temperature and W/F_o ratio with a different amount of catalyst and different feed rates, information was obtained regarding the controlling step in the reaction. The experimental results of these runs with 26.48 grams of catalyst are given in Table III. Data for the two different amounts of catalyst at 271 and 281°F were taken from Tables II and III, respectively, and fitted with least square fit equations. Figure 5 is a plot of these two equations. Referring to Figure 5, the curves for the two different amounts of catalyst are almost parallel and, within limits of experimental error, overlap. Hence, it was concluded that the reaction rate is the controlling step in the reaction.

The activation energy was calculated from the specific reaction rate constants using the Arrhenius Equation (2). Figure 6 shows a plot of ln k as a function of T^{-1}, where k is the specific reaction rate constant and T is the absolute temperature. Regression analysis was employed to obtain the best fitting straight line to the data points. From this plot, the apparent activation energy was determined to be 23,050 cals/mole. The activation energy reported by Kingman (11) for the reaction was 25,000 cals/mole. The disparity between the two values may be attributed to the differences in the experimental set-up and analysis.

Referring to Table II and Figure 4, at a temperature of 317.5°F conversions of 85% of hydrogen sulfide to products were obtained. However, the thermodynamic analysis given in Chapter II indicate that this degree of equilibrium conversion could only be attained at temperatures above 3000°F. One possible explanation for these high conversions at the low temperature is that some of the sulfur formed in the course of the reaction was removed from the sphere of the reaction thereby decreasing the back reaction to form hydrogen sulfide. Consider the basic reaction:

$$H_2S(g) \rightleftarrows H_2(g) + S(g)$$

if the hydrogen sulfide is completely converted to hydrogen and sulfur, that is, the back reaction is zero, then

$$p_S = y_S \times P_{sys} \qquad (2)$$

where p_S = partial pressure of S in vapor phase

y_S = mole fraction of S in vapor phase

P_{sys} = system pressure

therefore at equilibrium,

$$p_S = 0.5 \times 853.42$$

$$= 426.71 \text{ mm.Hg.}$$

From Table B-2, at 317.5°F, the vapor pressure of saturated sulfur is 0.3082 mm.Hg.

Since the partial pressure of sulfur in the vapor phase is far greater than the vapor pressure of sulfur at the reaction temperature, the sulfur formed due to the reaction will condense and be removed from the reaction system (the word "system" is used here in the thermodynamic context). This removal of sulfur from the reacting system would then tend to decrease the back reaction, thereby shifting the equilibrium towards more complete conversion of hydrogen sulfide to hydrogen and sulfur.

Experimental evidence in support of the removal of sulfur from the reaction system was:

1. After operating the reactor for some length of time, the reactor tube got plugged.
2. When the reactor tube was dismantled, formation of molybdenum sulfide was noticed in the reactor as soft, greyish black, shining crystals. However, the amount of hydrogen sulfide passed through the reactor, on a mole basis, was far greater than the amount of molybdenum present as the catalyst, without any noticeable decrease in the activity of the catalyst.

Hence, one may conclude from this that the high conversions obtained were due to the sulfur formed during the course of the reaction, dropping out of the reaction system.

Although the reaction was shown to be feasible, certain limitations of the reactor design and experimental procedure were noted; these are discussed below:

1. Space velocity limitations: The velocity of the gas in the reactor was too low to drive the sulfur vapors formed out of the system. This resulted in the sulfur vapors condensing at the cooler ends of the reactor thereby choking it up. This was a major limitation, since each time the reactor tube got choked, it had to be discarded and a new tube with fresh catalyst installed. In the equipment used in this study, the upper limit on the gas velocity was the capacity of the rotameter used to meter the inlet flow of gas. This could be rectified by using a larger capacity rotameter or a smaller diameter tube.
2. Temperature limitations: To determine the temperature of the reaction, the temperature of the gas should be measured at various points in the catalyst bed in the reactor and not only at the exit of the reactor, since the effluent gas was cooled some undetermined amount between the reactor and the thermocouple, due to the steep temperature drop between the reactor and the surroundings. Insulation was used between the thermocouple and the downstream end of the reactor; however, it did not prove to be very effective in reducing the heat leak. In addition, the temperatures at various points in the catalyst bed could not be measured because of the construction of the furnaces and the possibility of gas leaks.
3. Analytical limitations: The average error in the

experimental data was calculated and is shown in Tables I, II and III. The error in the material balance for each run was calculated and the average was between 5 and 10%, with a mean of 7.19%. This error is not large considering the scope of this work; however, it might be improved by using more precise methods of analysis.

Some of the possible sources of this error were:

1. Inaccuracy in weighing.
2. Inaccuracies in the balance and weights used.
3. Possible gas leaks in the connections between the "tygon" tubing and the flasks.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

The feasibility of the reaction has been amply demonstrated in the course of this study. It was shown that high conversions of hydrogen sulfide could be obtained using molybdenum as the catalyst. It was also shown that the reaction rate was the rate controlling step. The apparent activation energy was computed and it agreed reasonably well with the value obtained by Kingman (11). The study also highlighted certain drawbacks of the reactor design which have been discussed in Chapter V. For a more complete study of the reaction and the reactor design, further work would have to be done especially with regard to elucidating the mechanism of the reaction.

Plugging of the reactor after operating for some length of time was a major drawback. This was due (as postulated) to the condensation of sulfur at the cooler ends of the reactor. To help remedy this, it is recommended that in future work:

1. The reactor be constructed as a downflow fixed catalyst bed reactor--this would help the sulfur condensed during the reaction to flow out of the reactor by gravity.
2. Higher gas velocities be used in the reactor. This could be carried out by using a higher capacity rotameter or a smaller diameter reactor tube.
3. The reactor and preheater section be well insulated and

the heat leak from the system be reduced to a minimum.

To obtain more information about the reaction and the reactor design, the thermocouples used to measure the reaction temperature should be placed at various points in the reactor bed, instead of being located at the end of the reactor tube. Care will have to be exercised to see that the thermocouple connections are truly leakproof, more so at the high temperature of operation, since the products and the reactant are highly flammable.

In order to decrease the error in the material balance, it is recommended that valve G (see Figure 3) be a three-position valve. Also, a more precise balance and set of weights should be used in the analysis. Finally, it is recommended that a quantitative analysis of the exit gas from the wet test meter be made.

