6-30-1957

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Recommended Citation

Lamborn, Richard Gordon and Vogel, Carl Henry, "Measurement of the specific surface of perlite" (1957). *Theses*. 2531.
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MEASUREMENT OF THE SPECIFIC SURFACE OF PERLITE

THESIS

SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENT FOR THE
DEGREE OF
MASTER OF SCIENCE IN CHEMICAL ENGINEERING
AT THE
NEWARK COLLEGE OF ENGINEERING
BY
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AND
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MAY, 1957

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Approval of Thesis

For

Department of Chemical Engineering

Newark College of Engineering

By

Faculty Committee

Approved ______________________

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Newark, New Jersey
June, 1957

The authors express their sincere appreciation to Dr. Charles L. Mantell for assuming the responsibility of Thesis Advisor and guiding this work from inception to completion and to Perma-Rock Products, Inc., of Baltimore, Maryland for the financial assistance without which this work could not have been done.

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Abstract

A low temperature gas adsorption apparatus was designed for the purpose of measuring the total surface area of solids using the well known B.E.T. method.

The apparatus was designed so that several different gases could be used as adsorbates without evacuating the gas reservoirs each time a different adsorbate was to be used. This was accomplished by providing three gas reservoirs of sufficient capacity to allow several determinations to be made at moderate initial reservoir pressures of from two to three pounds per square inch. A separate bulb and manometer system was supplied for determining the saturation pressures of adsorbates used. Although the primary objective was to determine the surface area of expanded perlite, the gas burette volumes of the apparatus were designed large enough to permit the evaluation of surface areas over a wide range of values.

Several samples with known areas were checked using N_2 as the adsorbate to establish confidence factors and the values for the surface areas of several perlite samples are reported.

Statement of Problem

The purpose of the work undertaken in this report was to determine values for the surface area of Perlite.

Various methods of measurement were to be evaluated, and the best possible one was to be adopted, with the thought in mind not only of obtaining accurate results on Perlite, but with the possibility of applying this method of surface area measurement to other adsorbents.

The method of measurement chosen is to be described in detail as to design and operation.

Introduction

The importance of being able to measure accurately the surface areas of solids has long been recognized. Many methods have been devised and used for specific materials. The great progress made in the field of Catalysis would not have been possible were it not for the development of techniques which made the accurate measurement of catalysts and catalyst support areas possible. These measurements make it possible to design catalysts to perform in specific reactions. Therefore, manufacturing processes built around catalysts can be controlled with a greater degree of certainty. Knowledge of surface areas also aid in the study of molecular structures, reaction mechanisms, and reaction kinetics.

Much theory has been developed in the fields of adsorption and chemisorption in the past few years. These new developments are based on facts uncovered through knowledge of surface area determinations and accurate definitions of specific surface. The specific surface of porous adsorbents and catalyzers is one of their most important properties. A knowledge of the specific surface is necessary for calculating the amount and character of sorptions, surface energies, heat of adsorption, and for reaching basic conclusions regarding the character of a sorption process.

I. General Survey

A physical examination of expanded perlite revealed that this substance was of a friable and glassy nature. The particles viewed under a Leitz metallurgical microscope appeared to be hollow spheres and segments of spherical surfaces. Some particles had indentures or blow holes which perhaps developed when the raw perlite was popped or expanded. These indentures or pores do not have the regular pore diameter and orientation that is associated with the porous solids or adsorbents.

An analysis of perlite ore is given below. This is a typical analysis of an ore mined at the John-Claire Mine in Rosita, Colorado. (1)

Table I

SiO_2	69.79%
Al_2O_3	14.72%
Fe_2O_3	2.07%
MgO	1.08%
CaO	1.49%
Na_2O	2.75%
K_2O	3.98%
H_2O	4.0%
MnO_2	trace
SO_3	trace
Cl_2	trace

Surface areas can be roughly approximated by establishing a shape or surface factor which would enable one to calculate the specific surface when the characteristic dimension had been measured. Sieving, elutriation, and sedimentation methods have been developed to accomplish this. It was felt that because of the friable nature of perlite and the irregular shapes of the particles these methods would not yield accurate or reproducible results.

The electron microscope, photometric or light extinction techniques based on absorption of light by solid particles dispersed uniformly in a fluid medium would be of dubious value when employed to measure the surface of a translucent material such as perlite. Pidgeon and Dodd (2) measured the area of quartz powder by the microscopic and gas adsorption method. The areas calculated by gas adsorption techniques were consistently higher than those obtained by the electron microscope. This was perhaps due to the ability of the gas to penetrate cracks and fissures that were overlooked in the microscopic study. Pidgeon and his co-worker Dodd also reported lower areas were obtained by H_2O permeability methods. This seems to indicate the cracks and fissures were inaccessible to the flowing fluid. Therefore, the buoyant, pock-marked surface and spherical shells of perlite would not lend themselves to permeability, fluid flow, or microscopic method of surface measurement.

The result of heat of wetting, adsorption from solution, gas adsorption, and electron microscope surface area measurements for ZnO_2 pigments are reported by Ewing and Rhoda (3). They reported that the electron microscope method yielded inconsistent results due to the uncertainty of observing the edges of the crystal or optical illusion attributable to refraction of light and poor resolution power of the microscope. A better correlation of surface areas of ZnO_2 was obtained when gas adsorption and adsorption from solution techniques were employed. Daxad #11, a surface wetting agent of the alkyl-aryl type was employed, and the amount adsorbed from solution was measured with an interferometer. The irregular shapes of the perlite particles, "non-wettable" pores and poor adsorption properties indicated that adsorption from solution would not be a feasible method for surface area measurement.

Literature Survey

The method of Brunauer, Emmett, and Teller (4) for the measurement of surface area using gas adsorption has become well known and widely used. They postulated the theory that the adsorption of a gas on an adsorbent causes a film of adsorbed molecules to be deposited on the surface of the adsorbent. This film is multimolecular in character. Plotting an adsorption isotherm of the volume of gas adsorbed V_{ads} vs relative pressure P_r/P_o, where P_r is the pressure in mm of Hg at which V_{ads} was deposited, yields a typical "S" shaped curve. The deposition or adsorption of a unimolecular layer of gas molecules is said to be complete at the first pronounced change in the slope of the curve. This point of change in slope was called point "B". From the value of V_m at this point, the number N of gas molecules can then be calculated. The area of a gas molecule is found from equation (1).

$$(1) \quad A = 4(0.866)\left[\frac{M}{4\sqrt{2}Ad}\right]^{2/3}$$

where M is the molecular weight of the gas used, A is Avogadro's number (6.02×10^{23} molecules /g mol), and d is the density. This formula assumes two dimensional close packing of the gas molecules on the surface of the adsorbent. Therefore, the total area $\sum A = NA$.

However, in the measurement of the surface area of most adsorbents, the whole adsorption isotherm had to be plotted in order that point "B" could be ascertained with certainty. This experimental method was long and tedious.

In 1938 Brunauer and his co-workers proposed a refinement to their original method.

The equation $$\frac{P_r}{V_{ads}(P_o-P_r)} = \frac{1}{V_m C} + \frac{C-1}{V_m C}\frac{P_r}{P_o}$$

where P_r = pressure at which gas is adsorbed

P_o = saturation pressure of the gas

V_m = ml. gas required for monolayer

V_{ads} = ml. gas adsorbed at $\frac{P}{P_o}$

C = constant related to heat of liquefaction

$\frac{P_r}{P_o}$ = 0.05 - 0.35 for important B point range

is the equation of the isotherm of the multimolecular adsorption theory for adsorption taking place on a free surface. It is a linear equation, i.e., the plot of the function $\frac{P_r}{V_{ads}(P_o-P_r)}$ vs. $\frac{P_r}{P_o}$ gives a straight line if the theory is obeyed.

The intercept of the straight line is $\frac{1}{V_m C}$ and the slope is $\frac{C-1}{V_m C}$. Thus, one can obtain the two constants V_m and C from the experimental data. Having found V_m, the area can easily be calculated. (5)

Advantages and limitations of the surface area determination by gas adsorption are:

1. The experimental technique is simple. If the gas used is inert, such as nitrogen or argon, the adsorbate surface area is unchanged and the adsorbate can be recovered intact.
2. The method is accurate. Comparison with results obtained with visual methods shows relative surface areas agreeing within 10%.
3. The method is universal. It can be applied to any porous or finely divided adsorbent. (6)
4. The gas adsorption method is limited to finely divided substances. It cannot measure the surface of coarse particles accurately. This is due to the inaccuracy in measuring the dead space and large dead space corrections must be applied to small adsorption values.
5. In order to get the true surface of an adsorbent by gas adsorption, one should use the smallest gas molecules. A and N_2 molecules are larger than He, Ne, and H_2, but the former are more inert. No chemisorption complicates the determination of their van der Waals adsorption isotherms. If the adsorbent has exceedingly fine pores, N_2 gives erroneous results. Emmett (7) found that 50% dehydrated chabasite adsorbs

H_2 at 77° K, but practically no N_2 entered the pores at that temperature.

