

ABSORPTION OF HYDROGEN SULFIDE BY BENZONITRILE
AND TRIETHYLENE GLYCOL

By

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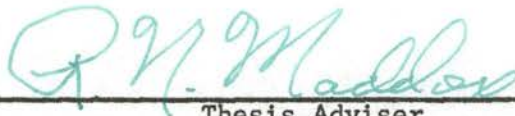
June, 1963

Submitted to the faculty of the Graduate School of
the Oklahoma State University
in partial fulfillment of the requirements
for the degree of
MASTER OF SCIENCE
May, 1965

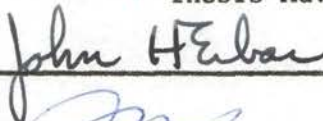
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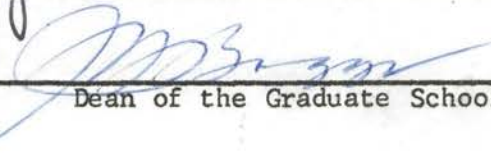
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Thesis Adviser





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PREFACE

Liquid-gas equilibrium data of benzonitrile with hydrogen sulfide and carbon dioxide, both separately and as a mixture, were obtained. The same procedure was repeated by using triethylene glycol instead of benzonitrile. The comparison of both solvents in their absorption of the mentioned gases was accomplished.

The work done is by no means the result of a single person's efforts. During the course of this study a great deal of help was obtained from Eugene McCroskey and D. H. Knoebel in the construction of the apparatus. The help of the staff and the graduate students of the School of Chemical Engineering is appreciated. I am also thankful to Louis Wilson, from the Department of Chemistry, for his aid in explaining the chemical reactions involved. Finally I am deeply grateful to Professor R. N. Maddox for his kind guidance in all phases of this work.

The financial aid necessary to pursue my graduate studies was generously supplied by ATAS Refinery of Socony Mobil in Turkey.

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CHAPTER I

THEORY

Gas absorption is an operation by which certain components of a gas mixture are dissolved in the liquid as a result of gas-liquid contact. There are two kinds of gas absorption, namely physical and chemical. In physical absorption gas is absorbed physically by the liquid. However in chemical absorption gas is absorbed as a result of a reaction between the two phases.

Gas can be absorbed by the liquid until equilibrium is established between two phases at constant temperature and pressure. If we fix the temperature and vary the pressure, the amount absorbed will increase as the partial pressure of the gas component increases. A typical curve for that situation is shown in Figure 1. If the liquid in question was ideal and the gas obeyed the ideal gas law, the curve would then be a straight line starting from the origin. Deviation from a straight line represents

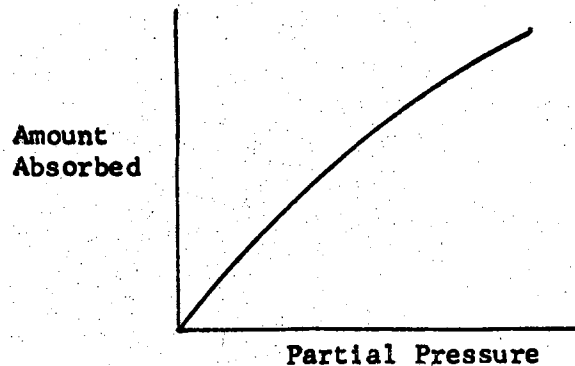


Figure 1. Amount absorbed versus partial pressure

departure from this ideality (12).

Gas absorption is applied in plants where it is wanted to recover from the gas some valuable components or eliminate the ones which are objectionable. In industry it is a great concern to eliminate acidic gases such as hydrogen sulfide or carbon dioxide (7). This is because of their corrosive nature, poisoning of the catalyst, and pollution of atmosphere. Therefore to eliminate them from liquids or gases various absorption processes have been developed. A few names that can be mentioned are: Girbitol, Glycol-amine (9), and sulfinol processes (3).

Generally the operation consists of absorbing the gases in a tray or packed tower, and then stripping them at relatively high temperatures. The choice of the solution used depends on its absorption capacity and on how well it can be regenerated after the gases are stripped.

CHAPTER II

INTRODUCTION

Before transferring natural gas by pipelines, it is essential that acidic gases should be removed. Among these gases carbon dioxide is an odorless component which is noncorrosive in absence of water. Therefore it is not necessary to remove it if there is no water in the system.

In July 8, 1963 issue of "The Oil and Gas Journal," (5), an article on the removal of hydrogen sulfide and water from gas streams has been published. The solution used for this removal is a mixture of benzonitrile and triethylene glycol. The process is claimed to be most applicable to systems where other sulfur compounds are in relatively low quantities. The solution is said not to absorb carbon dioxide.

However another source (8) after performing separate experiments on both chemicals has reported that benzonitrile absorbs carbon dioxide. It is also mentioned that there is no need for benzonitrile because triethylene glycol showed to be as good an absorber of hydrogen sulfide as benzonitrile.

The purpose of this work has been to investigate the claimed properties of benzonitrile. The investigation was done by obtaining equilibrium data for both chemicals with the two gases. As a result it was then possible to compare their absorption capacities for hydrogen sulfide, and find out whether or not they absorb carbon dioxide. To determine the effect of each gas on the absorption of the other both chemicals were treated by a

mixture of carbon dioxide and hydrogen sulfide. All of the experiments were performed at the same temperature and at various partial pressures of gases.

CHAPTER III

DESCRIPTION OF APPARATUS

The apparatus used can be classified as follows:

1. Gas treatment system
2. Constant temperature bath
3. Sampling system
4. Chromatographic analysis system

Gas Treatment System

The apparatus is shown in Figure 2. The gases were in cylinders which were equipped with pressure controllers. A model 11-330 Matheson regulator was used for hydrogen sulfide. For carbon dioxide an Airco regulator, whose connection number was 320, was used. The two cylinders were connected by a three-way stopcock, the third way of which led to a mercury manometer. One end of the manometer was open to the atmosphere and the other end was in contact with the gas line. The gas line ended at the point where it was connected to the glass bomb. The connections were made by neoprene rubber hoses. To prevent a reaction between the mercury of the manometer and hydrogen sulfide, water of height 1 cm. was kept over the mercury in both legs of the manometer. A scale, divided into one tenth inch increments, was used for measurement of pressure changes.

The glass bomb, Figure 3, was made of pyrex glass which had two stopcocks joined to it. The bomb was in the form of a cylinder whose two ends