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APPENDIX A

TABLE A-I

CALIBRATION OF CROSBY PRESSURE GAUGE 1

Pressure According to Weights psia	Pressure According to Pressure Gauge		
	Wts. Increasing psia	Wts. Decreasing psia	Mean Value psia
5.0	5.0	5.0	5.0
10.0	10.0	10.1	10.05
15.0	15.0	15.2	15.1
20.0	20.4	20.4	20.4
25.0	25.4	25.5	25.45
30.0	30.5	30.5	30.5
35.0	35.5	35.5	35.5
40.0	40.5	40.6	40.55
45.0	45.5	45.6	45.55
50.0	50.5	50.5	50.5
55.0	55.5	55.5	55.5

TABLE A-II

CALIBRATION OF CROSBY PRESSURE GAUGE 2

Pressure According to Weights psia	Pressure According to Pressure Gauge		
	Wts. Increasing psia	Wts. Decreasing psia	Mean Value psia
10.0	10.7	10.6	10.65
15.0	15.4	15.6	15.5
20.0	20.6	20.7	20.65
25.0	25.6	25.6	25.6
30.0	30.4	30.6	30.5
35.0	35.4	35.5	35.45
40.0	40.1	40.2	40.15
45.0	44.6	44.6	44.6
50.0	49.2	49.4	49.3
55.0	54.0	54.1	54.05
60.0	59.1	59.1	59.1