From surface area measurements, the average particle size may be determined.

$S = \frac{6}{\rho_s d}$ where S = specific surface

ρ_s = density of adsorbent

d = average particle diameter

In 1944 Harkins and Jura (8) pointed out that the mean area occupied by the gas molecules used as adsorbates was not known accurately. Some areas reported for the N_2 molecule are 13.8 and 16.2 sq. Å per molecule at -198.5°C. Harkins and Jura developed a method by which the area of the adsorbent could be measured by vapor adsorption without the assumption of a molecular area for the adsorbate.

$$\log \frac{P_r}{P_o} = B - \frac{A}{v^2}$$

A plot of $\log \frac{P_r}{P_o}$ vs. $\frac{1}{v^2}$ yields a straight line over a considerable range of the plot. The slope increases as the surface area increases. The area then is given by the equation $\sum A = kS^{\frac{1}{2}}$ where S is the slope of the straight line and k is a constant for a given adsorbed vapor at a given temperature. The value of k for N_2 is 4.06 at -195.8°C.

This above theory is based on monomolecular adsorption in the vapor phase. If the vapor phase condensed film is not present, the temperature of the isotherm must be lowered until this two dimensional phase exists.

Below in table II are values obtained by Harkins and Jura on several metallic oxides using their proposed method and the BET modified procedure. (8)

TABLE II

Sample	H & J	BET
TiO_2	13.8 m^2/g	13.9 m^2/g
$ZrSiO_4$	2.9	2.8
SiO_2	3.2	3.2
Al_2O_3	9.6	9.5

Livingston (9) states that a Harkins and Jura plot gives a straight line at from 0.2 - 0.4 relative pressures.

In 1947 Zettlemoyer and Walker (10) measured the surface areas of activated magnesias using N_2 adsorption isotherms. They employed both the BET and H&J methods using 15.25 sq. Å for N_2 area on porous solids recommended by H&J. Activated magnesias yielded type II and IV isotherms. The electron photomicrographs showed the XP magnesia to have a surface of porous plates.

TABLE III

Surface Area of Magnesia m^2/g (10)

	Sample	B-point	BET	H&J
Experimental	XP	198	230	209
	2642	142	154	140
-8 + 28 mesh granular	2652	123	125	109
	2641	74	71	81.5
Burned	2661	0.73	0.79	0.74

Gabriel and Cooley used a homologous series of aliphatic acids on a non-ionic adsorbent.

Blocker, Craig, and Orr (11) developed a steady gas flow method for surface determination. The areas obtained in twenty minute runs were in good agreement with values obtained by the conventional BET method. However, poor agreement was found with substances which were "slow" adsorbers.

Emmett (12) found in a critical review of the method of Harkins and Jura that the value of k in the equation $\text{Area} = k\ S^{\frac{1}{2}}$, obtained by heat of wetting or immersion in water, was questionable. He states that the agreement of values for surface areas obtained by the Harkins and Jura method on TiO_2 and the BET values was only fortuitous.

Askey and Feachem (13) reported as early as 1938 that the extrapolation of an "S" shaped isotherm to zero pressure or $\frac{P_r}{P_o} = 0$ yielded an intercept point V_m which gave values

10 - 30 per cent smaller than those obtained by the BET point "B" method.

One objection to the use of the BET method on porous media is found repeatedly throughout the literature. A correct assumption must be made of the molecular area of the adsorbate molecule in order to convert the volume of gas adsorbed at the completion of the monolayer into total surface area. The area of the N_2 molecule, used almost universally in a BET determination, has been reported as varying from 13.6 to 16.9 $\AA^2$ with peaks at 14.05, 15.25, and 16.05 $\AA^2$.

Additional references consulted in this literature search together with short abstracts can be found in the appendix of this report.

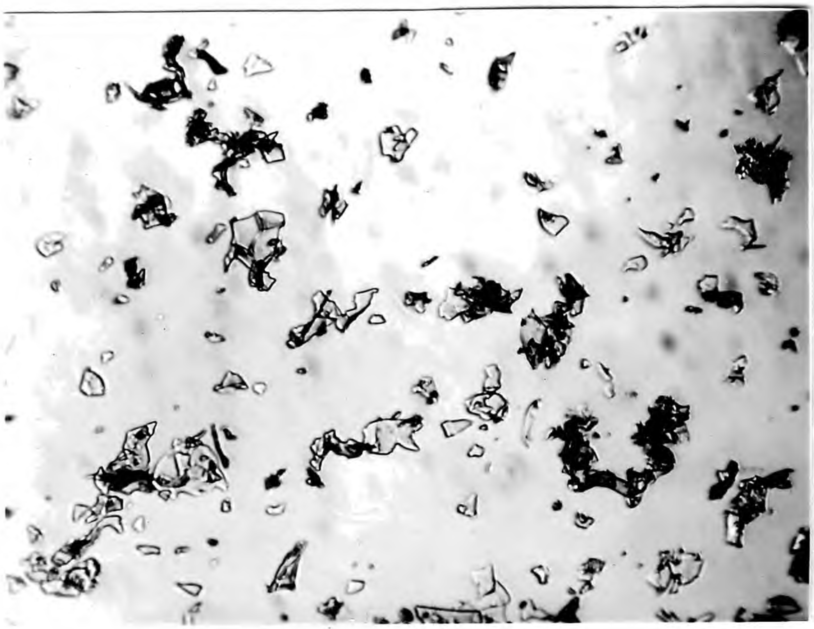
II. Experimental Program

A study of the various known methods of surface area determination shows that surface areas are obtainable directly only by the BET and Permeability methods. In all other methods, areas are obtained using other parameters. It would appear that the BET gas adsorption technique is the most convenient and accurate method yet devised. The other methods require a greater number of experimental points and more tedious calculations without producing more significant or reliable area values than can be obtained by the BET or point "B" methods.

The above reasons, substantiated by the physico-chemical nature of perlite and the inherent problems that would be encountered using the known methods of surface area determination as listed in section I, indicated that gas adsorption techniques would be a more logical approach to the problem.

Therefore, it was decided to use the BET low temperature gas adsorption technique and to design apparatus that could be used for a variety of materials and with several different gases.

The samples of perlite furnished by the Perma-Rock Corporation were examined under a Leitz metallurgical microscope. It was felt that #100 and #300 filter aids would



Perlite--#300 Filter Aid

Magnification 80X

have surface area values in the same order of magnitude and that the spherical surfaces would afford dead space values within the limits of the equipment. Dead space values are discussed elsewhere in this report.

Samples of Celite filteraid, Darco S-51 decolorizing carbon, and Silex sand were obtained. The Johns-Manville filter aids and the Silex sand had reported surface areas measured at the Johns-Manville research center using the BET method with N_2 as the adsorbate.

All samples were degassed at 110°C in an oven at atmospheric pressure for 24 hours and for one hour at 26"Hg vacuum prior to the test. Attempts to degas the sample in the sample tube while it was attached to the high vacuum apparatus were unsuccessful. The sample tube collapsed on one occasion. Also, the heat from the adsorption bulb on sample tube was conducted to stopcock "A", causing the Apiezon grease to melt. This resulted in a leak either at the stopcock or at the ground tapered joint connecting the adsorption bulb to the system.

No other attempt was made to activate or clean the surfaces of the samples. Jack Eigler, a graduate student working at the Newark College of Engineering on the adsorption of substances on Perlite, found that chemical treatment did not increase the adsorptivity or the amounts adsorbed.

The samples and the apparatus were pumped down to a vacuum of 5.0 x 10^{-4} mm or lower prior to the dead space determination. It was felt that this vacuum was sufficient and that the number of molecules of gas left in the system at that pressure would introduce little error to the surface area determinations. Evacuation of the Helium used in the dead space determinations was done at room temperature at a pressure of less than 5.0 x 10^{-3} microns.

