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# Adsorption as a means of separation: adsorbent screening

Elizabeth C. White  
*Lehigh University*

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ADSORPTION AS A MEANS OF SEPARATION:

ADSORBENT SCREENING

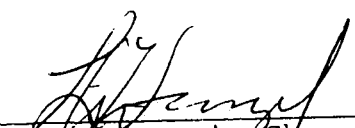
Adsorption as a Means of Separation:  
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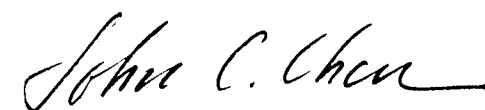
by  
Elizabeth C. White

A Thesis  
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in Candidacy for the Degree of  
Master of Science  
in  
Chemical Engineering

This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science

April 3, 1985  
(date)

  
Professor in Charge

  
Chairman of Department

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## Summary

This report describes the development of a continuous flow system for the screening of adsorbents using the volumetric method. By this technique the amount of gas adsorbed is calculated from a knowledge of the void volume of the adsorbent and a measurement of the total volume of the gas in the void volume and adsorbed on the bed. The measurement of the total volume may be made in any convenient manner. For this study, a thermal conductivity detector was calibrated such that a gas passing through the sample side will give a total mass determination. The design of the equipment for this technique is discussed.

Initial testing was done using triethylamine and diethylamine on zeolite Y. The operating procedure is covered in detail and the data reported. Improvements to the system, based upon the problems encountered, are discussed.

## Introduction

Adsorption has the potential of being a useful separating technique for gas mixtures. It is based on a developed process technology, and there are many different adsorbents commercially available. However, any adsorption separation is chemically specific, that is its effectiveness depends on the specific chemical system to be separated and the choice of adsorbent. It is precisely this latter fact that makes the capability to screen adsorbents with a particular process stream on a small scale essential. This project has developed such a capability.

The experimental equipment consists of an adsorption column, a thermal conductivity detector for measuring the concentration of the flowing gases, a syringe pump for constant flow of liquid feed and the other devices necessary for monitoring and controlling the process.

## Technical Background

Adsorption on zeolites shows great potential for separation of gas mixtures. This is because zeolites are high capacity, highly selective adsorbents on which physical adsorption is completely reversible. (1) Much of the current research effort in the field is directed toward the development of a theory of adsorption which can be used to predict mixed gas adsorption equilibrium compositions.

We will briefly discuss here some important aspects of adsorption: the techniques used to measure equilibrium isotherms, a short explanation of the specificity of zeolites, and an overview of current mixed gas adsorption theories.

### Techniques

Most of the techniques for adsorption measurement fall into one of two categories: volumetric or gravimetric. The volumetric method is the oldest and most widely used. The volume of gas adsorbed is determined from pressure - volume - temperature measurements, frequently by measuring the change in pressure due to adsorption of a gas onto an adsorbent contained within a known volume chamber. The principle can be used for either static or dynamic systems. The main source of error is in the determination of the volume in and around the adsorbent bed. This so called "dead volume" is calculated by admitting a known amount of a gas (usually helium) which will not be adsorbed and



noting the pressure change. The corresponding volume is found from an equation of state.

A variation of the volumetric method measures the volume adsorbed not by the change in pressure in the adsorption chamber itself but by desorbing the gas and collecting it in a chamber of known volume (or by measuring the volume in any desired manner). This adds an additional source of error in that the desorption must be complete.

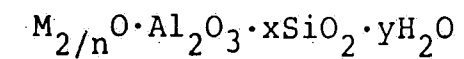
The gravimetric method determines the amount of gas adsorbed by attributing it to the increase in weight of the adsorbent after the gas is allowed to come in contact with the adsorbent. A buoyancy correction is required at high pressures. This correction factor is the main difficulty with the gravimetric technique.

The principles of the gravimetric method can also be used for dynamic measurements. Then the adsorbent is weighed at frequent intervals as the adsorbate is passed over it.

#### Adsorbents

There are many kinds of adsorbents which exhibit ultraporosity and are used for separation of gas mixtures. Included in this category are activated carbons, activated clays, inorganic gels such as silica gel and activated alumina, and the crystalline aluminosilicate zeolites. Of these, only the zeolites have an ordered crystal structure and thus a uniform pore size.

Zeolites are crystalline, hydrated aluminosilicates of group I and group II elements (higher ions can be introduced by ion exchange). The structure is that of an infinitely extending three-dimensional network of  $AlO_4$  and  $SiO_4$  tetrahedra linked by sharing of the oxygen atoms. The empirical formula is:



where n is the valence of cation M. The framework contains interconnecting channels and voids occupied by the cation and water molecules. Upon dehydration, those zeolites which retain their structure contain a large void volume and uniform molecular-sized channels making them high capacity, selective adsorbents.

#### Theories of Mixed Gas Adsorption

The literature contains many discussions of the theoretical treatment of the adsorption of single components. However, simultaneous adsorption of two or more components is not as extensively analyzed. Thus, when an adsorption system is designed, experimental work is required to obtain the mixed gas adsorption data. Direct measurement of multi-component data is both complicated and tedious; measurement of single component data is relatively convenient. It is therefore desirable to be able to predict adsorption from a mixture using parameters which characterize single component adsorption.

Sircar and Myers (2) have noted that there are presently three different approaches to mixed gas adsorption:

1. Extensions of pure gas equations (both localized and mobile adsorption models)
2. Thermodynamic methods
3. Potential theory

The first approach is attractive because the multi-component isotherms have analytic expressions. However, when the pure component isotherms do not conform to the simple equations of the model, the values of the derived constants are indefinite. Also, the method is not thermodynamically consistent unless all of the gases have the same value of monolayer coverage (moles adsorbed per gram of adsorbent). (2)

Perhaps the single most important equation in the field of adsorption is that of Langmuir. (3,4) The form of the equation can be derived from kinetics, as Langmuir did; from the thermodynamics considerations following Volmer (5); or from a statistical derivation as shown by Fowler. (6) The two assumptions in the theory are that the forces of interaction between the adsorbed molecules are negligible and that the adsorption is unimolecular. The equation derived by Langmuir is:

$$V = \frac{V_m bp}{1+bp}$$

where:  $V$  = volume adsorbed at pressure  $p$   
 $b$  = adsorption coefficient, a function of temperature  
 $V_m$  = volume of unimolecular surface coverage

When the Langmuir isotherm is extended to mixed gas unimolecular adsorption, the equations for a binary mixture are:

$$V_1 = \frac{V_m^{(1)} b_1 p_1}{1+b_1 p_1 + b_2 p_2} \quad \text{and} \quad V_2 = \frac{V_m^{(2)} b_2 p_2}{1+b_1 p_1 + b_2 p_2}$$

The Langmuir theory implies that the only effect that the presence of a second component can have is to decrease the area available for adsorption of the first component.

The Langmuir isotherm has been derived and modified for localized multimolecular layer adsorption for both pure and mixed gases. (7,8,9,10) The result - the "BET" equation - is not applicable to zeolites since zeolites exhibit a Type I (3) isotherm (unimolecular adsorption).

The models of mobile adsorption are based on the theory that the adsorbed phase is characterized by a two-dimensional equation of state. The parameters for mixed monolayers are then expressed in terms of the pure adsorbate parameters by using mixing rules. The analysis usually proceeds from statistical mechanics. (11,12,13)

The second category of equations, the thermodynamic methods, are independent of any particular adsorption model. Given any theory for adsorption of a pure gas, the extension to mixtures follows from an application of Raoult's law for the adsorbed mixture.

