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ABSTRACT

The Stefan diffusion-tube was utilized to determine the rate of mass transfer in three ternary systems in which two gases were simultaneously diffusing into a third stagnant gas. Constant evaporating binary liquid mixtures were used. The experimentally determined values are compared with the quantities predicted by an exact solution of the Stefan-Maxwell equations.

Results of these and related comparisons indicate that: (a) unless the interacting influence of the two diffusing gases upon each other is considered, any predictive method will be seriously in error: (b) the validity of the Stefan-Maxwell equations has been established under experimental conditions involving significant interaction among simultaneously diffusing components.

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INTRODUCTION

In the years since their formulation the differential equations which describe isothermal, isobaric diffusion in ideal multicomponent gas mixtures, the Stefan-Maxwell equations,^{1,2} have been solved exactly for only a few restrictive cases in ternary systems.³⁻⁸ A number of approximate solutions have been proposed for use in practical engineering design calculations.⁹⁻¹³ However, up to the present time, little experimental work has been done in verifying the Stefan-Maxwell equations themselves, or in comparing the predictions of the various approximate relationships. Experimental verification was obtained by Fairbanks and Wilke⁴ for the limited case of diffusion of one gas through two or three stagnant gases. This paper describes work completed by the authors upon ternary gas systems with only one gas stagnant and compares the experimentally measured values with those predicted by the Stefan-Maxwell equations.

THEORY

The original Stefan-Maxwell equations for unidirectional diffusion, which have since been derived more rigorously from kinetic theory by Hirschfelder, Curtiss, et. al.,^{15,16} when applied to a mixture of n components take the form

$$-\frac{dp_i}{dx} = \sum_{j \neq i}^n \alpha_{ij} c_i c_j (u_i - u_j), \quad (1)$$

where

$$\alpha_{ij} = \alpha_{ji} = \frac{R^2 T^2}{D_{ij} P} \quad (2)$$

and

$$i, j = 1, 2, \dots, n.$$

Equation (1) can be easily transformed into

$$-\frac{P}{RT} \frac{dy_i}{dx} = \sum_{j \neq i}^n \frac{N_i y_j - N_j y_i}{D_{ij}} \quad (3)$$

A similar expression can be written for each component in a multi-component mixture although, as pointed out by Toor,¹² for n components, there are only $n-1$ independent equations since we have $\sum_{i=1}^n y_i = 1$, and the total pressure is assumed constant. However, by adding another restriction--for example, letting $N_i = 0$ for one of the components, as in the case of a stagnant gas--the system becomes determinate.

Gilliland's exact solution of the diffusion equations³ for a ternary system with one stagnant component takes the form of two complex simultaneous equations and requires a trial-and-error solution, making it rather cumbersome for general engineering purposes. A number of approximation techniques have been proposed in recent years. Wilke¹⁰ has utilized a constant effective diffusion coefficient and the film pressure factor concept of Hougan and

Watson⁹ to obtain two solutions of differing complexity. Toor¹² simplified the Gilliland result by assuming $D_{AC} = D_{BC}$, while Shain¹³ has developed an iterative approximation which yields results comparable in accuracy to those of the Gilliland expressions.

EXPERIMENTAL PROCEDURE

Theory of the Stefan-tube Apparatus

The theory of the Stefan capillary-tube method for measuring diffusion coefficients in binary systems is reviewed in detail by Lee and Wilke.¹⁷ The apparent diffusion coefficient, D_A , obtained by this technique must be corrected for end and surface curvature effects by the relation

$$\frac{1}{D_A} = - \frac{\Delta x}{D} \frac{1}{x_A} + \frac{1}{D} \quad (4)$$

Hence the true diffusion coefficient, D , is determined from a plot of $1/D_A$ versus $1/x_A$ extrapolated to $1/x_A = 0$. In the present work, the technique of Lee and Wilke was used to measure the binary coefficients of the different organic substances in air.

Rigorously, the binary diffusion coefficient, D , depends to a very slight extent upon concentration and hence will vary across the path length. However, this variation with concentration is extremely small, and particularly in this work with small concentration changes across the film, the average value of D is essentially the same as the value at each end of the path.