The sample tubes were tapped on a hard surface while being filled in order to obtain close packing and to keep the dead space volume at a minimum.

Nitrogen gas was used as the adsorbate in all runs. It was felt that N_2 gas would not chemisorb and give step shaped isotherms in the region of 0.1 to 0.3 relative pressured. N_2 is used more universally than any of the other gases in BET surface area determinations. It has been found that better correlations are obtained with results of other methods of surface area determinations with BET gas adsorption areas when N_2 gas was used. No phase change has been reported to occur for N_2 at -195°C over the relative pressures range of 0.05 to 0.2.

Liquid nitrogen, commercial grade, supplied by Hoffman Laboratories, Newark, N. J., was used for the low temperature batch, to provide isothermal conditions for the adsorption runs. Saturation pressure determinations were made at the conclusion of every run to determine the temperature of the liquid N_2 bath and corrections were applied when calculating the values of the adsorption isotherm points.

III. Theory

If one could completely cover the surface of a solid with a monolayer of gas molecules and count the molecules, it would be possible to determine the total area of the solid since the area covered by a single molecule of a gas can be determined from known physical constants of the gas.

The volume of gas which will be adsorbed on any given surface is dependent on both the temperature and pressure of the gas. If the temperature decreases, the amount of gas adsorbed decreases; conversely, if the pressure increases, adsorption of the gas increases. In determining an adsorption isotherm and the surface area from an adsorption isotherm, the temperature is kept constant and the amount of gas adsorbed is controlled and measured by changing the pressure of the gas in a closed system.

In order to minimize variables introduced by gas behavior, the temperature used for gas adsorption should be at or near the normal boiling point of the gas adsorbate.

The basic assumption upon which the BET theory is based is that several layers of gas can be adsorbed on a solid surface, that is, that adsorption is multimolecular in nature. Brunauer, Emmett, and Teller (14) developed the following formula (a) for multimolecular adsorption on a free surface:

$$V_{ads} = \frac{V_m \, P_r C}{(P_o - P_r)\left[1 + (C - 1)\,\frac{P_r}{P_o}\right]} \qquad \text{(a)}$$

where V_{ads} is the volume of gas at 0°C and 1 atmosphere pressure, P_r is the pressure, P_o is the vapor pressure of the gas at the temperature of the adsorbent, C is a constant related to the heat of adsorption and V_m is the volume of gas (stp) necessary to form a single layer of molecules over the entire area. The development of the Langmuir equation and the BET modification is covered with great detail in the reference mentioned above.

The quantities P_r, P_o, and V_{ads} are determined experimentally and V_m and C can be obtained by rearranging the above equation into a linear form and plotting $\frac{P_r}{V_{ads}(P_o-P)}$ vs $\frac{P_r}{P_o}$

$$\frac{P_r}{V_{ads}(P_o-P_r)} = \frac{1}{V_mC} + \frac{C-1}{V_mC}\left(\frac{P_r}{P_o}\right) \qquad \text{(b)}$$

$$\text{therefore the slope } S = \frac{C-1}{V_mC} \qquad \text{(c)}$$

$$\text{and the intercept } I = \frac{1}{V_mC} \qquad \text{(d)}$$

For most isotherm data plotted in this manner, a straight line is obtained for only the lower portion of the isotherm between relative pressures $\frac{P_r}{P_o}$ of 0.05 to 0.30. The equations (c) and (d) can be solved for V_m and C. $\therefore V_m = \frac{1}{S+I}$ and $C = \frac{S}{I} + 1$.

V_m can therefore be calculated from the slope and intercept of the straight line portion of the curve. Once V_m is known, the total area is easily obtained.

$$\text{Area } (m^2) = 4.38\ V_m \text{ (ml stp)}$$

The constant 4.38 was derived by condensing the conversion factors into one number. If the average area covered by an N_2 gas molecule at -195°C is 16.25 Å^2 per molecule, assuming close packing, (1) one ml. of gas at standard conditions would cover an area of 4.38 m^2.

$$\text{Area of 1 ml } N_2 \text{ gas in } m^2 =$$

$$\frac{(16.25)\ \text{Å}^2/\text{molecule}\ (6.02)\text{x}(10^{23})\ \text{molecules}\ (1)\text{ml}\ 1\text{x}10^{-20} m^2/\text{Å}^2}{22{,}400\ cm^3\ (0.99997)\ ml/cm^3}$$

$$= 4.38\ m^2/\text{ml } N_2 \text{ stp.}$$

Brunauer, Emmett and Teller (15) arrived at the following formula for the area of an adsorbed gas molecule, assuming induced dipoles in two successive layers and close packed spheres.

$$A = 4(0.866)\left[\frac{M}{4\sqrt{2}\ Ad}\right]^{2/3}$$

where 0.866 is a constant derived from geometric considerations of close two dimensional packing, M is the molecular weight, A is Avogadro's constant, and d is the density of the gas in the solid phase.

The volume of the capillary tubing from the zero on the gas burette to the stopcock A and the zero reading of the manometer is found in the following fashion. (16)

$PV = k$

$V = V_o + V_{tB}$, where V_o is the volume of the capillary tubing and V_{tB} is the volume of gas contained in the gas burettes. Substituting $V_o + V_{tB}$ for V, we get

$$PV_o + PV_{tB} = k$$

rearranging, $PV_{tB} = k - PV_o$

$$\frac{d(PV_{tB})}{dP} = -V_o$$

A plot of PV_{tB} vs P should give a straight line, the slope of which is the negative of the free volume V_o.

Discussion of Results

The surface area values obtained from samples run on the equipment and procedures outlined in this report are not consistent with the reported surface areas. The best correlation is within 9% deviation exhibited by Celite 403 with a reported area of 21.0 m^2/g. The measured area was found to be 19.1 m^2/g or 1.9 m^2/g less than the reported area.

However, the opposite situation exists with the samples of Silex sand. The measured area was larger than the reported area by a considerable amount. The samples used as standards had no certification of correctness. It is not known whether we duplicated the same desorption, activation, drying, or degree of vacuum prior to the adsorption runs as was used in the reported surface area determinations.

It is generally felt throughout the field of surface area measurement by gas adsorption that an agreement within 6 - 8 m^2/g on nonporous media is rather good. All of the determinations made on our apparatus fall well within this range.

The anomalous results obtained for 100 and 300 Perlite filter aid cannot be as readily explained. These screened fractions were not classified by the experimentors. The photomicrographs show a decided range of particle size. Therefore, one cannot conclude from the facts at our disposal, that is, the manufacturing techniques, sizing, and screening history of the Perlite samples, that 300 filter aid should necessarily be composed of smaller particles and, therefore, have a greater external or internal surface area. Considering the confidence factor established through the results of the runs on known materials, one might conclude that the areas of 2.56 and 3.16 m^2/g for 300 filter aid and 10.7 and 7.2 m^2/g for 100 filter aid are reasonably correct. Perhaps if a more fortuitous choice of slope of the curve were made on the plots of P_r/P_o vs $P_r/V_{ads}(P_o-P_r)$ the values of surface area for any particular material would agree more closely with each other.

The magnitude of that part of the experimental error resulting from the selection of sample size and sample tube size (resulting in low dead space values) was kept as small as possible in every run. If the surface by physical inspection is known to be large a small sample should be taken, however, for materials of small surface areas it is necessary to take large samples and use adsorption tubes with large dead space values. The spread of the values obtained for 100 Perlite filter aid may be partially attributed to the difference in sample sizes used. The dead space values were 58.2 and 73.1 ml. Any errors in the pressure readings taken during the runs or deviations from the perfect gas laws would show a marked error in the final results. Loebenstein and Deitz (17) have shown that the percentage error can vary from 0.1 to 10.4% depending on the sample size and dead space V_a used.

Conclusion

It was felt that the apparatus as designed and constructed worked well and that satisfactory experimental results were obtained. However, when the apparatus was used, several weak points in design and manipulation manifested themselves. These will be discussed along with proposed corrections later in this section.

Listed below are important points that should be considered in using this BET apparatus.