Hill (14) and Myers and Prausnitz (15) used the analogy between vapor-liquid and gas-adsorbate systems to develop a thermodynamic treatment of gas adsorption. Using basic thermodynamics, Myers and Prausnitz developed an expression for the chemical potential of the components in the adsorbed phase as a function of composition and activity coefficients. At equilibrium, the chemical potentials of the gas and adsorbed phase are equated:

$$pY_i = \gamma_i P_i^\circ(\pi) X_i$$

where:  $Y_i$  = mole fraction of  $i$  in gas phase

$p$  = pressure

$\gamma_i$  = activity coefficient of  $i$  in the adsorbed phase

$X_i$  = mole fraction of  $i$  in the adsorbed phase

$P_i^\circ(\pi)$  = pure adsorbate vapor pressure for  $i$  at the temperature and spreading pressure  $(\pi)$  of the mixture

The spreading pressure of the mixture,  $\pi$ , is calculated from experimental adsorption isotherm data of the pure components.

For a pure component:

$$\frac{\Pi_i^{\circ} A}{RT} = \int_0^P \frac{N_t}{P} dp$$

For a binary mixture, at constant P,

$$-\frac{A}{RT} d\pi + \sum N_i d \ln_i = 0$$

$$\text{or } \frac{A}{RT} \Delta\Pi = \int_1^{Y_1} N_t \frac{x_1 - Y_1}{Y_1(1 - Y_1)} dy_1$$

$$\text{and } \Pi = \Pi_1^{\circ} + \Delta\Pi$$

This assumes the gas phase is an ideal solution.

For high pressures:

$$\phi_i P Y_i = \gamma_i f_i^{\circ}(\Pi) X_i$$

where  $\phi_i$  = the fugacity coefficient of the gas phase

$f_i^{\circ}(\Pi)$  = fugacity of the adsorbed phase

Both  $P_i^{\circ}(\Pi)$  and  $f_i^{\circ}(\Pi)$  are calculated from the pure gas adsorption equilibrium data.

It is frequently assumed that  $\gamma_i=1$  (ideal solution for the adsorbed phase). (15,17) Costa et. al. (16) have used the Wilson and UNIQUAC equations for vapor-liquid equilibrium to calculate the activity coefficients. In their calculations,  $\gamma_i$  is close to one only when the species are similar. Otherwise, the activity coefficients show a clear deviation from ideality.

The third category is the Polanyi potential theory. This is an old empirical theory for adsorption which has been modified several times (18-25) for use with both gas mixtures and single components on activated carbons and silica gel. Dubinin (26,27,28) developed the theory of volume filling for vapor adsorption on microporous substances. This form of the Polanyi relationship gives good results in correlating adsorption on zeolites.

Polanyi potential theory states that every adsorbate-adsorbent pair has a "characteristic curve" of the volume of adsorption space,  $W$ , plotted against the work of adsorption  $A$ .

For vapors:

$$A = RT \ln P_s/p$$

$$\text{and } W = av^*$$

where  $P_s$  = saturation vapor pressure at temperature  $T$

$a$  = amount adsorbate, moles/gram

$v^*$  = molar volume of the adsorbate

Dubinin gives an equation of the characteristic curve:

$$W = W_0 \left[ - \exp k \frac{A^2}{\beta^2} \right]$$

where  $k$  = the distribution function of the pores

according to sizes, a constant

$\beta$  = affinity coefficient of the characteristic curve, determined experimentally

Then the equation of the isotherm of adsorption of vapors on a microporous substance is:

$$a = \frac{W_0}{V^*} \exp B \frac{T^2}{\beta^2} (\ln P_s/P)^2$$

The constants  $W_0$  and  $B$  are found from experimental data.

For a binary mixture:

$$a_1 + a_2 = \frac{W_0}{N_1 \bar{V}_1 + N_2 \bar{V}_2} \exp \left[ \frac{-B T^2}{(N_1 \bar{\beta}_1 + N_2 \bar{\beta}_2)^2} \ln^2 \frac{P_{s12}}{P_{12}} \right]$$

where:

$N_1, N_2$  = molar fractions of components in adsorption phase

$\bar{V}, \bar{\beta}$  = partial molar values

$P_{12} = P_1 + P_2$  (sum of the partial pressures of components)

$P_{s12}$  = pressure of saturated vapor over the bulk solution (at the composition of the vapor at equilibrium with the adsorbate)

These equations are valid only for the region where the characteristic curve is invariant with temperature. The equations may also be used to calculate the basic thermodynamic properties of adsorption equilibrium,  $\Delta s$  and  $Q$ , within their region of validity.



## General Design

It was decided to use a volumetric method for gas adsorption because of its versatility and the ease with which one can achieve equilibrium. A constant composition gas was to be passed over the adsorption bed until equilibrium was reached. The adsorbed gases were then to be driven off and analyzed. For single component gases the analysis would consist of the determination of the total amount of gas adsorbed. This could be found quite easily with the use of a calibrated thermal conductivity detector. When mixed gases are used, it will then be necessary to collect the desorbed gas in a chamber of known volume and measure the change in pressure of the chamber to determine the total amount of gas which had been adsorbed. Also, it will be necessary to take samples of the desorbed gas and determine the composition using a gas chromatograph, already calibrated for this purpose.

For the volumetric method to give reliable results, two criteria must be met:

1. The adsorption-desorption cycle must be reversible.
2. The composition of the feed gas must be known and constant.

The former is a chemical consideration, while the latter must be solved through the equipment design and operation. The reversibility of the adsorption-desorption cycle depends on the type of adsorption occurring. In physical

adsorption, the forces between the adsorbate molecules and the adsorbent surface are similar to van der Waals forces between molecules. In chemisorption, the forces between adsorbent and adsorbate are much like the forces which lead to the formation of chemical compounds. Physical adsorption is usually reversible, chemisorption is not. Gas adsorption on zeolites is a completely reversible type of physical adsorption. Thus the volumetric method is acceptable if the feed gas composition can be kept constant and is accurately known. This was to be achieved by using a syringe pump to pump the liquid amine and by assuming constant helium flow as determined using a calibrated variable area flowmeter.

The volumetric method also requires that the void volume in the adsorbent bed and surrounding tubing be known. This was calculated by noting the pressure change resulting from the addition of a known mass of helium, since helium is not detectably adsorbed at room temperature.

Another design consideration was the need to determine that equilibrium existed between the flowing gas and the adsorbent bed. This could be monitored by comparing the gas stream before and after the adsorbent bed using the thermal conductivity detector. Since the thermal conductivity of a gas mixture varies with small changes in composition, this procedure would show when the composition of the feed and effluent were the same - the achievement of equilibrium.

The gas temperature was monitored using copper-constantan thermocouples, which were calibrated against a platinum resistance thermometer. The system uses both heating tape which is insulated with fiberglass, and a heated fluidized sand bath for heat sources.

## Apparatus

A diagram of the apparatus and one of the panel board are shown in the appendix in Figure A-1.

The amine is pumped as a liquid by the syringe pump into a heated section of tubing 20 feet long (the amine pre-heater). This vaporizes the amine. It is then mixed with the helium, which also has been heated. The two gases pass into a mixing chamber designed to ensure a homogeneous gas mixture throughout the rest of the system. The mixture then flows to the reference side of the thermal conductivity cell, through the adsorption bed and back to the sample side of the thermal conductivity cell. The gas exiting the sample side is passed through a  $H_2SO_4$  absorber and vented into the hood.

