OPERATING CHARACTERISTICS OF MEMBRANES

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It appears inevitable that membrane research will eventually lead to the establishment of the gaseous-diffusion process as one of industry's basic unit operations. This process permitted successful large-scale separation of isotopic uranium hexafluorides and was thus of *paramount* importance in the atomic bomb program; it continues to play just as important a role in peacetime developments of atomic energy. Industry has at its disposal literally hundreds of different porous materials which ultimately will be investigated and may be used as membranes for the separation of gases and vapors.

During the past two years a number of studies have been reported which utilized strictly microporous as well as plastic membranes for the separation of gases and vapors (*1, 2, 5, 8, 11, 13*). One of the microporous membranes of more than passing interest is porous glass (*3, 4, 5, 7, 8, 9, 12*), because the pore diameters of this material are in the range of 20 to 60 Å. and therefore are considerably smaller than the mean free paths of most gases at atmospheric temperature and pressure [about 500 to 3,000 Å. (*6*).] Five porous-glass membranes were studied, four of which were prepared at different times by the Corning Glass Works. Two of the membranes were taken from the same group of samples. Average wall thicknesses, that is, membrane thicknesses, were determined by a mercury - displacement method. Actual measurements of wall thicknesses would have required breaking of the membranes. The average value for the thicknesses of the five membranes is 0.114 cm. Thickness measurements on five other broken membranes which were available averaged 0.114 ± 0.017 cm.

Equilibrium time, rates of permeation, and ratios of the permeability rates which indicate the direction and degree of separation to be expected were determined for the five membranes at the same time and under identical conditions. The gases used were oxygen, nitrogen, and carbon dioxide at 25°C. over a range of 3.72 to 7.80 atm. absolute upstream and atmospheric downstream pressure.

APPARATUS AND PROCEDURE

The diagram of the apparatus is presented in Figure 1, and photographs are shown in Figures 2 and 3. The test gases were obtained from commercial sources and were not further purified. However, each gas was dried by being passed through silica gel before it entered the testing unit. The purity of the gases, as quoted by the suppliers, was 99.6% for oxygen, 99.0% for nitrogen, and 99.5% for carbon dioxide. Before the five membranes were installed in the top flange, they were "normalized" at 510°C. for 12 hr. to remove adsorbed organic vapors and water vapor. This procedure was rigidly followed before each run even though the same gas was being used. To determine the time required for membrane equilibrium at different pressures, it was necessary to desorb the membrane of the gas which had been passing through it during the previous run. All runs were made at essentially 25°C. Porous glass is normally consolidated to Vycor at 1,100° to 1,200°C. (*10*), which is far enough above the normalizing temperature for the pores to be assumed unaltered during the normalizing operation. Rates of permeation through the membranes were essentially the same before and after normalizing. Before a run was started, the valve in the top flange was opened and the system was purged at approximately 1.3 atm. abs. (5 lb./sq.in. gauge) for 2 min. The valve was then closed and the system immediately subjected to the desired operating pressure. The gas which permeated the membrane was discharged into the atmosphere except when a sample was collected in the burette to determine the rate of permeation, which was measured by displacement of a saturated sodium chloride solution from a 100-cc. gas burette, connected to the membrane by a rubber tube. The time required to collect samples was determined with a stop watch which could be read to within ± 0.25 sec. Atmospheric pressure was maintained on the downstream side of the membranes by means of a leveling bulb connected to the bottom of the gas burette. After a run was completed, either the rubber tubing leading to the gas burette was removed from the membrane or the three-way stopcock in the collection line was opened to the atmosphere. Diffusion of gas through the rubber tubing was considered negligible, as essentially atmospheric pressure was maintained on both sides of the tubing, the wall of which was 2 mm. thick. The time required for runs was quite short, namely, about 20 to 200 sec. The temperature of the room in which the experiments were conducted was constant within ± 0.5°C. during each run, and consequently it was not considered necessary to place the unit in a constant-temperature bath.