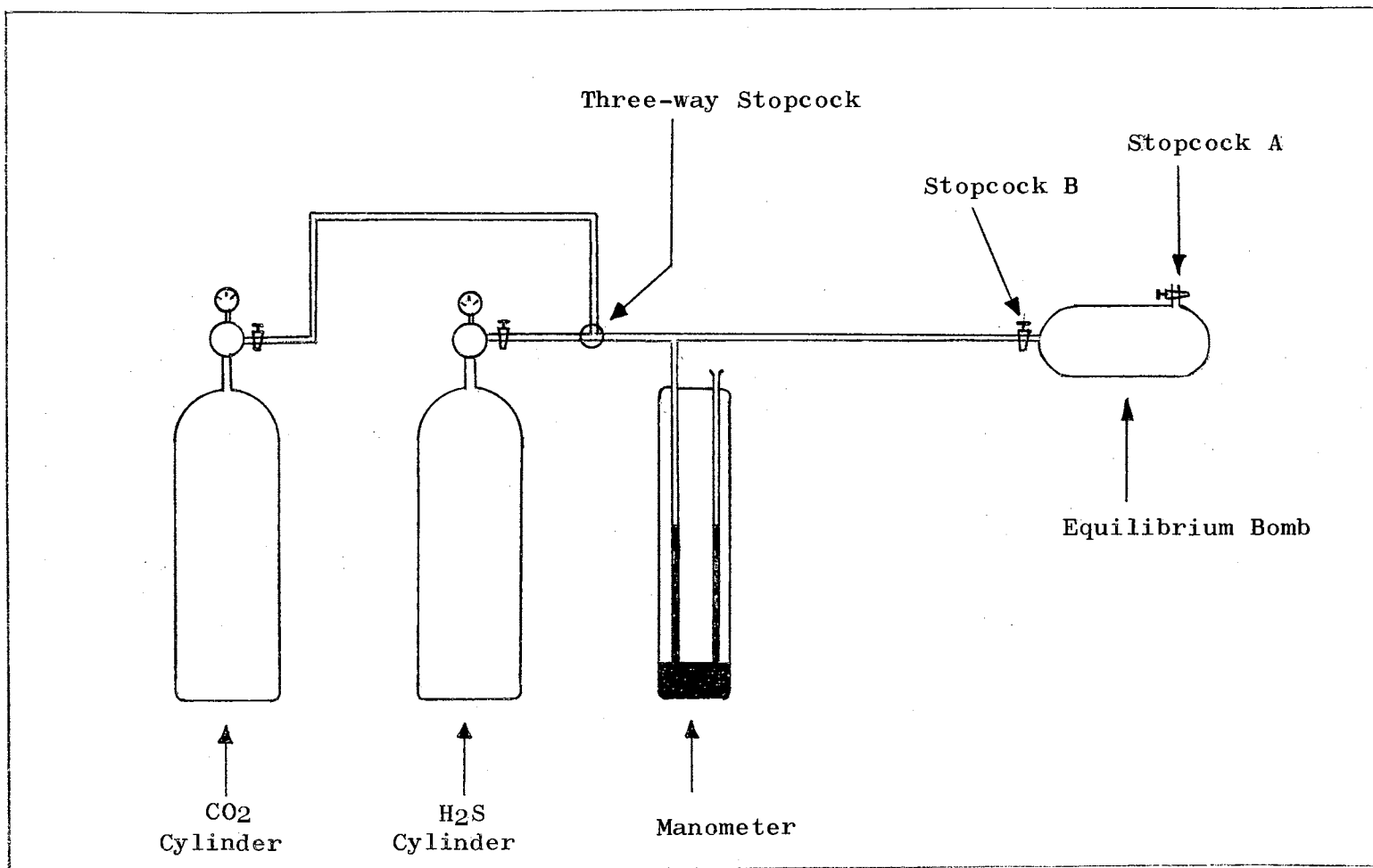


Figure 2. Gas Treatment System

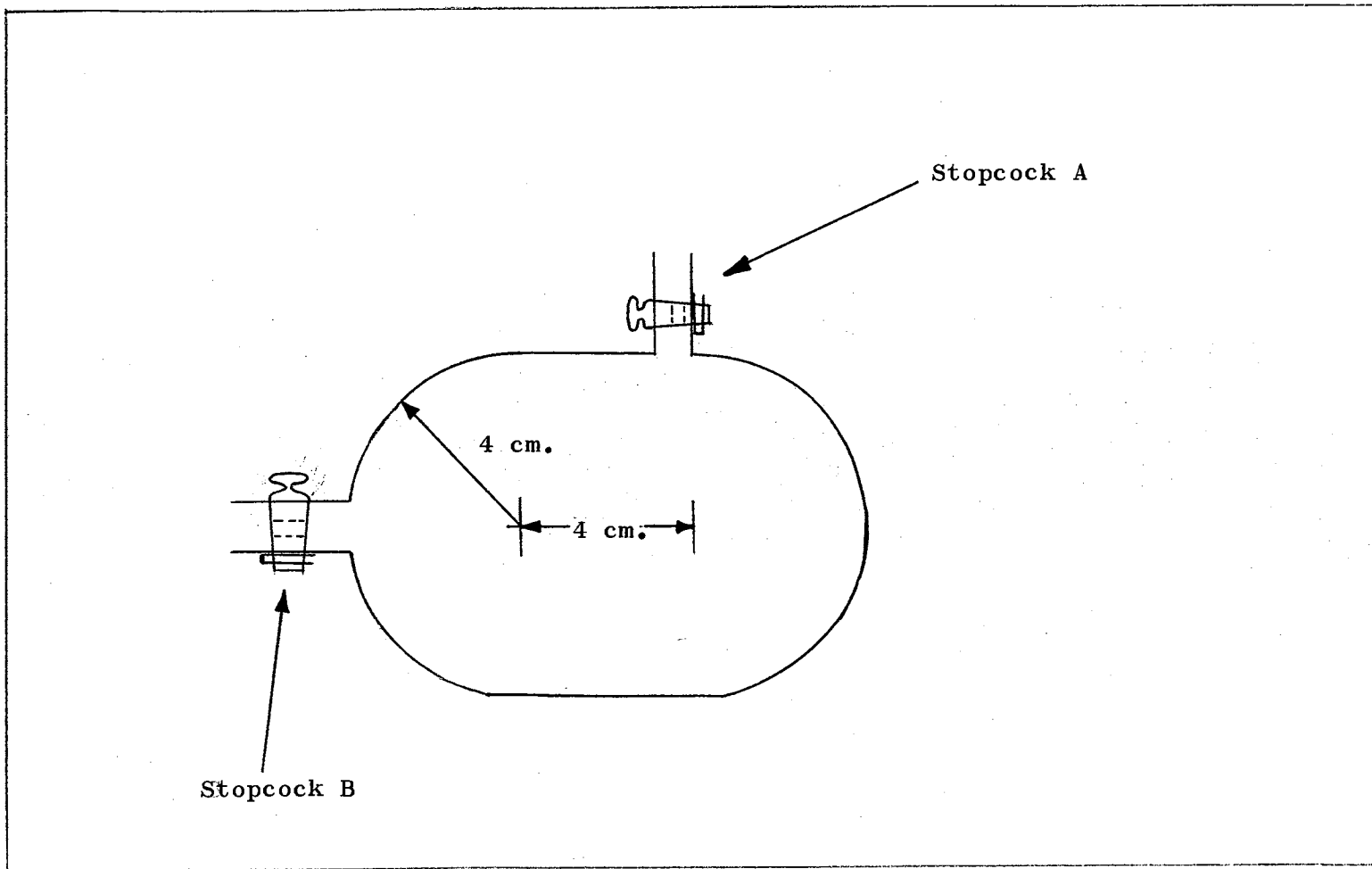


Figure 3. Glass Bomb

were covered by hemispheres. The diameter of the cylinder was 8 cm. and the total length, with the hemispheres included, was 12 cm. One stopcock was joined to the top of a hemisphere and another was joined to a point where the cylinder and the other hemisphere were joined together.

Constant Temperature Bath

Since all the points were to be determined at the same temperature, a constant temperature bath was necessary. The bath is shown in Figure 4. The bath was lined with copper. It was 60 cm. long, 35 cm. wide, and 35 cm. deep. The bath liquid was water and the temperature was kept at 30° C. Water was put into the bath through a line from the tap water system and was drained by an outlet valve connected to the bottom of the bath. A mercury thermometer was used to measure the temperature of the bath. It was divided into one degree Centigrade increments. The temperature was controlled by a Model 63 Thermistemp temperature controller made by the Yellow Springs Instrument Company. The heat source for the system was a 500 watt Vulcan electric heater. The voltage of the heater was controlled by a Type 116 variac made by the Superior Electric Company. For the cold sink tap water was circulated through copper coils put into the bath. The coils were $\frac{1}{4}$ " OD tubing. To keep the temperature uniform in every part of the bath a 20 watt 60 cycle stirrer made by Precision Scientific Company was used.

In the bath the bombs were tied to a bar by means of springs. The bar was shaped to fit the contours of the bombs. It was large enough to carry three bombs. Both ends of the bar were mounted on bearings to allow it to rock freely around its axis. The rocking was accomplished by a

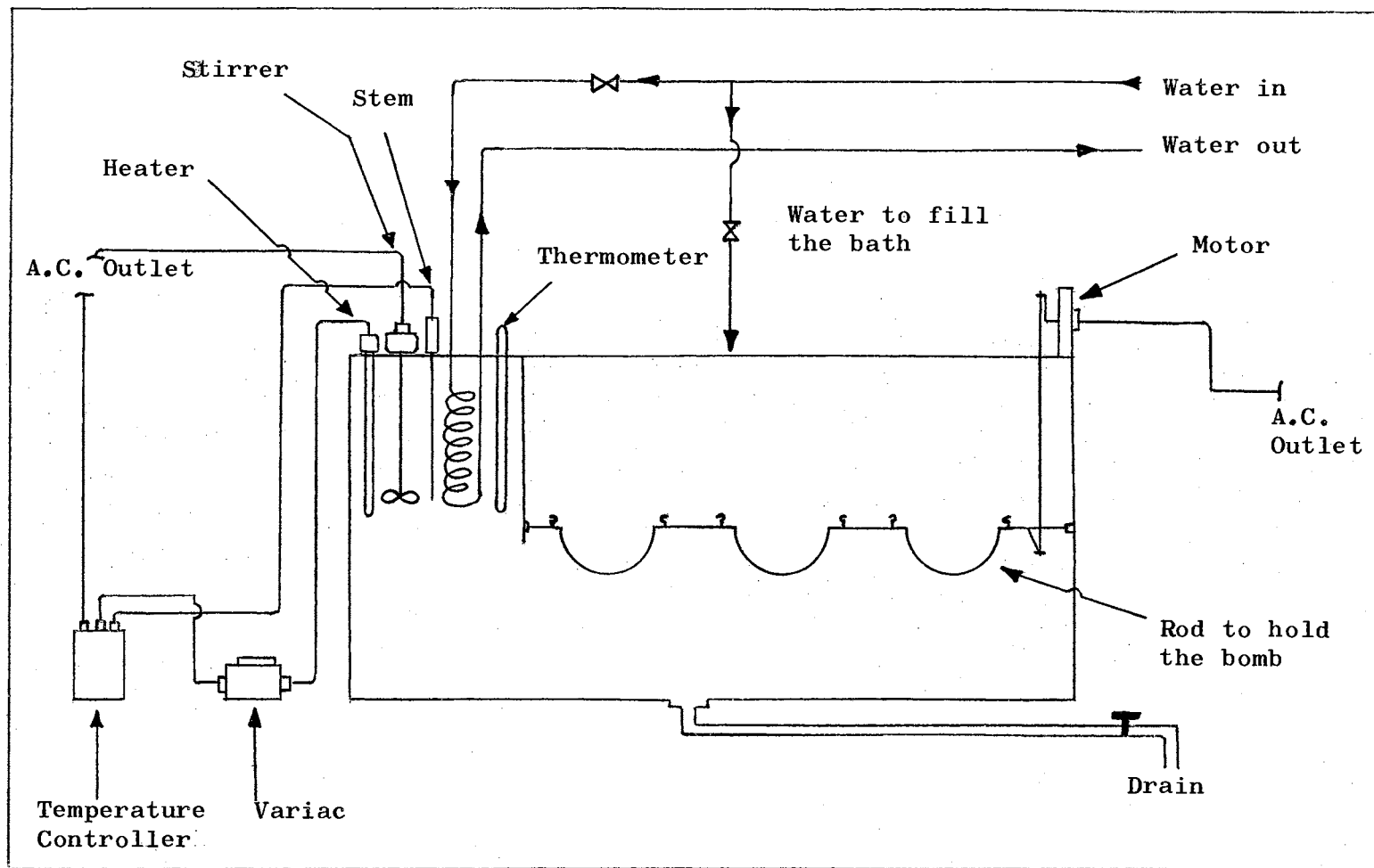


Figure 4. Constant Temperature Bath

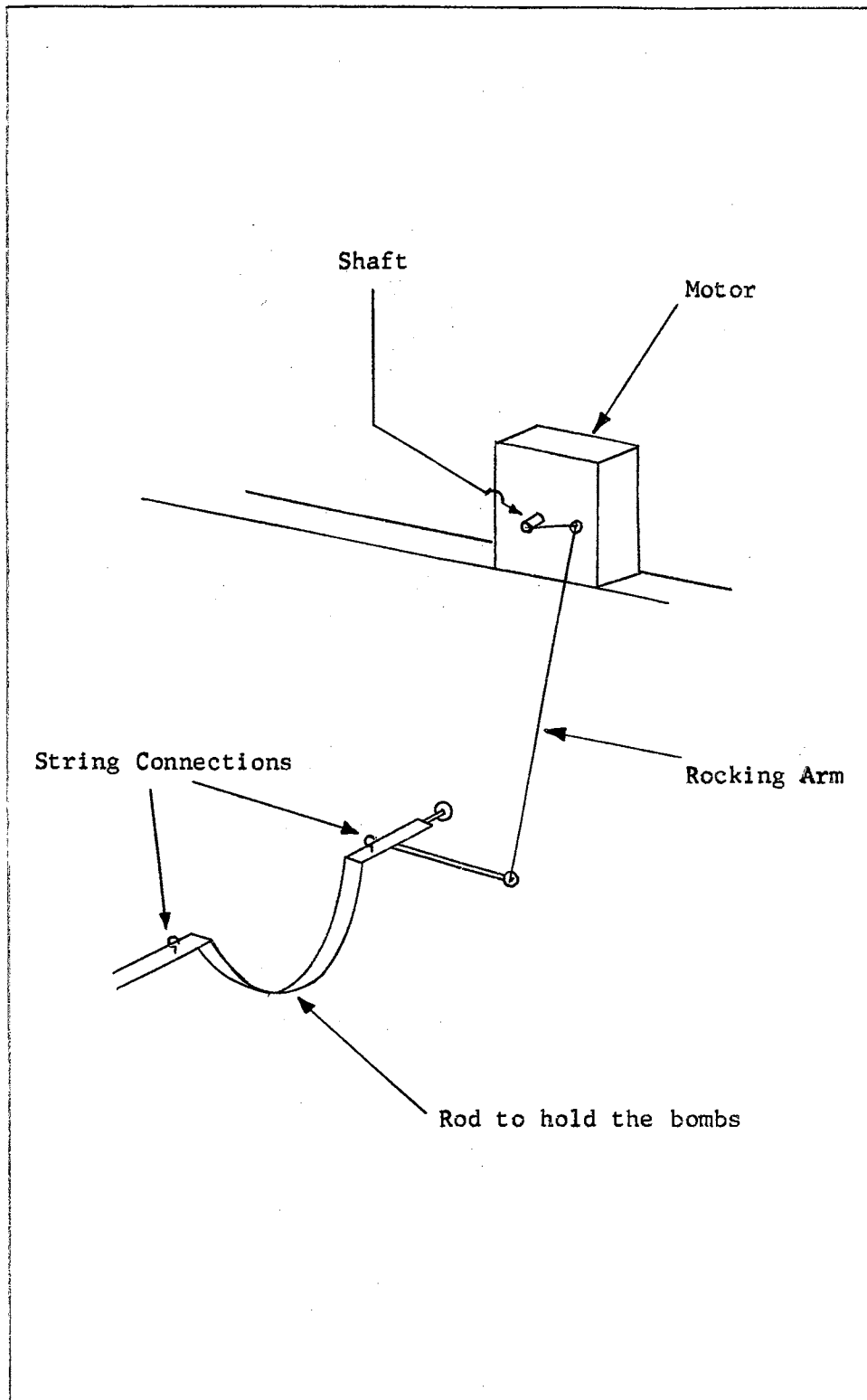


Figure 5. The Rocking System

shaded pole motor that had a speed of 90 rpm. Every time the shaft made a complete turn the bombs made an angle of 30° with the horizontal. The connections of the motor to the bar are shown in Figure 5.

