Isothermal Permeabilities from Thermo-Osmosis Experiments

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Synopsis

With the use of strain-gauge pressure transducers, an improved constant volume apparatus has been constructed for the measurement of isothermal permeabilities of gases from thermo-osmosis experiments. Permeabilities are reproducible to about ±5% accuracy and compare favorably with those obtained by alternative techniques. Revised values are presented for the permeability coefficients of the noble gases in rubber.

INTRODUCTION

The permeability of membranes to gases is commonly measured by one of the following isothermal techniques: variable volume, variable pressure, or variable concentration.[1] In 1952, Denbigh and Raumann[2] proposed an alternative nonisothermal technique that enables one to obtain isothermal permeabilities from experiments on thermo-osmosis. Using their method, they measured the permeability coefficients of N_2, CO_2, H_2, and H_2O,[3] and Bearman[4] measured the permeability coefficients of the noble gases. However, neither the apparatus of Denbigh and Raumann nor that of Bearman was designed for accurate permeability measurements, and the reproducibility of their results was relatively poor. We initiated the present studies in the belief that it would be possible to obtain more accurate measurements with an improved apparatus.

The major problem in designing the apparatus lies in measuring pressure accurately without disturbing the system. Denbigh and Raumann used wide-bore mercury-in-glass manometers which introduced undesirable thermal gradients and necessitated continual adjustments of the mercury levels in order to maintain constant volume. The effects of these disturbances on the results are not clearly understood. Bearman used fragile capillary-with-reservoir mercury manometers which required uncertain corrections for capillary effects. In order to avoid such difficulties, we have used strain-gauge pressure transducers. The transducers can be entirely submerged in constant temperature baths. With appropriate calibration, their accuracy is better than ±0.1 mm. Hg in the pressure range of 0–1 atm. Furthermore, with their use, the volume of the system remains essentially constant during an experiment.

Using our improved apparatus, we have remeasured the permeability coefficients of the noble gases (except radon) and carbon dioxide through a natural rubber membrane.[5]   Our measurements are reproducible within $\pm 5\%$, which is comparable to the accuracy claimed by most investigators who obtain their values under isothermal conditions.   Agreement with values obtained isothermally is of the order of $\pm 10\%$ and is well within the variations of the permeability coefficients determined by different isothermal methods.[1,6]   We have also studied, in selected cases, the dependence of permeability on mean pressure, mean temperature, and the temperature difference.   Studies were made on the reproducibility of measurements when we vary the membrane thickness, the membrane itself, and the design of the apparatus.   Special attention was paid to the effects of degassing, adsorption, and leakage on the values of the permeability.

THERMO-OSMOSIS

In the thermo-osmosis experiment, two vessels containing a gas under investigation are separated by a thin membrane and are maintained at constant temperatures different from each other in such a way that almost all of the temperature change takes place across the membrane thickness. It is then found that the temperature gradient causes the gas to pass through the membrane at an appreciable rate.   In the course of time, a concentration gradient is built up which causes the gas to flow in the opposite direction.   When the flow of gas caused by the temperature gradient just balances the opposing flow due to the concentration gradient, a steady-state pressure difference is built up across the vessels.   The temperature difference is usually so adjusted that this pressure difference has a magnitude of several centimeters of mercury.

By considering thermo-osmosis as partly due to the heat of solution of the gas in the membrane and partly due to the existence of a thermal diffusion process inside the membrane, Denbigh and Raumann[2] derived the following equations to describe, respectively, the steady state and the rate of approach to the steady state:

$$\ln (p_2/p_1)_\infty = -Q^*/R \ (1/T_1 - 1/T_2) \tag{1}$$

and

$$\frac{\ln (p_2/p_1)_\infty - \ln (p_3/p_1)_0}{\ln (p_2/p_1)_\infty - \ln (p_2/p_1)_t} = kt \tag{2}$$

where

$$k = 4ApP/nV_0l \tag{3}$$

Here, $\dot{T}_1$, T_2 are the temperatures of the two surfaces of the membrane; $(p_2/p_1)_t$ is the pressure ratio at time t; R is the gas constant; Q^* is the heat of transport; p is the mean gas pressure of the system; nV_0 is the total volume of gas under standard conditions; l is the thickness of the membrane; A

is the area of the membrane; and P is the mean isothermal permeability of the membrane.