1. Small adsorption or small differences in adsorption will magnify errors in relatively large measured volumes. Large gas volume adsorptions and small dead space volumes are therefore desirable.
2. Diffusion of the adsorbate gas may be slow. A range of 10 - 20 minutes may have to be allowed for equilibrium to come about at any given pressure of P_r/P_o depending on what portion of the isotherm the particular point lies and the amount of gas adsorbed. This is particularly true of a substance such as Perlite. The adsorption isotherm plot of Run 13 shows inaccuracies in the points obtained below a relative pressure of 0.1, presumably due to the fact that equilibrium conditions were not attained.
3. A system of finely divided solid and adsorbate gas at

low pressure has poor heat conductivity and the heat of adsorption must be dissipated in order to maintain isothermal conditions. The liquid level of the N_2 bath surrounding the adsorption bulb should be checked frequently and the level be kept well above the adsorbent level.

4. At low pressure, P_r/P_o and low volumes of gas adsorbed, adsorption isotherm contours may be sensitive to the nature of the adsorbing surface. Step shaped isotherms may result. Perlite isotherms indicate that the tenacity or binding forces existing at low pressures are not great. This may result in gas molecules being adsorbed and desorbed simultaneously, shifting at random, resulting in unstable equilibrium conditions.

5. With reference to volumes of gas to be adsorbed and dead space values incurred, a sample of 5-10 g should have an area of at least $1m^2$. For areas $<1m^2$, use a gas for an adsorbate having so low a saturation pressure that the number of molecules left in the gas phase during measurements is of the same order of magnitude as those actually adsorbed on the surface being measured.

6. Maintain constant temperature conditions in the

laboratory housing the equipment. Sudden changes in temperature introduce errors in the volume readings.

7. Outgas or pump down the equipment to the same degree of vacuum each time prior to commencing an actual surface area determination.

8. In conjunction with items 2, 3, and 4, be sure to return the mercury level in the manometer to the zero point after each addition of gas. This zero point should be in the small capillary section of the tubing. The zero point used in this work was in the large tube just below the small capillary portion. It was felt that this increased the error in readings below $P_r/P_o=0.20$.

9. Ascertain whether the sample is sintering, decrepitating or breaking down in any manner when subjected to a high vacuum. Surface areas may change in this manner.

Results **Table IV**

Sample Designation	Run No.	B.E.T. Area Calc. m^2/g	B.E.T. Area Reported m^2/g
Darco S-51	9	1,654.	1,600-1,800
Silex Sand (C-46739)	18	18.1	12.5 ± 0.6
" " "	20	16.8	12.5 ± 0.6
Filter Cell (C-46585)	12	12.75	19.2
" " "	11	15.62	19.2
#300 Perlite Filter Aid	13	2.56	------
" " "	15	3.16	------
#100 Perlite Filter Aid	16	10.7	------
" " "	17	9.3	------
" " "	19	7.2	------
Celite 403	14	19.1	21.0 ± 1.0

The point "B" method was used to determine the area of several samples to obtain a correlation between both methods. The results are tabulated below.

Table V

Run Number	B.E.T. Pt "B" m^2/g	B.E.T. st.line plot m^2/g
9	1610	1654
12	11.8	12.75
13	3.06	2.56

DESIGN OF EQUIPMENT

The design of the particular system used in this series of experiments was adopted to a large degree from that which is described by Barr and Anhorn (5).

Certain modifications were initiated, and these will be discussed in detail later in this section.

Good and bad features were disclosed, in actually working with this apparatus, and these too will come under detailed scrutiny.

Of prime importance in constructing any equipment of this nature is a good glass blower. We were fortunate in obtaining the services of Otto Greiner Company of Newark, New Jersey to act in this capacity. With one exception, work was well laid out; and assembled in a manner which provided for ease of operation, and minimum trouble from the standpoint of leaks.

Mechanical Vacuum Pump

In choosing the mechanical vacuum pump, thought must be given to obtaining one of sufficient capacity to do the job, but not one so large as to be far beyond the range of the mercury diffusion pump delivering gases to it. An oversize pump will, of course, be as effective, and might lower the start-up time; but once the mercury diffusion pump is in operation, the mechanical pump can only remove what it receives from the diffusion pump. A large excess capacity is not justifiable.

The Kinney KC-2 pump chosen for this application has performed well under operating conditions. Time required to bring the system down to 1.0 micron pressure is usually about thirty minutes--providing there are no leaks and the system is free of moisture.

Mercury Diffusion Pump

At pressures lower than 1.0 micron, the molecules of gas remaining in the system are too widely dispersed to effect further evacuation by means of the mechanical pump alone. It is here that the mercury diffusion pump enters the picture.

In effect, the diffusion pump traps the relatively small quantity of slow moving gas molecules in mercury vapor. As this vapor is condensed, the number of molecules are concentrated and their movement accelerated so that the mechanical vacuum pump can remove them from the system.

The diffusion pump used on this particular apparatus is of a three stage glass design, fabricated by Otto Greiner Company. The ultimate vacuum obtainable, according to the manufacturer, is about 1 x 10^{-8} mm. This value was never reached during operation--due probably to small leaks which were not detectable or measurable with our equipment.

A liquid nitrogen cold trap was installed in line between the mercury diffusion pump and the rest of system to prevent the moisture vapors from entering pump.

The McLeod Vacuum Gauge

A McLeod gauge capable of reading a vacuum as low as 1.0×10^{-3} microns was constructed for this BET apparatus.

Consider the formula, $P = 10{,}000\ h \left(\frac{v}{V}\right)$, and figure (1).

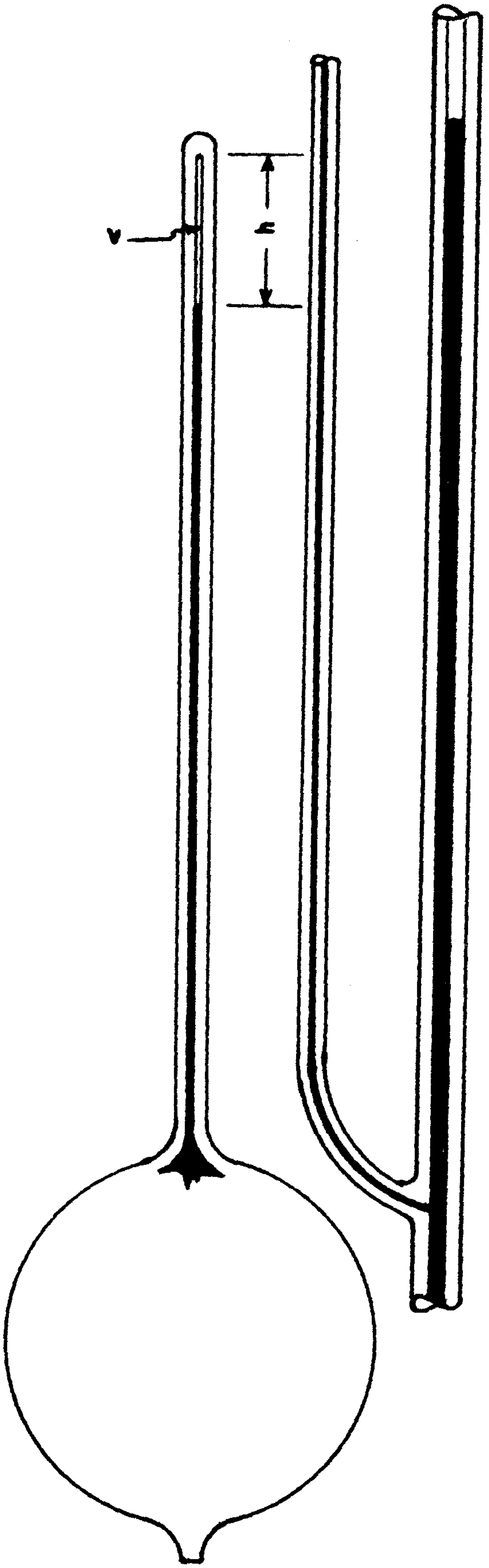
If v is the volume in ml of gas in the top of the closed capillary, V is the total volume in ml. of the bulb and capillary, and h is the difference in level in cm of mercury between the closed capillary and the side arm capillary, then P will be the pressure in microns in the system being measured.

For example, the volume of the large bulb including the capillary is 250 ml and if the volume v of the gas in the closed capillary is 0.0250 and h is 1 cm,

$$P = 10{,}000\ (1)\ \frac{0.0250}{250}$$

$P = 1.0$ micron or 1.0×10^{-3} mm mercury and the pressure will be 1.0 microns for each cm difference in level.

The constants 10,000/250 can be divided and a simple formula evolved to facilitate rapid pressure readings. Thus, $P = h(v)40$ is the formula to be used for the McLeod gauge on this apparatus.



