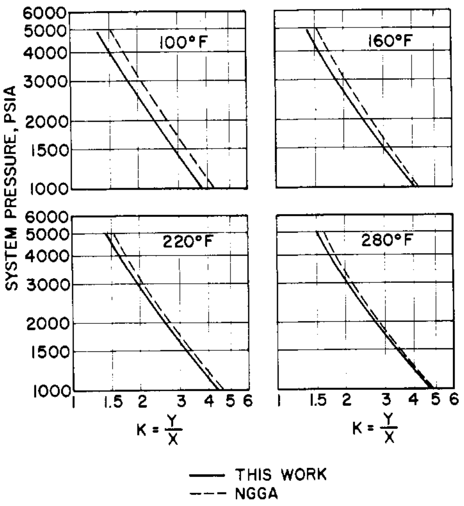

Figure 6. Experimental *K* values of methane compared to N.G.A.A. charts

variation in *K* values for nitrogen and methane as a function of the mole fraction of nitrogen and methane respectively in the vapor phase.

The *K* values for methane at 0% methane in the vapor phase were obtained from the *K* charts similar to Figure 3. These values have no physical meaning in the ternary system, but they are the *K* values for methane at a convergence pressure equal to the critical pressure of the normal decane-nitrogen system at the corresponding temperature. For each isotherm these *K* values were plotted against the pressure on a log-log paper as shown in Figure 5. At temperatures of 100, 160, 220, and 280° F. critical pressures for the binary system of *n*-decane-nitrogen were estimated by the method suggested by Grieves and Thodos (*4*). These values are 22,000, 20,000, 19,000, and 18,000 respectively.

The *K*-values for methane at these convergence pressures were evaluated from N.G.A.A. data book and are plotted in Figure 6 along with individual curves of Figure 5 at the corresponding temperature.

MATERIAL USED

The methane and normal decane were Phillips Petroleum Co. research grade chemicals with a minimum purity as follows: methane, 99% (the principal impurity was nitrogen); *n*-decane, 99.43%. The nitrogen used was OP grade with a minimum purity of 99.9%.

ACKNOWLEDGMENT

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Adsorption Rates in a Vapor-Solid System

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The adsorption rates of carbon dioxide and seven light hydrocarbons on microporous glass have been measured. The pressure and temperature dependence of the rates was studied, and comparison of the equilibrium adsorption values with those of other investigators is made. An equation is presented which allows straight-line correlation of the rate data over practically the entire time range.

TO STUDY the general behavior of the adsorption rates of hydrocarbons on porous solids the investigation of the following items was considered essential: sorption rate curves for CO_2 and seven light hydrocarbon vapors on microporous glass, the generalized effect of pressure and temperature on these curves, approximate equilibrium times for this series of adsorbates, comparison of equilibrium adsorption values obtained with those of other investigators using similar conditions and adsorbent, correlation of the rate data by an equation which would allow straight-line plotting of the data over practically the entire time range, and suggestion of the possible mechanistic scheme of adsorption.

EXPERIMENTAL

Adsorbent. The adsorbent used throughout the investigation was microporous Vycor glass No. 7930, an intermediate in the manufacture of 96% silica glass by the Corning Glass Works, Corning, N. Y. The adsorbent was in the form of disks 5 mm. thick and 50 mm. in diameter, each weighing approximately 15 grams. From one to five glass disks were used in the adsorbent chamber depending on the adsorbate and the temperature of the experiment. The properties of No. 7930 Vycor glass are given in detail by Nordberg (*10*). Some properties of Vycor disks very similar to the ones used are presented by Egger (*4*).

Free access of vapor to all surfaces was possible as the Vycor samples were spaced by 0.025 inch copper screen. A spacer was also employed between the bottom disk and the sample container.