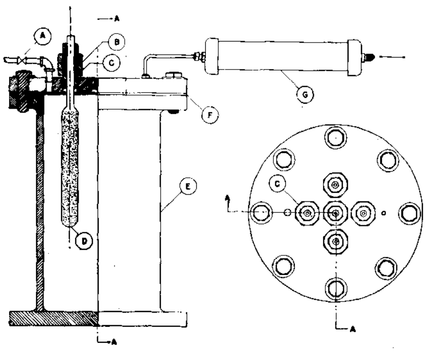

FIG. 1. DIAGRAM OF APPARATUS:

A, ⅛-in. stainless steel needle valve; *B*, ¾-in. O.D. by ¼-in. I.D. by ¾-in. rubber gasket; *C*, brass packing-gland-type adapter to hold porous-glass membrane; *D*, porous-glass membrane; *E*, standard pipe 6-in.-diam. by 12-in. long with 8-in.-diam. by ½-in. long steel flanges welded to both ends; the top flange, which contains the five adapters, was bolted on with eight ⅝-in. S.A.E. hexagonal bolts; *F*, ⅛-in. Garlock rubber gasket; *G*, 1½- by 10-in. standard pipe, silica-gel–filled gas drying unit.

DISCUSSION OF RESULTS

The dimensions of the five porous-glass membranes studied are presented in Table 1. Typical rates of permeation at equilibrium, G_{eq}, and the time required for equilibrium to be established within the membranes, t_{eq}, under various operating conditions for nitrogen, oxygen, and carbon dioxide are presented in Tables 2 through 4. The equilibrium rate of permeation, G_{eq}, is defined as the number of standard cubic centimeters of

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TABLE 1.—DIMENSIONS OF POROUS-GLASS MEMBRANES TESTED

Sample*	Length, mm.	Outside area, sq. cm.	Thickness, cm.
1	145	78.57	0.109
2	47	21.0	0.107
3	84	39.8	0.122
4	143	75.4	0.112
5	141	75.87	0.121
			Avg. 0.114

*Membranes 1 and 5 were from the same group of samples received from the Corning Glass Works.

TABLE 2.—NITROGEN EQUILIBRIUM PERMEABILITY DATA

G_{eq} = rate of permeation at equilibrium, std. cc./(sec.) (sq. cm.) -10^3
t_{eq} = time required for equilibrium to be established, hr.
p = 1.0 atm. abs. downstream pressure

Porous glass	1		2		3		4		5	
Upstream pressure conditions, π, atm. abs.	G_{eq}	t_{eq}	G_{eq}	t_{eq}	G_{eq}	t_{eq}	G_{eq}	t_{eq}	G_{eq}	t_{eq}
				A						
≈1.3 to 3.72	7.70	57.9	7.33	51.5	12.47	57.9	8.81	67.6	6.97	57.9
≈1.3 to 3.72	6.94	56.0	6.46	45.6	10.9	15.9	7.87	45.7	6.56	51.1
≈1.3 to 3.72	7.17	55.7	6.67	49.8	11.19	49.8	8.27	49.8	6.81	55.9
≈1.3 to 3.72	6.84	61.0	6.44	58.0	10.78	34.6	7.84	58.2	6.53	63.0
≈1.3 to 5.08 to 3.72	7.02	0.25	6.56	2.22	10.82	13.7	8.10	0.25	6.64	0.25
≈1.3 to 5.08 to 3.72	6.98	0.25	6.50	0.25	10.77	0.25	8.01	0.25	6.60	0.25
≈1.3 to 7.80 to 3.72	7.07	0.92	6.47	0.92	10.81	0.25	7.96	0.92	6.62	0.92
≈1.3 to 7.80 to 3.72	7.25	0.87	6.61	1.5	10.98	0.87	8.18	0.25	6.75	0.25
				B						
≈1.3 to 5.08	10.28	43.5	9.71	51.1	16.15	17.0	11.78	34.8	9.73	38.4
≈1.3 to 5.08	10.47	34.3	9.66	36.8	16.05	13.3	11.78	27.6	9.73	25.2
≈1.3 to 3.72 to 5.08	10.28	0.25	9.62	0.25	15.92	2.25	11.78	0.25	9.73	0.25
				C						
≈1.3 to 6.44	14.36	3.50	13.39	7.67	22.29	1.06	16.34	5.50	13.74	7.67
≈1.3 to 3.72 to 6.44	14.31	0.25	13.21	2.47	22.22	0.25	16.48	0.25	13.43	0.25
				D						
≈1.3 to 7.80	17.94	1.0	16.23	0.25	27.86	4.0	20.31	4.0	17.10	1.0
≈1.3 to 3.72 to 7.80	17.94	0.25	16.53	0.25	27.86	0.25	21.04	0.25	17.18	0.25
≈1.3 to 6.44 to 7.80	17.94	0.25	17.61	0.25	28.47	0.25	20.74	0.25	16.93	0.25