It should be noted here that in this approach plug flow is assumed in the diffusion tube (i.e., no radial concentration gradients). A recent study made by Heinzelmann, Wasan and Wilke¹⁹ confirms this assumption.

APPARATUS

The apparatus used for this work is shown schematically in Fig. 1.

Air enters the system at room temperature at a positive pressure of some 7 to 7-1/2 in. of water from three parallel tanks of the compressed gas. A constant flow rate of 0.0668 cfm (1890 cc/min) is maintained by three pressure regulators. (The method of determination of this flow rate is discussed in the following section, Procedure). It then passes through a specially built 6-in. column of magnesium perchlorate to eliminate any trace quantities of water from the gas. After passing through a flowrator, the air is heated preliminarily to within approximately one degree of the bath temperature by a heating element consisting of two heating tapes wrapped about numerous coils of the 1/4-in. copper tubing through which the gas flows and surrounded by several layers of asbestos insulating strips. The air is then directed by the tubing into the constant-temperature bath, where it passes through a 40-ft coil to bring its temperature to exactly 35° C before entering the suspended diffusion unit. The air and dilute organic vapors are then lead by tubing from the bath to a blower and exhausted outside the building.

The heart of the apparatus is the diffusion unit. Figure 2 is a drawing of this unit and Fig. 3 is a photograph of the unit and adjacent submerged peices of copper tubing. The air entering the unit first passes through copper straightening vanes spaced 2 mm apart to eliminate all turbulence accompanying the transition into the chamber, before it flows over the capillary diffusion tube itself. All parts of the unit other than the entrance and exit tubing and the vanes are constructed of brass to achieve the combination of strength and good thermal conductivity.

The diffusion tubes and corresponding tube holders were built so as to be interchangeable. The dimensions of the tubes are:

	<u>Large tube</u>	<u>Small tube</u>
Inside diameter	2.40 cm	1.44 cm
Wall thickness	0.031 in.	0.031 in.
Depth	15.25 cm	15.31 cm

The tube holders were built to leave a minimum of clearance (0.001 in.) between the holder and tube, but still permit the latter to be removed when desired. The thickness of the diffusion tube holders is 0.123 in.

Later in the experimental work, it was decided to condense and analyze at least one vapor sample to confirm the assumption of equal vapor and liquid composition. Therefore two acetone-dry-ice glass traps were located in series just after the second thermometer well. Connections between the traps and the copper tubing of the original system were designed so that this latter unit could be removed from the apparatus without halting the air flow whenever an analysis was desired.

In all cases, gravimetric calibration curves of the depth, x_A , versus the combined weight of the sample and diffusion tube were utilized. In this manner a given sample could be weighed at the beginning and at the end of each run and a mean x_A chosen for calculation purposes.

Compositions of the binary liquid were determined from indexes of refraction. Initially, calibration curves showing the index of refraction versus mole fraction were determined for the narrow range of interest in each case. During the experiment, readings were taken on the binary samples immediately before and after each run, and a mean composition was determined.

The calibration curves were also used for the condensed-vapor analysis. These curves are given in Appendix C of the reference thesis.¹⁴

Benzene, carbon tetrachloride, methyl alcohol, and chloroform were A.C.S. standard purity chemicals. The chloroform was vacuum distilled over magnesium perchlorate to minimize the water content. The ethyl alcohol was vacuum distilled over sodium to decrease the water content to 0.425 weight percent. The compressed air had a maximum water content of 220 ppm.

PROCEDURE

Before full-scale experimental work was initiated, the proper operating conditions were determined. The bath temperature of 35.0°C was chosen as a compromise between a desire to obtain high vapor pressures for the pure components and the mixtures--thus increasing the rate at which material is lost and hence shortening the required running time--and the difficulties involved in constructing the proper heater for the air stream and in locating reliable partial-pressure data for several common binary mixtures. Such data has been published by Scatchard and his co-workers for mixtures of methyl alcohol-benzene,²¹ methyl alcohol-carbon tetrachloride,²² and ethyl alcohol-chloroform²³ at 35°C , and these values were used for this work. From this data partial pressure-versus-concentration curves were drawn up¹⁴ and the values shown in Table I were read for use in the calculations. It is felt that the uncertainty introduced in plotting the data and reading the curves for use in the calculations is of the order of 0.5 mm Hg.