The glass samples were originally prepared by boiling for eight hours in concentrated nitric acid, boiling for two hours in distilled water, and standing for twenty-four hours in distilled water. The last two steps were repeated four times. The specimens were then heated at 300° C. for six hours in a muffle furnace, and thereafter further dried in a vacuum desiccator for one week under a vacuum of 10 to 20 microns. Between runs the Vycor samples were desorbed (10 to 20 microns) at 90° C. for one hour, then in excess of 65° C. for two hours, and finally at 30° C. for 6 to 12 hours (until the next run).

Adsorbates. Specifications of the adsorbates which were used as follows:

Gas	Grade	Purity
Carbon dioxide	Bone dry	99.8%
Methane	Technical	96%
Ethane		95%
Ethene	C.P.	99.5%
Propane	Instrument	99.9%
Propene	C.P.	99.0%
n-Butane	Instrument	99.9%
1-Butene	C.P.	99.9%

Apparatus. The constant pressure adsorption rate apparatus is shown schematically in Figure 1 and pictorially by Figure 2. The basic principle of the apparatus, that is maintaining a constant pressure during adsorption, is that of the recording sorption kinetics apparatus of Rosen (*11*).

With reference to Figure 1, the system is pressurized with adsorbate through valve 6–2, while valves 6–4 and 6–5 are closed and valve 6–3 is open. The run begins when the adsorbent cell is brought to pressure through valve 6–4, and valve 6–5 is opened after closing valve 6–3. As the sample beings to adsorb gas, the differential pressure decrease created at constant volume is detected by the relative mercury heights in the arms of differential manometer 9. When this occurs, the mercury in the right arm makes contact with the electrical lead in that arm. This contact, by means of relay box 11, signals solenoid valve 7, and permits mercury flow from the mercury well into tube 12. The rise of mercury in this tube compresses the gas volume until the original pressure is restored. By this method then, the incoming mercury automatically replaces the volume of gas adsorbed by the sample while maintaining pressure equality with the constant initial pressure trapped in reference pressure vessel 8.

The adsorption measuring tube was assembled from a 48 mm. O.D.–44 mm. I.D. glass tube to which two 4 mm. 3-way stopcocks were attached through No. 10 rubber stoppers. All glass-rubber and metal-rubber connections were sealed with Armstrong's A-14 epoxy resin. The aluminum adsorbent vessel 16 and the steel mercury well 3, although of greatly different volumes, were of the same construction, being composed of a cylindrical shell fitted between two circular recessed plates. The shell was held tightly between the recessed area of each plate by eight ½-inch bolts connecting the two plates; sealing was accomplished by means of rubber "O"-rings seated in grooves on the ends of the shell. Reference pressure vessel 8 consisted of an American Instrument Co. (Aminco) stainless steel pressure bomb of approximately 1500 cc. whose volume was reduced to about 600 cc. by insertion of a length of brass round. Differential manometer 9 was built of ¼-inch and $\frac{9}{16}$-inch Aminco tubing, ells, and tees. The electrical probe extending into the right arm (right side, facing picture) of the manometer was simply a bare iron wire. The wire was sealed and insulated by use of a ⅜-inch, one-wire thermocouple gland from the Conax Corp. All external electrical leads were heavy copper wire.

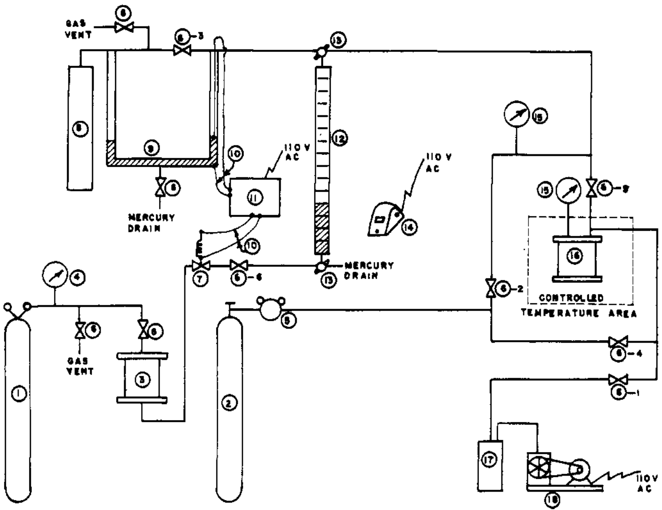

Figure 1. Constant pressure sorption rate apparatus

Legend for Figures 1 and 2.