TABLE 3.—OXYGEN EQUILIBRIUM PERMEABILITY DATA

G_{eq} = rate of permeation at equilibrium, std. cc./(sec.) (sq. cm.) -10^3
t_{eq} = time required for equilibrium to be established, hr.
p = 1.0 atm. abs. downstream pressure

Porous glass	1		2		3		4		5	
Upstream pressure conditions, π atm. abs.	G_{eq}	t_{eq}	G_{eq}	t_{eq}	G_{eq}	t_{eq}	G_{eq}	t_{eq}	G_{eq}	t_{eq}
				A						
≈1.3 to 3.72	6.30	25.3	5.92	42.7	9.86	6.0	7.27	49.5	5.98	31.0
≈1.3 to 3.72	6.53	20.5	6.17	35.5	10.13	5.5	7.46	20.5	6.21	5.5
≈1.3 to 3.72	6.50	23.5	6.10	23.5	10.04	23.5	7.46	23.5	6.21	27.2
≈1.3 to 7.80 to 3.72	6.57	1.75	6.19	1.25	10.22	0.25	7.65	5.0	6.35	3.5
≈1.3 to 7.80 to 3.72	6.57	1.5	6.12	1.5	10.13	0.25	7.55	0.25	6.21	0.25
				B						
≈1.3 to 5.08	9.78	11.8	9.24	20.3	15.10	6.5	11.10	22.6	9.26	12.8
≈1.3 to 3.72 to 5.08	9.92	2.5	9.04	1.0	15.06	2.5	11.11	1.0	9.27	1.0
≈1.3 to 3.72 to 5.08	9.83	0.5	9.20	2.5	15.06	0.5	11.33	0.5	9.34	0.5
≈1.3 to 3.72 to 5.08	9.72	1.4	9.03	2.5	15.00	2.5	11.11	0.5	9.27	0.5
				C						
≈1.3 to 6.44	13.46	2.25	12.59	5.0	20.78	5.0	15.29	2.25	12.70	14.75
≈1.3 to 5.08 to 6.44	13.46	1.25	12.37	1.25	20.44	1.25	15.31	1.25	12.70	1.25
≈1.3 to 5.08 to 6.44	13.46	1.50	12.30	2.25	20.64	2.25	15.31	2.25	12.70	0.5
				D						
≈1.3 to 7.80	16.62	2.5	15.58	4.5	25.90	4.25	19.26	2.0	16.17	8.5
≈1.3 to 7.80	16.78	2.3	15.60	3.75	25.90	4.0	19.31	1.75	16.25	6.75
≈1.3 to 6.44 to 7.80	16.87	1.12	15.61	0.25	26.22	0.25	19.31	0.25	16.07	0.25
≈1.3 to 6.44 to 7.80	16.87	1.50	15.67	1.5	25.92	1.5	19.31	0.25	16.22	0.50

gas per second per square centimeter passing through the membrane. The time, t_{eq}, required for equilibrium to be established within the membrane is in hours.

The membranes were operated in three ways. In the first case after the system was purged (at 5 lb./sq.in. gauge) the membranes were brought rapidly from purging pressure to the initial operating pressure, which in this case was also the final operating pressure. In the second case the initial operating pressure was higher than the final operating pressure. When essentially constant rates of permeation were established for all the membranes at the initial operating pressure, the final operating pressure was produced by suddenly decreasing the pressure on the membranes to the desired value. This type of operation was initiated to save time in establishing membrane equilibrium, because during several trial runs it was observed that the membrane came to equilibrium considerably faster at higher than

TABLE 4.—CARBON DIOXIDE EQUILIBRIUM PERMEABILITY DATA

G_{eq} = rate of permeation at equilibrium, std. cc./(sec.) (sq. cm.) -10^3
t_{eq} = time required for equilibrium to be established, hr.
p = 1.0 atm. abs. downstream pressure