It should be noted that the three systems utilized were chosen from a group of mixtures for which data were available at 35°C . Each of the three presents a slightly different situation for study. The methyl alcohol-benzene-

air system is one in which both the pure diffusion coefficients into air and the partial pressures above the constant evaporating mixture are widely different. In the methyl alcohol-carbon tetrachloride-air system, the partial-pressure difference is greatly reduced, but the two diffusion coefficients are even further apart. The ethyl alcohol-chloroform-air system is representative of the case in which the diffusion coefficients are much more nearly equal but the partial-pressure difference is extremely large.

The proper air flow rate was determined by experimentation. Too low a value will produce stagnation in the gas chamber of the diffusion unit, hence reducing the driving force and consequently the rate of mass transfer through the film. Conversely, too large a value will increase the turbulence in the film until turbulent diffusion becomes the chief mode of mass transport. The flow rate thus selected produced a velocity of 73.4 cm/sec and a Reynolds number of 95.5 in the copper vanes of the diffusion unit. This rate also fixed the operating pressure of the system at 7 to 7-1/2 in of water above atmospheric pressure, since the blower vacuum was found to be of negligible influence. It should be noted that air was treated as a single component in this work, since exact calculations showed that there was essentially no difference between the diffusion coefficients of the various species into oxygen and nitrogen.

Once the proper operating conditions were selected, the true binary coefficients for the diffusion of each of the five organic compounds into air were measured by the technique of Lee and Wilke for the small diffusion tube, and several of the values were checked by experimentation with the large tube. All later work utilized the small tube exclusively, however, because generally the results obtained from its application were more consistent with calculated values (to be demonstrated later) and included a smaller total Δx effect.

As mentioned earlier, an important factor in the work with ternary systems (binary liquid evaporating and diffusing into stagnant air) is the constant-evaporating mixture, because the composition of the vapor leaving the diffusion unit must be known in order to ascertain how much of each liquid component has been lost. In addition, for steady-state operation a constant driving force is needed for each component.

A constant-evaporating mixture is one in which the combination of diffusion coefficients and partial vapor pressures of the two components is such that the liquid composition is always constant for the case of evaporation by molecular diffusion, and is therefore equal to the composition of the vapor leaving the mixture.

In this work, approximate component ratios for the constant-evaporating mixtures were calculated for the three systems (i.e., methyl alcohol-benzene, methyl alcohol-carbon tetrachloride, and ethyl alcohol-chloroform) by using Wilke's second method. The exact values were then approached by experimentally observing the direction and size of composition changes indicated by the refractometer analysis for various initial compositions within the range of expectation. Once the value for a given mixture was correctly ascertained, a series of 10 to 12 runs was conducted and the next mixture was considered. In this manner, results were obtained for all three mixtures.

Finally, since the constant-evaporating mixtures could not always be exactly located and maintained, and also as a matter of scientific interest, it was decided to attempt to condense and analyze the vapors leaving the diffusion unit. For this operation, the equipment described earlier was used.

RESULTS

As mentioned earlier, the first quantities determined experimentally in this work were the true diffusion coefficients for the organic compounds in air. These values were obtained by plotting the reciprocal of D_A against $1/x_A$ for each run, according to the technique of Lee and Wilke, and extrapolating to infinite x_A as shown in Fig. 4 for the methyl alcohol-air system. A least-squares treatment was employed to accurately locate the dotted lines and the true diffusion coefficient in all cases. The results of this work are shown in Table II, where the figures in the last column are the values obtained by calculations based upon the equation of Hirschfelder, Bird, and Spotz, including the constant coefficient correlation presented by Wilke and Lee¹⁸ (the modified Hirschfelder, Bird, and Spotz equation). The values for the "collision diameters", r_0 or σ , for each of the substances involved were taken from the tables in Hirschfelder, Curtiss, and Bird.²⁴