At the start of the experiment, the pressure on each side of the membrane may be adjusted to any convenient value. As the thermo-osmosis proceeds, the changes in pressure with time are recorded. The system is considered to be at steady state when the pressure ratio remains constant to three significant figures for over 24 hr. The permeability, P, in units of square centimeters per second per atmosphere, is obtained through the use of eq. (2).

EXPERIMENTAL

Apparatus

The system consists of the cell proper (17 cc. in volume) and a vacuum line (1250 cc. in volume) which is necessary for the addition of gases to the cell and for the calibration of the pressure transducers. The cell (Fig. 1) consists of the membrane assembly, M, two pressure transducers, T_{18} and T_{17}, connectors B_1 and B_2, and the valves, V_1, V_2, V_3, and V_4. Valves V_1 and V_2 separate the membrane assembly from the rest of the system and are used during calibrations of the transducers. Valves V_3 and V_4 are toggle valves which separate the cell from the vacuum line.
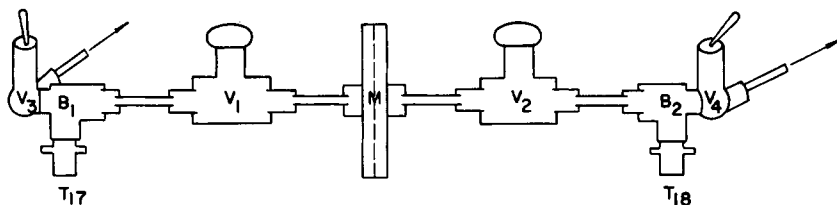

Figure 1.

Our membrane assembly (Fig. 2) is essentially the same as that of Bearman[7] with the following exceptions: (1) instead of aluminum housings, solid bronze housings are used to eliminate the pitting of the aluminum due to corrosion; (2) molded nylon screws and threaded nylon rods and nuts are used in place of metal screws to reduce the heat transferred between the two constants (±0.05°C.) temperature baths; (3) three pairs of thermocouples, rather than one, are placed at different regions of the surfaces of the membrane and the porous bronze; (4) the design of the Lucite rings holding the housings allows easy separation of the assembly from the baths.

The system, including the cell and membrane, is relatively vacuum-tight. Thus, starting with a pressure of less than 0.1 μ Hg, the pressure increases to 0.05 mm. Hg in a 12-hr. period without pumping.