Sampling System

The apparatus used is shown in Figure 6. One end of the equilibrium bomb was connected to a gas cylinder and a manometer by a three-way stopcock (A). The other end was connected to the sampling system which was made of pyrex glass. It had a bulb whose volume was determined to be 13.600 cc. Two 1 mm stopcocks, (D) and (E), were joined to the bottom and to the side of the bulb. The bulb was connected to a spherical liquid trap through a line of 7 mm OD. The length of the line was 20 cm. The spherical trap, with an outside diameter of 4.5 cm, had a delivery tube coming out of its other end. The line contained stopcock (F) and led to the precipitating tubes. These were 3 cm diameter, 25 cm long tubes sealed at one end. Their open ends were closed by two-hole rubber stoppers. One of the holes was used for the entering gas which went down the tube by a tygon tube. The other hole carried the gas from that tube to the next one. All the tubes were connected in series and their number varied for different experimental work. The last tube had a syringe connected to the outlet hole.

During the experiments tubes were filled with ammoniacal zinc solution or with barium hydroxide (13) depending on the gas to be precipitated.

Chromatographic Analysis System

For gas analysis a FM - 500 type chromatograph was used. The column was $\frac{1}{2}$ " OD copper tube and was 60 cm long. A 100-120 mesh silica gel was

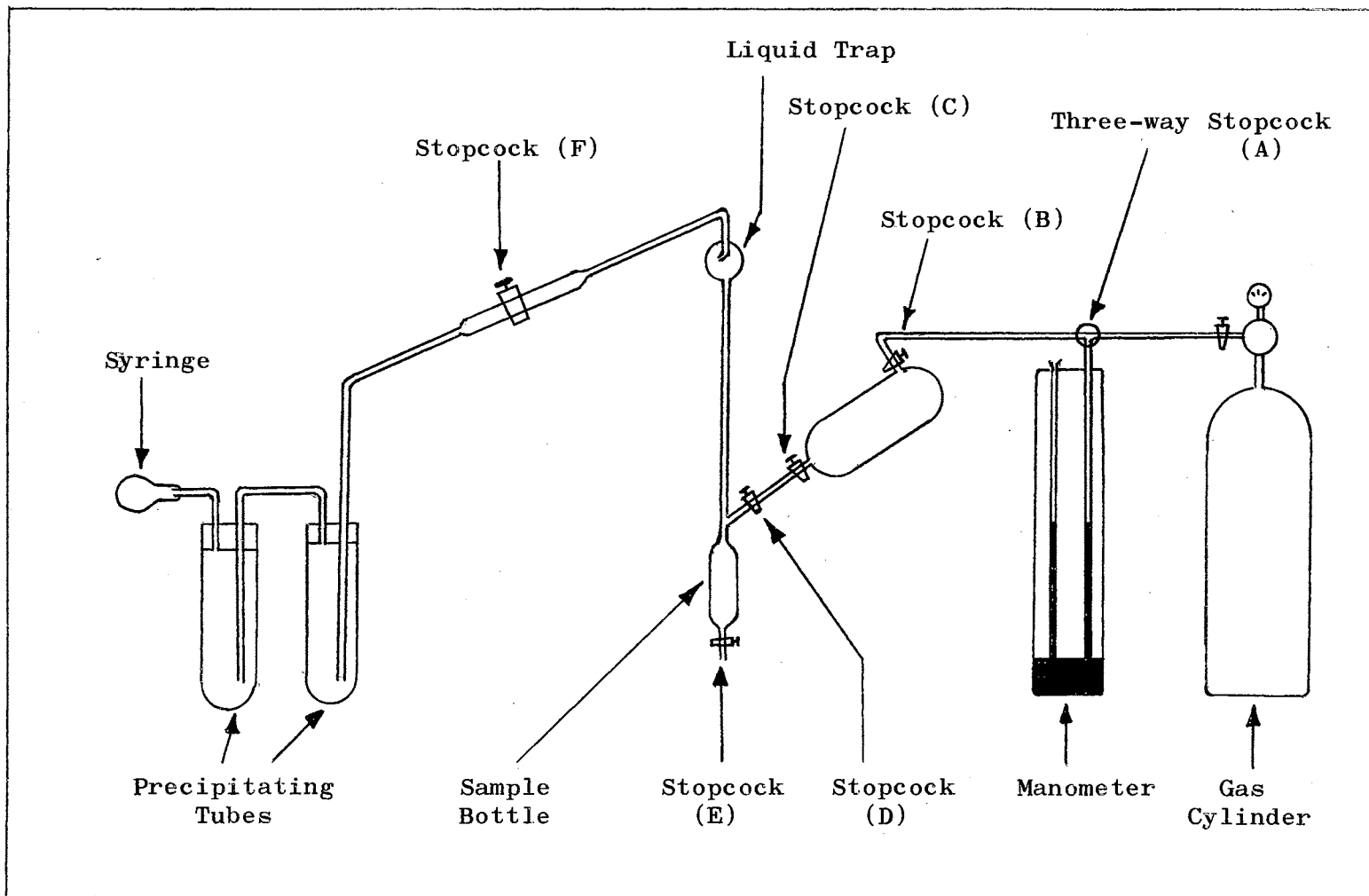


Figure 6. Sampling System

used for packing. The temperature of operation was 100° C. The gas samples were injected into the chromatograph by a 5.00 cc teflon syringe.

CHAPTER IV

EXPERIMENTAL PROCEDURE

Data were taken for four different cases which can be listed as follows:

- A. Gas-liquid equilibrium of pure solvents with hydrogen sulfide and carbon dioxide at different partial pressures.
- B. Determining how well benzonitrile can be regenerated.
- C. Determining the quantity of the absorbed gas which can be driven out of the solution by heat.
- D. Determining the effect of one gas on the absorption of the other.

Gas-Liquid Equilibrium of Pure Solvents With Hydrogen Sulfide and Carbon Dioxide at Different Partial Pressures

When a gas and an absorbent are put in a closed vessel a drop in pressure is observed as a result of absorption. If the amounts of gas and solvent are initially known, the quantity of absorbed gas can be determined from the measured pressure drop. This was the basic principle used in determining the liquid-gas equilibrium between the mentioned gases and the solvents. For this reason the volume of the bomb was calibrated before the experiments, and each time a known amount of solvent was used.

The procedure used in this set of experiments was as follows.

Initially the bomb was cleaned with water and ethanol. Then it was evacuated by means of a vacuum pump so that the solvent could be treated with the desired gas. After purging the lines with the gas, the connections shown in Figure 2 were made. Since one end of the manometer was open to atmosphere, before every gas treatment the atmospheric pressure was read from a barometer. The mercury height necessary to obtain the desired pressure in the bomb was calculated. The bomb was then filled with the gas by opening stopcock B. When the desired pressure was reached, stopcock B was closed and the bomb was disconnected from the system. Before putting the solvent into the bomb an equal amount (25.00 cc) of the gas was removed to keep the conditions unchanged with the addition of the solvent. This was accomplished by covering the outlet of the stopcock on the bomb with a rubber diaphragm and taking the gas with a syringe. The solvent was put in the bomb in the same way. The bomb was then put into the constant temperature bath shown in Figure 4. The motor connected to the shaft was started to rock the bombs. This made it possible to increase the rate of absorption. The rocking was carried on from fifteen minutes to one half an hour.

The apparatus shown in Figure 2 was used to measure the pressure drop caused by absorption. When the bomb was connected to the system, the three-way stopcock was arranged such that the bomb was open only to the manometer. Then stopcock B was opened. The mercury height was recorded, and immediately the three-way stopcock was arranged to open the bomb both to the manometer and the gas cylinder. Gas was let into the bomb until the initial mercury level was obtained. The bomb was again put into the constant temperature bath. This procedure was repeated until there was no pressure drop caused by absorption, which meant that

equilibrium was obtained.

A certain amount of gas would be absorbed while feeding the gas into the bomb. The pressure drop that would be caused by this quantity was not measured. To make this error negligible, gas was delivered to the bomb as quick as possible.

During the experiments three different pressures were used. These were 0.50, 1.00, and 1.50 atmospheres. At 1.00 and 1.50 atmospheres pressure the bombs were first evacuated and then filled with gas. However at 0.50 atmospheres the bombs were first brought to 0.50 atmospheres by the addition of argon and then the desired gas was put in up to a total pressure of 1.00 atmospheres. In this way it was possible to have a partial pressure of 0.50 atmospheres for the gas and also experimental difficulties such as leaks were eliminated. However to find out whether this was a valid procedure or not, benzonitrile was treated with hydrogen sulfide and also with a mixture of hydrogen sulfide and argon where the partial pressure of hydrogen sulfide was 0.50 atmospheres. The results showed that equal amounts of hydrogen sulfide were absorbed by benzonitrile in both cases.

All the experiments were done in a room where the temperature varied between 30° C and 35° C. Since the constant temperature bath was at 30° C an error was introduced in taking the bombs out of the bath for gas treatment. Because the thermal conductivity of glass and the temperature gradient were very small, this effect was neglected. Besides, the whole procedure of gas treatment was accomplished within two minutes which was not a sufficiently long time to bring any significant changes to the system.

During the experiments the vapor pressures of the solvents were not

taken into account. This was due to their very low values. At 30° C benzonitrile is reported (4) to have a vapor pressure of 1 mm Hg. Triethylene glycol has a vapor pressure less than 1 mm Hg. Since these values are beyond the limit of accuracy of pressure measurement, their effects were not considered.

Determining How Well Benzonitrile Can Be Regenerated

The second group of experiments were carried out to find out how much hydrogen sulfide can be absorbed by benzonitrile after it has been regenerated. All the work was done at a partial pressure of 1.00 atmospheres of hydrogen sulfide.

First the gas was treated with the solvents as explained in the Case A experiments. When equilibrium was reached the solvent was transferred to an open beaker and heated until the gas was driven out. Benzonitrile was then cooled to 30° C and the previous gas treatment was repeated. After equilibrium was again reached, the absorbed gas was again driven out by heat and the solvent was cooled for another gas treatment. The experiments were terminated at the end of the third gas treatment.

Determining the Quantity of the Absorbed Gas Which Can Be Driven Out of the Solution by Heating

Experiments were done to determine the quantity of the absorbed gas that can be driven out of the solvents by heat. Two basic analyses were made in this group of experiments. These were determination of hydrogen sulfide and carbon dioxide. In both cases volumetric quantitative methods were used.

Hydrogen sulfide was precipitated by ammoniacal zinc solution. The

precipitate was treated with hydrochloric acid. The reaction made hydrogen sulfide evolve. The evolution might have caused some loss of gas that could lead to wrong results. Therefore a known amount of standardized potassium iodate-iodide solution was added immediately. This captured all the hydrogen sulfide and stopped its loss. The solution was then back titrated with standard sodium thiosulphate until the dark blue color formed by starch indicator disappeared (1).

Barium hydroxide was used in precipitating carbon dioxide. A known amount of standardized solution was put in the precipitating tubes. After precipitating carbon dioxide, the amount left was determined by titrating it with standard hydrochloric acid (13). In the experiments current quantitative methods were used to standardize the solutions (14).

The steps followed were:

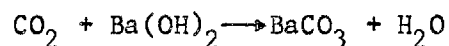
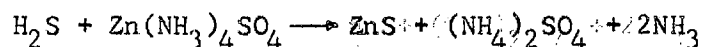
1. Gas treatment
2. Reaching equilibrium
3. Sampling
4. Analysis

The solvents were treated with gas in exactly the same way as in Case A until equilibrium was reached.

When equilibrium was reached, a known amount of sample was taken from the bottle and analyzed. The apparatus is shown in Figure 6. The reason for connecting the bomb to the gas cylinder and the manometer was to keep the pressure constant while sample was being taken. This was done by pressuring the line to the pressure of the bomb. Then stopcock B was opened. Since both pressures were equal, no change

in levels was observed on the manometer. Then stopcock C was opened. This made the solvent flow to stopcock D. Meanwhile stopcock E was closed and stopcock F was opened. To let the liquid enter the bulb, a very small opening was made by stopcock D. This small opening made it possible to control the liquid flow. Sometimes the flow stopped because of the pressure inside the bulb. This was eliminated by a syringe connected to the last precipitating tube. Its function was to take some of the gas out through the precipitating tubes and hence reduce the pressure in the apparatus. As a result, the flow of liquid continued into the bulb.