The above flow scheme is used for the adsorption saturation step. Valves numbered 1-4, 6, 7, 9-11 are open; 5, 8, 12 and 13 are closed.

The liquid amine flow is constant to 0.005 ml/min, and the pressure is constant to within the readability of the pressure gauges. The helium flow is constant to 2-4 percent of the total flow as measured by a calibrated variable area flowmeter. The mixing chamber is a 75 ml cylinder, packed with stainless steel helices.

For desorption, the syringe pump is not in use and is shut out of the system with valve 1. The adsorption bed itself is closed off from the main flow stream with valves

7 and 9 closed, valve 8 open. The lines are flushed with helium until all the amine has been removed. The adsorption bed is now opened to the flow stream and the bypass closed, allowing the adsorbed gas to desorb and flow through the sample side of the thermal conductivity detector. For mixed gases, the desorbed gas is collected in a chamber and its' composition determined by gas chromatography; for pure components the gas is vented to the hood. Prior to a desorption run with pure components, the thermal conductivity detector is calibrated. Thus, when the desorbed gas and the gas from the void volume pass through the thermal conductivity detector, the calibration can be used to determine the amount of amine in the gas. Since the composition of the gas in the void volume and the void volume are known, the amount of amine which has been desorbed from the zeolite can be calculated.

All the equipment was chosen for amines service. The tubing is type 316 stainless steel, 1/8 inch O.D. The valves and pressure gauges are also stainless steel. The flowmeter is glass. The thermal conductivity detector, Gow-Mac number 24-150, has stainless steel tubing with rhenium-tungsten filaments. The wetted parts of the syringe pump are Teflon, stainless steel and sapphire. The adsorbent bed is contained in a piece of 1/4 inch O.D., type 316 stainless steel tubing.

## Calibration

Several pieces of equipment required calibration:

A variable area flowmeter was used to determine the helium flowrate. This had standard calibration tables provided by the manufacturer (appendix Tables A-1 to A-10). Flowrates for pressures between the calibration tables were determined by linear interpolation.

The copper-constantan thermocouples were made in the laboratory. The millivolt readings were obtained using a potentiometer with a galvanometer indicator. The thermocouple-potentiometer system was calibrated against a Platinum resistance thermometer which was read on a Mueller bridge. The thermometer had been certified as a satisfactory standard of the International Practical Temperature Scale by the National Bureau of Standards.

The thermocouple calibration data were fitted to a second order polynomial of the form:

$$(\text{degree Celsius}) = a_0 + a_1 * (\text{mvolt}) + a_2 * (\text{mvolt})^2.$$

The analytical expression for the calibration has an accuracy of  $\pm 0.5$  celsius degree. Table A-11 in the appendix gives the values of  $a_0$ ,  $a_1$  and  $a_2$  for all five thermocouples.

The thermal conductivity detector was calibrated in order to be used to determine the total amount of a pure amine passing through the sample side. Weighed samples of liquid amine were injected into a heated, flowing helium

stream before the sample side of the thermal conductivity cell. When the vaporized amine passed through the cell, the area of the resulting peak was assigned a numerical value by the integrator. The calibration consisted of this numerical area vs. grams amine relationship. The thermal conductivity of the mixture exhibits a linear relationship with concentration over the range used. The grams amine-area number relationship is therefore also linear, within experimental error. The calibration "curve" was fit to a straight line equation of the form:

$$[\text{Area}] = b_0 + b_1 * (\text{gram amine}).$$

Table A-12 in the appendix contains the values for  $b_0$  and  $b_1$  for both triethylamine and diethylamine. As mentioned above, there is experimental error in the calibration, most conspicuously seen in the fact that  $b_0$  is not zero. A graph of the triethylamine calibration is also in the appendix; Figure A-2. This shows that there is scatter in the data. The most probable cause of the scatter is fluctuations in the helium flow, which varied by 4ml/min or 5 percent of the total flow. Since a thermal conductivity detector is sensitive to flow changes, this could have caused the scatter. Note also that the scatter is at higher area values and grams of amine. Since at larger amounts of amine the fluctuations of the area number are larger numerically (2 percent of the total value), this also would contribute to the scatter of the data points.

By standard error analysis (29,30), it was determined that the calibration will give a mass value of  $\pm 0.004$  grams. When the calibration was checked before desorption runs, the result showed the calibration to be reproducible.



## Calculations

The adsorbent bed was packed with zeolite in the laboratory. The zeolite was crushed and dried to a constant weight of  $\pm 0.002$  gram. The bed was packed and glass wool plugs held the zeolite in place. The remaining zeolite was again dried to a constant weight of  $\pm 0.002$  gram. The amount of zeolite in the adsorption bed is the difference in the two weights, accurate to  $\pm 0.003$  gram (29).

In order to calculate the amount of amine adsorbed, it is necessary to know the volume of the tubing around the adsorbent bed - the void volume, and the composition of the feed gas mixture passing through the bed. The void volume was determined using helium, since it is not appreciably adsorbed at room temperature. First the volume of the tubing between valves 11 and 9 (valve 10 open, 8 closed) was calculated by injecting 25.00 ml of air into the evacuated system and noting the pressure change on gauge #5. The mean value was:  $83.2 \pm 0.3 \text{ cm}^3$ . To determine the void volume, the entire system, including the bed, was evacuated, then valves 7 and 9 closed to shut off the bed from the system. Helium was let into the system and the resulting pressure noted on gauge #5. Valves 11 and 8 were then closed. When valve 9 was opened, helium flowed into the adsorbent bed chamber and the resulting pressure change noted on gauge #5. The void volume was calculated from this pressure change using the ideal gas relationship. The calculation

of the void volume for the bed used in this study gave a value of  $9.4 \pm 0.4 \text{ cm}^3$ .

The composition of the feed gas mixture was calculated from the known flowrates of the liquid amine and helium, assuming a homogeneous mixture. The helium flowrate was measured on a variable area flowmeter. The flowmeter, together with the standard calibration charts, gave an accuracy of 3 percent for readings between 10 and 100 percent of full scale. For a typical flowrate of 70 ml/min, this amounted to 2 ml/min. The amine flowrate was set on the syringe pump to an accuracy of 0.005 ml/min, or 6 percent of a typical flowrate of 0.08 ml/min. With these typical values, by standard error analysis, (29), the error in the composition was 6.7 percent (or mole fraction of a typical run  $0.165 \pm 0.11$ ), roughly the variation seen in the baseline fluctuations from the thermal conductivity detector.

The partial pressure of the amine was given by  $P_{\text{amine}} = X_{\text{amine}} P_{\text{total}}$  for an ideal gas mixture. For the low pressures here, this ideal gas assumption was acceptable.

The amount of a pure component (amine) adsorbed was found upon desorption of the adsorbent bed. Prior to the desorption, the thermal conductivity cell calibration was checked. In all cases, it agreed with the original calibration. When the helium passed through the void volume and the saturated adsorbent, it flushed out the amine

present. Since the amine then passed through the detector, the total grams of amine present in the void volume and adsorbed on the adsorbent was known. The amount of amine in the void volume was known from the composition of the feed gas. The rest of the amine was the amount adsorbed by the zeolite. For a typical run,  $0.864 \pm 0.004$  gram triethylamine was adsorbed. The largest source of error in this number is seen to be due to the calibration (29). This order of magnitude represents, then, the lower limit of the working range of the system.