AIR GAP

STAINLESS STEEL BATH

NYLON NUT

NYLON THREADED ROD

$\frac{3}{4}$" LUCITE

NYLON BOLT AND NUT

O RING

LAYER OF SILICONE GREASE

NYLON SCREW

SMALL LUCITE RING

BUTYL O RING

AIR GAP

POROUS BRONZE

$\frac{1}{8}$" COPPER TUBING

SWAGELOK CONNECTOR

SOLID BRONZE

RUBBER MEMBRANE

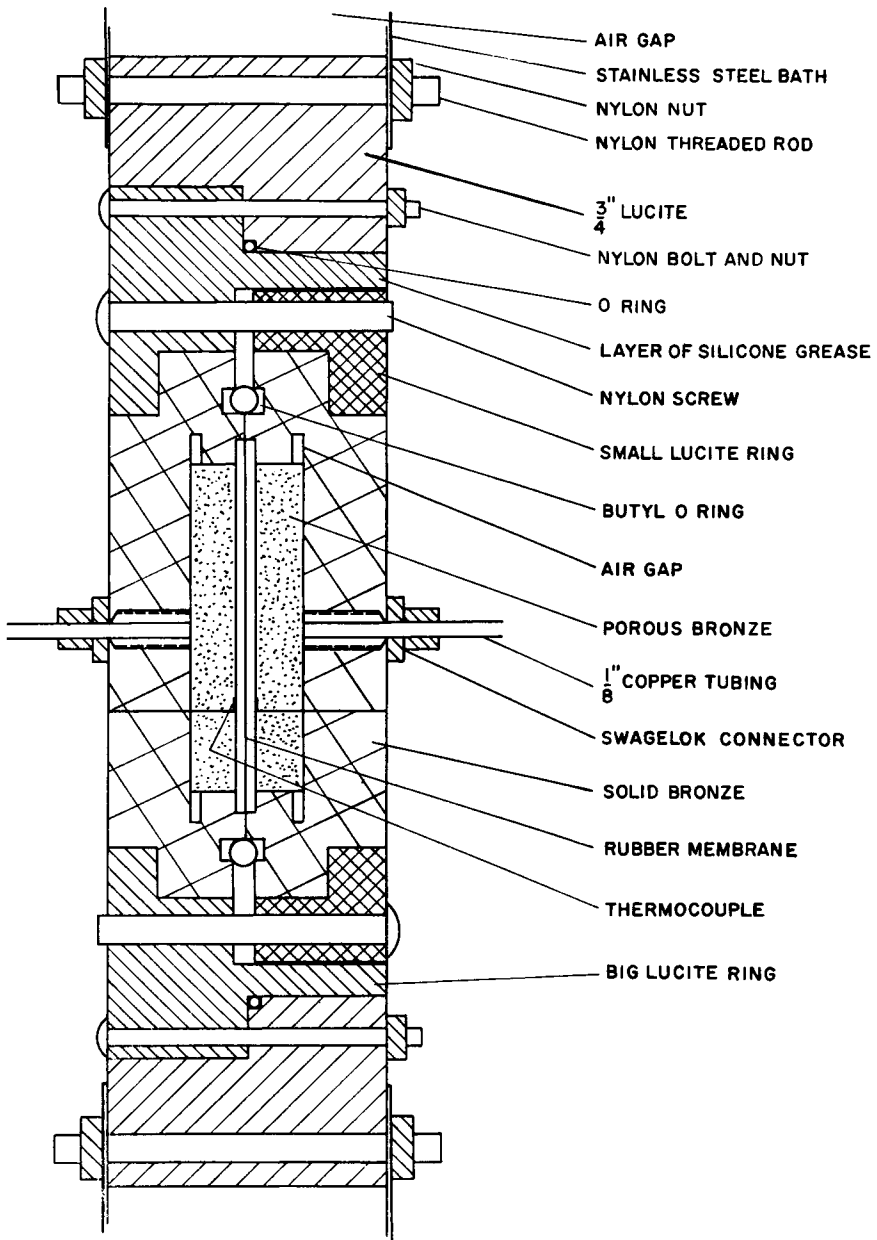THERMOCOUPLE

BIG LUCITE RING

Figure 2.

Pressure Measurement

We measure the pressure of our cell by the use of 15 psi absolute pressure transducers (type 4-312A) manufactured by Consolidated Electrodynamics Corporation. These are small ($^1/_2$ in. diameter, $^3/_4$ in. nominal length),

light-weight (2 g.), diaphragm-type pressure pick-ups operable over a temperature range of −350 to +300°F. This variable resistance-type transducer has unbonded strain-gauge windings connected in a four-arm bridge as the sensing elements. Output and input connections with the transducers are made through four numbered pin-type terminals which fit an electrical connector, requiring no soldering for attachment. When the electrical leads are properly protected, the cell may be submerged completely in thermostats. Each transducer has an output of about 22 mv. full range at 80°F. which we amplify 80 times in order to measure the full range of pressure to within 0.1 mm. Hg on a Leeds and Northrup K-3 potentiometer. We use a 6-v. storage battery as the source of the 5-v. input. In order to have a reproducible output, it is necessary to have a constant input. However, the input tends to change slightly whenever the applied pressure changes. Thus, just before each measurement, we adjust the input to 5.0000 ± 0.0001 v.

Our greatest problem in using the transducers lies in their calibration. We find that the relationship between pressure and emf is parabolic. There is a definite zero-shift with the change of temperature. In addition, a hysteresis effect is observed. That is, whenever a sudden large change in pressure is experienced by the transducer's diaphragm, a calibration change is noted. These shifts are random and are of the order of 0.1 mm. However, if no sudden large change in pressure occurs, the calibration is stable within ±0.1 mm. Hg over a prolonged period. In order to achieve the accuracy we desire, we calibrate the transducers after each run.