When the bulb was full up to the calibration mark, the flow was stopped by closing stopcocks C, D, B, and the needle valve of the cylinder. The syringe and the bomb were disconnected. To drive out the gas absorbed by the solvent the bulb was heated slowly under a low flame. As the gas came out it passed through the precipitating tubes where it was precipitated. The reactions occurring for the two cases were:



The heating continued until the bubbling of the gas through the tubes ceased. Then stopcock E was closed to stop any solution from climbing back through the line because of the low pressure formed inside the apparatus upon cooling. When the bulb became cool, a connection was made to an argon cylinder through stopcock D. Meanwhile stopcock E was opened very slowly and argon started to go through the line. The purpose of this was to drive out any gas that was left in the lines. Since argon passes through the solution without precipitating, only the other gas coming out was precipitated. This went on until no more precipitation would occur. Then stopcock E was opened to drain out the solvent. The

precipitate in the tubes was transferred to flasks and the tubes and lines were washed with deionized water, which was also added to the flasks. The precipitate was then analyzed.

For each experiment care was taken to have sufficient number of precipitating tubes. Otherwise some gas would go out without being trapped. In the experiments it was observed that the last one or two tubes contained clear solutions which meant that complete precipitation was obtained.

Determining the Effect of One Gas on the Absorption of the Other

The fourth group of experiments were performed by using mixtures of gases instead of pure components. All the points were taken at 30° C and at a total pressure of one atmosphere. The only variable was the partial pressures of gases.

The steps followed were:

1. Gas treatment
2. Obtaining equilibrium
3. Gas analysis
4. Sampling
5. Liquid analysis

Gas treatment and reaching equilibrium were accomplished in the same way as in Case A. The only differences were that the solvents were put in the bombs before the bombs were evacuated, and also mixtures of gases were fed to the bombs. The feeding was done by arranging the three-way stop-cock C of Figure 2.

As soon as equilibrium was reached, the composition of the gas in the bomb was determined. This was accomplished by taking the gas sample with a syringe and injecting it to the chromatograph. The gas analysis was

followed by sampling which was done as described in Case C.

In these experiments the precipitating tubes were divided into two groups. The first group contained ammoniacal zinc solution and the second group contained barium hydroxide. The two groups were connected in series as before. While the mixture of gases was passing through these solutions, hydrogen sulfide was precipitated by the first and carbon dioxide by the second group of precipitating tubes respectively. Carbon dioxide was not precipitated by ammoniacal zinc solution because it first had to react with water to ionize and then form the precipitate. However the time it stayed in that solution was not sufficient to bring the ionization (7). Therefore it was captured by barium hydroxide.

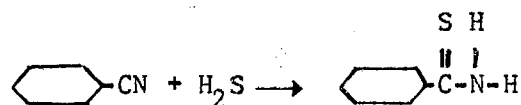
CHAPTER V

DISCUSSION OF RESULTS

Liquid-gas equilibrium data were obtained for benzonitrile and triethylene glycol with hydrogen sulfide at different pressures. The results are shown in Figures 7 and 8.

When the absorption capacities of the solvents for hydrogen sulfide are compared, it is seen that benzonitrile absorbs approximately 60 per cent more hydrogen sulfide by weight than triethylene glycol. This indicates that in a solution of both solvents most of the absorption is not due to the presence of triethylene glycol as claimed by a previous investigator (8).

Benzonitrile was checked for its absorption of hydrogen sulfide after regeneration. A sharp decrease was observed in the amount of hydrogen sulfide absorbed (Table III). Also, in treating benzonitrile with hydrogen sulfide, the color of benzonitrile turned to yellow. When the solvent was heated the yellow color remained. These suggested that an irreversible reaction was taking place between benzonitrile and hydrogen sulfide. The reaction is given in the literature (6) as:



This reaction is said to be reversible with cupric or lead oxides. Since such a compound was not used while heating, complete reversibility was unlikely to take place.

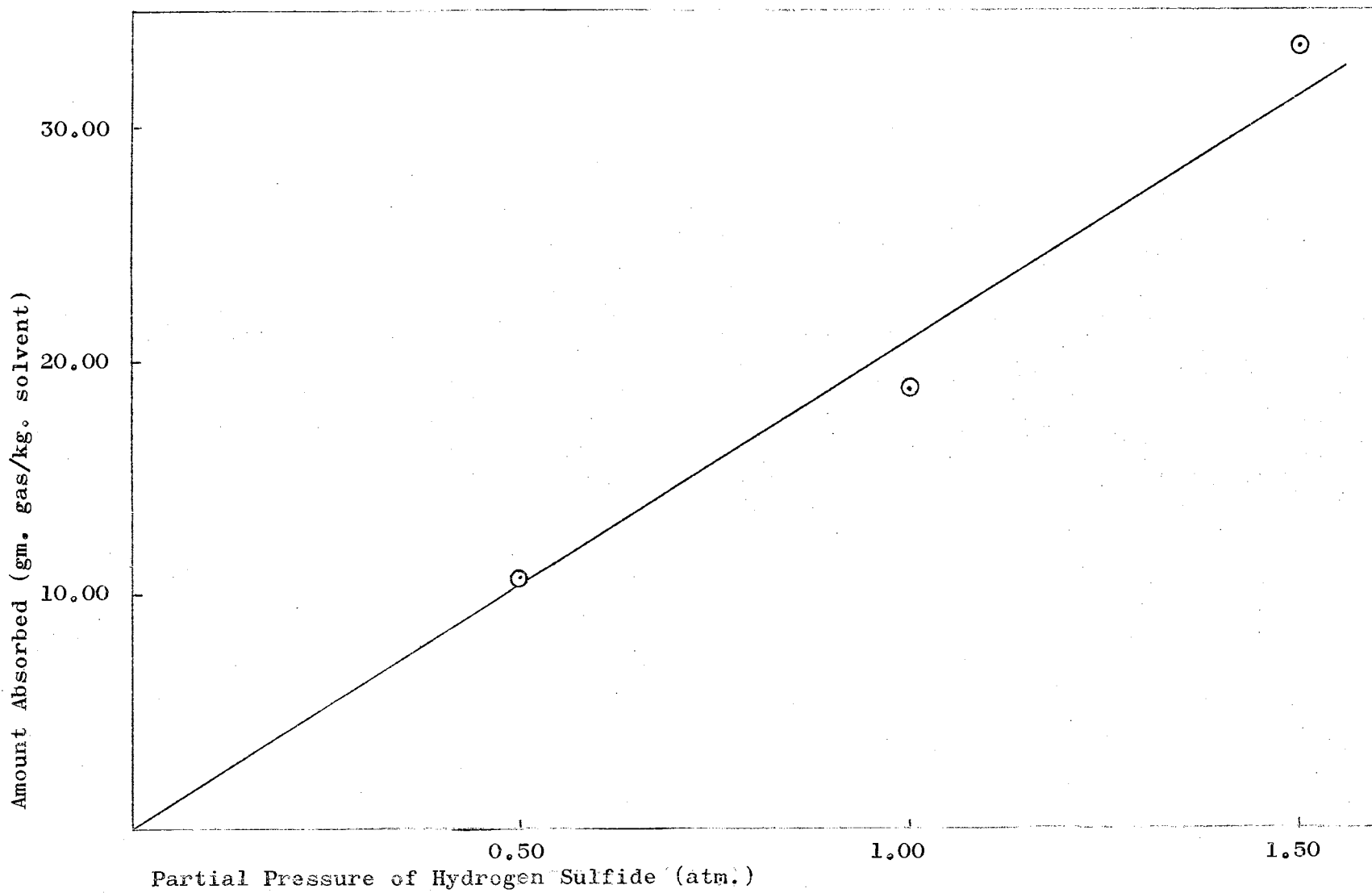


Figure 7. Gas-Liquid Equilibrium Between Hydrogen Sulfide and Benzonitrile

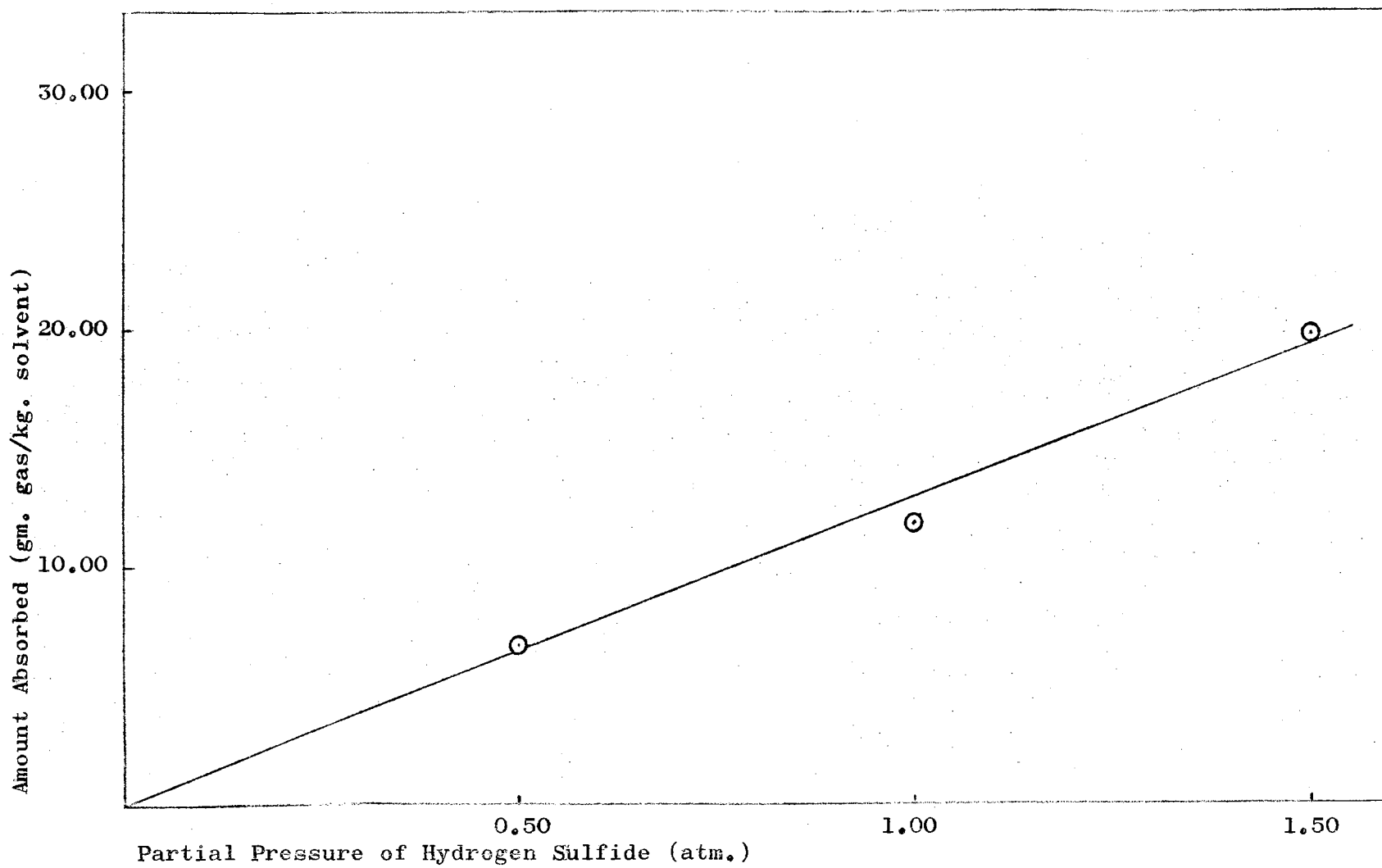


Figure 8. Gas-Liquid Equilibrium Between Hydrogen Sulfide and Triethylene Glycol

After determining the presence of a reaction, it was decided to test how much of the absorbed gas could be driven out of the solvents by heating them. Results obtained from the third group of experiments show that some hydrogen sulfide is staying with both of the solvents after they are heated. The comparisons of the absorbed gas with the gas driven out by heat are shown in Figures 9 and 10.