1. Pressurizing gas cylinder (nitrogen)
2. Test gas cylinder
3. Mercury well (570 cc.)
4. Pressure gage (500 p.s.i.g.)
5. Victor pressure regulator (used only as pressure reducer)
6. ¼ inch needle valve
7. ⅛ inch normally-closed solenoid valve
8. Reference pressure vessel (approx. 600 cc.)
9. Differential manometer system
10. Electrical leads
11. Relay box for solenoid valve
12. Adsorption measuring vessel (approx. 600 cc.)
13. 4 mm. three-way stopcock
14. Electric timer
15. Pressure gage (30 inch vacuum to 100 p.s.i.g.)
16. Adsorbent vessel (empty volume approx. 80 cc.)
17. Cold trap (acetone-CO_2)
18. Vacuum pump
19. Thermoregulator
20. Agitator
21. 250 watt blade heater
22. Relay box for blade heater

The ¼ inch needle valves were procured from the American Instrument Co. and Autoclave Engineers, Inc.; the ⅛ inch, normally-closed solenoid valve, which was rated for mercury use at 1500 p.s.i., came from Airmatic Valve, Inc. All connections between valves, gages, vessels, and gas cylinders were made with ¼ inch stainless steel tubing.

For the 0° C. runs the temperature control of the sample vessel was maintained with an ice-water mixture; the 30° C. temperature was controlled by a 250 watt blade heater and a thermoregulator system.

Recording of data was done visually by means of an electric timer and a scale located behind tube 12, dividing it into divisions of $\frac{1}{20}$ inch. The volume adsorbed at any time was determined directly as the volume of mercury added to tube 12 up to that time. With this method it was possible to read to 0.1–0.2 of one division (0.005 to 0.01 inch) or 0.18 to 0.36 cc. The cross-sectional area of the adsorption measuring tube was determined at various points and an average value was used in calculating adsorption volumes. Automatic recording is possible with

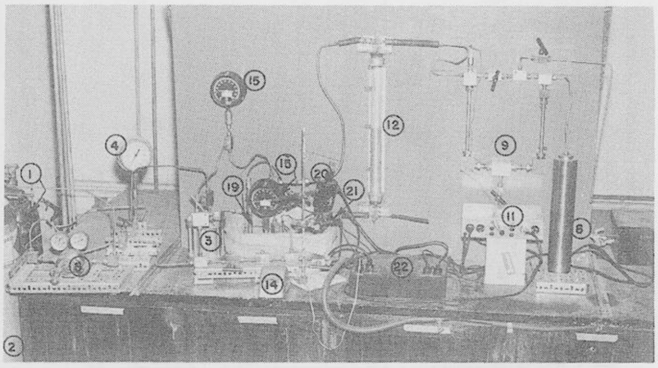

Figure 2. Pictorial view of constant pressure sorption rate apparatus

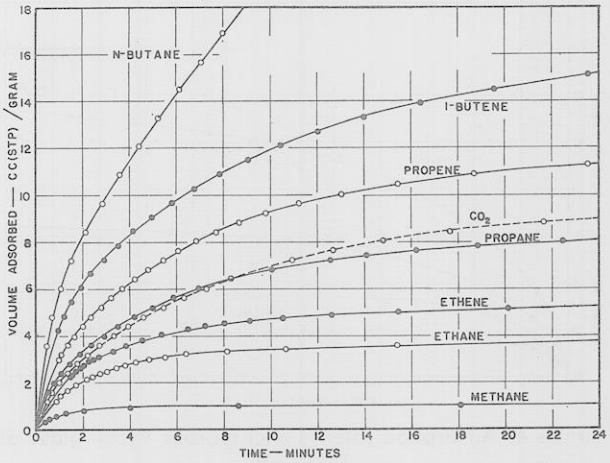

Figure 3. Adsorption rate on microporous Vycor glass at 0° C. and 15 p.s.i.a.

this apparatus and a description of such a system is given by Haman (*6*).