In addition to these values, the interaction coefficients, D_{AB} , between the two organic vapors were needed for calculations with the ternary systems. By combining the measured binary coefficients listed in Table II with the force constant, ϵ/k , for air (97.0°K), the corresponding ϵ/k values for each of the organic compounds were calculated from the modified Hirschfelder, Bird, and Spotz equation. Once these values were known, it was possible to use this equation to determine an experimental D_{AB} for each of the three vapor interactions. The values thus obtained are:

$$D_{\text{CH}_3\text{OH}-\text{C}_6\text{H}_6} = 0.0809 \text{ cm}^2/\text{sec}$$

$$D_{\text{CH}_3\text{OH}-\text{CCl}_4} = 0.0676$$

$$D_{\text{C}_2\text{H}_5\text{OH}-\text{CHCl}_3} = 0.0566$$

For the ternary systems, the approximate constant-evaporating mixtures located at 35.0° and 756 mm Hg in this work were 0.651 mole fraction methanol in benzene, 0.618 mole fraction methanol in carbon tetrachloride, and 0.122 mole fraction ethanol in chloroform. Once these values had been determined, standard mixtures were made up and liquid composition changes were kept at a minimum, hence ensuring an accurate knowledge of the vapor composition. Small changes in liquid composition frequently occurred, since the figures listed above are only approximate (probably ± 0.002) and since the standard mixtures were not of exactly these compositions. However, such variations seldom exceeded 0.001 mole-fraction units. Therefore there was always a negligible concentration gradient in the liquid and always an essentially constant and known partial pressure of each component at the gas-liquid interface.

A further assurance of the accuracy of this method of determining the vapor composition was offered by the condensation analysis described earlier. The system methanol-carbon tetrachloride-air was chosen for this check. The results of the run considered showed that the mean liquid composition was 0.613 mole fraction methanol, and the composition of the condensed vapor was 0.596 methanol.

Since the main objective of this investigation was to test the validity of the Stefan-Maxwell equations it was decided to compare the experimental molar losses of components A and B for each ternary run with a path length sufficiently long enough to minimize errors in measurement with the corresponding losses predicted by the theory. Therefore, the latter equations were solved for all cases in which the path length exceeded 6 cm, and the resulting predicted values of molar quantities evaporated are compared with the experimental values in Table III.

The true values of x used in the above calculations and those to follow were determined by subtracting the mole-fraction average of the total Δx correction factors for components A and B in the ternary mixtures from x_A in each case.¹⁴ These factors were obtained by a method similar to that of Lee and Wilke for binary mixtures.

DISCUSSION

A complete discussion of the possible sources of error in this experimental work appears in the reference thesis.¹⁴ It should be pointed out that the runs with the longest path lengths yield the most reliable results. This fact was kept in mind in the selection of sample runs for the comparisons described earlier.

The binary coefficients measured in the first portion of this experiment and recorded in Table II are self-consistent although they are larger (from 2 to 10%) than those predicted by the modified Hirschfelder, Bird, and Spotz equation.¹⁸

Experimental results for the ternary (binary liquid) systems are summarized in Table III and compared with the Gilliland solution of the Stefan-Maxwell equations. It can be seen from Table III that the agreement is satisfactory and within the limits of experimental error. It is of interest to note that predicted values of moles lost by evaporation in each case is too large for component A and too small for component B. This phenomenon may be due in part to either experimental error or some inherent feature of this particular physical arrangement, and its significance is subject to question.

For a further comparison with the values predicted by the various approximate equations described earlier the reader is referred to the reference

thesis.¹⁴ It is sufficient to note here that a comparison with values calculated on the assumption of diffusion of A and B independently into a stagnant binary mixture showed clearly that the evaporation rates of both components are greatly enhanced by their parallel diffusion.

The results of this work, therefore, appear to support the following conclusions:

- a. In this type of experiment, interaction does occur between the simultaneously diffusing molecules, and some method of accounting for its effect is required.
- b. The validity of the Stefan-Maxwell equations has been established under experimental conditions more stringent than any that have been employed previously. These results, together with earlier data for stagnant gas mixtures, provide some proof of the applicability of the Stefan-Maxwell equations to diffusion in complex multi-component gas systems.