Porous glass Upstream pressure conditions, π atm. abs.	1 G_{eq}	1 t_{eq}	2 G_{eq}	2 t_{eq}	3 G_{eq}	3 t_{eq}	4 G_{eq}	4 t_{eq}	5 G_{eq}	5 t_{eq}
				A						
≈1.3 to 3.72	10.09	47.6	7.12	53.1	11.81	53.1	9.35	53.1	8.52	57.6
≈1.3 to 6.44 to 3.72	9.74	2.75	6.96	2.75	11.60	2.75	9.28	2.75	8.34	2.75
≈1.3 to 7.80 to 3.72	10.47	0.5	7.20	0.5	11.85	0.5	9.50	8.5	8.59	6.00
≈1.3 to 7.80 to 3.72	10.66	0.75	7.35	0.75	12.11	0.75	9.82	0.75	8.98	0.75
				B						
≈1.3 to 5.08	15.6	38.2	10.8	46.3	17.80	34.6	14.40	46.3	13.10	40.5
≈1.3 to 3.72 to 5.08	15.70	4.25	10.91	9.25	17.83	0.25	14.36	0.75	12.98	0.50
				C						
≈1.3 to 6.44	20.55	32.2	14.49	42.2	24.22	25.7	19.00	42.2	16.68	28.9
≈1.3 to 5.08 to 6.44	20.9	0.5	14.69	18.25	24.5	18.25	19.63	4.75	17.70	7.0
				D						
≈1.3 to 7.80	26.9	10.1	18.72	10.1	30.5	8.58	24.5	10.1	22.46	10.08
≈1.3 to 7.80	26.9	9.08	18.89	9.08	30.9	9.08	24.5	9.08	22.9	9.08
≈1.3 to 6.44 to 7.80	26.9	0.42	18.4	3.42	30.9	0.42	24.5	0.42	22.46	4.67

at lower operating pressures. In the third case the initial operating pressure was greater than atmospheric, but less than the final operating pressure. When the rates of permeation through all the membranes were nearly constant, the pressure was increased rapidly to the final operating level.

The upstream pressure side conditions, represented by Π, in Tables 2 through 4, have been arranged so that the first pressure listed corresponds to the purging pressure on the membrane. After the system was purged, the pressure was suddenly increased to the second pressure listed and equilibrium was established within the membranes at this pressure. In cases where a third pressure is given, the pressure was rapidly changed from the second to the third value and equilibrium was again attained. All tables list G_{eq} and t_{eq} only for the last upstream pressure condition.

In all instances the rate of permeation through membrane 3 was definitely greater than through the other membranes. To some extent this is also true for membrane 4. The membrane thicknesses, listed in Table 1, differ by less than 14% and therefore the differences in flow cannot be explained by thickness variations. Previous investigations (*3, 9, 12*) have repeatedly demonstrated that the average pore size of porous glass is at most about 60 Å. and also that the pore-size distribution is rather sharp; therefore, it is unlikely that a difference in pore size or in distribution was responsible for the different flow rates. Support for this contention is given by the data in Table 5, where average permeability are shown to be essentially constant for all five porous-glass membranes. This is proof that all flow experiments were made in the molecular flow region.

Table 5 also gives ratios of permeabilities of N_2 and O_2. These ratios were found to be constant for the individual runs, and therefore only average values are reported. This fact signifies that all membranes behaved exactly alike in molecular flow. Consequently the conclusion is justified that the differences in flow rates were due to variations in the number of pores per unit surface area, that is, differences in void volume. This is a perfectly logical explanation.

In designing industrial diffusion units, it is important that the variation in flow rates per unit area and the variation in separation characteristics for different membranes constructed of the same basic material should be well defined. Without such knowledge, the design engineer faces the possibility of over- or underdesigned units.