The results of the experiments concerning the treatment of the solvents with carbon dioxide show that both benzonitrile and triethylene glycol absorb carbon dioxide. However benzonitrile absorbs 125 per cent more carbon dioxide by weight than triethylene glycol. Although in the literature (5) it is reported that benzonitrile does not absorb carbon dioxide, the results of this study show that this information is not correct.

The amount of absorbed carbon dioxide was compared with the values determined by heating the solvents. For both solvents practically all the gas absorbed could be driven out by heat. The gas-liquid equilibrium relations of benzonitrile and triethylene glycol with carbon dioxide are shown in Figures 11 and 12.

To find out the effect of one gas on the absorption of the other, experiments were performed with mixtures of the gases. The analysis were made by driving the gases out of the solvents with the application of heat. Because the absorbed carbon dioxide could be driven out by heat, this analysis showed the true absorption of carbon dioxide. The results indicate that much less carbon dioxide was absorbed in the presence of hydrogen sulfide.

Since the true absorption of hydrogen sulfide could not be determined by heating the solvents, nothing can be said about how much hydrogen

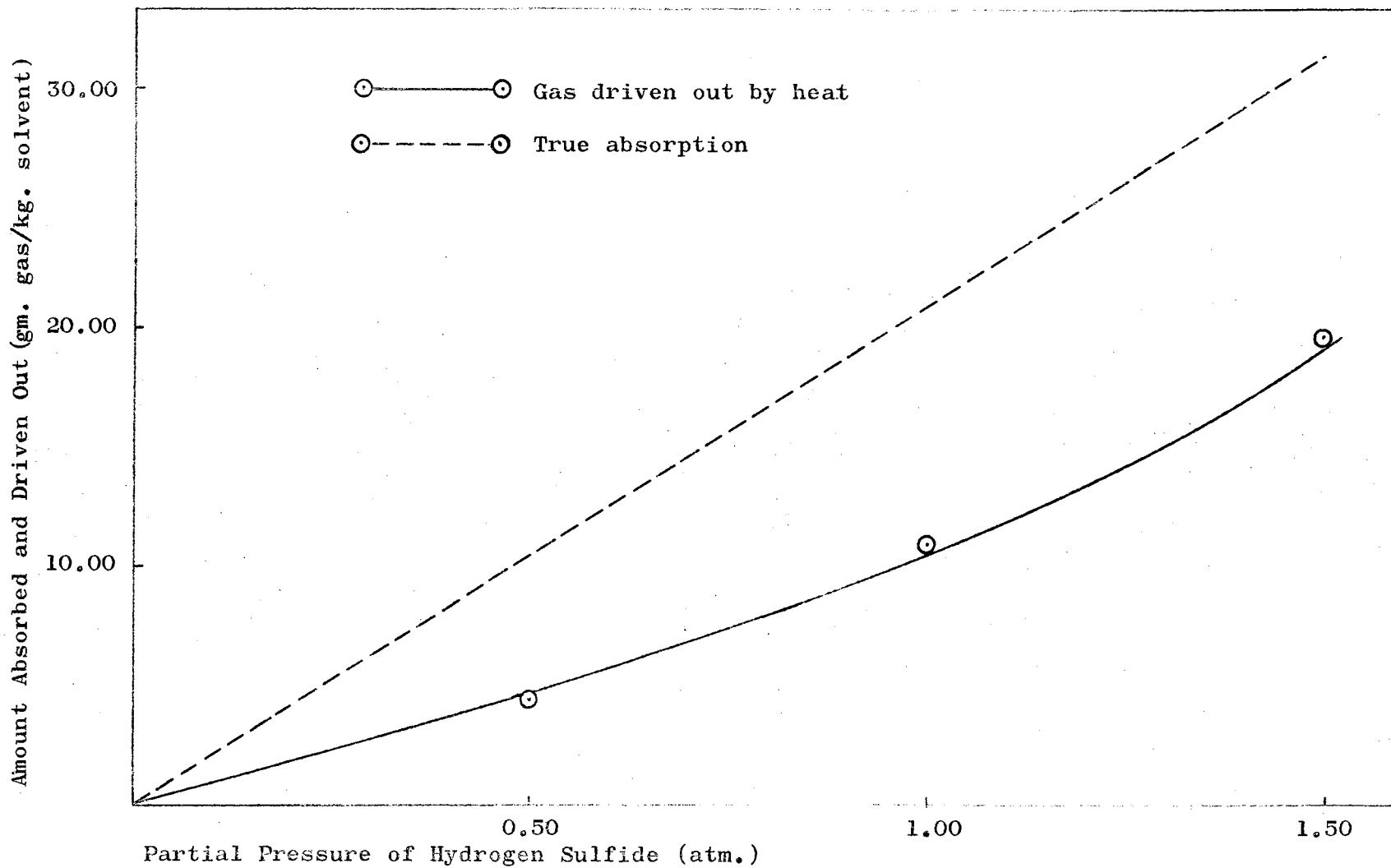


Figure 9. Comparison of Absorbed Hydrogen Sulfide With the Hydrogen Sulfide Driven Out of Benzonitrile by Heating

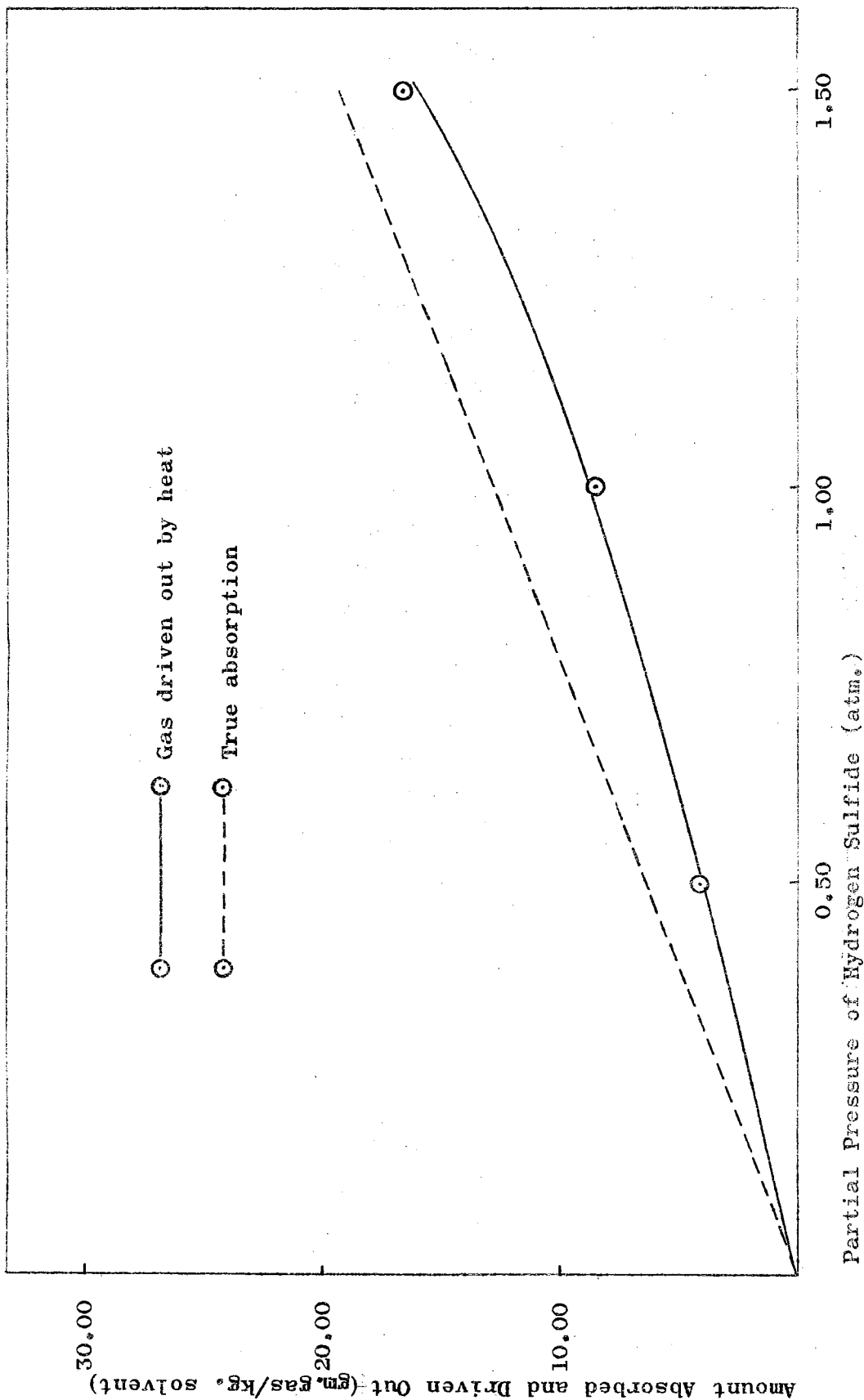


Figure 10. Comparison of Absorbed Hydrogen Sulfide With the Hydrogen Sulfide Driven Out of Triethylene Glycol by Heating