NOMENCLATURE

c_i	gas-phase concentration of component i
D_A	apparent diffusion coefficient in binary system
D_{ij}, D	true binary diffusion coefficient
N_i	diffusion flux for component i
P	total pressure
P_i	partial pressure of component i
R	gas constant
T	absolute temperature
u_i	gaseous diffusional velocity of component i molecules
x	film thickness
x_A	apparent film thickness
Δx	total correction for film thickness (= $x_A - x$)
y_i	mole fraction of component i in gas phase

FOOTNOTES AND REFERENCES

* Work done under the auspices of the U. S. Atomic Energy Commission.

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Table I. Partial Pressure Data for the Binary Mixtures.

Mixture		Mole Fraction	P _A	P _B
A	B	of A	(mm Hg)	(mm Hg)
CH ₃ OH	- C ₆ H ₆	0.651	170.2	122.3
CH ₃ OH	- CCl ₄	0.618	170.0	155.0
C ₂ H ₅ OH	- CHCl ₃	0.122	32.3	272.5

Table II. Experimental and calculated results at 35.0°C.

System	Tube	Δx (cm)	D(measured) (cm ² /sec)	D ^a (cm ² /sec)	D(calculated) ^b (cm ² /sec)
C ₆ H ₆ - air	L	0.22	0.114	0.113	0.101
	S	0.35	0.107	0.107	
CCl ₄ - air	L	0.20	0.0931	0.0926	0.0877
	S	0.15	0.0898	0.0894	
CH ₃ OH - air	L	1.36	0.193	0.192	0.173
	S	0.35	0.192	0.191	
C ₂ H ₅ OH - air	S	0.19	0.145	0.144	0.136
CHCl ₃ - air	S	0.05	0.101	0.101	0.0985

^a Measured D corrected to 760 mm Hg.^b At 760 mm Hg.

Table III. Comparison of calculated and experimental results.

Mixture 1. Methyl alcohol (A) - Benzene (B) - Air (C)						
Values calculated from Gilliland Eqs.			Experimental values			Deviation in total moles evaporated ^a (%)
Run No.	Moles of A evaporated ($\times 10^2$)	Moles of B evaporated ($\times 10^2$)	Total Moles evaporated ($\times 10^2$)	Moles of A evaporated ($\times 10^2$)	Moles of B evaporated ($\times 10^2$)	
34	1.328	0.654	1.982	1.229	0.665	+ 4.6
35	1.733	0.851	2.584	1.635	0.882	+ 2.7
37	2.16	1.062	3.222	2.030	1.100	+ 2.9
38	1.492	0.734	2.226	1.413	0.762	+ 2.3
40	1.313	0.645	1.958	1.241	0.672	+ 2.4
41	1.280	0.629	1.909	1.192	0.650	+ 3.6
Avg.						+ 3.1
Mixture 2. Methyl alcohol (A) - carbon tetrachloride (B) - Air (C)						
43	1.663	0.984	2.647	1.635	1.027	- 0.6
45	1.362	0.806	2.168	1.289	0.807	+ 3.4
47	1.297	0.765	2.062	1.215	0.772	+ 3.8
49	2.29	1.302	3.592	2.090	1.317	+ 5.4
Avg.						+ 3.0

Table III. (continued)

Mixture 3. Ethyl alcohol (A) - chloroform (B) - air (C)						
Run No.	Moles of A evaporated ($\times 10^2$)	Moles of B evaporated ($\times 10^2$)	Total Moles evaporated ($\times 10^2$)	Moles of A evaporated ($\times 10^2$)	Moles of B evaporated ($\times 10^2$)	Total Moles evaporated ($\times 10^2$)
52	0.118	0.805	0.923	0.114	0.826	0.940
53	0.214	1.454	1.668	0.202	1.470	1.672
55	0.145	0.984	1.129	0.138	0.989	1.127
57	0.221	1.504	1.725	0.212	1.520	1.732
59	0.266	1.811	2.077	0.257	1.841	2.098
60	0.255	1.758	2.013	0.248	1.785	2.033
61	0.248	1.709	1.957	0.240	1.732	1.972
					Avg.	
						- 0.7

Deviation in
total moles
evaporated^a
(%)