The data presented in Tables 2 through 4 are pertinent to the start up of a diffusion process. Probably

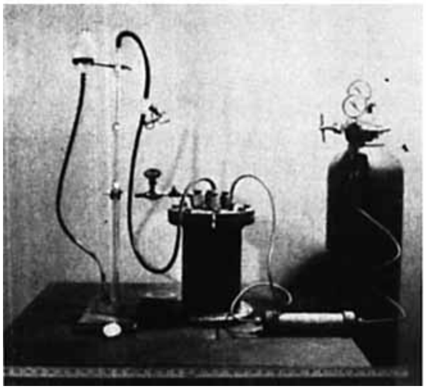

FIG. 2. ASSEMBLED TESTING APPARATUS.

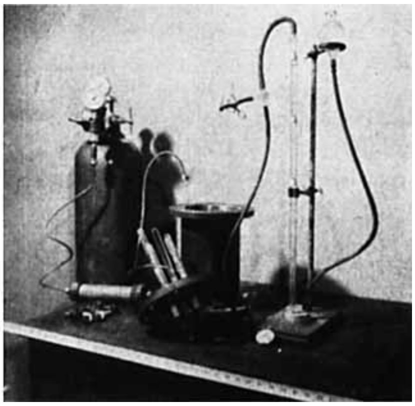

FIG. 3. MEMBRANES AND DISASSEMBLED TESTING APPARATUS.

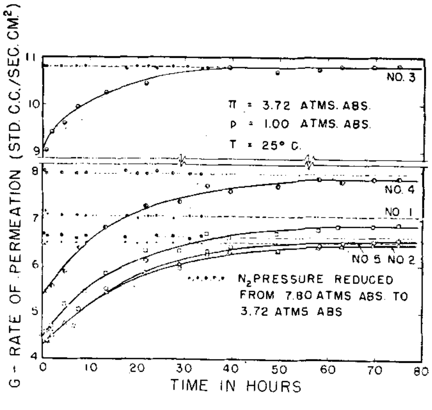

FIG. 4. NITROGEN PERMEATION RATES × 10^3 THROUGH POROUS-GLASS MEMBRANES.

all membranes require a certain length of time to reach membrane equilibrium. The data indicate that equilibrium can be obtained in anywhere from a fraction of an hour to several hours at high pressures, compared with as much as several days or more at low pressures. One would expect that similar phenomena would be exhibited by ceramic and metallic membranes which have pore diameters up to approximately 500 Å. For pore diameters greater than 500 Å., the adsorbed molecular layers should not contribute appreciably to the total rate of flow through the capillary. It would be expected that the smaller the pore diameter, the greater would be the length of time required for membrane equilibrium. Very slow adsorption of gas within the pores is believed to account for the time lag observed in attaining membrane equilibrium; therefore, the smaller the pore diameter, the greater would be the influence of the adsorbed layers on the effective pore diameter and the greater would be the effect which adsorption contributed to the flow and separation mechanism.

Some variation in reproducibility of t_{eq} was observed (Tables 2 through 4) when the membranes were studied under the same conditions. It was definitely established, however, that t_{eq} is always smaller at higher upstream pressures than at the lower upstream pressures. In all cases it was consistently found that on reducing the upstream pressure to a lower final operating pressure, the time required for membrane equilibrium was considerably reduced. It is recommended that membrane equilibrium be attained by initially subjecting the membrane to a high upstream pressure for a short period of time and then either suddenly or slowly decreasing it to the final operating pressure. The typical data presented in Figure 4 justify this recommendation.

For the data presented in Figure 4, nitrogen was placed on the outside of membranes at 7.80 atm. abs. (100 lb./sq. in. gauge) for just 1/2 hr. and the membranes did not necessarily reach equilibrium before the pressure was suddenly decreased to 3.72 atm. abs. (40 lb./sq.in. gauge). When the membranes were operated in this manner, it was found that reduction of the upstream pressure resulted in very rapid establishment of flow equilibrium as compared with the approximately 2 days needed when the pressure was increased from the purging pressure to 3.72 atm. abs. Similar results were obtained for oxygen and carbon dioxide under identical operating conditions, and the rates of permeation were of the same order of magnitude as the G_{eq} values obtained when the upstream pressure was increased from purging pressure (about 1.3 atm. abs.) to 3.72 atm. abs.

The experimental data in Tables 2 through 4 are also significant in that t_{eq} was observed to be greater in all cases when the upstream pressure was increased from essentially atmospheric conditions to the intermediate rather than to the higher final pressures.