A cathetometer, with an accuracy of ±0.01 mm. is used to read a $^3/_4$-in. bore mercury manometer, which acts as the primary standard for the calibration of the transducers. Our calibration procedure requires two persons to read simultaneously the voltage output of the transducers and the mercury levels. The manometer readings are converted to 25°C. according to the equation: $h_{25} = h_T + d_T/d_{25}$ (where h denotes heights and d is density of mercury). The corrected manometer data for each transducer with its corresponding voltage output are then fitted by the method of least squares to a parabola. Each calibration has at least seven points to cover the pressure range (about 1.5–3 cm. Hg) encountered in a run. The root-mean-square deviation of the calibrated points from the fitted curve is taken as a measure of the accuracy of our pressure measurements in the course of a run. We have found that this deviation varies between 0.05–0.1 mm. of mercury. The outputs of the transducers for a run are converted analytically to pressures from the coefficients of the equation for the parabola.

The advantages of using the transducer as a pressure-measuring instrument are as follows. It can be completely submerged in a constant-temperature bath. Its output can be followed by a recording potentiometer for continuous reading in kinetic runs. The system can be completely glass-free and thus can be used for helium work. The transducer has a very small volume and is resistant to corrosion. It is less fragile than a glass

manometer and requires no corrections for thermal gradients, volume changes, or for capillary effects. Moreover, these transducers are available for great varieties of pressure ranges. Its greatest disadvantage to us is the necessity of calibration after each run. However, this is probably not necessary if less accurate results are desired.

Materials

In the course of our investigation, the parameters of our system were varied. For system I, a pure gum rubber membrane of thickness 0.0325–0.0350 cm. (membrane type A) was used with a Parker O-ring 2-228 of Parker compound B278-7 (a special butyl compound). Brass toggle valves with synthetic rubber seats were used with this assembly. For system III, a type B membrane of thickness 0.058–0.059 cm. was used with Parker butyl O-ring 2-228. The type A and type B membranes were manufactured by the same company and were supposed to have the same composition. However, the type A membranes appear to be darker and not so spongy. All-metal Monel diaphragm-type toggle valves were used in system III. Some auxiliary work was done in system II which had Teflon O-rings and Monel toggle valves. Table I lists the details for the various systems used.

The specifications of gases used are as follows.

Xenon was obtained from Matheson Scientific, Inc. in a 100-ml. Pyrex flask at 755 mm. Hg pressure. It was certified to have impurities of less than 80 ppm.

Neon was obtained from Matheson in a 7-liter tank at a pressure of 250 psig. It was certified to have impurities of less than 80 ppm.

Krypton was obtained from Matheson in a 7-liter tank at a pressure of 250 psig. It was certified to have impurities of less than 170 ppm.

Argon was obtained from Linde Air Products Co. in a steel tank with a guaranteed minimum purity of 99.995%.

Helium was obtained from Linde in a steel tank with a guaranteed minimum purity of 99.99%.

Two tanks of CO_2 were used. The first tank was of unknown origin, and the purity of the gas was not specified. The second tank was obtained from Matheson and had a guaranteed minimum purity of 99.99%.

TABLE I
Details for the Various Systems

System	Toggle valves	O-ring material	O-ring Dimensions, cm.			Membrane material	Membrane thickness, cm.
			I.D.	Height	Width		
I	Brass	Butyl	5.715	0.348	0.349	Type A	0.0325–0.0350
IIa	Monel	Teflon	5.715	0.344	0.375	Type A	0.0360–0.0440
IIb	Monel	Teflon	5.715	0.358	0.374	Type B	0.0270–0.0280
IIc	Monel	Teflon	0.715	0.346	0.355	Type B	0.0275–0.0285
III	Monel	Butyl	0.715	0.361	0.343	Type B	0.0580–0.0590

Procedure

(1) The thermostats are set to the desired temperatures. A period of at least 4 hr. is allowed for the system to come to thermal equilibrium with the baths.

(2) Previous to the first run with any new gas, the system is degassed by pumping for at least 36 hr. The pump is turned off and the system is first flushed and then filled with the gas to be used. A waiting period of 2–5 hr. after the addition of the gas permits most of the adsorption by the system to occur before the start of a run. The pressure on either side of the membrane is adjusted to suitable values. Normally, we start the experiment with equal pressures on the two sides of the membrane. However, in order to obtain permeability data, for the gases with a small pressure difference at steady state (Ar and Kr), we started the run with either the hot side or the cold side having a higher pressure.