- 1.8

- 0.2

+ 0.2

- 0.4

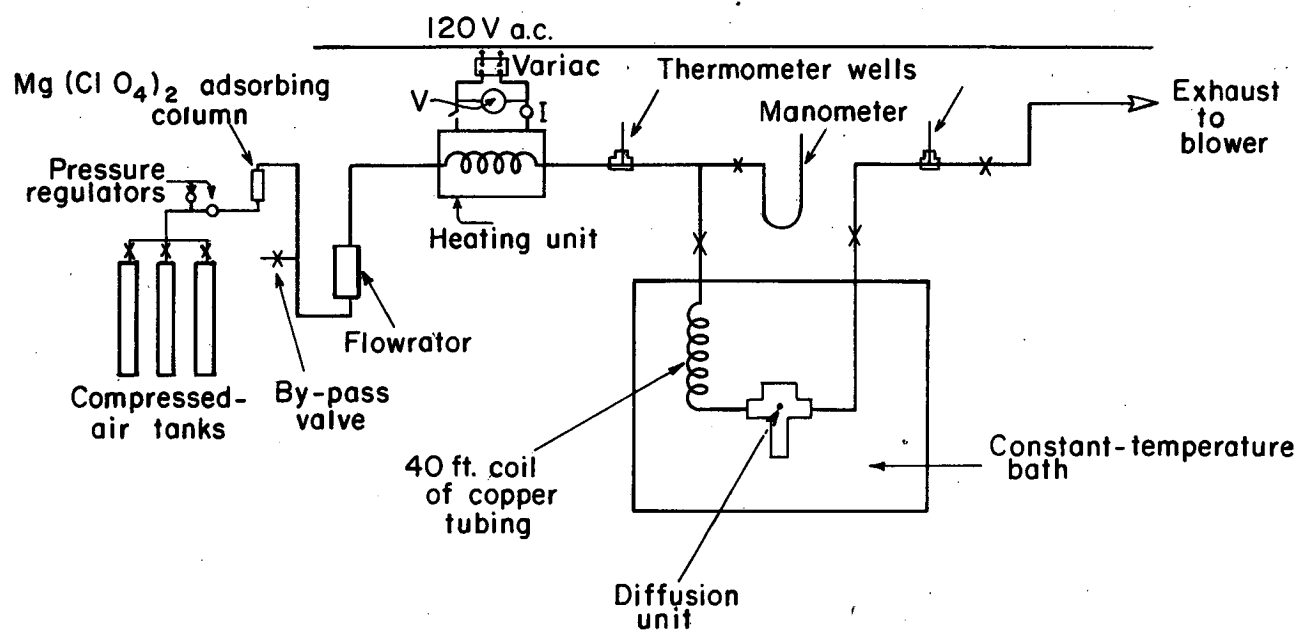
- 1.0

- 1.0

- 0.8

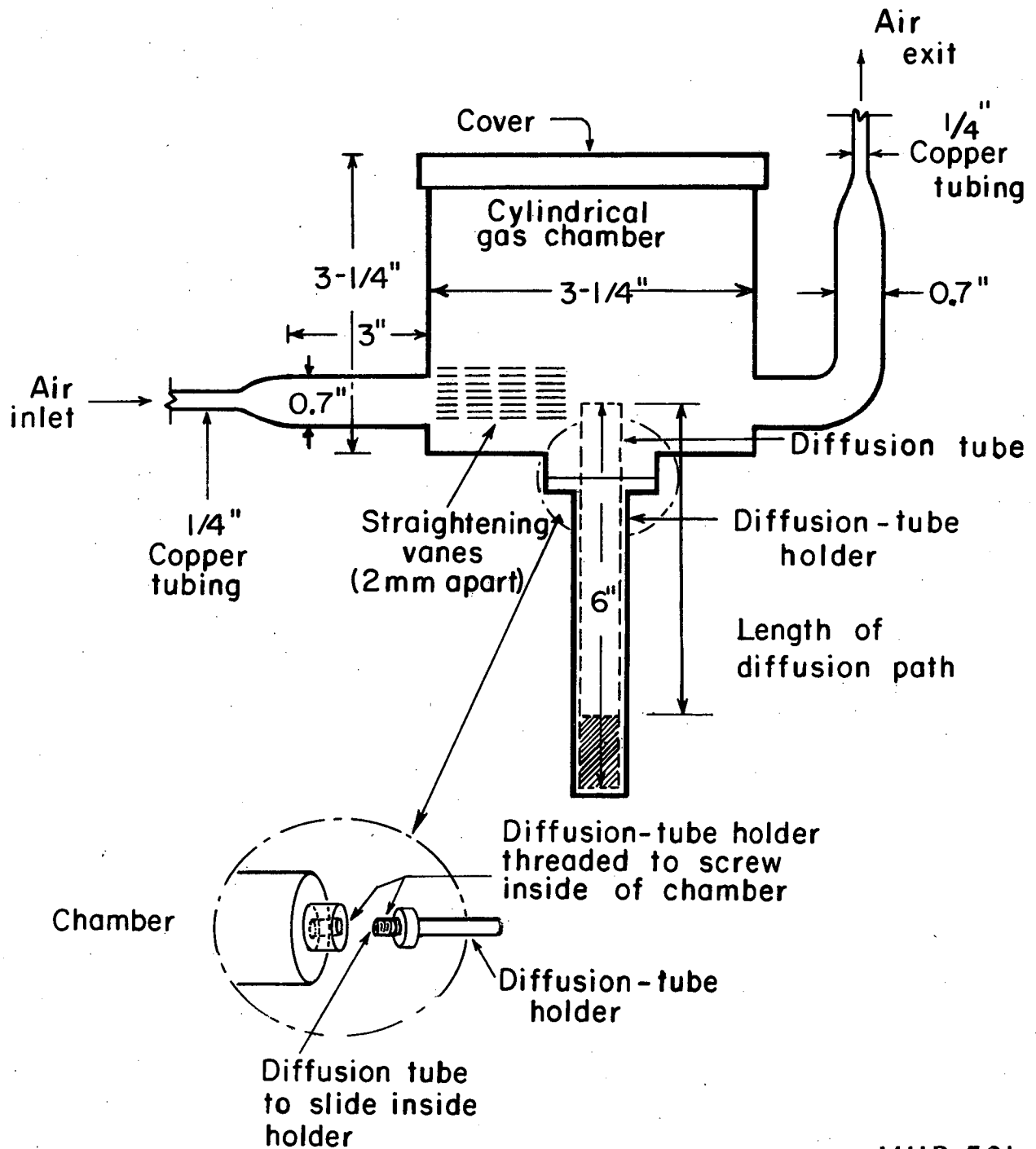
- 0.7

^a Deviation of values predicted by Gilliland Eqs. from actual experimental results.