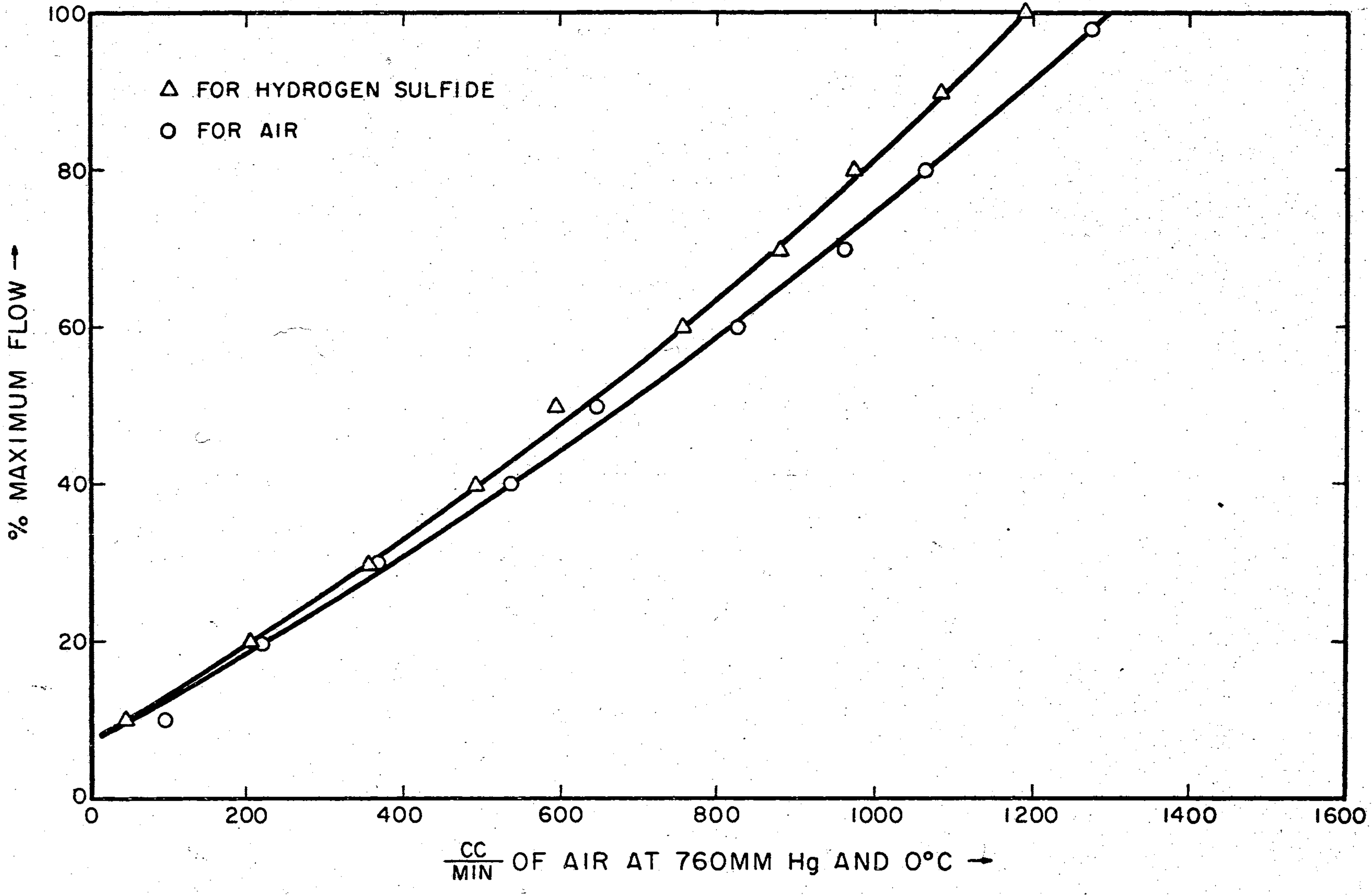

Figure 7. Calibration Curves for "Sho-Rate" Rotameter for Air and H_2S

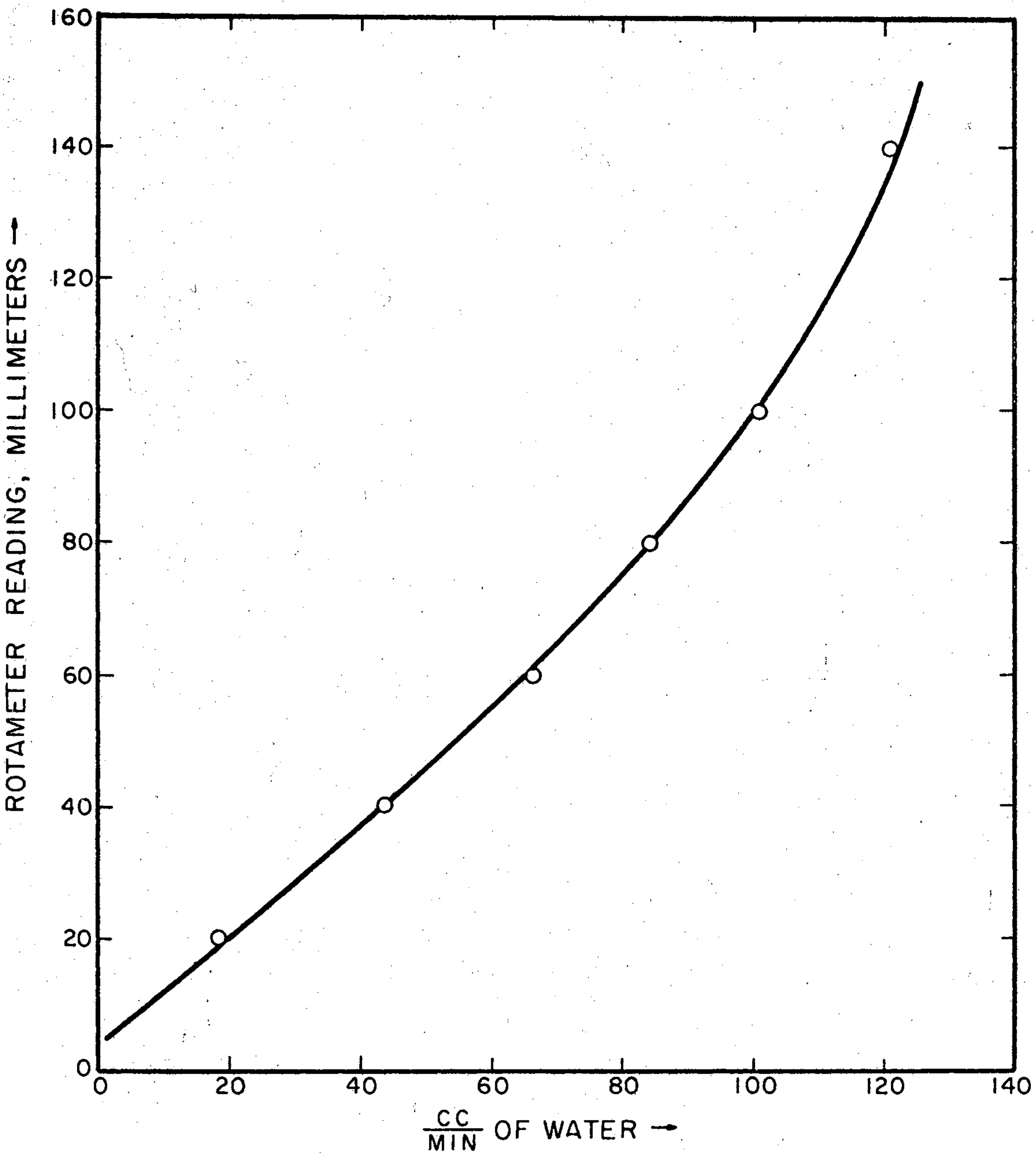

Figure 8. Calibration Curve for 603 Rotameter for Water

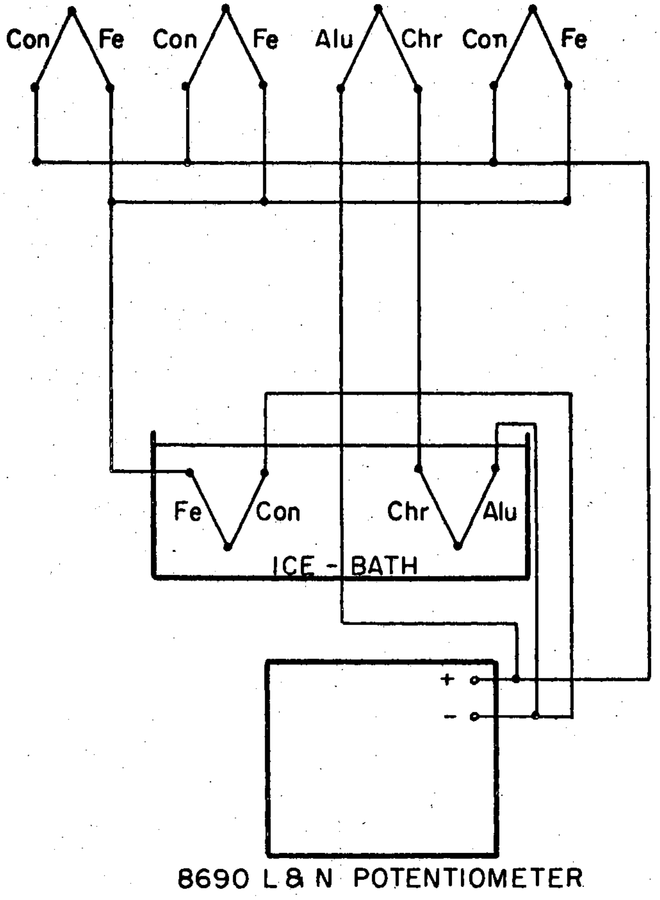

Figure 9. Circuit Diagram for Thermocouple Connections

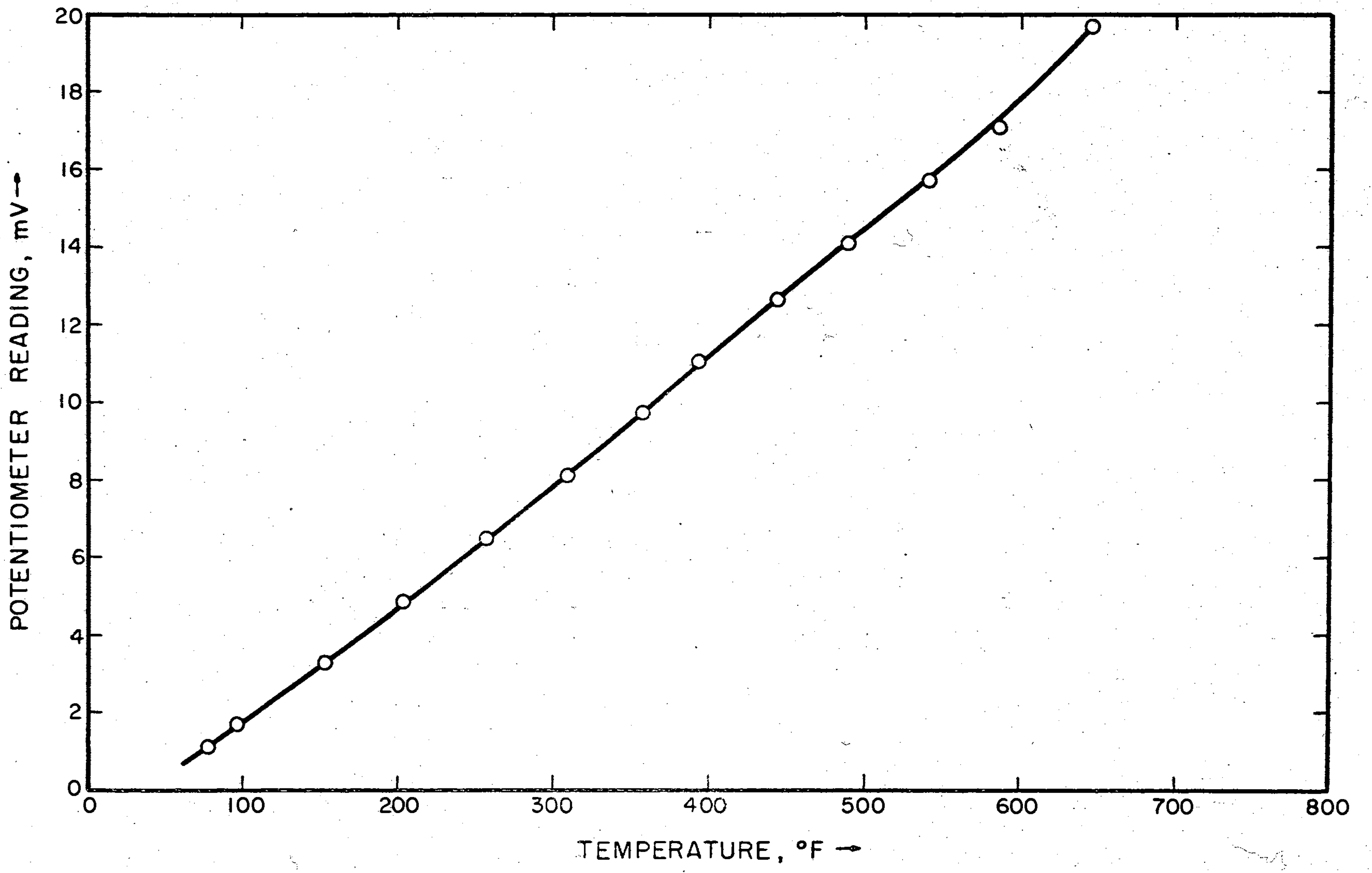