Consequently, the lower the upstream operating pressure, the more important it becomes to direct attention to the best method for obtaining membrane equilibrium as rapidly as possible.

TABLE 5.—AVERAGE PERMEABILITIES AND PERMEABILITY RATIOS

Gas	Average permeability*: $P \times 10^3$ Membrane number 1	2	3	4	5	
Nitrogen	0.285	0.260	0.494	0.334	0.297	
Oxygen	0.265	0.242	0.461	0.310	0.279	
Carbon dioxide						
Δp: 2.72 atm.	0.411	0.282	0.531	0.392	0.382	
: 4.08 atm.	0.417	0.285	0.532	0.394	0.385	
: 5.44 atm.	0.415	0.287	0.545	0.398	0.386	
: 6.80 atm.	0.430	0.293	0.552	0.403	0.402	
Based on N_2 and O_2 the CO_2 flow should be	0.227	0.207	0.394	0.266	0.237	
	Actual permeability ratios α values					Theoretical ratio†
N_2/O_2	1.076	1.076	1.070	1.077	1.065	1.069
N_2/CO_2‡	0.688	0.916	0.922	0.848	0.773	1.253
O_2/CO_2‡	0.635	0.852	0.860	0.787	0.727	1.172

$$\text{* Permeability } P = \frac{\text{(Std. cc.) (cm. thickness)}}{\text{(sq. cm. area) (sec.) } (\Delta p \text{ atm. pressure drop})}$$

$$\text{†Theoretical ratio: } \frac{P_{N_2}}{P_{O_2}} = \sqrt{\frac{M_{O_2}}{M_{N_2}}} = \sqrt{\frac{32}{28}} = 1.069$$

‡Average P_{CO_2} values at Δp of 2.72, 4.08, 5.44 atm. are used. At 6.80 atm. condensed flow begins to increase noticeably with pressure.

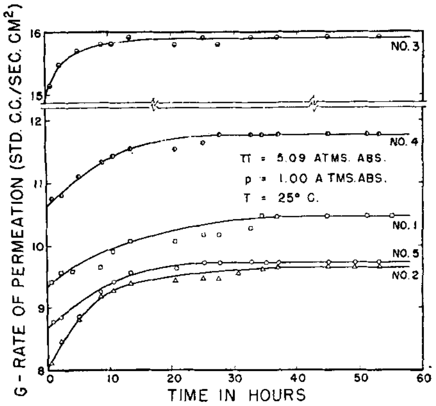

FIG. 5. NITROGEN PERMEATION RATES × 10^3 THROUGH POROUS-GLASS MEMBRANES.

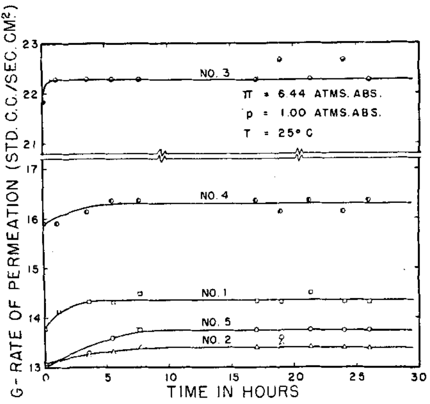

FIG. 6. NITROGEN PERMEATION RATES × 10^3 THROUGH POROUS-GLASS MEMBRANES.

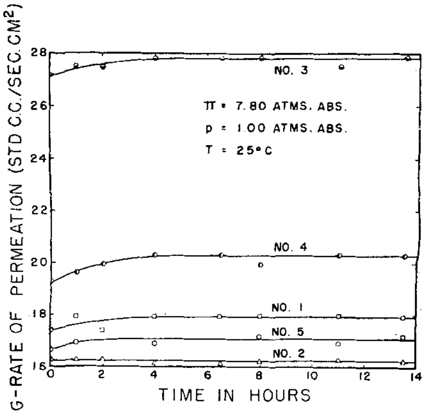

FIG. 7. NITROGEN PERMEATION RATES × 10^3 THROUGH POROUS-GLASS MEMBRANES.