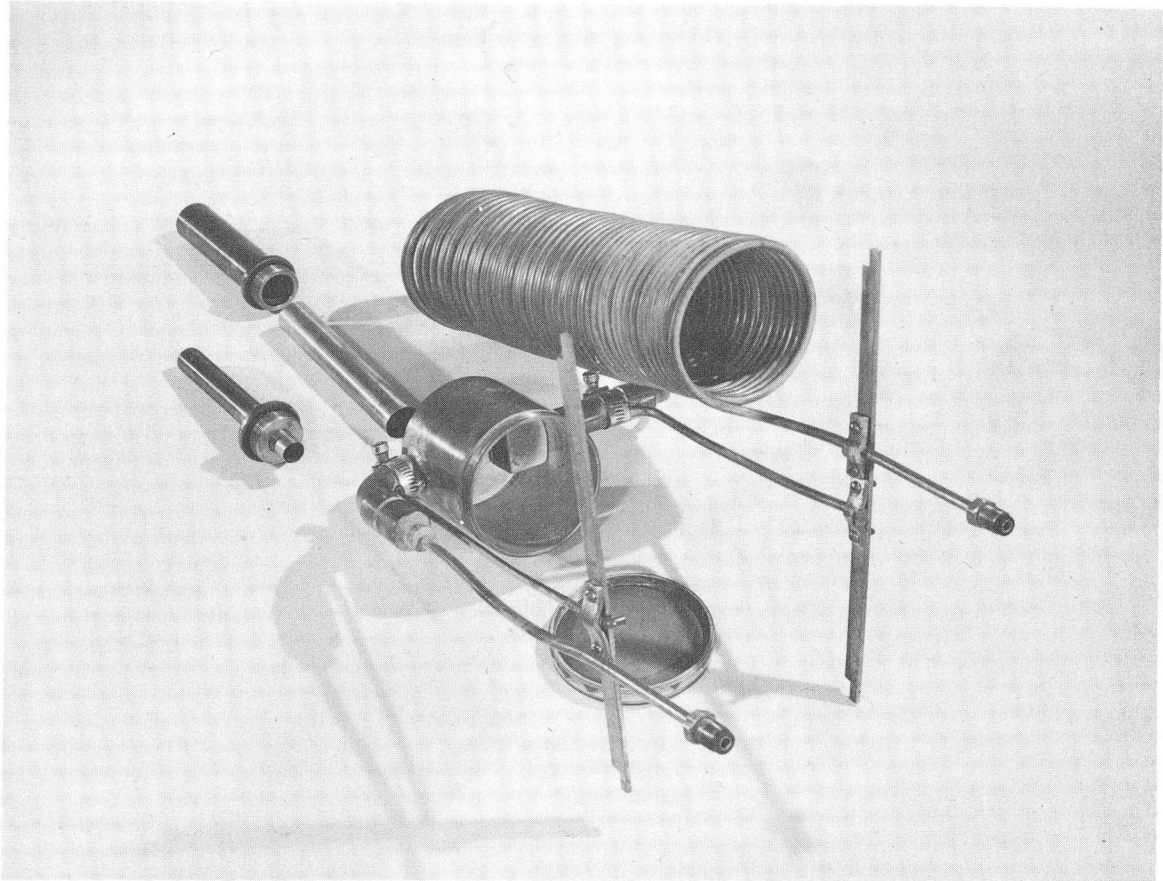
MUB-702

Fig. 1. Schematic diagram of the apparatus.



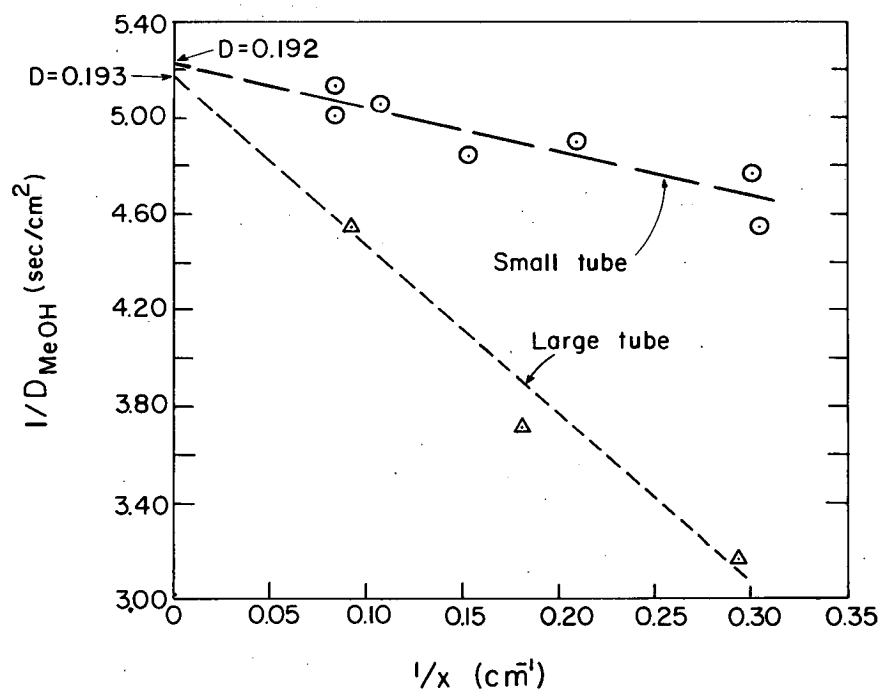
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Fig. 2. Diffusion unit.



ZN-3085

Fig. 3. Components of the diffusion unit.



MU-25629

Fig. 4. Experimental diffusion coefficients for methyl alcohol in a methyl alcohol-air system.

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