Figure 10. Calibration Curve for Thermocouple 1

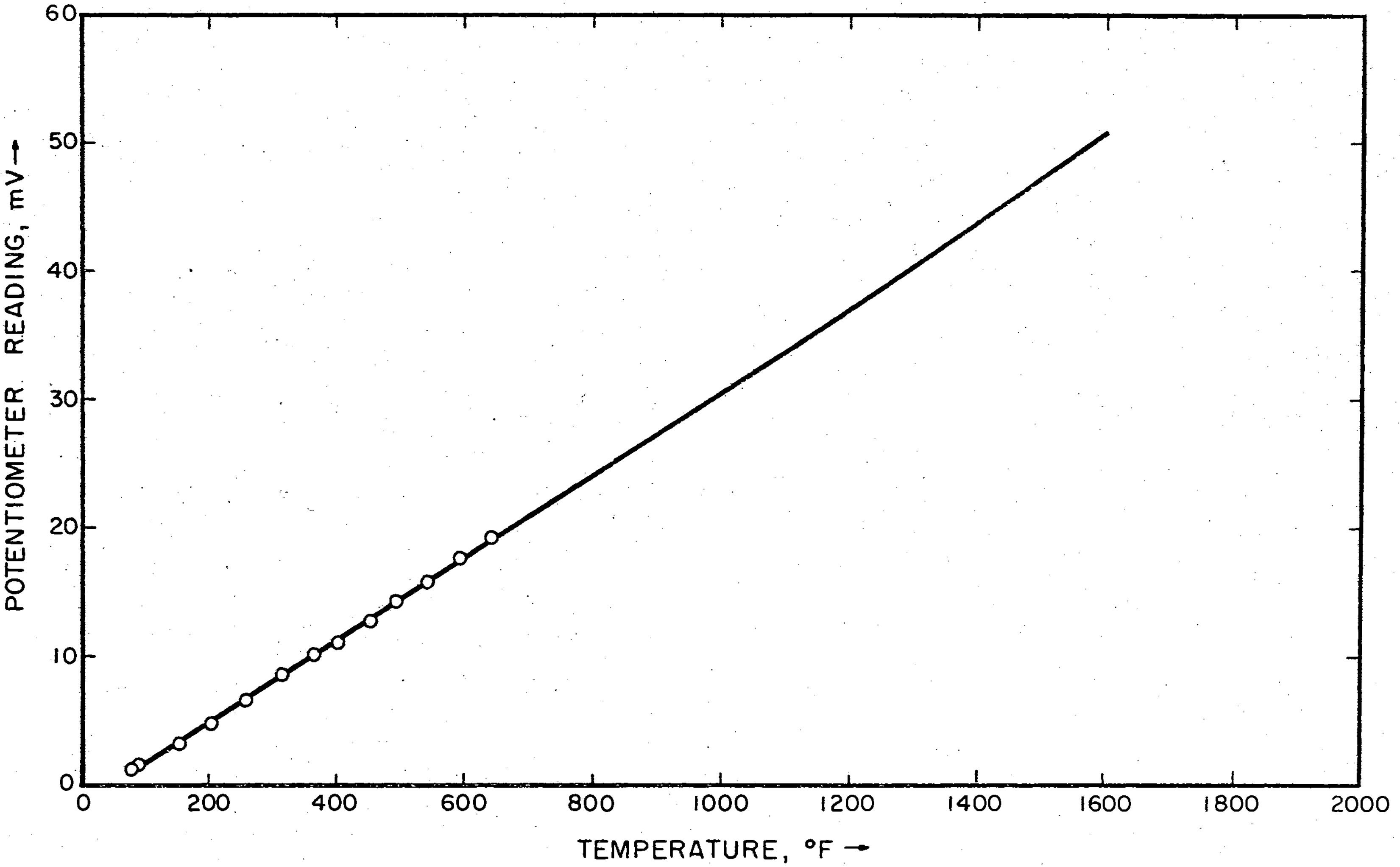

Figure 11. Calibration Curve for Thermocouple 2

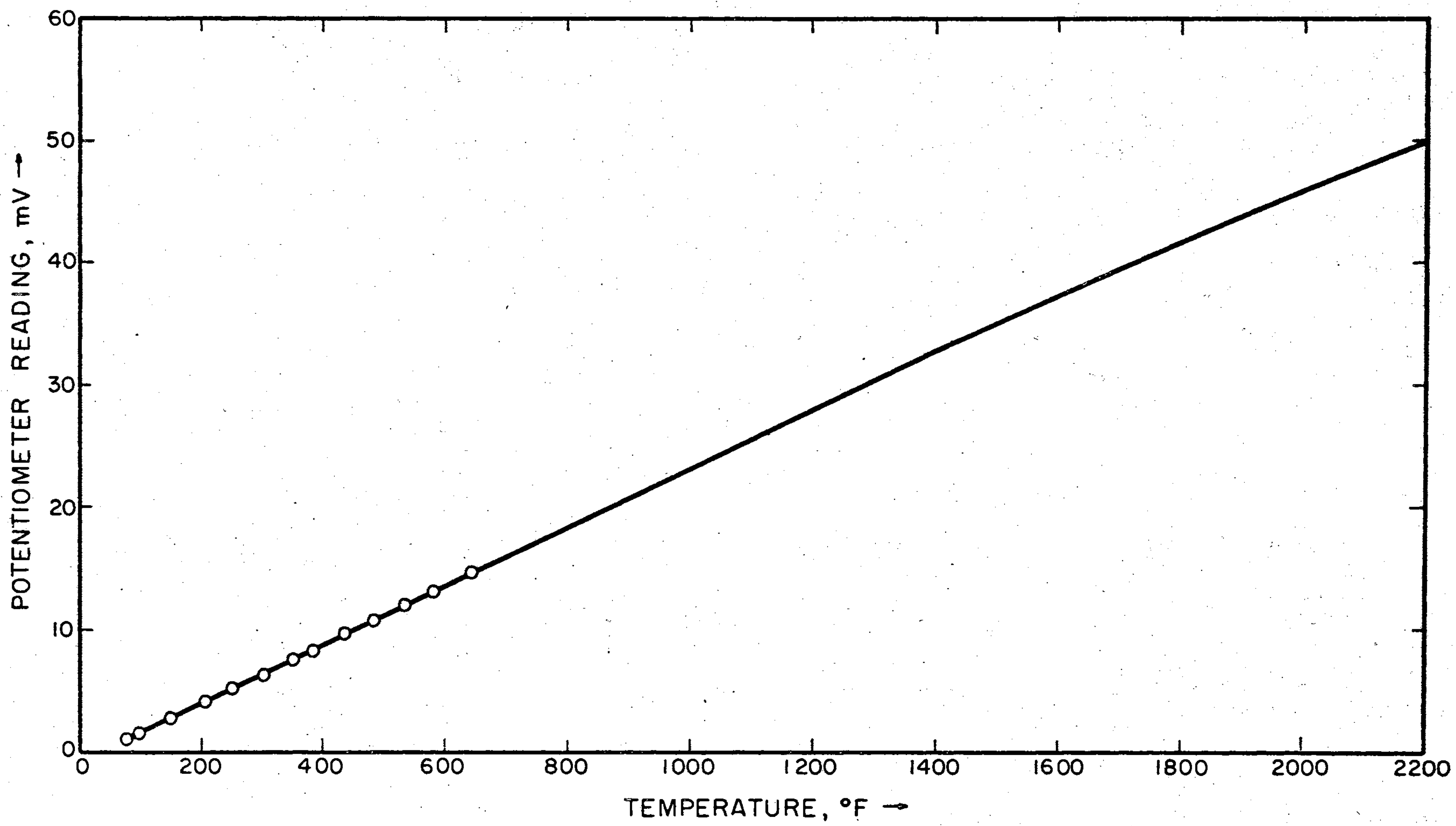

Figure 12. Calibration Curve for Thermocouple 3

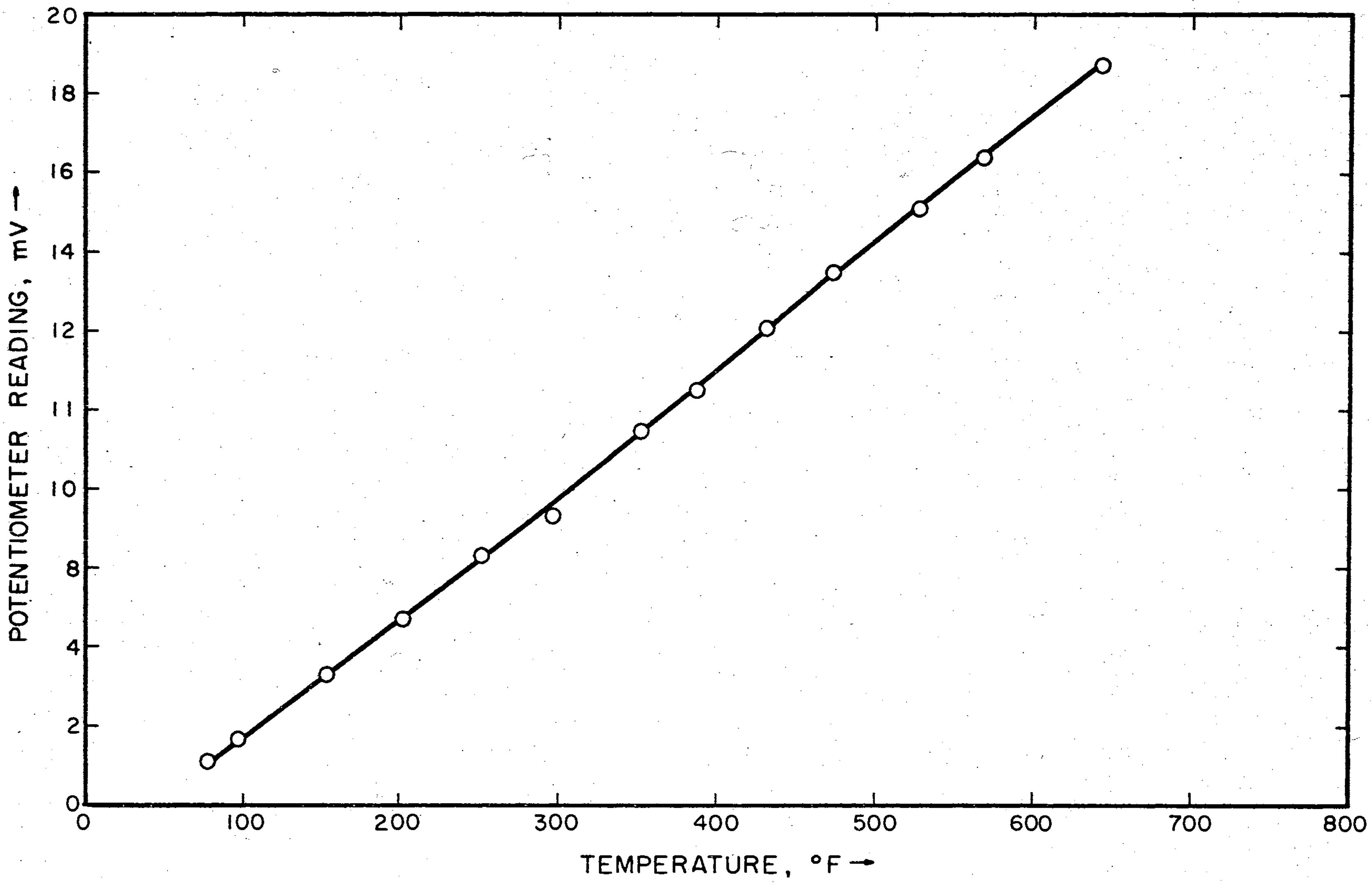

Figure 13. Calibration Curve for Thermocouple 4

APPENDIX B

TABLE B-I

CRITICAL CONSTANTS AND HEAT AND FREE ENERGY OF FORMATION

Substance	T_c, °K	P_c, Atm.	z_c	ΔH_f @25°C kcal/mole	ΔG_f @25°C kcal/mole
Sulfur	1313.0	116.0	-	+31.02	+19.36
Hydrogen	33.3	12.8	0.304	0	0
H_2S	373.6	88.9	0.284	-4.77	-7.85

Note: The above values were taken from Perry (17) and Hougen and Watson (10).