PROCEDURE

When switching from one adsorbate to another, the entire system was evacuated; the system exterior to the adsorbent vessel was then flushed twice with new adsorbate and repressurized, while desorption continued in the adsorption cell.

After the sample had been desorbed and brought to the desired adsorption temperature, the following procedure was carried out.

The vacuum pump was cut out of the system by closing valve 6–1, and the mercury reservoir was pressurized by opening the valve on gas cylinder 1 and then adjusting the pressure regulator on the cylinder to 50 to 100 p.s.i.g., depending on the expected value of total adsorption. The mercury level in the adsorption measuring tube was adjusted to the zero point by allowing mercury to flow through the solenoid valve using manual operation of the relay box. The entire system with the exception of the adsorption vessel was brought to the adsorption pressure by opening the test gas cylinder and allowing adsorbate gas to flow in through valve 6–2. Valve 6–2 was close when the system reached the desired pressure; valve 6–3 across the differential manometer was then closed, thus establishing a reference pressure in vessel 8.

In the actual run, the adsorbent cell was brought to system pressure as quickly as possible (1 to 2 seconds maximum) through valve 6–4, the electric timer was started, and the adsorbent vessel was opened to the system by means of valve 6–5. When relay box 11 began to click, mercury levels in the measuring tube were recorded at regular height intervals along with the corresponding time values in seconds. No points were recorded at the very beginning when mercury flowed in continuously because in this region the mercury flow had not quite caught up with the adsorption. To minimize the length of this lag period the mercury well was pressurized to about 100 p.s.i.g., and needle valve 6–6 was opened fully at the outset of a run. As a run progressed valve 6–6 was adjusted to increase sensitivity until near equilibrium it was practically closed; decreasing the pressure on the mercury well was also used to increase sensitivity after a run had progressed. Data were recorded until the time between pulses of the solenoid became excessive—greater than 100 seconds. Due to the increased sensitivity in this region one pulse of the solenoid corresponded to only about 0.2 to 0.3 cc. After 1 to 2 hours the adsorption ceased or became practically undetectably for almost every gas (except for *n*-butane which at 0° C. is extremely close to its condensation temperature under 15 p.s.i.a.). This volume was then recorded as the equilibrium value.

RESULTS

The major portion of the work was concerned with determining the constant pressure adsorption rate curves for CO_2 and the seven hydrocarbons. Runs were carried out for all eight gases at a constant pressure of 15 p.s.i.a. and temperatures of 0° and 30° C.; single runs for 1-butene at 20 and 25 p.s.i.a. and 30° C. were also undertaken to study the effect of pressure on rate of adsorption. Comparative rate curves for the eight adsorbates are given in Figure 3. It is interesting to note that the rate curves of CO_2 and propane at 0° C. cross over.

Equilibrium times for the adsorbates tested varied from about ½ to 1 hour for the 30° C. runs and 1 to 2 hours for the 0° C. tests. Increasing the adsorption pressure also appears to extend the equilibrium time.

The rate of adsorption at any time can be determined directly as the slope of the rate curve (total volume adsorbed *vs.* time) at that particular time coordinate. Figure 4, which contains a sample of the data obtained, clearly indicates greater slopes for the 0° C. curves than for the respective 30° C. curves. Consequently, at constant pressure a decrease in temperature results in an increase in the rate of adsorption. Similar results were obtained for all adsorbates tested.

With reference to the three isobars of Figure 5, it can be noted that the slope of the rate curve, at any point, increases as the pressure is raised from 15 to 20 and then to 25 p.s.i.a. This signifies that at constant pressure the rate of adsorption increases with increasing pressure.