### Operating Procedure

The zeolite bed was activated (dehydrated) by passing dry, hot helium through it for 4 hours. The helium was heated to 200°C and set at a flowrate of 200 ml/min. Prior to an adsorption run, the adsorbent bed was evacuated, then filled with hot, dry helium at the pressure and temperature to be used during the run. It was then closed off from the rest of the system.

The general procedure for taking data on this system begins with the adsorption step. The system was heated to the run temperature and the desired helium flowrate and pressure achieved. The vaporized amine was added to the helium flow. Once a homogeneous mixture was obtained, as determined by a stable baseline on the thermal conductivity detector, the adsorption bed was opened to this mixture. The detector was used to indicate the saturation of the bed by the return of the baseline. The adsorbent bed was then shut off from the system and the amine line closed. When the system was flushed of the amine, again indicated by the return of the baseline on the detector, the adsorption step was completed.

The desorption step began with a check of the calibration of the detector. The calibration conditions were set on the system and amine injected into the sample side of the thermal conductivity detector, as during the calibration. Once it was determined that the calibration was

still valid, desorption could begin. At the same conditions of temperature, pressure and flowrate as used during calibration, the adsorption bed was opened to the rest of the system. The amine in the void volume and that which had been adsorbed on the zeolite now passed through the detector. The total amount of amine passing through the detector was given by the calibration. Since the void volume and the composition of the gas in the void volume was known, the amount of amine adsorbed on the zeolite could be determined.

During initial testing it was found that the detector takes 4-5 hours to heat up to the desired temperature. The constant temperature bath and the heating tapes require only about 45 minutes to heat the gas to any desired temperature. Flowrates and pressures can be achieved rapidly and easily using the micrometering valves, numbers 2 and 10, at the inlet and outlet of the system.

During the adsorption saturation step the amine is added to the helium as a vapor. The amine is pumped as a liquid, therefore the amine preheater must completely vaporize it. In order to ensure that the preheater was hot enough and was actually vaporizing the amine, with valve 1 closed the amine was pumped to the atmosphere at 2-3 ml/min until the emerging amine was a vapor and thermocouple 4 indicated that the amine was hot enough to be vaporized. The valve to the atmosphere was then closed

and the flowrate on the pump was turned down to the one to be used for the run. When the run pressure was reached in the amine preheater line, valve 1 was opened and the vaporized amine mixed with the heated helium at the desired pressure and temperature.

When the amine was first added to the helium, the adsorption bed, already at the run temperature and pressure, was closed off from the flow of gases (valves 7 and 9 closed, bypass valve 8 open). The gas mixture passed through the reference side of the detector, through the bypass and back to the detector on the sample side. When the amine mixture first reached the reference side of the detector, the recorder pen made a decisive swing, indicating that the thermal conductivity, and thus the composition, of the gases on the reference side and the sample side of the detector were not the same. And the gas mixture had passed through the system and back to the sample side of the detector, the recorder pen returned to the baseline, within the 6 percent fluctuations of the gas mixture composition. This indicated that the composition of the gases on the two sides of the detector were the same. When this baseline had been achieved, the valves to the adsorbent bed, numbers 7 and 9, were opened and bypass valve 8 was closed. Again, as the amine from the feed gas mixture was adsorbed onto the zeolite, the compositions of the gases passing through the reference and sample sides

of the detector were not the same and the recorder pen moved decisively. When the zeolite became saturated with amine, the feed gas mixture passed through the bed with its' composition unchanged. At this time the recorder pen had returned to the baseline and the adsorption saturation step was complete. The bed was closed off (valves 7 and 9 closed, valve 8 opened) from the main flow. The amine pump was shut off and valve 1 closed. Helium continued to flow and flushed out the amine remaining in the system.

As mentioned above, the calibration of the detector is checked before desorption. Desorption was carried out using the same conditions of temperature, pressure and helium flowrate as during calibration. The integrator was turned on; valves 7 and 9 were opened, valve 8 closed. As the helium passed through the adsorption bed, the amine was desorbed. Using the calibration curve, the total amount of amine in the void volume and that which was adsorbed was determined.

All the equipment can be turned off without any special preparations. The amine preheater should not be left with hot amine in it as when it cools the tubing can become plugged with an oxidation product. It is best to clean out the preheater with isopropyl alcohol, which is the preferred storage solvent for the pump.

## Results

The results for the adsorption of triethylamine and diethylamine on 1.43 grams of zeolite y are given in Table

1.

Table 1

	Run	T(°C)	Mole Fraction	Partial Pressure	Grams Adsorbed
Triethylamine	1	151	0.172	7.2	0.86
	2	145	0.180	9.8	0.86
	3	141	0.168	9.2	0.86
	4	154	0.082	4.3	0.17
Diethylamine	1	150	0.150	8.0	0.045
	2	150	0.169	8.9	0.076
	3	152.5	0.168	9.8	0.063

Within the experimental error and the accuracy of both the composition determination and the calibration of the detector, the results for triethylamine runs 1, 2, and 3 become one data point. Number 4 was not repeated. For diethylamine these were the only data taken. Again, numbers 2 and 3 are, within the accuracy possible, one data point.

There has not been enough data taken to draw any conclusions about the usefulness of the adsorption of triethylamine and diethylamine on zeolites for separation of gas streams. However there has been enough runs done to see that changes to the system may make the job of gathering data an easier one.



The two primary problems at this time are the calibration accuracy and the composition accuracy. For these small amounts of amine adsorbed a more accurate calibration is needed. However, this will make necessary a more stable composition before the accuracy of the data will be improved.

To improve the calibration is not an easy task since the present calibration is accurate to 0.004 grams. However, it is a necessary project if the data is to be more accurate. The best solution would be to use a gas sampling valve to deliver the sample injection during calibration. This would give a sample whose volume would be known more accurately that it can be weighed under the experimental conditions.

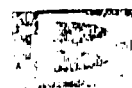
The composition may be more constant if a different method of mixing the helium and amine gases is used. One idea is to bubble the helium through the amine, achieving the saturation pressure of amine in the helium flow. Any given composition is calculated from a temperature measurement. This method has the added advantage of eliminating one of the secondary problems of the system, that of the syringe pump failing occasionally. It may also eliminate the plugging of the lines with the product of the oxidized amine.

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Appendix



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HATFIELD, PENNSYLVANIA 19440

Table A-1

CUSTOMER <i>Lehigh</i>	CUSTOMER ORDER NUMBER 904532	BROOKS SERIAL NUMBER 8303H 62311
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MAX. FLOW 0.295	MIN. FLOW 0.015	UNITS STD. LIT./MIN.	METERED FLUID HELIUM	DATE 05 JAN 1979
MAX. PRESS. DROP		ATMOS. PRESSURE		
MODEL NUMBER		METERING TEMPERATURE 70.00 DEG.F		
TUBE NUMBER	R-2-15-AAA	METERING PRESSURE 14.70 PSI A		
FLOAT NUMBER	SPHERICAL	METERING DENSITY 0.0001660 GMS/CC		
FLOAT MATERIAL	TANTALUM	FLOAT DENSITY 16.60 GMS/CC		
STD. CONDITIONS	1 ATMOS. & 70°F	DENSITY AT STD. COND. 0.0001660 GMS/CC		
PERF. CURVE NO.	0010	METERING VISCOSITY 0.01970 CP.		