(3) The toggle valves V_3 and V_4 are shut. The time of shutting of the second toggle valve is considered the start of the run. The pressure of the rest of the line is reduced to insure positive sealing of the seats of the one-way toggle valves.

(4) Measurements of the transducer outputs, thermocouple outputs, and the time are recorded at suitable intervals. Such measurements are taken until after the steady state has been reached.

(5) Calibration of the transducers follows immediately. The valves V_1 and V_2 are shut. They prevent the nitrogen gas used for the calibration from entering the membrane assembly. They also prevent pressure variations at the transducers caused by the flow of gas between thermostats. The pressure of the rest of the line is adjusted by the addition of the cali-brating gas to approximately the average pressure of the system. The toggle valves, V_3 and V_4, are then opened so that the transducers will not suffer any sudden large change in pressure.

(6) At the completion of the calibration, the line is evacuated with V_1 and V_2 shut and then with V_1 and V_2 open. This prevents the contamination of the membrane assembly by the calibrating gas.

RESULTS

The permeability coefficients are obtained through the use of eq. (2), i.e.,

$$\log (X'_\infty - X'_t) = -kt/2.303 + \log (X'_\infty - X'_0)$$

where $X' = \log (p_H/p_C)$ and $k = 4ApP/nV_0l$. The quantities A, p, l, and nV_0 can all be measured. Thus, we can obtain P if we know the value of k. To obtain k, we first plot $\log (X'_\infty - X'_t)$ versus t and discard by graphical inspection initial and final data points which are subject to large errors. The remaining points are fitted by the method of least squares to a straight line.

TABLE II

Gas	Date	p_{mean}, cm.	T_{mean}, °C.	$P \times 10^8$, cm.²/sec.-atm.	System
Xenon	2/20/62	45.48	30.6	63.7	I
	2/22/62	36.86	30.4	61.6	I
	2/25/62	27.54	30.5	62.2	I
Neon	3/28/62	44.93	31.1	11.5	I
	4/3/62	47.16	30.9	11.1	I
Argon	4/10/62	52.61	30.8	17.6	I
	4/15/62	52.66	30.5	17.3	I
Krypton	6/8/65	54.01	29.3	24.6	III
	6/28/65	57.04	29.7	22.1	III
Helium	5/21/62	64.76	31.2	26.2	I
	7/17/62	54.48	31.2	27.6	I
	12/13/61	51.78	30.6	28.6	I
	12/21/61	51.59	30.9	27.6	I
	6/11/62	34.92	30.9	27.1	I
	6/18/62	34.85	31.1	27.1	I
	6/15/62	25.15	31.0	26.6	I
	11/27/64	54.39	29.9	23.2	III
Carbon dioxide(I)	7/27/62	51.36	30.5	116.2	I
	11/26/64	54.75	29.8	105.5	III
Carbon dioxide(II)	11/23/64	52.83	29.7	98.6	III
	2/10/65	55.35	29.9	98.4	III

TABLE III

Gas	Date	p_{mean}, cm.	T_{mean}, °C.	ΔT, °C.	$P \times 10^8$, cm.²/sec.-atm
He	7/1/62	24.688	30.6	11.1	26.3
	6/27/62	24.947	30.1	8.5	26.9
	6/15/62	25.153	31.0	8.0	26.6
	6/21/62	25.003	29.3	6.5	23.7
	7/10/62	24.630	28.5	4.4	24.6
CO₂	2/24/65	58.810	29.2	12.6	102.1
	3/8/65	57.647	28.8	10.1	104.2
	2/10/65	55.350	29.9	9.1	98.4
	2/22/65	59.611	28.5	7.6	103.1
	3/11/65	58.542	28.9	5.3	100.0

Table II lists the results for the various gases. We note the following: (1) comparison of the results of similar runs with the same gas shows that our reproducibility is of the order of ±5%; (2) permeability values do not appear to be a function of the mean pressure; (3) there is no significant change in the value of P after an elapse of 7 months, as shown by the results for the helium gas; (4) permeability values are lower for system III than for system I.