McLeod Gauge

Figure 1

Vacuum Reservoirs

In place of the vacuum reservoir tanks used quite frequently in maintaining control of the various mercury columns, it was decided to make use of a second mechanical vacuum pump. Vacuum reservoir tanks must be quite large to be effective. (On the order of 20 to 25 liter capacity.) This would mean a considerable amount of extra space since two such tanks would be required. In addition, these tanks have to be pumped down periodically. Their only advantage lies in the vacuum reserve available in case of electrical power failure.

The vacuum pump used in this instance was a small Cenco pump loaned to us by Metals Disintegrating Company, of Elizabeth, New Jersey.

Gas Reservoirs

Containers must be incorporated in the apparatus for storage of gases used in making the runs.

These are installed in such a manner as to permit being completely evacuated prior to admitting gases, and are equipped with small indicating manometers to check relative gas pressure.

It was decided to install three such units each of one liter capacity in the apparatus to permit storage of a wider variety of gases, and to make the apparatus more flexible in its use.

Mercury Seals

If the need should arise for storing gas in the gas reservoirs at pressures lower than atmospheric, mercury seals are provided. These insure no contamination of the gases by air (due to leaky stopcock) when operating the reservoirs at the lower pressures. When operating at above atmospheric pressure with Nitrogen or Helium, it is not essential to operate these seals since any leakage will be from the inside.

When operating the cut-offs in the open position, the mercury level is maintained just below the end of the tube from the gas reservoir. In the closed position, the mercury is raised to the upper stopcock which is then closed. To open again the mercury is lowered to the original level. In the closed position there is no possibility of gas coming in contact with stopcock to vacuum section of system, and no chance of air leaking into the gas reservoir.

Gas Burettes

Basically these are composed of a series of calibrated bulbs connected by capillary tubing with engraved reference marks.

The purpose is to measure the volume of adsorbate gas before admitting it to adsorption bulb. With this in mind, it was decided to install two such burettes to insure a wide range of gas volumes (from 8.125 ml to 259.125 ml).

Since volume changes with temperature, these burettes were water jacketed, and provision was made for a thermometer well for temperature measurements. To eliminate temperature fluctuations such as were encountered during the runs reported, a constant temperature bath should be installed.

Manometers

Manometers were constructed of 10 mm glass tubing to lessen error due to meniscus effects of the mercury. Mercury level must be adjusted to zero point on the pressure leg of manometer to maintain a constant volume in the system. To avoid errors due to capillary effects the zero point should have the same diameter as opposite leg; and to maintain a minimum dead space in system, zero point should be established as close to capillary tube as possible.

In order to avoid errors in reading the manometer due to parallax, the use of scales engraved on a mirror is of definite advantage.

Adsorption Bulb

Since size of sample will vary with type of material being evaluated, no set design can be used.

In some systems the adsorption bulb is filled, and then sealed to apparatus. When run is over, tubing must be broken to remove adsorption bulb. This necessitates a fair amount of skill in glass blowing to insure a leak-free joint.

In order to avoid this situation, it was decided to construct the adsorption bulbs with a ground glass connector for easy connection and removal from system.

Gas Purification Bulbs

This is a series of bulbs of about 250 ml capacity each. In order of use they contain copper wire, glass beads, and activated charcoal. Purpose is the removal of oxygen and moisture from the gases used in making a run. These gas purification bulbs should be considered as a refinement to the basic system. Unless very critical determinations are in order, the authors do not believe the use of these bulbs to be essential.

Original installation prevented use of heater on the bulb containing the copper wire, and use of liquid nitrogen baths on bulbs containing glass beads and charcoal. This condition should be corrected by wider spacing of these bulbs if it is desired to use them in the future.

Tubing

Capillary tubing used in adsorption section of system has a 2.0 mm bore. Tubing of 10.0 mm bore was used throughout rest of apparatus with the exception of the high vacuum header. Tubing of 25.0 mm bore was selected for high vacuum header to insure minimum resistance of flow of the gas molecules being removed by the vacuum pumps.

Stopcocks

Both two way and three way stopcocks are of tapered construction. Two way stopcocks have diagonal bore to minimize possibility of leakage. If it is necessary to regrind stopcocks, a dispersion of fine alundum in water works quite well. This can be applied with a piece of soft cotton. Authors recommend use of Apiezon N grease for all stopcocks. This grease is manufactured by Metropolitan-Vickers Electrical Co. Ltd. of England and is quite resistant to all gases normally encountered in the equipment.

DESCRIPTION OF EQUIPMENT

Mechanical Vacuum Pump (D)

Manufacturer - Kinney Manufacturing Division
The New York Air Brake Company

Model---------- KC-2

Type----------- Compound

Ultimate Pressure------- 0.2 Micron, McLeod Gauge

Free Air Displacement-- 2.0 CFM

RPM-------------------- 755

Motor H.P.------------- ¼ (110 volt)

Motor RPM-------------- 1800

Oil Capacity----------- 6 oz.

Cooling---------------- Air

Shaft Diameter--------- 3/4 inch

Inlet Connection------- 3/4 inch Screwed

Valve Type------------- Feather

Net Weight, Complete--- 70 pounds

Mercury Diffusion Pump (F)

Manufacturer - Otto Greiner Company

Type----------- Three Stage

Ultimate Pressure------- 1 x 10^{-8} mm

Mercury Capacity------- 4 pounds

Heating---------------- Resistance wound coil controlled
by rheostat

Cooling---------------- Water

McLeod Gauge (H)

Manufacturer - Otto Greiner Company

Bulb Volume - 250.0 cc Mercury

Ultimate Reading - 10^{-8} microns

Accuracy - 1×10^{-8} microns

Gas Purification Trap (J)

Contents - Reduced copper wire

Purpose - Operated at about 350 degrees Centigrade to remove all but approximately 0.05% of the oxygen from gases being used in the system. Oxidized copper wire can be regenerated by passing Hydrogen gas through trap.

Overflow Bubbler (K)

Contents - Mercury

Purpose - Regulates maximum pressure at which gas may be stored in the gas reservoir (N).

Gas Purification Trap (L)

Contents - Glass Beads

Purpose - Operated in a bath of liquid nitrogen to remove moisture from gases used in system.

Gas Purification Trap (M)

Contents - Activated charcoal

Purpose - Operated in a bath of liquid nitrogen to remove all but the inert gases from Helium.

Gas Reservoirs (N)

Capacity - 1 liter

Purpose - Storage of purified gases for subsequent use.

Indicating Manometers (O)

Purpose - To indicate pressure of gases stored in reservoirs.

Mercury Cutoffs (P)

Purpose - To prevent gases stored in reservoirs from dissolving in stopcock grease, and thereby contaminating system or causing leaks to occur. This system also allows storage of gases at below atmospheric pressures since there is no problem of air contamination due to a leaky stopcock.

Gas Bulb Burettes (Q)

Purpose - To measure volume of gas admitted to adsorption bulb.

Capacity - 1, 3, 5, 7, 10 ml. (Small burette).
5, 15, 25, 50, 130 ml. (Large burette).

Temperature Control - Water jacketed to provide for close temperature control. Constant temperature can be achieved by circulating water in a closed system.

Temperature Measurement - By means of thermometers accurate to 0.1 degree Centigrade.

Adsorption Bulb (R)

Purpose - To hold sample of material undergoing test.

Capacity- Various sizes, each of which can be connected to system by means of a tapered ground glass connector.

Manometer for Adsorption Bulb (S)

Purpose - To measure pressure of gas in adsorption bulb.

Capacity- To measure 1,000 mm of pressure.

Vapor Pressure Bulb (T)

Purpose - To determine vapor pressure of gas being used as an adsorbate.

Compression Bulb for Vapor Pressure (U)

Purpose - To store gas for use in determining vapor pressure.

Manometer for Vapor Pressure (V)

Purpose - To measure pressure of gas in vapor pressure bulb.

Capacity- To measure 1,000 mm of pressure.

Meter Scale (W)

Manufacturer - Scientific Glass Apparatus Company

Type - Engraved glass mirror.

General Operation

The equipment was designed to be used in the following manner.

A known weight of material (adsorbent) in a closed system is to be subjected to a vacuum sufficient to remove all adsorbed gas molecules.

A known volume of adsorbate gas is to be admitted to this closed system, and adsorbed under isothermal conditions.

The equipment was designed to permit the measurement of pressure, volume, and temperature relationships necessary to facilitate the calculations of the precise amount of adsorbate gas adsorbed on the sample.

A detailed description of the operational procedure and calculations are to be found on pages 41-56 of this report.