STD. LIT./MIN.

SCALE READING	FLOW	SCALE READING	FLOW	INSCRIPTION			
SCALE READING	FLOW	SCALE READING	FLOW	SCALE READING	FLOW	SCALE READING	FLOW
148.5--	0.295	66.0--					
146.4----	0.29	63.5----	0.09				
144.2--		61.1--					
142.1----	0.28	58.1----	0.08				
139.9--		54.6--					
137.8----	0.27	51.1----	0.07				
135.6--		47.6--					
133.8----	0.26	44.0----	0.06				
132.2--		40.3--					
130.5----	0.25	36.6----	0.05				
128.9--		32.8--					
127.2----	0.24	28.6----	0.04				
125.6--		23.1--					
123.9----	0.23	17.6----	0.03				
122.2--		12.4--					
120.6----	0.22	7.4----	0.02				
118.7--		2.4--	0.015				
116.7----	0.21						
114.8--							
112.8----	0.20						
110.8--							
108.8----	0.19						
106.8--							
104.8----	0.18						
102.8--							
100.9----	0.17						
98.9--							
96.9----	0.16						
94.9--							
92.9----	0.15						
91.0--							
88.7----	0.14						
86.1--							
83.5----	0.13						
80.9--							
78.3----	0.12						
75.7--							
73.3----	0.11						
70.8--							
68.4----	0.10						



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HATFIELD, PENNSYLVANIA 19440

Table A-2

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH	904536	8305H64787

MAX. FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
690	40	STD. CU. CM/MIN.	HELIUM	13 MAY 1983
MAX. PRESS. DROP			ATMOS. PRESSURE	14.70 PSI
MODEL NUMBER			METERING TEMPERATURE	70.00 DEG. F
TUBE NUMBER R-2-15-AAA			METERING PRESSURE	20.00 PSI G
FLOAT NUMBER SPHERICAL			METERING DENSITY	0.0003908 GMS/CC
FLOAT MATERIAL TANTALUM			FLOAT DENSITY	16.60 GMS/CC
STD. CONDITIONS 1 ATMOS. & 70°F			DENSITY AT STD. COND.	0.0001660 GMS/CC
PERF. CURVE NO. 0010			METERING VISCOSITY	0.01972 CP.

STD. CU. CM/MIN.

INSCRIPTION									
SCALE READING	FLOW	SCALE READING	FLOW	SCALE READING	FLOW	SCALE READING	FLOW	SCALE READING	FLOW
149.7--	690	81.3--							
147.9--		78.8--							
146.1--		76.3--							
144.2--		74.0--							
142.4--	650	71.8--	250						
140.5--		69.6--							
138.7--		67.5--							
136.8--		65.3--							
135.0--		63.2--							
133.6--	600	61.0--	200						
132.1--		58.4--							
130.7--		55.4--							
129.3--		52.4--							
127.8--		49.5--							
126.4--	550	46.5--	150						
125.0--		43.4--							
123.5--		40.3--							
122.1--		37.1--							
120.7--		34.0--							
119.1--	500	30.8--	100						
117.4--		26.6--							
115.7--		22.0--							
114.0--		17.5--							
112.3--		13.1--							
110.6--	450	8.9--	50						
108.9--		4.8--	40						
107.2--									
105.5--									
103.8--									
102.2--	400								
100.6--									
99.0--									
97.4--									
95.8--									
94.1--	350								
92.5--									
90.9--									
88.9--									
86.4--									
83.9--	300								



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HATFIELD, PENNSYLVANIA 19440

Table A-3

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH	904536	8305H64787

MAX FLOW	MIN FLOW	UNITS	METERED FLUID	DATE
1040	60	STD. CU. CM/MIN.	HELIUM	13 MAY 1983
MAX PRESS. DROP			ATMOS. PRESSURE	14.70 PSI
MODEL NUMBER			METERING TEMPERATURE	70.00 DEG. F
TUBE NUMBER	R-2-15-AAA		METERING PRESSURE	40.00 PSI G
FLOAT NUMBER	SPHERICAL		METERING DENSITY	0.0006157 GMS/CC.
FLOAT MATERIAL	TANTALUM		FLOAT DENSITY	16.60 GMS/CC
STD. CONDITIONS	1 ATMOS. & 70°F		DENSITY AT STD. COND.	0.0001660 GMS/CC
PERF. CURVE NO.	0010		METERING VISCOSITY	0.01972 CP.

STD. CU. CM/MIN.

SCALE READING		FLOW	SCALE READING		FLOW	INSCRIPTION		SCALE READING		FLOW	SCALE READING		FLOW
148.9--		1040	47.1--										
146.3--			43.2--										
143.7----	1000		39.1----	200									
141.1--			35.1--										
138.4--			31.0--										
135.8--			25.7--										
133.7--			19.8--										
131.7----	900		14.1----	100									
129.7--			8.8--										
127.7--			3.6--	60									
125.8--													
123.8--													
121.6----	800												
119.8--													
117.6--													
115.4--													
113.1--													
110.9----	700												
108.7--													
106.4--													
104.2--													
101.8--													
99.4----	600												
97.0--													
94.7--													
92.3--													
89.9--													
87.0----	500												
84.0--													
81.1--													
78.1--													
75.2--													
72.5----	400												
69.8--													
67.1--													
64.4--													
61.8--													
58.7----	300												
54.8--													
51.0--													



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HATFIELD, PENNSYLVANIA 19440

Table A-4

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH	904536	8305H64787

MAX FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
1360	80	STD. CU. CM/MIN.	HELIUM	13 MAY 1983
MAX. PRESS. DROP			ATMOS. PRESSURE	14.70 PSI
MODEL NUMBER			METERING TEMPERATURE	70.00 DEG. F
TUBE NUMBER	R-2-15-AAA		METERING PRESSURE	60.00 PSI
FLOAT NUMBER	SPHERICAL		METERING DENSITY	0.0008401 GMS/CC
FLOAT MATERIAL	TANTALUM		FLOAT DENSITY	16.60 GMS/CC
STD. CONDITIONS	1 ATMOS. & 70°F		DENSITY AT STD. COND.	0.0001660 GMS/CC
PERF. CURVE NO.	0010		METERING VISCOSITY	0.01972 CP.

STD. CU. CM/MIN.

SCALE READING		FLOW		SCALE READING		FLOW		SCALE READING		FLOW	
148.5--	1360	74.1--									
146.4--		72.0--									
144.3--		70.0--									
142.3--	1300	68.0--	500								
140.2--		66.0--									
138.2--		64.0--									
136.1--		62.0--									
134.3--		60.0--									
132.7--	1200	57.1--	400								
131.1--		54.3--									
129.6--		51.4--									
128.0--		48.6--									
126.4--		45.7--									
124.9--	1100	42.8--	300								
123.3--		39.8--									
121.7--		36.8--									
120.2--		33.9--									
118.4--		30.9--									
116.7--	1000	27.1--	200								
114.9--		23.0--									
113.2--		18.8--									
111.4--		14.7--									
109.7--		11.0--									
108.0--	900	7.2--	100								
106.2--		3.5--	80								
104.4--											
102.6--											
100.7--											
98.8--	800										
96.9--											
95.1--											
93.2--											
91.3--											
89.4--	700										
87.2--											
85.0--											
82.8--											
80.6--											
78.4--	600										
76.2--											





# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO.  
HATFIELD, PENNSYLVANIA 19440

Table A-5

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH	904536	8305H64787

MAX. FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
1960	100	STD. CU. CM/MIN.	HELIUM	13 MAY 1983
MAX. PRESS. DROP			ATMOS. PRESSURE	14.70 PSI
MODEL NUMBER			METERING TEMPERATURE	70.00 DEG. F
TUBE NUMBER	R-2-15-AAA		METERING PRESSURE	100.0 PSI G
FLOAT NUMBER	SPHERICAL		METERING DENSITY	0.001288 GMS/CC
FLOAT MATERIAL	TANTALUM		FLOAT DENSITY	16.60 GMS/CC
STD. CONDITIONS	1 ATMOS. & 70°F		DENSITY AT STD. COND.	0.0001660 GMS/CC
PERF. CURVE NO.	0010		METERING VISCOSITY	0.01972 CP.