For the sake of simplicity, the G_{eq} and t_{eq} data have been presented in tabular form in Tables 2 through 4. To show typical detailed rates of permeation data obtained, one complete set of data has been presented for nitrogen at various upstream pressures in Figures 4 through 7. It is seen in Figure 4 that the initial rate of permeation may vary by as much as 30% from the rate of permeation at equilibrium, G_{eq}. As the upstream pressure was increased (Figures 5, 6 and 7), the initial rates of permeation more closely approached the rates at equilibrium, G_{eq}. This should be expected, for adsorption takes place much more rapidly at the higher pressures. Membranes 2 and 3 diverged the most in regard to rates of permeation. On increase of the upstream pressure, the percentage variation in rates of permeation remained approximately the same for all membranes, but when it is interpreted as difference in actual flow rates the higher the upstream pressure, the greater the difference in actual flow rates.

Uniform separation characteristics of individual membranes are just as important as uniform flow rates through the membranes. Similarly, the effect of pressure on establishing membrane equilibrium and the time required for membrane equilibrium is of paramount importance. It would indeed be undesirable to construct a number of diffusion stages without knowing whether all the membranes used possessed identical separation characteristics.

The separation factor, α, for ideal gases through ideal membranes is equal to the square root of the inverse ratio of the molecular weights of the individual components. This is in accordance with the separation predicted by Graham's equation(*5*), which is

$$\frac{y_A}{1-y_A} = \sqrt{\frac{M_B}{M_A}}\left[\frac{\pi x_A - p y_A}{\pi\,(1-x_A) - p\,(1-y_A)}\right]$$

where y_A and y_B are the mole fractions of the components on the low-pressure side of the membrane, x_A and x_B are the mole fractions of the components on the high-pressure side, M_A and M_B are the molecular weights, and II and p are the upstream and downstream pressures, respectively. Graham's equation in this form assumes that the feed composition remains virtually constant. When experimental permeability data for individual components and for identical operating conditions are available, they may be used in place of $\sqrt{M_B/M_A}$, usually expressed in terms of $\alpha = G_A/G_B$. The α values are an index to the direction and degree of separation of the components. The separation characteristics of the five membranes studied are presented in Table 5 in terms of α values which were computed from observed rates of permeation data, and the α values are also compared with $\sqrt{M_B/M_A}$ values. As stated previously, the constancy of the α values for N_2-O_2 is proof that the separation characteristics of all five membranes for nitrogen-oxygen separation were quite uniform even though the equilibrium rates of permeation through the five membranes varied as much as 30% under identical operating conditions. The consistent α values for nitrogen-oxygen indicate that the pore diameters of the five membranes are relatively uniform, but the number of pores per unit area probably varied.

This accounts for the marked difference in the observed rates of permeation at equilibrium. The α values for nitrogen-oxygen also were in good agreement with the theoretical values.

When the carbon dioxide data in Table 5 are analyzed, it is obvious that carbon dioxide permeated at a much higher rate than would be predicted from N_2 and O_2 flow. At 25°C. oxygen and nitrogen are considerably above their critical temperatures, which are —118.8° and —147.1°C. respectively. The critical temperature of carbon dioxide is 31.1°C.; consequently, oxygen and nitrogen were actually gases at 25°C., whereas carbon dioxide could be considered a vapor. Experimental evidence has been reported which shows that vapor mixtures and gas-vapor mixtures cannot be expected to separate in the direction predicted by the square root of the inverse ratio of the component molecular weights(*5, 8*), and the α values calculated here for nitrogen–carbon dioxide and oxygen–carbon dioxide indicate that these gas-vapor mixtures do not separate in inverse proportion to the square root of the molecular weights.

The flow rates of carbon dioxide were found to be greater than predicted by Graham's law. The anomalous flow behavior of carbon dioxide through various membranes has also been reported by other investigators(*8*). This observation is in accordance with the concept that "condensed" flow rates of vapors are generally greater than flow rates of gases. Substantiating evidence of condensed flow phenomena was reported in the separation study of azeotropic vapors (*5*). It is of further interest to note that the α values for the gas-vapor mixtures studied showed considerable variation between the five different membranes. The membranes which gave the highest flow rates, that is 3 and 4, also exhibited a greater proportion of condensed flow, a result that agrees with the contention that these membranes possessed a higher void volume, or pore surface.

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