EQUIPMENT NOMENCLATURE

A - Stopcock to Adsorption Bulb

B - Stopcock to Gas Manifold Line

C - Stopcock on McLeod Gauge By-Pass Line

D - Mechanical Vacuum Pump

E - Oil Trap, for Mechanical Vacuum Pump

F - Mercury Diffusion Pump

G - Cold Trap, for Mercury Diffusion Pump

H - McLeod Gauge

I - Tapered Groove Stopcock

J - Gas Purification Trap

K - Overflow Bubbler

L - Gas Purification Trap

M - Gas Purification Trap

N - Gas Reservoirs (3)

O - Indicating Manometers (3)

P - Mercury Cutoffs (3)

Q - Gas Bulb Burettes with Water Jackets and Thermometer Wells (2)

R - Adsorption Bulb

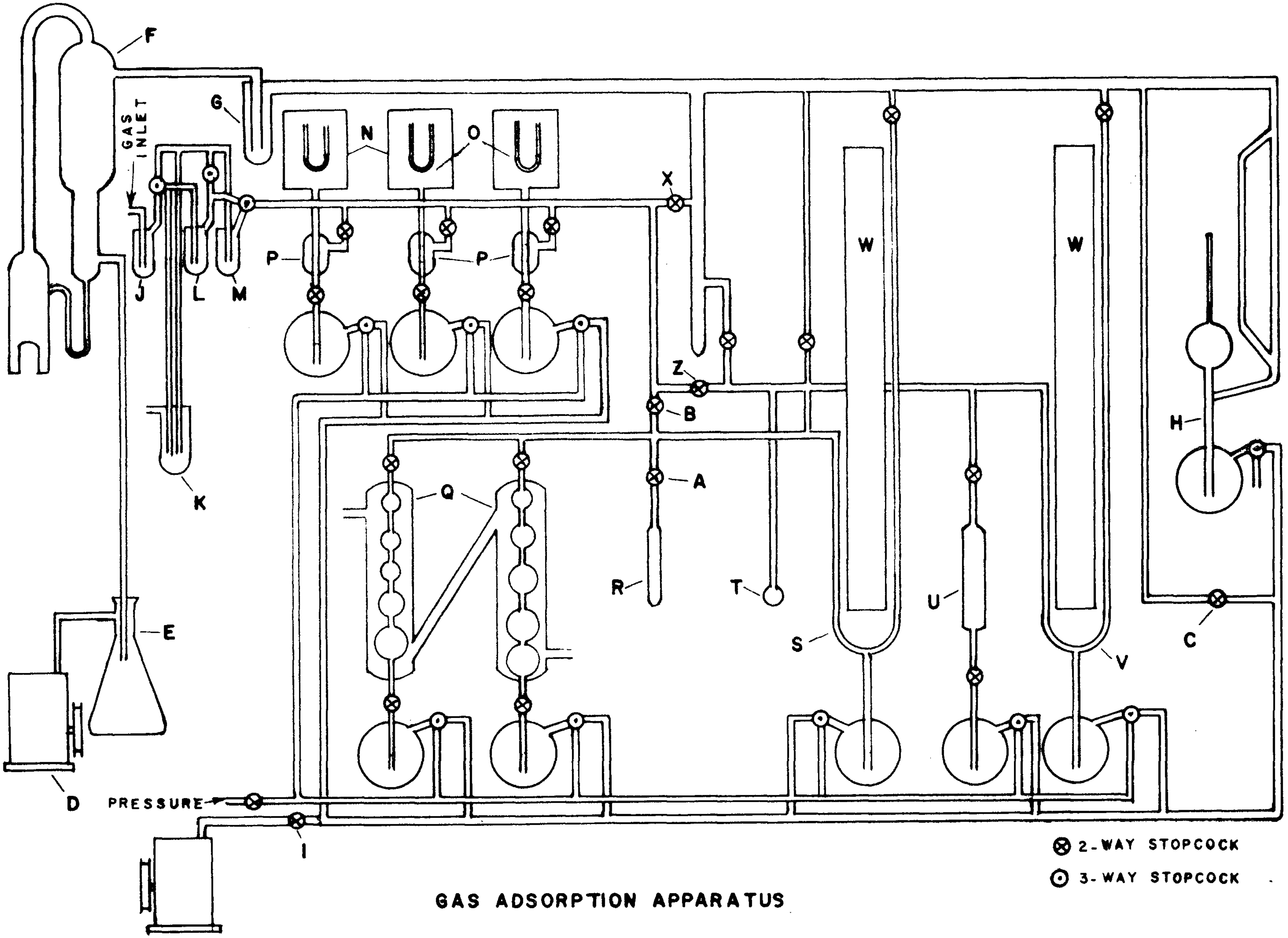
S - Manometer for Adsorption Bulb

T - Vapor Pressure Bulb

U - Compression Bulb for Vapor Pressure

V - Manometer for Vapor Pressure

W - Meter Scales (2)



GAS ADSORPTION APPARATUS

OPERATING INSTRUCTIONS

A - Start-Up Procedure - The following five steps are to be followed in starting up equipment, preparatory to making a run.

1 - Open all stopcocks to the vacuum line with the exception of stopcock "A" which leads to the sample bulb. Make particular note of opening stopcock "C" located on the by-pass line around the McLeod gauge. (This permits equalization of pressure on the mercury in the McLeod gauge and reservoirs, and prevents the mercury from bubbling.)

2 - Start mechanical vacuum pump. As soon as pump returns to normal, close stopcock "C".

3 - As soon as reading is obtainable on McLeod gauge, start mercury diffusion pump--making sure to fill cold trap on diffusion pump with liquid nitrogen, and setting rheostat at a reading of between 55 and 60.

4 - Open stopcock "A" slowly, and allow sample to evacuate.

5 - Continue evacuating until a reading of $1x10^{-4}$ to $1x10^{-6}$ mm is obtained on the McLeod gauge. Initially this may take several hours due to the presence of water vapor on the walls of the apparatus. This initial pump down time can be reduced by heating the glass walls (not the stopcocks) with a wide-spread flame from a Bunsen burner.

B - Testing for Leaks - The following procedure should be adopted to determine presence of possible leaks in the system.

1 - When optimum vacuum is reached, shut off all stopcocks to the vacuum line.

2 - Allow apparatus to remain this way for about fifteen minutes.

3 - Open stopcocks to various parts of the system, one at a time, checking the vacuum in each instance by means of the McLeod gauge. The presence of a leak will be indicated by a lower vacuum reading on the McLeod gauge.

4 - If leaks are present, they are usually found in the joints and stopcocks, and can usually be located by means of a high frequency vacuum tester (Scientific Glass Co. No. P9470), a white spark discharge indicating presence of gas ions and therefore a leak.

Leaks in joints and tube walls can be corrected by heating and refusing that particular area.

Leaks in stopcocks can be corrected by releasing vacuum and removing stopcock, which is then ground, regreased and replaced.

5 - Procedure must be repeated when any leaks are detected.

C - Filling Gas Reservoirs - In filling the gas reservoirs "N" the following steps should be observed.

1 - Flush gas through purification system (J,K,L,M) allowing it to escape through outlet on "K".

2 - Admit gas to its respective reservoir until the gauge on tank reads between 2 and 3 psig.

3 - After one gas is stored and before another one can be admitted, the gas maniford section of system must be evacuated to prevent mixing of the gases. For example, in adding Nitrogen and Helium, Nitrogen is added first in the prescribed manner and manifold section is evacuated. Helium is added last since it will be the first gas used in determining the free space of the system. Therefore, a second evacuation is not necessary.

D - Measuring Free Space - The free space or volume "Vo" of the adsorption system must be determined. This is the volume of the space between the stopcock "A" just above the sample tube, the zero point of the gas burette, and the zero point of the manometer. This particular value is determined only once for any given system; since, unless breakage occurs, will remain constant.

1 - With system evacuated, stopcock "A" closed, mercury at zero point in manometer and mercury at zero point in gas burette; gas (Helium or Nitrogen) is admitted through stopcock "B" to give a pressure reading on manometer of between 700 and 800 mm.

2 - Stopcock "B" to gas maniford is then closed and the pressure determined with mercury at zero point in gas burette.

3 - Mercury is lowered to the first reference point in burette and pressure again measured.

4 - This procedure is repeated at each reference point until gas burette has been emptied of mercury.