Equilibrium adsorption values for the complete series of adsorbates at 15 p.s.i.a. and 0° and 30° C. are presented in Table I, and comparison with the data of others using a similar adsorbent is made. With the exception of the present data, all 0° C. data were pressure interpolated, and all 30° C. values were pressure interpolated and temperature cross-plotted from 0°, 25°, and 40° C. data. The starred (*) values of Egger indicate average values of two separate investigations on approximately the same mass of Vycor glass. It is to be expected that different batches of porous glass can have varying adsorptive capacities

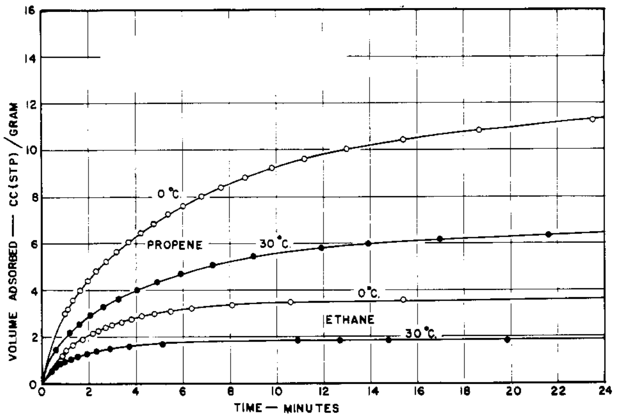

Figure 4. Adsorption rate on microporous Vycor glass at 15 p.s.i.a.

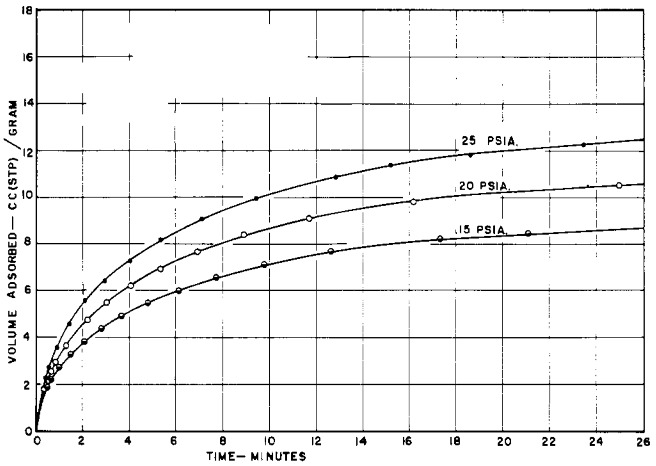

Figure 5. Adsorption rate of 1-butene on microporous Vycor glass at 30° C.

(*i.e.*, surface areas), and therefore comparison of data becomes somewhat difficult.

Sources of Error. Certainly, the start-up procedure contributed the major source of error. In the time required to pressurize the sorbent cell, that is 1 to 2 seconds, a certain amount of adsorption took place which was not accounted for. However, in this short period of time the vapor has not had time to diffuse extensively into the inner structure of the sorbent (Figure 7). This is the probable explanation why the equilibrium sorption values were not noticeably affected (Table I).

The void volume of the sample cell was made as small as possible. Cell 16 was of such dimensions that the appropriate number of glass disks together with the required size metal blank did fit in the cell with a clearance of about $\frac{1}{32}$ inch on top and around the perimeter of the disks. Thus any slight variation from the system pressure in setting the pressure in the adsorption cell resulted in an insignificant change in the resultant system pressure, as the void volume of the sorbent chamber was very much less than that of the overall system.

Table I. Equilibrium Adsorption Values at 15 P.S.I.A.

Vapor	Temperature °C.	Volume Adsorbed Cc. (STP)/Gram Adsorbent Present Data	Egger's Data (*4*)	Miller's Data (*9*)
Methane	30	0.637	0.65*	
	0	1.09	1.3*	
Ethane	30	1.85	1.9	
	0	3.70	2.4	
Ethene	30	3.84	3.8*	3.7
	0	5.50	5.4*	5.6
Propane	30	3.82	3.8	
	0	8.67	6.6	
Carbon dioxide	30	6.24	6.1*	6.4
	0	9.99	10.4*	8.3
Propene	30	6.68		
	0	12.18		
1-Butene	30	9.18		
	0	17.09		
n-Butane	30	9.66		

* Average values of 2 separate investigations.