Table III shows the variation of P with ΔT. We see that P appears to be independent of ΔT.

TABLE IV

Gas	Present study		Bearman[4]		Denbigh and Raumann[3]		van Amerongen[6]		Norton[8]	
	T_{mean}, °C.	$P \times 10^8$, cm.^{2}/ sec.-atm.	T_{mean}, °C.	$P \times 10^8$ cm.^{2}/sec.-atm.	T_{mean}, °C.	$P \times 10^8$, cm.^{2}/ sec.-atm.	T_{mean}, °C.	$P \times 10^8$, cm.^{2}/ sec.-atm.	T_{mean}, °C.	$P \times 10^8$, cm.^{2}/ sec.-atm.
He	31.0°	27.0	34	34			35	33	25	17
							25	23		
Ne	31.0°	11.3	34.5	9						
Ar	30.7	17.5	34.5	22						
Kr	30.5	19.3	34.5	48						
Xe	30.5	62.5	34.5	73.5						
CO_2	29.8	100.8			30.9	216	35	145	25	33
					30.9	175	25	102		

Fig. 3. Plot of P as a function of mean temperature: (B) data of Bearman;[4] (V) data of van Amerongen;[6] (N) data of Norton;[8] ($\times$) present study.

Table IV compares our results of systems I and III with those of other workers.[3,4,6,8]   Our results are, on the whole, lower than those of Bearman[4] but a minimum in the permeability of the rare gases is also observed by us. Our results for CO_2 are considerably lower than those of Denbigh and Raumann[3] but agree within $\pm 10\%$ with those of van Amerongen.[6]   The reproducibility of the measurements of Denbigh and Raumann is about $\pm 8\%$, while our reproducibility for carbon dioxide is better than $\pm 4\%$.

Our results for He also agree within $\pm 10\%$ with those of van Amerongen.[6] In Figure 3 the P values for helium are plotted as a function of mean temperature.   We see that Bearman's value[4] is high and Norton's value[8] is

low, while van Amerongen's values[6] lie almost within the scatter of our measurements.

ERRORS

We have studied quite extensively the various sources of error in the experiment, and will summarize our results with a few comments, mostly qualitative in nature.   The most important errors are associated with the measurements of the membrane temperature and of the membrane area and also with the disturbing side effects of degassing, adsorption, and leakage.

Temperature

The permeability varies with temperature.   In the present apparatus, the temperature is, of course, nonuniform, and, at first sight, it is not obvious at what temperature the permeability has been evaluated.   We have, however, presented evidence in Table III that, at a constant arithmetic mean temperature, the calculated permeability is independent of temperature difference within experimental error.   Since, at zero temperature difference, the temperature of the system is uniform, it is evident that our permeability values apply to the arithmetic mean temperature.   In our experiments, we measured this temperature with an accuracy of about $\pm 0.15°C$.   The error arises both from temperature fluctuations during the experiment and from variability in temperature over each face of the membrane.

Area of the Membrane

In the theory of the experiment, it is assumed that the gas has free access to the membrane over the area A in contact with the porous bronze plates and no access at all elsewhere.   In practice, this assumption is not entirely valid in our membrane assembly (Fig. 4).   There is a solid bronze rim 0.125 in. wide between the porous bronze and the raised rim used for pinching the membrane.   Since our membrane is placed in direct contact with the solid bronze rim, the part of the membrane in contact with it can also serve as a pathway for gas to diffuse through the membrane. Since the gas does not have entirely free access to the solid bronze rim, the rate at which gas diffuses through this part of the membrane must be less than the rate through the remainder of the membrane which is in contact with the porous bronze.   The effective A is therefore greater than the area of the porous bronze and less than that of the outer edge of the solid bronze rim.   We have chosen, rather arbitrarily, the diameter of the porous bronze plus half the width of the solid bronze rim as our effective diameter in calculating A.   If we take the area of the porous bronze for A, our P will be larger by a factor of 1.148.   If we increase our He values by this factor, we find that our values are slightly higher than those of van Amerongen while our present values are slightly lower than those of van

Figure 4.

Amerongen. This leads us to believe that the best choice of A is probably between our present value and the area of the porous bronze.