TABLE B-II

VAPOR PRESSURE OF SATURATED SULFUR (24)

Temperature, °F	Pressure, Atm.
248	0.00004
284	0.00014
320	0.00044
356	0.00116
392	0.00279
428	0.00605
464	0.012
500	0.0226
536	0.0397
572	0.0663
608	0.1060
644	0.164
680	0.244
716	0.354
752	0.500
788	0.690
824	0.937
832.3	1.000
860	1.25
896	1.63
932	2.12
968	2.68
1004	3.40
1040	4.246
1076	5.255
1112	6.44
1148	7.821
1184	9.417
1194.8	10.00

APPENDIX C

SAMPLE CALCULATIONS

Sample calculations are shown for:

a. determination of the percentage conversion and residence time.

b. determination of the reaction order and specific reaction rate constant.

The experimental data for these calculations are given in Table II.

a. Calculations have been shown for Run 12 in Table II at 317.5°F.

Weight of catalyst = 75.49 grams
Input rate of H_2S gas at S.T.P. = 360 cc/min
= $3.60 \times 10^{-2}/2.2414 \times 10^{4}$
= 1.601×10^{-2} gmole/min
= $1.601 \times 3.408 \times 10^{-2} \times 10^{+1}$
= 0.547 grams/min
= 0.547×10.766 grams
= 5.88 grams
Weight of H_2S passed = 5.88 grams
Weight of H_2S undecomposed = 0.400 grams (from H_2S absorber flasks)
Weight of H_2S decomposed = 5.480 grams
G-moles of H_2S decomposed = $5.480 \times 10^{-1}/3.408$
= 1.605×10^{-1} gmoles
= $1.605 \times 2.2414 \times 10^{-1} \times 10^{4}$
= 3,600 cc

Volume of gas measured by WTM = 3,680 cc at 79°F (299.3°K) and 750 mm.Hg. abs.

$3680 \times 750/299.3 = V_2 \times 760/273.2$

$V_2 = 3680 \times 750 \times 273.2/760 \times 299.3$

$= 3310$ cc at 760 mm.Hg. and 0°C

Volume of hydrogen produced = 3600 cc

Volume of hydrogen collected in WTM = 3310 cc

Volume of gas unaccounted = 290 cc

Percent error = $290 \times 10^2/360 \times 10.766$

= 7.49%

Percent Conversion:

Volume of gas measured by WTM = 342 cc/min

Volume of gas at S.T.P. = 308 cc/min

G-moles of gas = $3.08 \times 10^{-2}/2.2414 \times 10^4$

= 1.370×10^{-2} gmoles/min

Conversion = $1.370 \times 10^{-2}/1.601 \times 10^{-2}$

= 0.856

Percent Conversion = 85.6%

Residence Time = 75.49/0.547

= 1.38×10^2 g-cat/g-H_2S/min

The other values were calculated in a similar fashion.

b. The order of the reaction and the specific reaction rate constant were calculated by using a modified differential analysis on the data, as outlined by Levenspiel (12) and by Smith (18).

A sample calculation at 317.5°F is given below:

From Table II

TABLE C-I

No.	1	2	3	4
Feed rate, g/min	0.547	0.980	1.439	1.766
$W/(F'_{H_2S})_o \times 10^{-2}$, $\frac{\text{g-cat}}{\text{g-}H_2S\text{/min}}$	1.380	0.760	0.516	0.419

TABLE C-I (Continued)

No.	1	2	3	4
$W/(F_{H_2S})_o \times 10^{-3}$, $\frac{\text{g-cat}}{\text{gmole } H_2S/\text{min}}$	4.710	2.620	1.760	1.430
X_{H_2S}, dimensionless	0.856	0.822	0.495	0.318
C_{H_2S}, g/min	0.037	0.030	0.553	0.942
$C_{H_2S} \times 10^{3}$, gmoles/min	1.085	0.880	1.620	2.760
Vol. rate at reactor exit, liter/min	0.495	0.889	1.312	1.610
$C_{H_2S} \times 10^{2}$, gmoles/liter	0.219	0.099	1.231	1.715

The values of X_{H_2S} and C_{H_2S} were plotted as a function of $W/(F_{H_2S})_o$ in Figures 14 and 15, respectively. From Figure 14, slopes were taken at various $W/(F_{H_2S})_o$ to obtain the differential rate, and from Figure 15, the corresponding concentrations were obtained for the same values of $W/(F_{H_2S})_o$. The values obtained are given in Table C-II.

TABLE C-II

No.	1	2	3	4
$W/(F_{H_2S})_o \times 10^{-3}$, $\frac{\text{g-cat}}{\text{gmole } H_2S/\text{min}}$	1.0	2.0	3.0	4.0
$r_{H_2S} \times 10^{4}$, $\frac{\text{gmole } H_2S}{(\text{g-cat})(\text{min})}$	2.16	3.70	0.464	0.222
$C_{H_2S} \times 10^{+2}$, $\frac{\text{gmole } H_2S}{\text{liter}}$	2.580	1.090	0.580	0.310