The question of constancy of adsorbent temperature does not arise in static adsorption measurement, as the sorbent is presumably left in contact with the adsorbate long enough to insure thermal equilibrium. In this instance only the final state of the adsorbent is important. As long as the sample is at the equilibrium temperature in the final state, the equilibrium adsorption value for that temperature will always be obtained. However, in measuring rates of adsorption if the adsorbent temperature builds up with time, the resultant rate data will be of little value.

In the present case, if the glass disks were assumed to be completely adiabatic, an appreciable temperature change could occur in some instances (*i.e.*, about 10° C. for CO_2 at 15 p.s.i.a. and 0° C.). Nevertheless, as mentioned previously, the aluminum sample cell (high thermal conductivity) was made as compact as possible. Thus, it was thought that the constant temperature reservoir could maintain the adsorbent at approximately constant temperature throughout a run. This was checked experimentally by locating a thermocouple on the sorbent surface. It was found that there was no detectable temperature rise accompanying the adsorption of CO_2 on porous glass at 0° C. and 15 p.s.i.a. This is not to say that no temperature rise was present, but only that it was not of such magnitude to cause a significant effect on the rate data.

Naturally, in all adsorption systems one tries to eliminate all adsorbing substances other than the sample. In the present system a very small amount of rubber and a minute amount of stopcock grease were present.

A No. 10 rubber stopper and a three-way stopcock were used on each end of measuring tube 12. The lower portion of the tube always contained some mercury, thus the lower stopper and stopcock need not be considered. Rubber "O"-rings were used in sealing the sample vessel; however, these exposed a minute surface to the adsorbing vapor. Blank tests were made which would check the sorption in the top rubber stopper, the "O"-rings of vessel 16, and in the stopcock grease of the upper stopcock. No detectable amount of adsorption was observed in an hour.

Leak tests were peformed with the apparatus by closing valve 6-3. No detectable leak was obtained in two hours, the period of the longest run.

CORRELATIONS

Three equations were tested in an attempt to correlate the rate data obtained.

Bangham and Burt (*1*) found that they could accurately

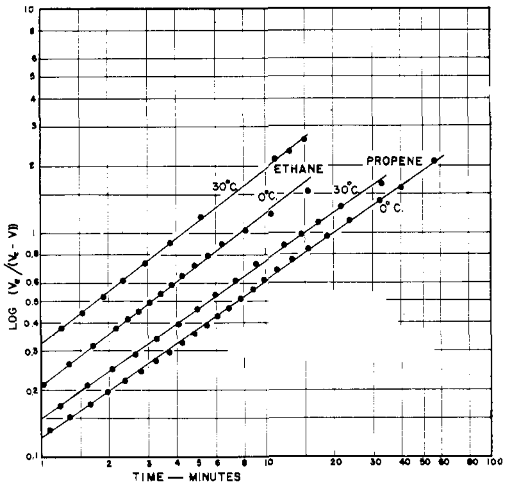

Figure 6. Correlation of adsorption rate data

express their results for the rate of sorption of CO_2 on fine glass wool at essentially constant pressure by:

$$V = Kt^{1/m} \tag{1}$$

where V is total volume adsorbed at any time t, and K and m are constants. Application of this equation to the data showed that correlation was possible for only a relatively short time period after the outset of adsorption, depending on the adsorbate and the temperature. However, it is clear from the form of the equation that it cannot be valid close to equilibrium, since it would give infinite adsorption for infinite time.

The theory of rate of adsorption on a free surface was first derived by Langmuir (*8*). Upon integration and rearrangement, Langmuir's rate equation becomes:

$$2.303 \log (V_e/(V_e - V)) = Kt \tag{2}$$

where V and V_e are the volumes adsorbed after time t and at equilibrium, respectively. Harned (*7*) found that this equation represented the vapor adsorption of chloropicrin and of carbon tetrachloride on charcoal at low pressures. When the data were correlated with this equation (Figure 8), agreement was obtained for only a slightly greater period of time than with Equation 1.