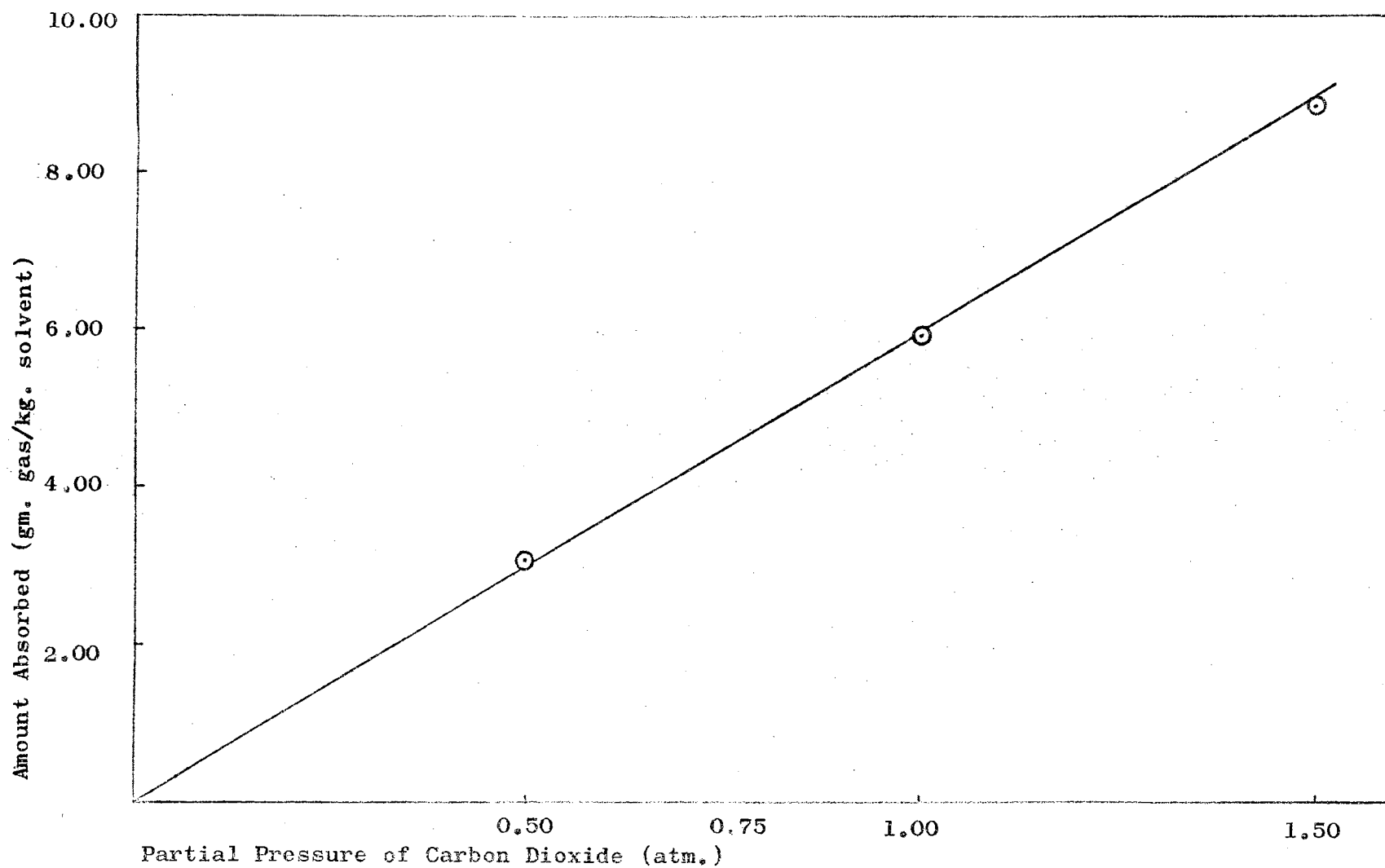


Figure 11. Gas-Liquid Equilibrium Between Carbon Dioxide and Benzonitrile

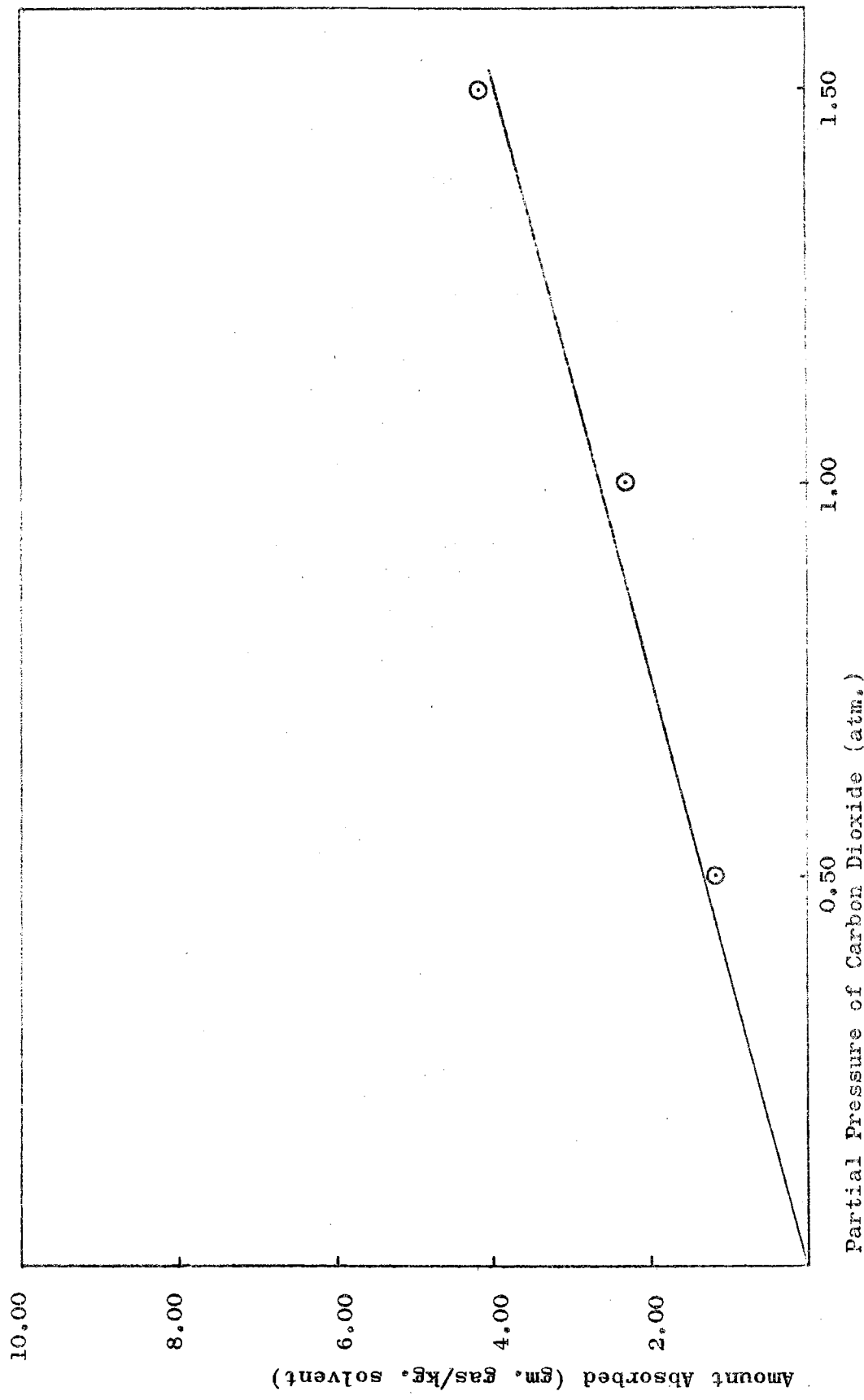


Figure 12. Gas-Liquid Equilibrium Between Carbon Dioxide and Triethylene Glycol

sulfide was absorbed in the presence of carbon dioxide. However the results indicated that more hydrogen sulfide can be driven out of the solvents when a mixture of gases is used. The relations obtained are shown in Figures 13, 14, 15, and 16.

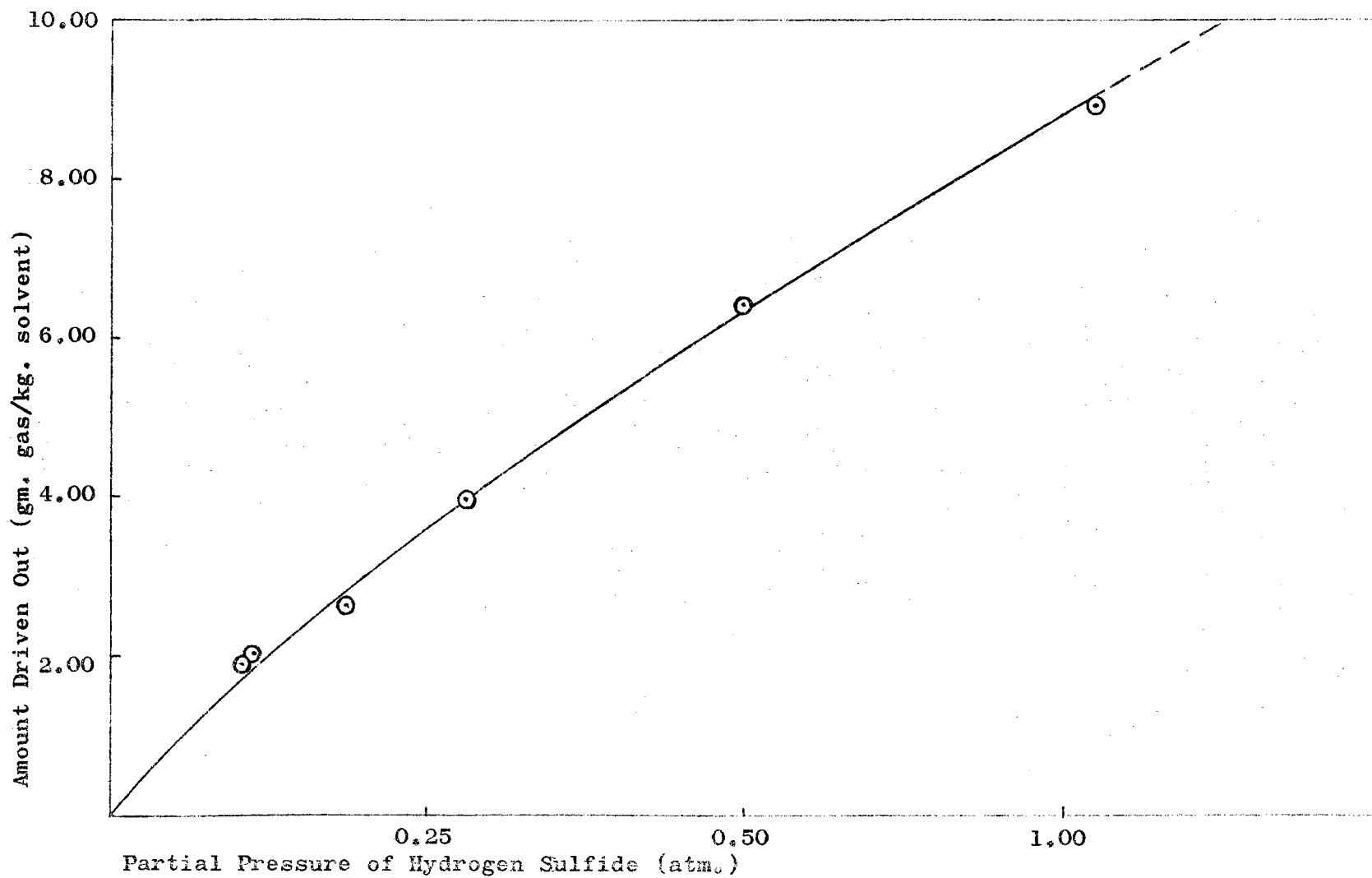


Figure 13. Hydrogen Sulfide Driven Out of Benzonitrile After the Solvent Was Brought to Equilibrium With a Mixture of Hydrogen Sulfide and Carbon Dioxide

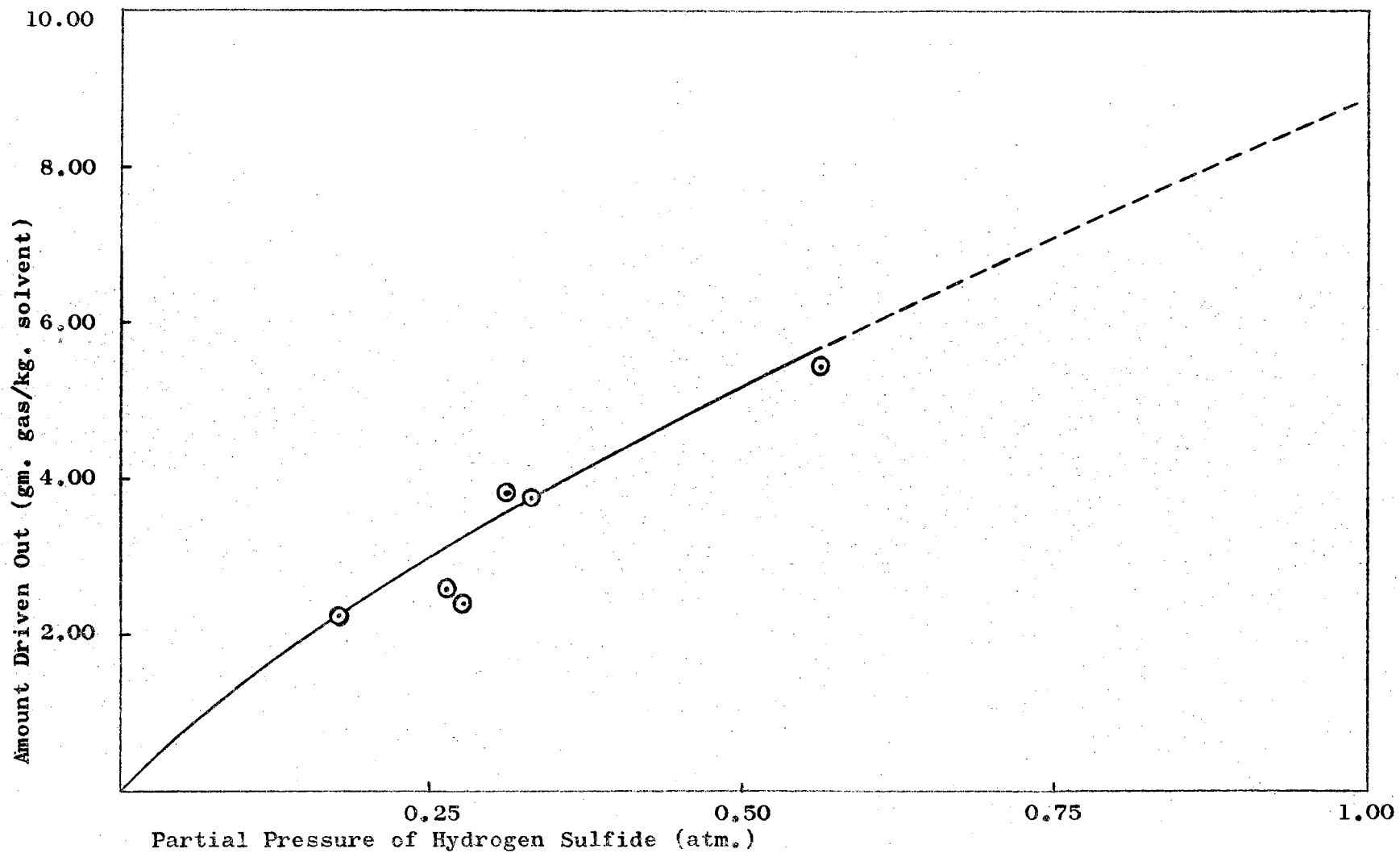


Figure 14. Hydrogen Sulfide Driven Out of Triethylene Glycol After the Solvent Was Brought to Equilibrium With a Mixture of Hydrogen Sulfide and Carbon Dioxide