5 - This method provides a series of pressure readings at known burette volumes (Vtb); and assuming run was made at constant temperature, and with a constant amount of gas the following relationship holds true:

$$PV = \text{constant} = K$$

$$V = Vo + Vtb$$

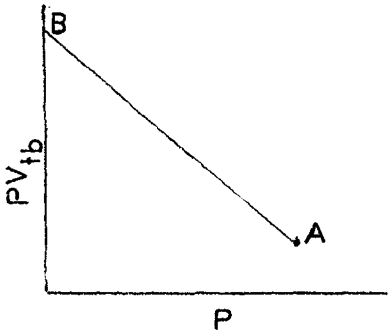
$$PVo + PVtb = K$$

$$PVtb = K - PVo$$

$$\frac{d(PVtb)}{dP} = -Vo$$

6 - A plot of PVtb versus P for each point should produce a straight line whose slope is the negative value of the free volume (Vo).

It follows that $\frac{A - B}{P} = -Vo$ or $\frac{B - A}{P} = Vo$ in ml.



E - Determination of Bulb Factors (f_v) - It is convenient at this point to construct a table of bulb factors (f_v) which are an aid in simplifying later calculations of volumes of nitrogen at standard conditions. The actual table appears on page 57 of this report, but the manner in which it is determined will be discussed here.

$$f_v = (Vtb + Vo)\frac{(273.2)}{(760)}$$

where:

Vtb is the measured volume of each bulb or combination of bulbs in the gas burette.

and:

Vo is the free space in the adsorption system as determined in section D and which remains constant.

Therefore:

In determining the actual volume of gas (Vt) at standard conditions from the measured volumes Vtb and Vo and measured pressure (P) and temperature (T, ^{o}K); the following relationship may be used.

$$Vt\ (N_2),\ stp = \frac{f_v \times P}{T}$$

F - Measurement of Free Volume in Sample Tube (Va) - Since this determination is made at liquid nitrogen temperature, a gas must be used which will not adsorb on the sample to be tested. Such a gas is helium.

1 - A large sample of helium is introduced into the burette system with stopcock "A" remaining closed. (Normally use the total volume of both burettes.)

2 - The pressure is measured (P) and the volume of helium is corrected to standard conditions (Vt_{He}).

3 - Place liquid nitrogen bath around sample tube, and allow it to come to temperature--about five minutes is usually sufficient. Make sure level of liquid nitrogen remains constant during determination.

4 - Open stopcock "A" and allow helium to enter sample tube. Take pressure reading (P_1) and record burette temperature (t, °C).

5 - Close stopcock "A". Open stopcock "B" and "X" to evacuate system. Then slowly open stopcock "A" and evacuate helium from sample tube, and remove nitrogen bath.

6 - This determination must be made for each run since the size of sample tube and sample contained will vary.

G - Making a Run

1 - After removing Helium from system, and reestablishing vacuum at original level; close stopcocks A,X,Z. (see drawing).

2 - Open stopcock from appropriate gas reservoir and allow a predetermined volume of gas to enter gas burette.

3 - Close stopcock B, adjust manometer S to zero point, and record pressure (P_X), and burette temperature.

4 - Immerse adsorption bulb R in liquid nitrogen bath.

5 - Open stopcock A, adjust zero point on manometer; readjust to zero point when equilibrium conditions are obtained.

6 - Read and record pressure (P_R) and burette temperature.

7 - Close stopcock A.

8 - Open stopcock B and admit a second charge of adsorbate gas at a greater pressure (P_X) than that of the previous charge.

9 - Close stopcock B; adjust manometer to zero point; read and record pressure (P_X) and burette temperature for second point.

10 - Open stopcock A and repeat step number 4.

11 - For additional points on adsorption isotherm, repeat steps 1 through 6.

When sufficient data has been obtained so that values of P_r/P_o can be calculated for the range of 0.05 to 0.4, the run is normally complete for the straight line, and point "B" methods.

If a complete adsorption isotherm is required, continue taking points until P_r/P_o reaches a value of 1.0.

At the completion of each run, P_o must be determined. This can be accomplished by admitting a large volume of adsorbate gas through stopcock Z to compression bulb U, and allowing gas to condense in vapor pressure bulb T which is submerged in a liquid nitrogen bath. Saturation pressure is the highest equilibrium pressure obtainable.

H - Shutdown - One can start shutting down equipment while vapor pressure determination is being made. This is accomplished in the following manner:

1 - Shut off mercury diffusion pump, but allow cooling water to continue passing through it.

2 - Open stopcocks X,B,A slowly in that order.

3 - Remove liquid nitrogen bath from adsorption bulb.

4 - After vapor pressure determination has been made, open stopcock Z, and evacuate vapor pressure section of system.

5 - Lower mercury in manometers into their respective reservoirs.

6 - Lower mercury in gas burettes into their respective reservoirs, and shut off stopcocks on both sides of burette.

7 - Open stopcock C and other stopcocks on mercury reservoirs to vacuum line. (This equalizes pressure in system.)

8 - Shut off mechanical vacuum pump as soon as diffusion pump is cool to the touch.

9 - Shut off water and all electrical connections.

SAMPLE CALCULATIONS

Nomenclature

V_T--------- Volume of adsorbate gas as measured in gas burettes and capillary tube. (ml)

$V_T(stp)$--- Volume of adsorbate gas corrected to standard conditions. (ml)

$V_A(stp)$--- Volume of adsorbate gas in dead space of adsorption bulb, corrected to standard conditions. (ml)

$V_R(stp)$--- Volume of adsorbate gas which is not adsorbed at each point during run, corrected to standard conditions. (ml)

$V_{ads}(stp)$- Cumulative volume of adsorbate gas adsorbed at any point during run, corrected to standard conditions (ml)

P_X--------- Pressure of each addition of adsorbate gas as measured on manometer. (mm)

P_R--------- Residual pressure of adsorbate gas (not adsorbed), as measured on manometer, after equilibrium conditions are obtained. (mm)

P---------- Actual pressure of adsorbate gas to system. (mm)

P_o--------- Vapor pressure of adsorbate gas. (mm)

$V_T(He)$----- Initial volume of helium gas admitted to system for determining dead space in adsorption bulb, as measured in gas burettes and capillary tube. (ml)

$V_R(He)$----- Residual volume of helium gas in system after admitting gas to adsorption bulb. (ml)

P_T(He)---- Pressure exerted by initial volume of helium gas, as measured on manometer. (mm)

P_A-------- Barometric pressure. (mm)

P_S-------- Standard pressure. (mm)

T--------- Standard temperature. (°K)

T_1-------- Gas burette temperature. (°K)

V_m-------- Volume of adsorbate gas which forms a monolayer over total surface of sample. (cc/gm)

s--------- Slope of curve.

I--------- Y intercept of curve.

W--------- Weight of sample. (gm)

P_R(He)---- Pressure exerted by residual volume of helium. (mm)

V_o-------- Actual volume of capillary tube. (ml)

VA(He)---- Volume of helium gas in dead (free) space of adsorption bulb. (ml.)

f_v-------- Gas burette bulb factor.

f_a-------- Adsorption bulb factor.

V_{TB}------- Actual volume of gas burettes bulbs. (ml) (Calibrated with mercury by Otto Greiner Company before installing.)

Determination of Free Space in Capillary Tube (V_o)

V_{TB} (ml)	P (mm)	PV_{TB}
1.0	382.5	382.5
4.0	288.0	1152.0
9.0	204.5	1840.5
16.0	145.0	2320.0
26.0	103.0	2678.0
31.0	90.0	2790.0

From a plot of PV_{TB} versus P, the following values for B and A (see operating instructions for discussion) were obtained.

B = 3500 A = 250

Therefore:

$$V_o = \frac{B - A}{P} = \frac{3500 - 250}{400} = 8.125 \text{ ml}$$

This value remains constant as long as no changes are made in the capillary section of system.