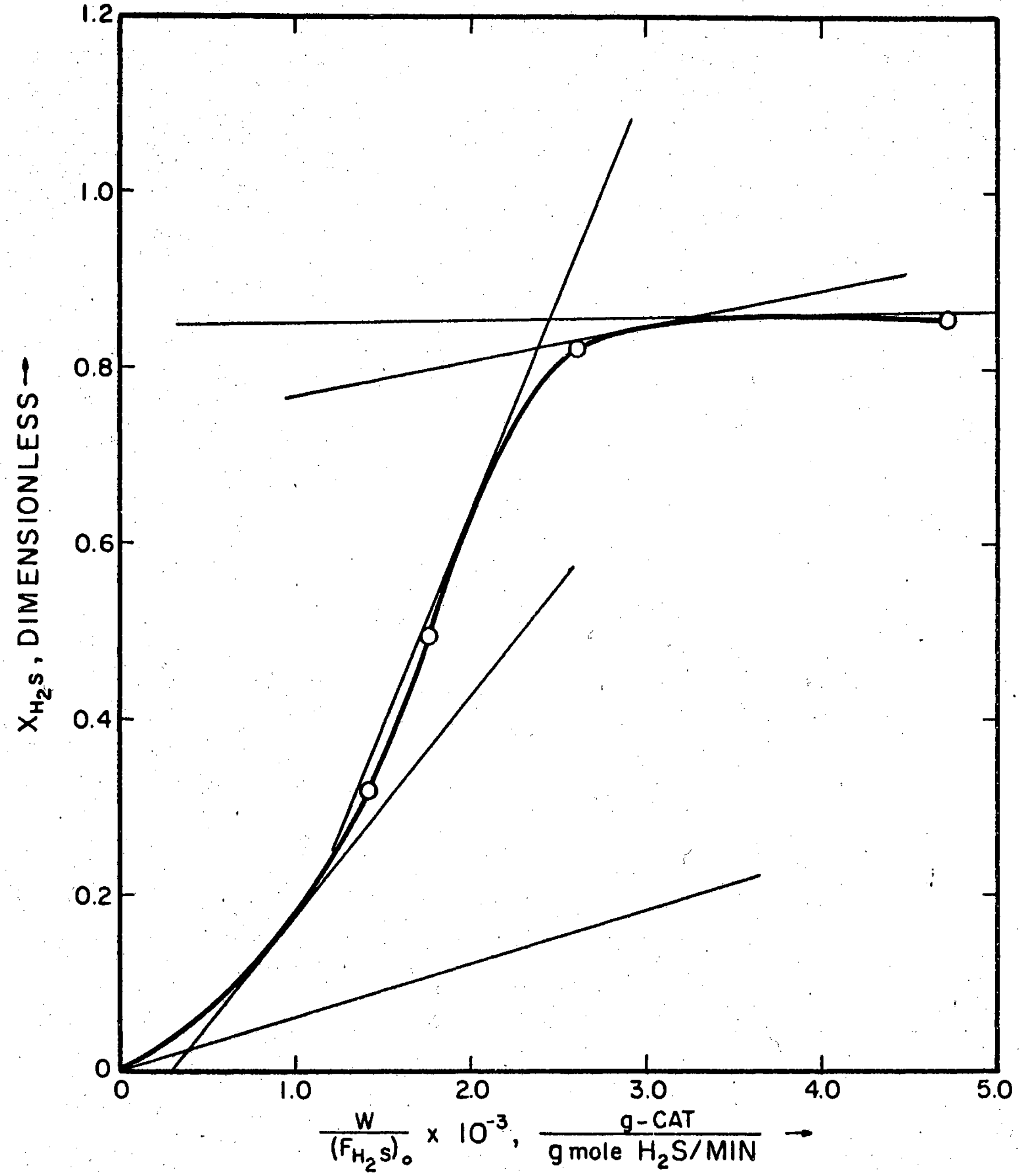

Figure 14. Determination of the Rate of the Reaction from Experimental Data

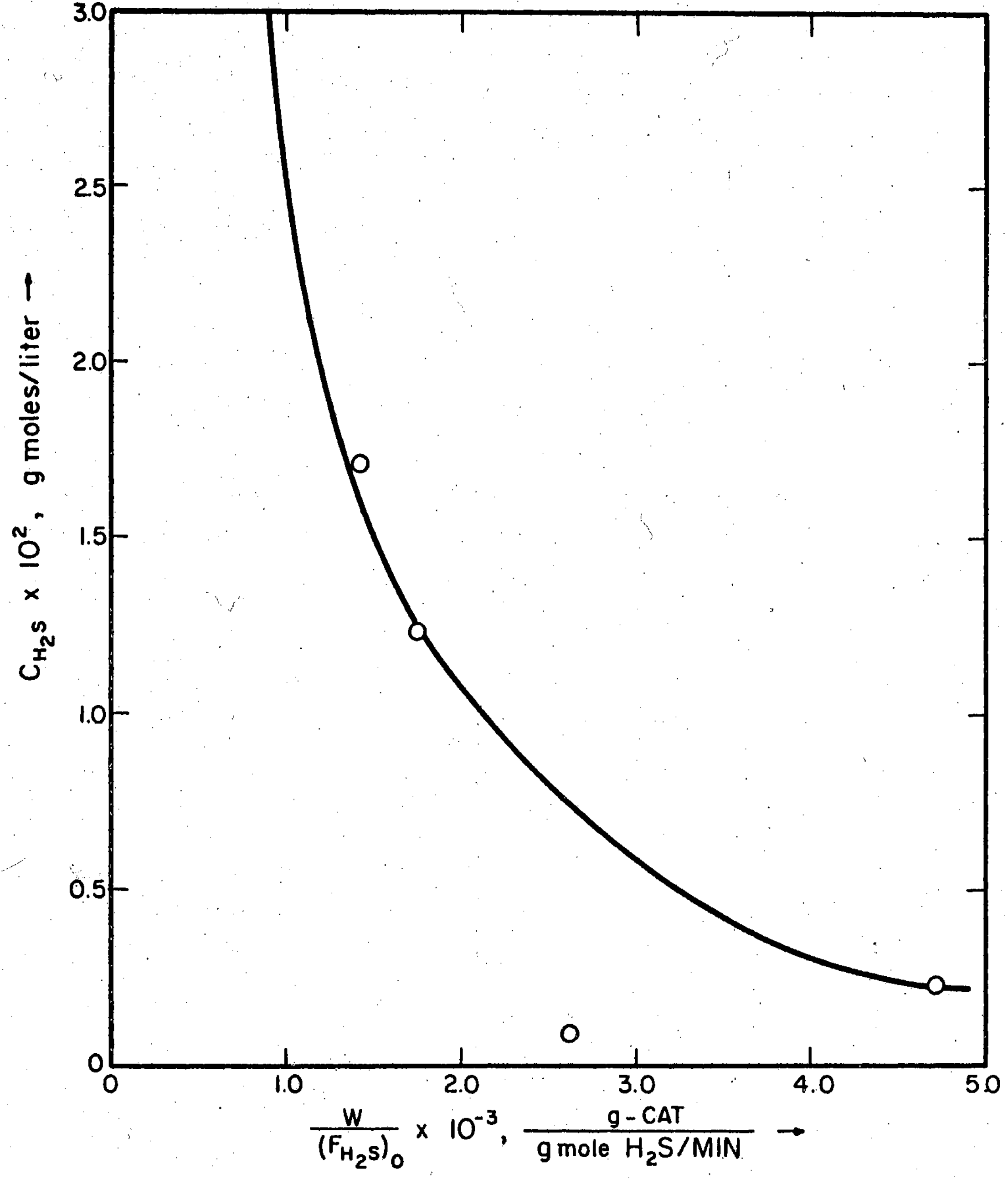

Figure 15. Concentration Variation with Residence Time for the Catalytic Decomposition Reaction