Bearman[7] used the diameter of the porous bronze in calculating P. Table V shows the comparison of our results with his if we change his effective A to correspond to our definition. We see that, except for krypton, the results compare quite well if we use the comparable A for the area of the membrane.

TABLE V

	$P \times 10^8$, cm.^{2}/sec.-atm.	
Gas	Present study	Bearman's (corrected)
He	27.3	29.6
Ne	11.3	7.8
Ar	17.5	19.2
Kr	29.5	41.8
Xe	62.5	64.0

Degassing

When we add a new gas into the system, the old gas in the system will degas independently and will contaminate the new gas. From a series of auxiliary experiments, we found that after pumping the system for 60

hr., degassing causes the pressure of the cell to increase a maximum 0.6 mm. of Hg during a 12-hr. period.   If we are working at 25 cm. Hg, we will then have a partial pressure of contaminant of only 0.3% in a twelve hour period.   The contribution to pressure differences arising from the thermo-osmosis of the impurities introduced will be of the order of 0.01 mm. Hg, which is well within the limits of error of our pressure measurements.

Adsorption

The process of adsorption is very prominent when a gas is first introduced into an evacuated system.   We found that in 20 hr. each half of the system can adsorb enough gas to cause the pressure to drop from 74 cm. to 72 cm. Hg at room temperature.   Furthermore, about half of the gas is adsorbed within $2^{1}/_{2}$ hr. after its introduction into the system.   Therefore, in order to avoid effects of adsorption, we prefer to start our runs after a gas has been in the system for several (2–5) hours.   We do not like to wait too long, for the longer we wait the more impurities will leak into the system.   Since our permeability data seem to be independent of the waiting period of 2–5 hr., we do not believe a longer waiting period is necessary.

Leakage

Leakage can be an important source of error.   Gases may leak into the system by diffusion through the O-ring used to seal the system and thence by lateral diffusion into the rubber membrane.   Gases may leak out of the system by the reverse process.   In addition, gas may leak around the membrane if it is not sufficiently pinched.   It is difficult to select an O-ring and a membrane so that the assembly is vacuum tight while the membrane is pinched sufficiently to prevent gas from flowing around the edges of the membrane.   However, with some care, it is possible to simultaneously seal the system and pinch the membrane.

Since the O-ring itself has a finite permeability to all gases, it does not prevent long-term leakage even if it does seal the system.   After numerous experiments, we concluded that if the O-ring material is properly selected, the effects of leakage are small.   Thus, we found that butyl rubber O-ring are quite adequate for all of the gases studied, while the Teflon O-rings in System II uniformly led to erratic results.

CONCLUSIONS

The method of thermo-osmosis has the advantage of finding simultaneously two quantities of interest, namely, the heat of transport and the permeability coefficient.

With proper care in the choice of O-ring, we can measure permeability thermo-osmotically with an accuracy equal to that obtained by other methods.   The irreproducibility of about $\pm 5\%$ under even the best of conditions is probably due chiefly to the combined effects of the residual leakage, degassing, and adsorption.   We are inclined to believe that these are also important sources of error in other techniques.

Our results for the permeabilities of the rare gases should supercede those obtained by Bearman.[4,9]

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Résumé

Par l'emploi d'un transducteur de pression à jauge de contrainte, on a construit un appareil perfectionné à volume constant permettant la mesure des perméabilités isothermes de gaz au départ d'expériences thermo-osmotiques. Les perméabilités sont reproductibles avec une précision d'environ 5% et peuvent être comparées favorablement avec celles obtenues par d'autres méthodes. Des valeurs corrigées sont présentées pour des coefficient de perméabilité de gaz rares au sein du caoutchouc.

Zusammefassung

Unter Verwendung von Dehnungsmesser-Druck-Umwandlern wurde eine verbesserte Apparatur zur Messung der isothermen Permeabilität von Gasen bei konstantem Volumen aus Thermo-Osmoseversuchen konstruiert. Die Permeabilität ist mit einer Genauigkeit von etwa ±5% reproduzierbar und stimmt gut mit den nach anderen Verfahren erhaltenen Werten überein. Revidierte Werte für den Permeabilitätskoeffizienten von Edelgasen in Kautschuk werden angegeben.