To correlate adsorption rate data for glass wool which extended into weeks and even months, Bangham and Sever (*2*) found it necessary to employ the following equation:

$$2.303 \log (V_e/(V_e - V)) = Kt^{1/m} \tag{3}$$

It is obvious that Equation 2 is simply a special case of Equation 3 when $m = 1$. If Equation 3 were fit a set of data, a plot of log $[\log[V_e/(V_e - V)]]$ *vs.* log t should closely approximate a straight line. Such a plot, using rate data obtained for two of the adsorbates, is given in Figure 6. In a few cases very good correlation was obtained even up to the final data point just before equilibrium. Where deviation does occur there is a strong possibility that the V_e determined for that run may have been slightly in error; thus, as equilibrium is approached V becomes nearly equal to V_e, and any slight change in V_e would cause a very large change in the term, $V_e/(V_e - V)$. The value of m in Equation 3 varied from about 2½ to 3. Not only was this value approximately the same for all the adsorbates, but it was practically identical for a particualr gas at two different temperatures (0° and 30° C.).

It is thus of considerable interest that the Bangham and Sever correlation worked well with the seven light hydrocarbons and an adsorbent appreciably different from glass wool.

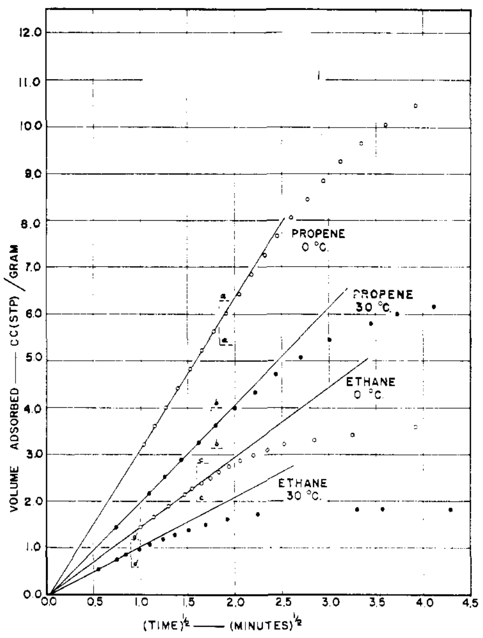

Figure 7. Diffusion correlation

Adsorption Mechanisms. It seems reasonable with the adsorbent used—microporous Vycor glass with an average pore diameter of 40 to 60 angstroms—that the following mechanistic scheme should occur:

1. The initial rate follows Fick's law, that is, the rate is controlled by diffusion through the pores of the adsorbent.
2. After the intial period, when the first molecules have diffused into the innermost regions of the adsorbent, the rate commences to be limited by adsorption on a free surface—Langmuir's law.
3. When a monolayer has built up on the surface, multilayer adsorption will occur in most cases.
4. When sufficient layers have been adsorbed on the walls of the smaller pores, the pore space will be bridged by a meniscus and condensation may occur in these capillaries. The pressure required to cause condensation of a particular vapor in pores of specified width can be determined from the Kelvin equation (*3*).

In Figure 7, a plot of total adsorption *vs.* the square root of time, a straight line is obtained which nearly passes through the origin and is obeyed for a definite length of time for each vapor tested (only two are used as examples). This is taken as correspondence with Fick's law of diffusion (*5*). The points at which the respective diffusion correlations begin to deviate are indicated by a–a, b–b, c–c, and d–d. Corresponding points are shown in Figure 8 by the same symbols, which in this figure indicate where the monolayer correlation becomes valid. Naturally, after a short period when a monolayer has formed on the surface, the Langmuir correlation no longer applies, as is evident from Figure 8.

The fact that the extrapolation in Figure 7 does not quite pass through the origin can probably be traced to the error in the start-up procedure. The extrapolation distance is 1½ seconds from the origin, which is precisely the average start-up lag time.