STD. CU. CM/MIN.

SCALE READING		FLOW		INSCRIPTION		SCALE READING		FLOW	
149.1--	1960	96.9--		32.4--					
147.6--		95.6--		30.5--					
146.1--		94.3--		28.1--					
144.7--	1900	93.0--	1100	25.6--	300				
143.2--		91.7--		23.0--					
141.7--		90.4--		20.5--					
140.2--		88.9--		18.0--					
138.7--		87.3--		15.4--					
137.2--	1800	85.8--	1000	13.1--	200				
135.7--		84.2--		10.8--					
134.4--		82.6--		8.5--					
133.2--		81.0--		6.3--					
132.1--		79.4--		4.0--					
130.9--	1700	77.8--	900	1.7--	100				
129.7--		76.2--							
128.6--		74.7--							
127.4--		73.3--							
126.3--		71.9--							
125.1--	1600	70.5--	800						
123.9--		69.1--							
122.8--		67.7--							
121.6--		66.3--							
120.4--		64.9--							
119.2--	1500	63.5--	700						
117.9--		62.0--							
116.5--		60.6--							
115.2--		59.0--							
113.9--		57.1--							
112.6--	1400	55.2--	600						
111.3--		53.4--							
110.0--		51.5--							
108.7--		49.6--							
107.4--		47.7--							
106.1--	1300	45.9--	500						
104.8--		44.0--							
103.5--		42.0--							
102.1--		40.1--							
100.8--		38.2--							
99.5--	1200	36.3--	400						
98.2--		34.4--							



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HARTFORD, PENNSYLVANIA 19440

Table A-6

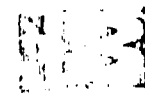
CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH UNIVERSITY	21595	8202H38443

MAX. FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
0.0445	0.0025	STD. LIT./MIN.	HELIUM	17 FEB 1981

MAX. PRESS. DROP	MODEL NUMBER	TUBE NUMBER	FLOAT NUMBER	FLOAT MATERIAL	STD. CONDITIONS	PERF. CURVE NO.	ATMOS. PRESSURE	METERING TEMPERATURE	METERING PRESSURE	METERING DENSITY	FLOAT DENSITY	DENSITY AT STD. COND.	METERING VISCOSITY	70.00 DEG.F	14.70 PSI A	0.0001660 GMS/CC	2.540 GMS/CC	0.0001660 GMS/CC	0.01970 CP.
	1110.	R-2-15-AAA	SPHERICAL	GLASS	1 ATMOS. & 70°F	0010													

STD. LIT./MIN.

SCALE READING		FLOW		SCALE READING		FLOW		SCALE READING		FLOW	
149.8--	.0445	100.2--		16.2--							
148.5----	.044	98.8----	.024	13.0----	.004						
147.1--		97.5--		9.8--							
145.7----	.043	96.1----	.023	6.7----	.003						
144.4--		94.7--		3.6--	.0025						
143.0----	.042	93.3----	.022								
141.7--		91.9--									
140.3----	.041	90.5----	.021								
139.0--		88.7--									
137.6----	.040	86.8----	.020								
136.2--		84.8--									
134.9----	.039	82.8----	.019								
133.8--		80.8--									
132.7----	.038	78.9----	.018								
131.6--		76.9--									
130.5----	.037	74.9----	.017								
129.3--		73.2--									
128.2----	.036	71.5----	.016								
127.1--		69.7--									
126.0----	.035	68.0----	.015								
124.9--		66.3--									
123.8----	.034	64.5----	.014								
122.7--		62.8--									
121.6----	.033	61.1----	.013								
120.4--		59.1--									
119.3----	.032	56.8----	.012								
118.0--		54.5--									
116.8----	.031	52.2----	.011								
115.6--		49.8--									
114.3----	.030	47.5----	.010								
113.1--		45.2--									
111.9----	.029	42.7----	.009								
110.6--		40.2--									
109.4----	.028	37.6----	.008								
108.2--		35.1--									
106.9----	.027	32.6----	.007								
105.7--		30.0--									
104.4----	.026	26.6----	.006								
103.0--		23.1--									
101.6----	.025	19.7----	.005								



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HARTFORD, PENNSYLVANIA 15440

Table A-7

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH UNIVERSITY	21595	8202H38443

MAX. FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
0.35	0.02	STD. LIT./MIN.	HELIUM	17 FEB 1981
MAX. PRESS. DROP			ATMOS. PRESSURE	14.70 PSI
MODEL NUMBER		1110.	METERING TEMPERATURE	70.00 DEG.F
TUBE NUMBER		R-2-15-AAA	METERING PRESSURE	100.0 PSI G
FLOAT NUMBER		SPHERICAL	METERING DENSITY	0.001288 GMS/CC
FLOAT MATERIAL		GLASS	FLOAT DENSITY	2.540 GMS/CC
STD. CONDITIONS		1 ATMOS. & 70°F	DENSITY AT STD. COND.	0.0001660 GMS/CC
PERF. CURVE NO.		0010	METERING VISCOSITY	0.01972 CP.

STD. LIT./MIN.

SCALE READING	FLOW	SCALE READING	FLOW	DESCRIPTION	SCALE READING	FLOW	SCALE READING	FLOW
148.9	0.35	82.0	0.15					
147.1		79.8						
145.3	0.34	77.5	0.14					
143.5		75.3						
141.7	0.33	73.2	0.13					
139.9		71.1						
138.0	0.32	69.0	0.12					
136.2		67.0						
134.6	0.31	64.9	0.11					
133.2		62.8						
131.8	0.30	60.8	0.10					
130.4		58.1						
129.0	0.29	55.2	0.09					
127.6		52.2						
126.2	0.28	49.3	0.08					
124.8		46.3						
123.4	0.27	43.3	0.07					
122.0		40.1						
120.6	0.26	37.0	0.06					
119.0		33.8						
117.3	0.25	30.7	0.05					
115.7		26.4						
114.0	0.24	21.7	0.04					
112.3		17.1						
110.6	0.23	12.7	0.03					
108.9		8.6						
107.3	0.22	4.4	0.02					
105.6								
103.9	0.21							
102.3								
100.6	0.20							
99.0								
97.3	0.19							
95.7								
94.0	0.18							
92.4								
90.7	0.17							
88.8								
86.5	0.16							
84.3								

# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HATFIELD, PENNSYLVANIA 19440

Table A-8

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH UNIVERSITY	21595	8202H38443

MAX. FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
0.65	0.03	STD. LIT./MIN.	HELIUM	17 FEB 1981
MAX. PRESS. DROP			ATMOS. PRESSURE	14.70 PSI
MODEL NUMBER 1110			METERING TEMPERATURE	70.00 DEG.F
TUBE NUMBER R-2-15-AAA			METERING PRESSURE	200.0 PSI G
FLOAT NUMBER SPHERICAL			METERING DENSITY	0.002402 GMS/CC
FLOAT MATERIAL GLASS			FLOAT DENSITY	2.540 GMS/CC
STD. CONDITIONS 1 ATMOS. & 70°F			DENSITY AT STD. COND.	0.0001660 GMS/CC
PERF. CURVE NO. 0010			METERING VISCOSITY	0.01972 CP.