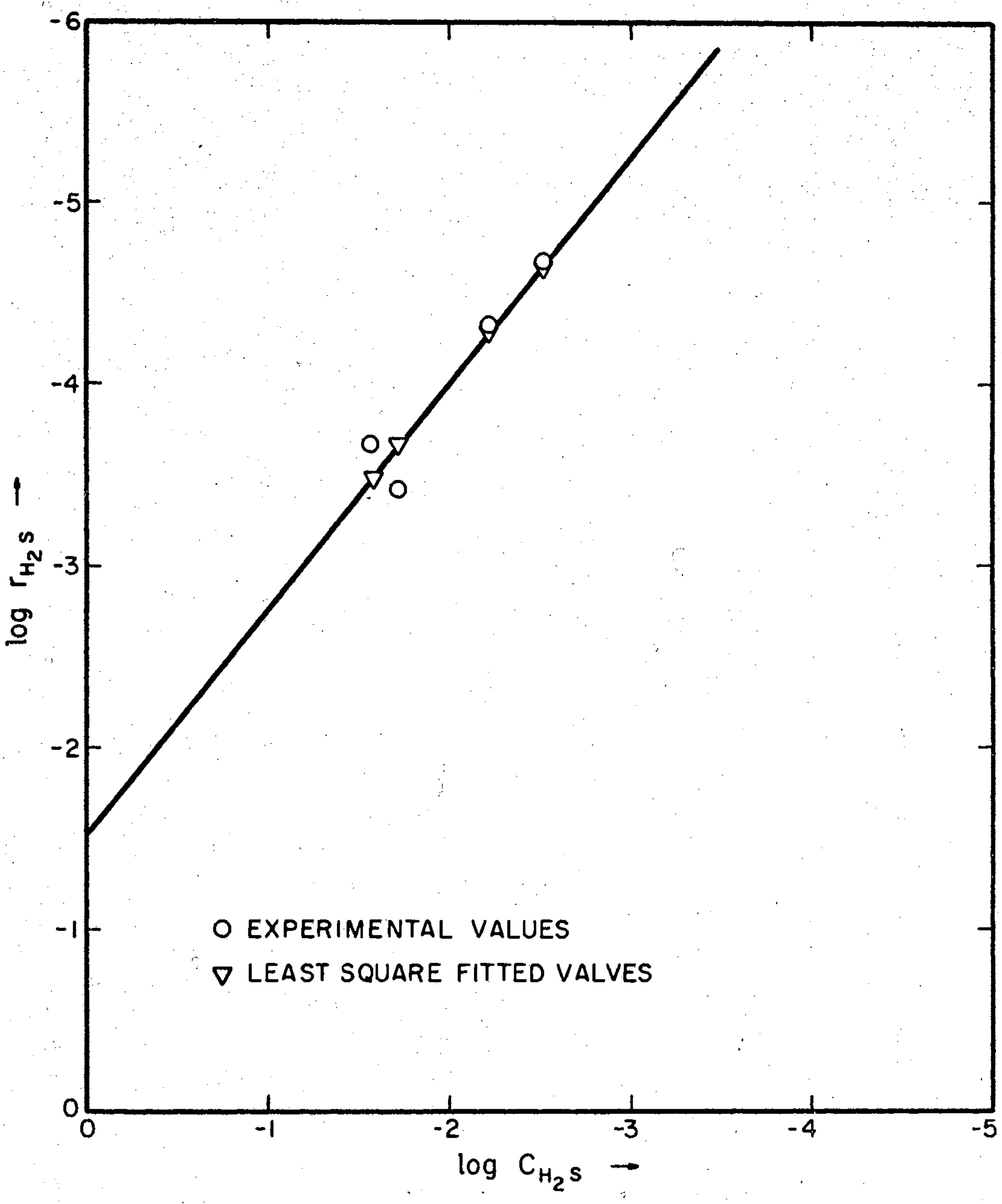

Figure 16. Determination of Order and Rate Constant for the Decomposition Reaction

Let $r_{H2S} = k(C_{H2S})^n$

where k = specific reaction rate constant

and n = order of the reaction

or, $\log r_{H_2S} = \log k + n\log C_{H_2S}$

TABLE C-III

No.	1	2	3	4
$\log r_{H_2S}$	-3.666	-3.432	-4.334	-4.654
$\log C_{H_2S}$	-1.588	-1.722	-2.236	-2.508

Figure 16 is the plot of log r_{H_2S} versus C_{H_2S}. A regression analysis was performed on the values to obtain the best fitting line. The order and the specific reation rate constant were calculated from the best fitting line and the equation thus:

$$n = \text{slope} = \frac{-5.24 - (-2.75)}{-3.0 - (-1.0)} = \frac{-2.49}{-2.0} = 1.245 \cong \underline{1.0}$$

$$\log k = -1.485449 \text{ (from the equation)}$$

$$k = 0.0326 \frac{\text{(liter)}}{\text{(g-cat)(min)}}$$

NOMENCLATURE

C_A - concentration of component A, gmole/liter

(F_A) - molar feed rate of component A, gmole/min

(F'_A) - mass feed rate of component A, gram/min

k - specific reaction rate constant, (liter)/(g-cat)(min)

K_a - equilibrium constant

P_c - critical pressure, atm.

t - temperature, °F

T - absolute temperature, °K

T_c - critical temperature, °K

W - weight of catalyst, grams

X_A - fractional conversion of component A, dimensionless

z_c - critical compressibility factor

Subscripts

o - initial value

Greek Letters

ΔG_f - free energy of formation, kcal/mole

ΔG^o - standard free energy change, kcal/mole

ΔH^o_f - heat of formation, kcal/mole

τ - residence time, (g-cat)(min)/(g-mole H_2S)

ϵ_A - fractional change in volume of A on reaction

Abbreviations

abs. - absolute

atm. - atmosphere

Alu - Alumel

cals - calories

cat - catalyst

cc - cubic centimeter

Chr - Chromel

Con - Constantan

Fe - Iron

g, gm - gram or grams

ga. - gauge

kcal - kilo calories

min - minute

mm.Hg. - millimeters of mercury

p.s.i. - pounds per square inch

psia - pounds per square inch absolute

psig - pounds per square inch gauge

S.T.P. - standard temperature and pressure (760 mm.Hg. and 0°C)

thru - through

vol. - volumetric

WTM - Wet test meter

Symbols

( ) - literature reference

[ ] - equipment reference

VITA

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