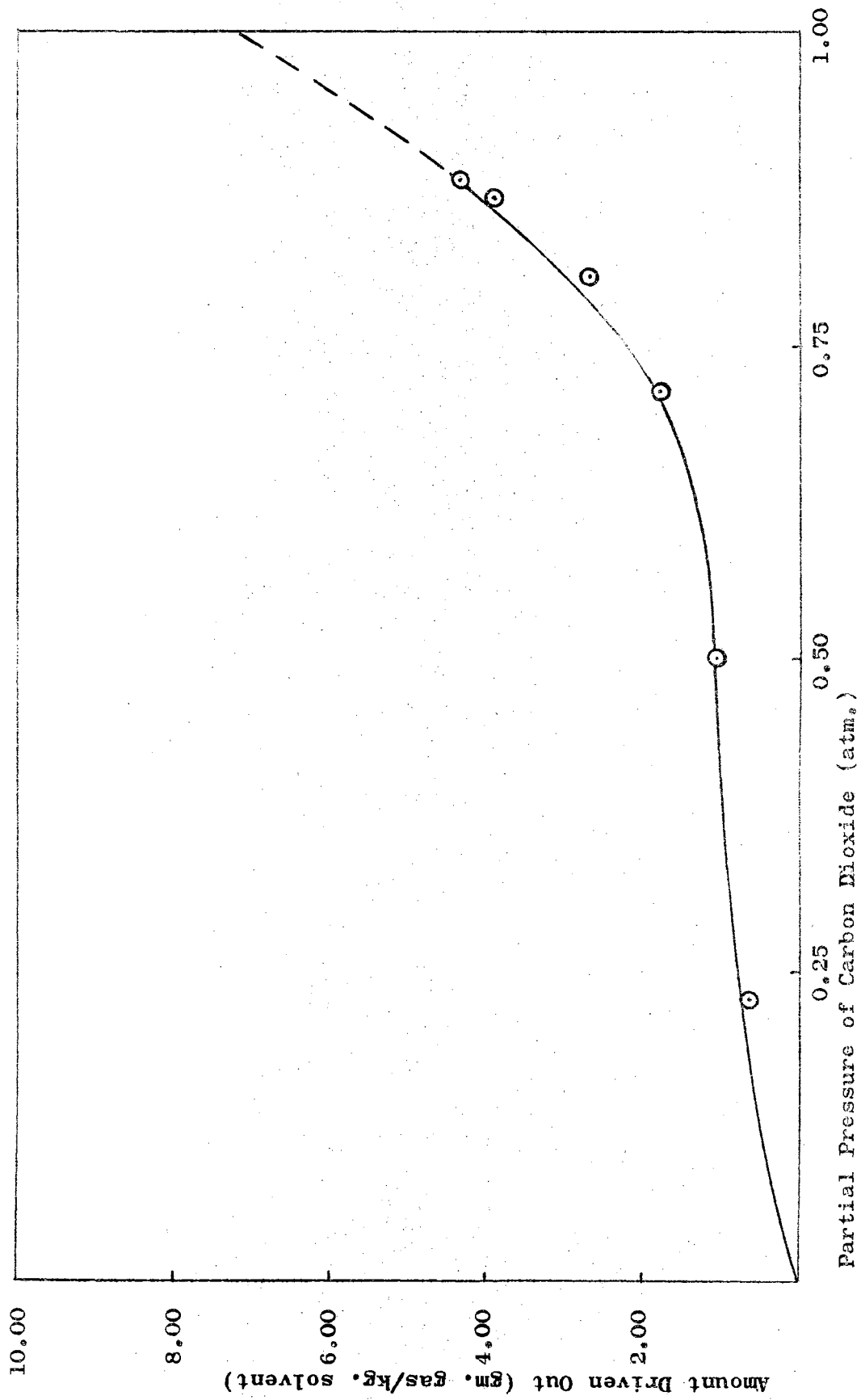


Figure 15. Carbon Dioxide Driven Out of Benzonitrile After the Solvent Was Brought to Equilibrium With a Mixture of Carbon Dioxide and Hydrogen Sulfide

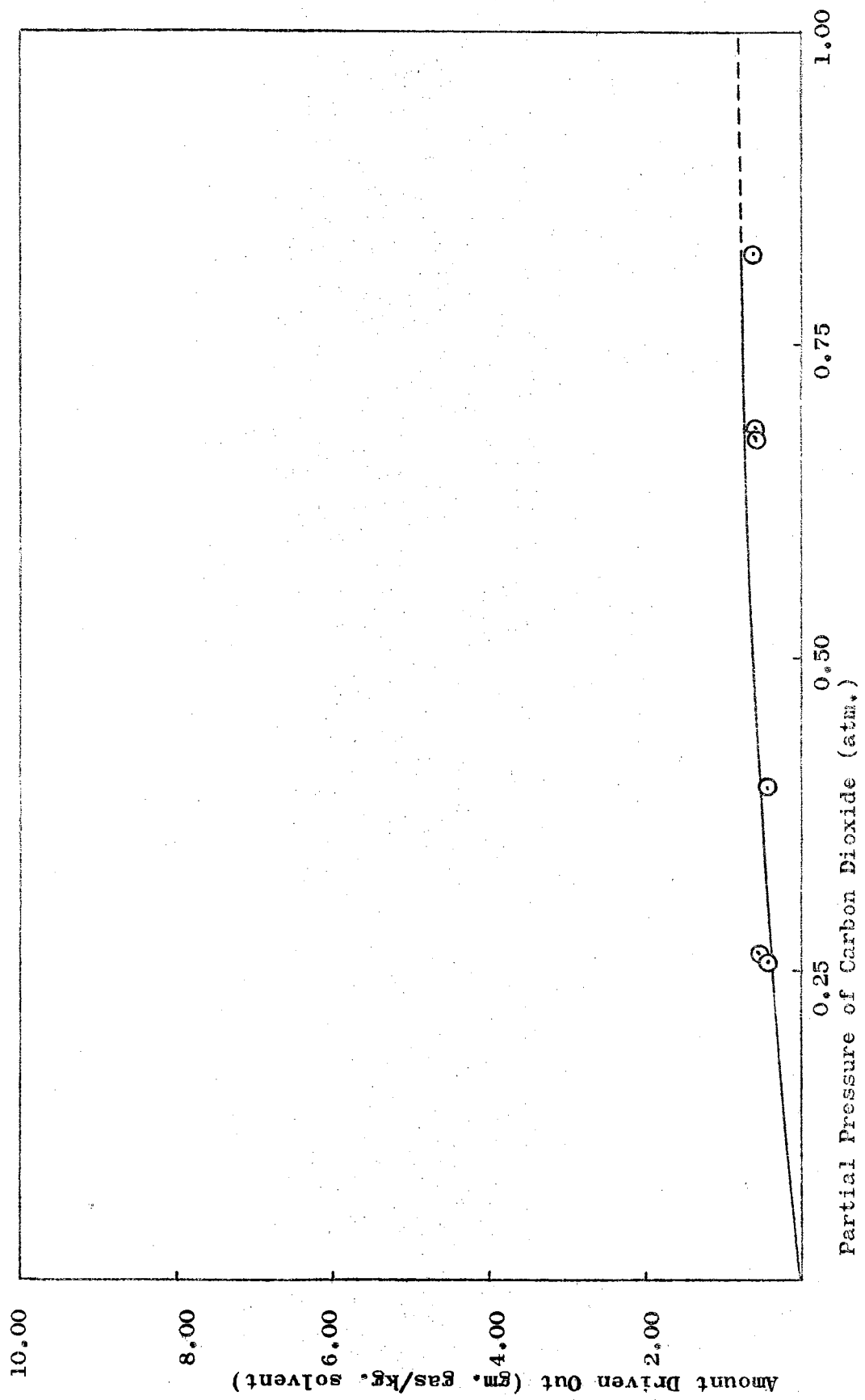


Figure 16. Carbon Dioxide Driven Out of Triethylsulfate Glycol After the Solvent Was Brought to Equilibrium With a Mixture of Hydrogen Sulfide and Carbon Dioxide

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

From the results obtained it can be concluded that benzonitrile absorbs more hydrogen sulfide than triethylene glycol. However both solvents have lower absorption capacity than any of the current absorbers. If it is compared with a 50 per cent solution of diethanol amine, we find that diethanol amine absorbs seven times more hydrogen sulfide by weight than benzonitrile at a partial pressure of one atmosphere and at 30° C. In addition to that, benzonitrile is more expensive than the other solutions.

One of the claimed characteristics of benzonitrile was that it did not absorb carbon dioxide. However the data shows that benzonitrile absorbs a considerable amount of carbon dioxide. Therefore it cannot be used as an absorbent only for the removal of hydrogen sulfide.

With the above conclusions it is believed that benzonitrile is not recommendable for use in processes to remove hydrogen sulfide without the removal of carbon dioxide.

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APPENDIX A
PRESENTATION OF DATA

TABLE I

Equilibrium data obtained for benzonitrile and hydrogen sulfide at 30° C and at three different pressures. The numbers represent gram of gas absorbed per kilogram of solvent.

PRESSURE: 0.50 atm.

SAMPLE	1 - 10.96
	2 - 10.50
AVERAGE	10.73
MAX. DEV.	2.19 %

PRESSURE: 1.00 atm.

SAMPLE	1 - 18.40
	2 - 19.15
AVERAGE	18.78
MAX. DEV.	2.02 %

PRESSURE: 1.50 atm.

SAMPLE	1 - 34.12
	2 - 32.80
AVERAGE	33.46
MAX. DEV.	1.73 %

TABLE II

Equilibrium data obtained for triethylene glycol and hydrogen sulfide at 30° C and three different pressures. The numbers represent gram of gas absorbed per kilogram of solvent.

PRESSURE: 0.50 atm.

SAMPLE 1 - 6.79

2 - 6.48

AVERAGE 6.63

MAX. DEV. 2.26 %

PRESSURE: 1.00 atm.

SAMPLE 1 - 12.46

2 - 11.80

AVERAGE 12.13

MAX. DEV. 2.72 %

PRESSURE: 1.50 atm.

SAMPLE 1 - 20.50

2 - 18.98

AVERAGE 19.74

MAX. DEV. 3.85 %

TABLE III

Data showing the amount of hydrogen sulfide absorbed by benzonitrile after repeated regenerations. The numbers represent gram of gas absorbed per kilogram of solvent.

PRESSURE: 1.00 atm.

TEMPERATURE: 30° C.