STD. LIT./MIN.

SCALE READING	FLOW	SCALE READING	FLOW	DESCRIPTION			
149.4	0.65	75.3	0.25				
147.5		73.0					
145.5		70.7					
143.6		68.4					
141.7		66.1					
139.7	0.60	63.8	0.20				
137.8		61.5					
135.9		58.8					
134.2		55.7					
132.7		52.5					
131.1	0.55	49.4	0.15				
129.6		46.2					
128.1		42.9					
126.6		39.6					
125.1		36.2					
123.6	0.50	32.9	0.10				
122.1		29.4					
120.6		24.5					
118.9		19.6					
117.1		14.7					
115.3	0.45	10.3	0.05				
113.5		5.9					
111.7		1.5	0.03				
109.9							
108.1							
106.3	0.40						
104.5							
102.8							
101.1							
99.4							
97.7	0.35						
96.1							
94.4							
92.7							
91.0							
88.9	0.30						
86.2							
83.5							
80.8							
78.0							



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HATFIELD, PENNSYLVANIA 19440

Table A-9

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH UNIVERSITY	21595	8202138443

MAX. FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
0.92	0.05	STD. LIT./MIN.	HELIUM	17 FEB 1981
MAX. PRESS. DROP			ATMOS. PRESSURE	14.70 PSI
MODEL NUMBER 1110.			METERING TEMPERATURE	70.00 DEG.F
TUBE NUMBER R-2-15-AAA			METERING PRESSURE	300.0 PSI G
FLOAT NUMBER SPHERICAL			METERING DENSITY	0.003509 GMS/CC
FLOAT MATERIAL GLASS			FLOAT DENSITY	2.540 GMS/CC
STD. CONDITIONS 1 ATMOS. & 70°F			DENSITY AT STD. COND.	0.0001660 GMS/CC
PERF. CURVE NO. 0010			METERING VISCOSITY	0.01972 CP.

SCALE READING		FLOW		DESCRIPTION		SCALE READING		FLOW	
149.5--	0.92	99.0--		25.2--					
148.0--		97.7--		21.8--					
146.6--	0.90	96.4--	0.50	18.5--	0.10				
145.1--		95.1--		15.1--					
143.7--		93.8--		12.1--					
142.2--		92.5--		9.1--					
140.8--		91.2--		6.1--					
139.3--	0.85	89.8--	0.45	3.0--	0.05				
137.9--		88.1--							
136.4--		86.4--							
135.0--		84.7--							
133.9--		82.9--							
132.8--	0.80	81.2--	0.40						
131.7--		79.5--							
130.6--		77.7--							
129.4--		76.0--							
128.3--		74.3--							
127.2--	0.75	72.8--	0.35						
126.1--		71.2--							
125.0--		69.7--							
123.9--		68.1--							
122.8--		66.6--							
121.7--	0.70	65.0--	0.30						
120.6--		63.5--							
119.4--		61.9--							
118.2--		60.4--							
116.9--		58.3--							
115.6--	0.65	56.1--	0.25						
114.4--		54.0--							
113.1--		51.8--							
111.8--		49.6--							
110.5--		47.4--							
109.3--	0.60	45.2--	0.20						
108.0--		42.9--							
106.7--		40.5--							
105.5--		38.2--							
104.2--		35.9--							
102.9--	0.55	33.6--	0.15						
101.6--		31.3--							
100.3--		28.5--							



# ROTAMETER CALIBRATION DATA



BROOKS INSTRUMENT DIVISION  
EMERSON ELECTRIC CO  
HATFIELD, PENNSYLVANIA 19440

Table A-10

CUSTOMER	CUSTOMER ORDER NUMBER	BROOKS SERIAL NUMBER
LEHIGH UNIVERSITY	21595	8202H38443

MAX. FLOW	MIN. FLOW	UNITS	METERED FLUID	DATE
1.18	0.06	STD. LIT./MIN.	HELIUM	17 FEB 1981
MAX. PRESS. DROP		ATMOS. PRESSURE		14.70 PSI
MODEL NUMBER 1110.		METERING TEMPERATURE		70.00 DEG.F
TUBE NUMBER R-2-15-AAA		METERING PRESSURE		400.0 PSI G
FLOAT NUMBER SPHERICAL		METERING DENSITY		0.004607 GMS/CC
FLOAT MATERIAL GLASS		FLOAT DENSITY		2.540 GMS/CC
STD. CONDITIONS 1 ATMOS. & 70°F		DENSITY AT STD. COND.		0.0001660 GMS/CC
PERF. CURVE NO. 0010		METERING VISCOSITY		0.01972 CP.

STD. LIT./MIN.

SCALE READING		FLOW	INSCRIPTION		SCALE READING	FLOW	SCALE READING	FLOW
149.5--	1.18	63.4--						
147.2--		61.1--						
144.8--		58.2--						
142.4--		54.8--						
140.1----	1.1	51.4----	0.3					
137.7--		48.0--						
135.4--		44.6--						
133.5--		41.1--						
131.7--		37.5--						
130.0----	1.0	34.0----	0.2					
128.2--		30.4--						
126.4--		25.5--						
124.6--		20.4--						
122.9--		15.3--						
121.1----	0.9	10.7----	0.1					
119.2--		6.1--						
117.3--		1.5--	0.06					
115.3--								
113.3--								
111.3----	0.8							
109.4--								
107.4--								
105.4--								
103.3--								
101.1----	0.7							
98.9--								
96.8--								
94.6--								
92.4--								
90.2----	0.6							
87.8--								
85.2--								
82.7--								
80.2--								
77.7----	0.5							
75.2--								
72.8--								
70.5--								
68.1--								
65.8----	0.4							

Table A-11

Thermocouple Calibration

$$[\text{degree Celsius}] = a_0 + a_1 * (\text{mVolt}) + a_2 * (\text{mVolt})^{**2}$$

Thermocouple	$a_0$	$a_1$	$a_2$
1	6.2084	23.198	-0.2389
2	6.5653	23.038	-0.2258
3	6.3863	22.948	-0.2236
4	6.2328	23.134	-0.2237
5	6.1528	23.124	-0.2283

Table A-12

Thermal Conductivity Detector Calibration

$$[\text{Area}] = b_0 + b_1 * (\text{gram amine})$$

Compound	$b_0$	$b_1$	Correlation
Triethylamine	0.0250	6.376	0.99573
Diethylamine	0.0227	5.624	0.99485

Calibration Conditions:

P: 31 psig

Detector T: 190°C      i = 100amp

Helium Flowrate: 67.2 ml/min

Attenuation x 4



FIGURE A-1  
DIAGRAM OF APPARATUS

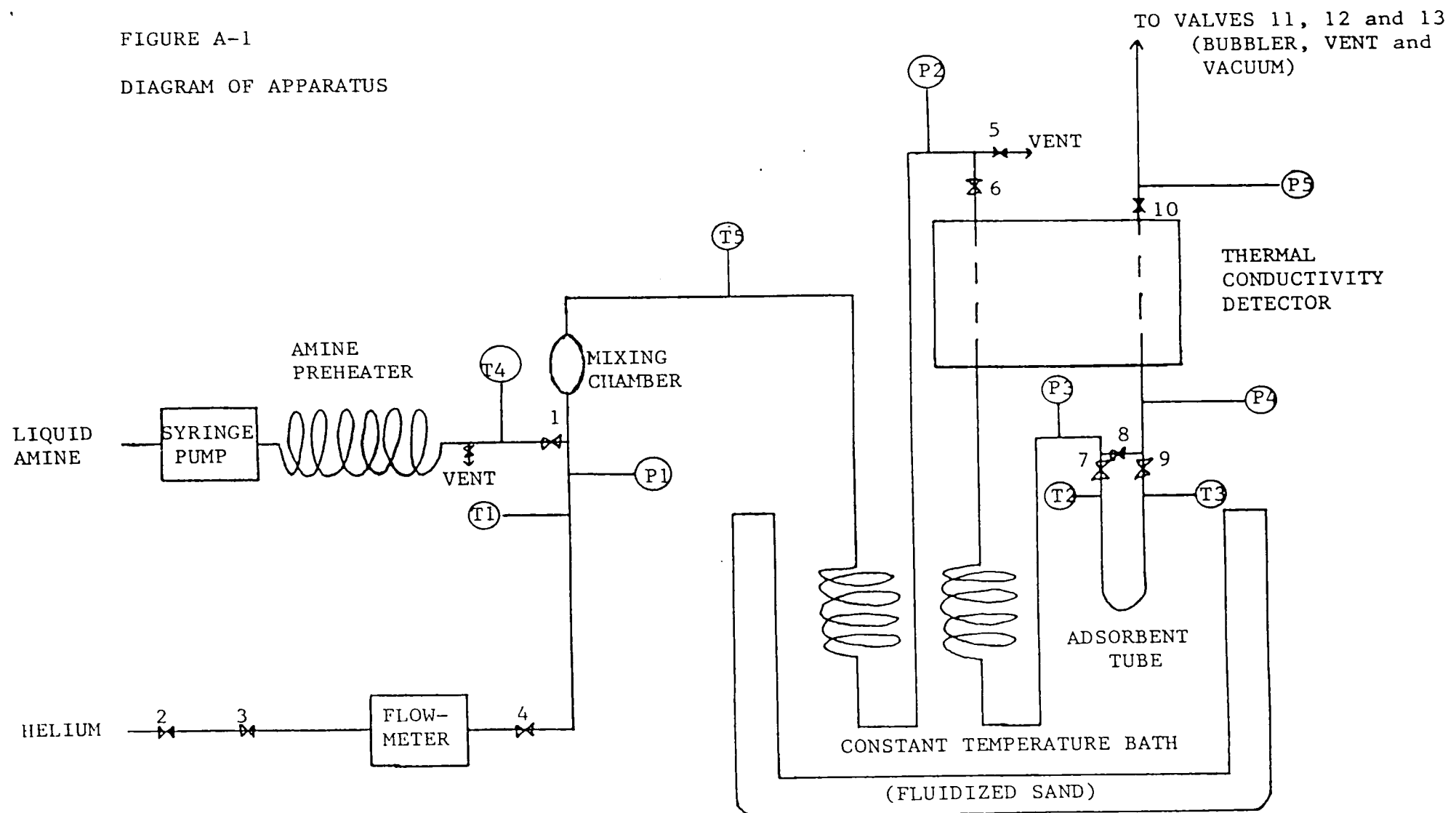
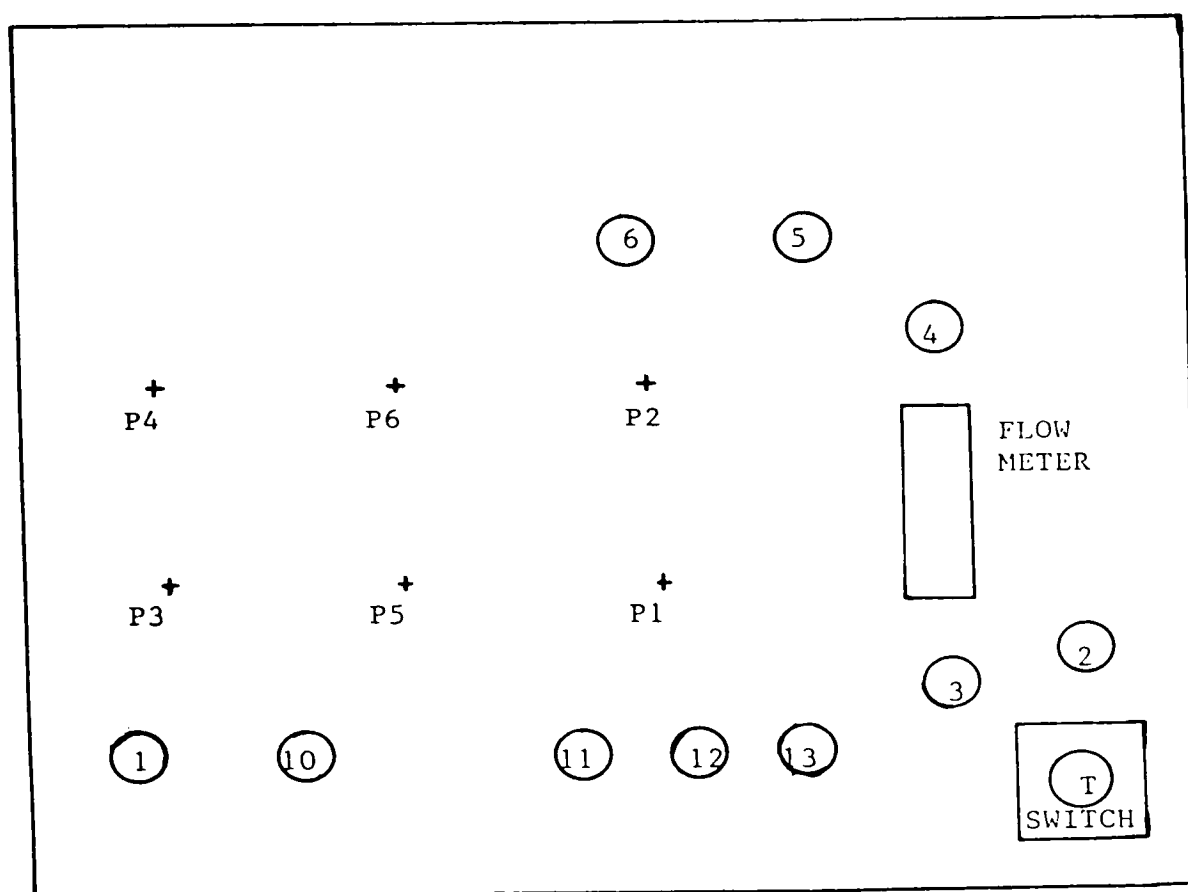


FIGURE A-1  
DIAGRAM OF PANEL BOARD



SYMBOLS:  
VALVE ○  
PRESSURE GAUGE +

FIGURE A-2  
THERMAL CONDUCTIVITY DETECTOR CALIBRATION  
TRIETHYLAMINE