The period of obeyance for the diffusion correlation appears to increase with increasing molecular size and decreasing temperature, *i.e.*, longer for propene than for ethane, and longer for 0° C. than for 30° C. These certainly are reasonable phenomena if diffusion through the pores is the controlling mechanism in this region, as larger molecules should diffuse through the same porous medium with

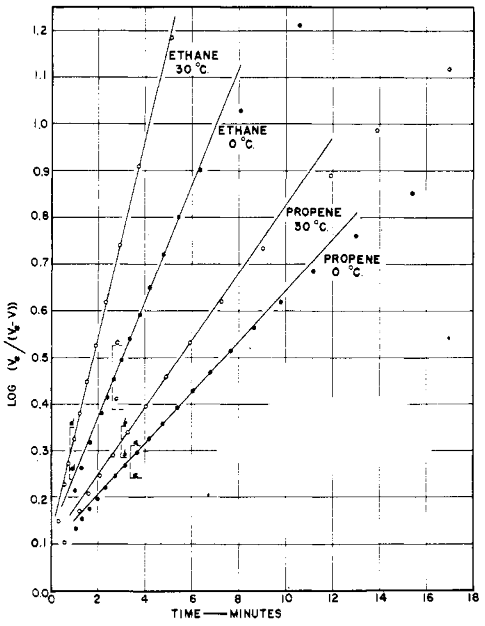

Figure 8. Monolayer surface adsorption correlation

more difficulty than molecules of lesser dimensions. Further, the lower the temperature, the less energetic is a gas molecule, *i.e.*, the lower its average translational motion.

ACKNOWLEDGMENT

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Phase Relationships for the $AgNO_3$-AgI-$AgIO_3$ System

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IN THE REPROCESSING of spent fuel elements from reactors, gaseous products are liberated which include radioactive iodine. To remove this iodine, a tower consisting of silver nitrate coated on ceramic packing is used (*6*). The reactions conditions are in the range of 200° C. and 1 atm.

The over-all equation in unbalanced form is:

$$AgNO_3 + I_2 \rightarrow AgIO_3 + AgI + NO_2 \qquad (1)$$

The reaction mechanism is uncertain. Blasewitz and Schmidt (*1*) report a series of reactions as follows:

$$2AgNO_3 + I_2 \rightleftarrows 2AgI + O_2 + 2NO_2 \qquad (2)$$

$$AgNO_3 + \tfrac{1}{2} I_2 + O_2 \rightleftarrows AgIO_3 + NO_2 \qquad (3)$$

$$AgIO_3 \xrightarrow{\Delta} AgI + \tfrac{3}{2} O_2 \qquad (4)$$

They further state that AgI is the thermodynamic stable end-product when the reaction is run at 200° C.

Lacksonen (*5*) has shown conclusively that $AgIO_3$ is stable in the presence of dry air or helium in the pressure range of 1 to 760 mm. of Hg and for temperatures as high as 330° C. No decomposition was noted in the presence of AgI or $AgNO_3$. Dupuis and Duval (*3*) report a decomposition temperature of 410° C. without melting. Silver iodide has a decomposition temperature of 552° C. without melting, and $AgNO_3$ melts at 211° C. without decomposition (*4*).

In studying the kinetics of the I_2 (v) – $AgNO_3$ (s or l) reaction, Lacksonen (*5*) experimentally measured the mole ratio of $AgIO_3$/AgI and compared the results with a theoretical maximum ratio of 0.5 if all of the oxygen liberated via Equation 2 reacts as in Equation 3 with Equation 4 not kinetically applicable. These experiments showed that the $AgIO_3$/AgI ratio was always lower than the maximum value of 0.5. It dropped to as low as 0.22 and was dependent on the physical state of the reacting zone. Hence, the temperature-composition phase diagram for the AgI-$AgIO_3$-$AgNO_3$ ternary was necessary in the interpretation of the kinetics of the reaction.

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