Untreated Benzonitrile: 20.32

First regeneration: 12.13

Second regeneration: 10.62

TABLE IV

Equilibrium data obtained for benzonitrile and carbon dioxide at 30° C and 1.00 atmosphere.

Gram of carbon dioxide absorbed per kilogram of benzonitrile: 5.65

Equilibrium data obtained for triethylene glycol and carbon dioxide at 30° C and 1.00 atmosphere.

Gram of carbon dioxide absorbed per kilogram of triethylene glycol: 2.88

TABLE V

Data showing the amount of hydrogen sulfide that was driven out of benzonitrile by heating after equilibrium. The equilibrium temperature was 30° C. The numbers represent gram of gas driven off per kilogram of solvent.

PRESSURE: 0.50 atm.

SAMPLE	1 - 4.30
	2 - 4.62
	3 - 4.32
AVERAGE	4.42
MAX. DEV.	4.69 %

PRESSURE: 1.00 atm.

SAMPLE	1 - 11.02
	2 - 10.86
	3 - 10.81
AVERAGE	10.90
MAX. DEV.	1.15 %

PRESSURE: 1.50 atm.

SAMPLE	1 - 19.96
	2 - 19.65
	3 - 19.29
AVERAGE	19.63
MAX. DEV.	1.74 %

TABLE VI

Data showing the amount of hydrogen sulfide that was driven out of triethylene glycol by heating after equilibrium. The equilibrium temperature was 30° C. The numbers represent gram of gas driven off per kilogram of solvent.

PRESSURE: 0.50 atm.

SAMPLE 1 - 4.08

2 - 4.25

3 - 3.78

AVERAGE 4.04

MAX. DEV. 6.32 %

PRESSURE: 1.00 atm.

SAMPLE 1 - 8.34

2 - 8.46

3 - 8.78

AVERAGE 8.53

MAX. DEV. 3.01 %

PRESSURE: 1.50 atm.

SAMPLE 1 - 16.47

2 - 16.25

3 - 16.66

AVERAGE 16.46

MAX. DEV. 1.29 %

TABLE VII

Equilibrium data obtained for benzonitrile and carbon dioxide at 30° C and at three different pressures, where carbon dioxide was determined by heating the solution. Numbers represent gram of gas absorbed per kilogram of solvent.

PRESSURE: 0.50 atm.	SAMPLE 1 - 3.10
	2 - 3.12
	3 - 3.07
	AVERAGE 3.10
	MAX. DEV. 0.77 %
PRESSURE: 1.00 atm.	SAMPLE 1 - 5.86
	2 - 5.90
	3 - 5.88
	AVERAGE 5.88
	MAX. DEV. 0.34 %
PRESSURE: 1.50 atm.	SAMPLE 1 - 8.76
	2 - 8.87
	3 - 8.93
	AVERAGE 8.85
	MAX. DEV. 1.05 %

TABLE VIII

Equilibrium data obtained for triethylene glycol and carbon dioxide at 30° C and three different pressures, where carbon dioxide was determined by heating the solution. Numbers represent gram of gas absorbed per kilogram of solvent.

PRESSURE: 0.50 atm.

SAMPLE	1 - 1.18
	2 - 1.22
	3 - 1.21
AVERAGE	1.20
MAX. DEV.	1.74 %

PRESSURE: 1.00 atm.

SAMPLE	1 - 2.33
	2 - 2.30
	3 - 2.37
AVERAGE	2.33
MAX. DEV.	1.50 %

PRESSURE: 1.50 atm.

SAMPLE	1 - 4.18
	2 - 4.28
	3 - 4.26
AVERAGE	4.24
MAX. DEV.	1.51 %

TABLE IX

Data showing the amount of absorbed gas mixture that was driven out of benzonitrile by heating after equilibrium. The equilibrium temperature was 30° C and the total pressure was 1.00 atmosphere. Numbers represent gram of gas absorbed per kilogram of solvent.

PARTIAL PRESSURE OF

HYDROGEN SULFIDE (atm.)	HYDROGEN SULFIDE	CARBON DIOXIDE
0.11	1.93	4.34
0.13	2.01	3.93
0.19	2.57	2.78
0.28	3.98	1.77
0.50	6.41	1.08
0.77	8.93	0.65

TABLE X

Data showing the amount of gas mixture that was driven out of triethylene glycol by heating after equilibrium. The equilibrium temperature was 30° C and the total pressure was 1.00 atmosphere. Numbers represent gram of gas absorbed per kilogram of solvent.

PARTIAL PRESSURE OF		
HYDROGEN SULFIDE (atm.)	HYDROGEN SULFIDE	CARBON DIOXIDE
0.18	2.24	0.61
0.26	2.60	0.46
0.27	2.40	0.53
0.32	3.80	0.60
0.33	3.74	0.60
0.61	5.45	0.45

APPENDIX B

SAMPLE CALCULATIONS

SAMPLE CALCULATIONS

Calculation of amount of hydrogen sulfide absorbed is shown below. The gas was obtained by heating the solvent. In the example given hydrogen sulfide was absorbed by triethylene glycol at 1.00 atmosphere, and 30° C.

Normalities of the standard solutions:

a - Potassium iodate-iodide. (0.1319 N)

b - Sodium thiosulphate. (0.1093 N)

Milliliters of potassium iodate-iodide solution: 100.00

Milliequivalents of potassium iodate-iodide solution: 100.00 x 0.1319
= 13.190

Milliliters of sodium thiosulphate solution: 52.13

Milliequivalents of sodium thiosulphate solution: 52.13 x 0.1093 = 5.698

Net milliequivalents: 7.492

Gram of hydrogen sulfide absorbed per kilogram of triethylene glycol:

$$(7.492 \text{ m.eq. H}_2\text{S}) \times \left(\frac{1}{1000} \frac{\text{eq. H}_2\text{S}}{\text{m.eq. H}_2\text{S}} \right) \times \left(\frac{1000}{13.60} \frac{\text{ml. TEG/lit. TEG}}{\text{ml. TEG}} \right) \times$$
$$\left(\frac{34.07}{2} \frac{\text{gm. H}_2\text{S}}{\text{eq. H}_2\text{S}} \right) \times \left(\frac{1.000}{1.125} \frac{\text{lit. TEG}}{\text{kg. TEG}} \right) = 8.34 \text{ gm. H}_2\text{S/kgm. TEG}$$

Calculation of amount of carbon dioxide absorbed is shown below. The gas was obtained by heating the solvent and precipitating it with barium hydroxide. In the example given carbon dioxide was absorbed by benzonitrile at 1.00 atmosphere and 30° C.

Normalities of standard solutions:

a - Barium hydroxide. (0.1214 N)

b - Hydrochloric acid. (0.2075 N)

Milliliters of barium hydroxide: 300.00

Milliequivalents of barium hydroxide: 300.00 x 0.1214 = 36.420

Milliliters of hydrochloric acid: 158.06

Milliequivalents of hydrochloric acid: 158.06 x 0.2075 = 32.797

Net milliequivalents: 3.623

Gram of carbon dioxide absorbed per kilogram of benzonitrile:

$$(3.623 \text{ m. eq. Ba(OH)}_2) \times \left(\frac{44,011}{2000} \frac{\text{gm. CO}_2}{\text{ml.eq. Ba(OH)}_2} \right) \times$$

$$\left(\frac{1000}{13.600} \frac{\text{ml. BN/lit. BN}}{\text{ml. BN}} \right) \times \left(\frac{1.000}{1.010} \frac{\text{lit. BN}}{\text{kgm. BN}} \right) = 5.86 \text{ gm. CO}_2/\text{kgm. BN}$$

Calculation of amount of carbon dioxide absorbed is shown below. The analysis was made by measuring the pressure drops. In the example given carbon dioxide was absorbed by benzonitrile at 1.00 atmosphere and 30° C. Similar calculations were carried for determining hydrogen sulfide.

Volume of the bomb: 433.60 ml.

Moles of gas that is present after 25.00 cc of liquid sample is put in:

$$N = \frac{P V}{R T} = \frac{(1.00) \times (408.60)}{(82) \times (303)} = 0.0164 \text{ moles}$$

Recorded pressure drop: 5.85 inches of Hg.

Gram of carbon dioxide per kilogram of benzonitrile:

$$(0.0164 \text{ moles CO}_2) \times (44.01 \text{ grams CO}_2/\text{mole CO}_2) \times$$

$$\left(\frac{1000 \text{ ml. BN/lit. BN}}{25.00 \text{ ml. BN}} \right) \times \left(\frac{1.000 \text{ lit. BN}}{1.010 \text{ kgm. BN}} \right) \times \left(\frac{5.85 \text{ inches of Hg.}}{29.92 \text{ inches of Hg.}} \right) =$$

$$5.65 \text{ gm. CO}_2/\text{kg. BN}$$

APPENDIX C

ERROR ANALYSIS

ERROR ANALYSIS

Expected error involved in determining the amount of gas absorbed is shown for the case of triethylene glycol and hydrogen sulfide, at 1.00 atmosphere and 30° C. The error introduced in standardizing the solutions is also considered.

Error involved in standardizing sodium thiosulphate:

$$\begin{aligned}
 \text{Weight of weighing bottle + KIO}_3 &= 23.9312 \pm 0.0008 \\
 \text{Weight of weighing bottle} &= 23.7530 \pm 0.0008 \\
 \text{Weight of KIO}_3 &= 0.1782 \pm 0.0016 \\
 \text{Weight of KIO}_3 &= 0.1782 \pm 0.90 \% \\
 \text{Milliliters of Na}_2\text{S}_2\text{O}_3 &= 45.69 \pm 0.12 \% \\
 \text{Normality of Na}_2\text{S}_2\text{O}_3 &= \frac{0.1782 \pm 0.90 \%}{35.668} \times \frac{1000}{45.69 \pm 0.12 \%} \\
 \text{Normality of Na}_2\text{S}_2\text{O}_3 &= 0.1093 \pm 1.02 \%
 \end{aligned}$$

Error involved in standardizing potassium iodate:

$$\begin{aligned}
 \text{Milliliters of KIO}_3 &= 25.00 \pm 0.03 \% \\
 \text{Milliliters of Na}_2\text{S}_2\text{O}_3 &= 30.17 \pm 0.12 \% \\
 \text{Normality of KIO}_3 &= \frac{(30.17 \pm 0.12 \%) \times (0.1093 \pm 1.02 \%)}{25.00 \pm 0.03 \%} \\
 \text{Normality of KIO}_3 &= 0.1319 \pm 1.17 \%
 \end{aligned}$$

Error involved in determining hydrogen sulfide:

$$\text{Milliliters of KIO}_3 = 4(25.00 \pm 0.03 \%)$$

$$\text{Milliliters of KIO}_3 = 100.00 \pm 0.12 \%$$

$$\text{Milliequivalents of KIO}_3 = (100.00 \pm 0.12 \%) \times (0.1319 \pm 1.17 \%)$$

$$\text{Milliequivalents of KIO}_3 = 13.190 \pm 1.29 \%$$

$$\text{Milliliters of Na}_2\text{S}_2\text{O}_3 = 52.13 \pm 0.18 \%$$

$$\text{Milliequivalents of Na}_2\text{S}_2\text{O}_3 = (52.13 \pm 0.18 \%) \times (0.1093 \pm 1.02 \%)$$

$$\text{Milliequivalents of Na}_2\text{S}_2\text{O}_3 = 5.698 \pm 1.20 \%$$

$$\text{Milliequivalents of KIO}_3 = 13.190 \pm 0.170$$

$$\text{Milliequivalents of Na}_2\text{S}_2\text{O}_3 = 5.698 \pm 0.068$$

$$\text{Net milliequivalents} = 7.492 \pm 0.238$$

$$\text{Net milliequivalents} = 7.492 \pm 3.18$$

$$\text{Gram of H}_2\text{S per kilogram TEG} = 8.341 \pm 3.54 \%$$

VITA

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