Determination of Free Space in Adsorption Bulb (V_A), and Determination of Adsorption Bulb Factor (f_a). Run 13

$$V_T(He) = (V_{TB} + V_o)\frac{(T)}{(T_1)}\frac{(P_T He)}{(P_s)}\frac{(P_A)}{(P_s)} = \text{ml (stp)}$$

$$V_T(He) = (233.125)\frac{(273.2)(366.0)(772.5)}{(295.6)(760.0)(760.0)} = 105.7 \text{ ml (stp)}$$

$$V_R(He) = (V_{TB} + V_o)\frac{(T)}{(T_1)}\frac{(P_R He)(P_A)}{(P_S)\ (P_s)} = \text{ml (stp)}$$

$$V_R(He) = (233.125)\frac{(273.2)(122.0)(772.5)}{(295.6)(760.0)(760.0)} = 35.2 \text{ ml (stp)}$$

$$V_A(He) = V_T(He) - V_R(He) = \text{ml (stp)}$$

$$V_A(He) = 105.7 - 35.2 = 70.5 \text{ ml (stp)}$$

$$f_a = \frac{V_A(He)}{P_R(He)} = \frac{70.5}{122.0} = 0.577$$

In this instance, a second determination was made, and another value for f_a was obtained.

$$f_a = 0.595$$

An average value for f_a of 0.586 was used in subsequent calculations in Run 13.

Each time the adsorption bulb or sample contained therein is changed, this determination must be made.

Determination of Gas Burette Bulb Factor (f_v)

$$f_v = (V_{TB} + V_o)\frac{(\ T\ )}{(P_s)}$$

$$f_v = (9.0 + 8.125)\frac{(273.2)}{(760.0)} = 6.15$$

These factors remain constant for any given combination of gas burette bulbs and capillary tubing as long as no changes (due to breakage) occur.

Pressure Determination of Each Addition of Nitrogen Gas (P_X)

Example: Based on second point established during Run 13.

$P_X = 432.0$ mm (Read directly from manometer.)

Residual Pressure of Nitrogen Gas (P_R)

Example: Based on second point established during Run 13.

$P_R = 9.0$ mm (Read directly from manometer.)

Actual Pressure of Each Addition of Nitrogen Gas (P)

Example: Based on second point established during Run 13.

$$P = P_X - P_R$$

$$P = 432.0 - 9.0 = 423.0 \text{ mm.}$$

Note: On the first point taken on any run $P = P_X$ since there is no residual pressure P_R. Each new addition of gas to the system however, exerts only that pressure P equal to the total pressure P_X less the residual pressure P_R from the preceding addition.

Volume of Nitrogen Gas Corrected to Standard Conditions $V_T(stp)$

Example: Based on second point established during Run 13.

$$V_T(stp) = \frac{f_V(P)}{T_1} = \text{ml}$$

$$V_T(stp) = \frac{6.15(423.0)}{298.8} = 8.85 \text{ ml}$$

Residual Volume of Nitrogen Gas Corrected to Standard Conditions $V_R(stp)$

Example: Based on second point established during Run 13.

$$V_R(stp) = \frac{f_V(P_R)}{T_1} = \text{ml}$$

$$V_R(stp) = \frac{6.15(9.0)}{298.8} = 0.186 \text{ ml}$$

Volume of Nitrogen Gas in Dead Space of Adsorption Bulb Corrected to Standard Conditions, $V_A(stp)$

Example: Based on second point established during Run 13.

$$V_A(stp) = f_a(P_R) = \text{ml}$$

$$V_A(stp) = 0.586(9.0) = 5.27 \text{ ml}$$

Volume of Nitrogen Gas adsorbed, Cumulative, Corrected to Standard Conditions, $V_{ads}(stp)$

Example: Based on second point established during Run 13.

$$V_{ads}(stp) = V_T - (V_R + V_A) = \text{ml}$$

$$V_{ads}(stp) = 16.02 - (0.186 + 5.27) = 10.57 \text{ ml}$$

Vapor Pressure of Nitrogen Gas, Corrected to Standard Conditions, $P_o(stp)$

$$P_o(stp) = \frac{P_o(P_A)}{P_S} = \text{mm}$$

$$P_o(stp) = \frac{804.0(772.5)}{760.0} = 819.0 \text{ mm}$$

Calculation of Surface Area from the Straight Line Plot

From the plot of $\frac{P_R}{V_{ads}(P_o - P_R)} \times 10^3$ versus P_R/P_o

$I = Y \text{ intercept} = 0.1 \times 10^{-3}$

$$S = \text{slope} = \frac{(16.0 - 0.1) \times 10^{-3}}{0.1} = 79.5 \times 10^{-3}$$

$$V_m = \frac{1}{S + I} = \frac{1}{(79.5 + 0.1)10^{-3} \times W} = 0.58 \text{ ml/g}$$

$$\text{Area} = 4.38\, V_m = 4.38(0.58) = 2.56 \text{ m}^2/\text{g}$$

Calculation of Surface Area by the "Point B" Method

From the plot of V_{ads} ml/g versus P_R/P_o, Point B was established, and the tangent to the curve at this point intercepts the Y axis. This point of interception is V_m.

For Run 13: $V_m = 0.70$

$\text{Area} = 4.38 V_m = 4.38(0.70) = 3.06 \text{ m}^2/\text{g}$

TABLE OF BULB FACTORS (f_v)

Bulb Volume	f_v
8.125	2.92
9.125	3.28
12.125	4.35
13.125	4.72
14.125	5.075
17.125	6.15
22.125	7.95
24.125	8.66
29.125	10.45
28.125	10.09
34.125	12.25
44.125	15.85
54.125	19.50
53.125	19.10
103.125	37.18
233.125	83.90
234.125	84.40
237.125	85.30
242.125	87.20
249.125	89.50
259.125	93.20

Note: This is only a partial listing, which includes those values most commonly used in the experiments.

DATA SHEET

DATE 1/19/57 RUN NO. 13

Barometric Pressure (mm)---------------- 772.5

Room Temperature (°C)------------------- 24.3

Vapor Pressure (P_o, mm)----------------- 819.0

Sample Designation---------------------- Perlite, #300 Filter Aid

Weight of Sample (gms)------------------ 21.70

Vacuum in System (mm)------------------- 9.8×10^{-6}

Volume of Helium (Vt, ml)--------------- 233.125

Initial Helium Pressure (P, mm)--------- 366.0

Residual Helium Pressure P_1, mm)-------- 122.0

Burette Temperature (°C)---------------- 22.4

Vt (N_2) ml.	Burette Temp. oC	Px mm.	Pr mm.	P=Px-Pr mm	Vt (N_2) ml (stp)	Vt (N_2) ml (stp)
17.125	25.6	343.5	0.0	343.5	7.17	7.17
17.125	25.6	432.0	9.0	423.0	8.85	16.02
"	"	460.0	18.0	442.0	9.25	25.27
"	"	478.0	30.5	447.5	9.37	34.64
"	"	541.0	46.0	495.0	10.33	44.97
"	"	480.5	62.5	418.0	8.75	53.72
"	"	437.5	78.0	359.0	7.50	61.22
"	"	560.0	89.0	471.0	9.86	71.08
"	25.3	451.0	105.0	346.0	7.23	78.31
"	25.1	520.0	116.5	404.0	8.45	86.76
"	25.0	491.5	131.0	360.5	7.55	94.31
"	24.9	502.0	143.5	359.0	7.52	101.83
"	24.8	445.0	157.0	288.0	6.04	107.87
"	24.8	433.5	167.5	266.0	5.58	113.45
"	24.6	463.0	177.0	286.0	6.00	119.45
"	24.6	526.0	188.0	338.0	7.10	126.55
"	24.6	529.0	200.0	329.0	6.90	133.45
"	24.6	453.0	213.0	240.0	5.05	138.50
"	24.5	505.0	216.0	289.0	6.07	144.57
"	24.4	542.0	224.5	318.0	6.70	151.27
"	24.3	549.0	235.5	314.0	6.60	157.87

DATE 1/19/57 RUN NO. 13

V_t (N_2) ml	Burette Temp. C	Px mm	Pr mm	P=Px-Pr mm	V_t (N_2) ml (stp)	V_t (N_2) ml (stp)
17.125	24.2	537.0	246.0	291.0	6.11	163.98
"	24.1	522.0	257.5	265.0	5.57	169.55
"	24.0	534.0	267.0	267.0	5.62	175.17
"	23.3	562.0	272.0	290.0	6.11	181.28
"	23.2	605.0	281.0	324.0	6.83	188.11
"	23.1	759.0	294.0	465.0	9.80	197.91
"	22.9	822.0	308.5	514.0	10.80	208.71
"	22.8	848.0	327.0	521.0	11.00	219.71
"	22.7	929.0	346.0	583.0	12.30	232.01
"	22.6	842.0	367.0	475.0	10.05	242.06
"	22.5	803.0	384.0	419.0	8.85	250.91
"	22.4	852.0	396.0	456.0	9.67	260.58
"	22.3	862.5	417.5	445.0	9.42	270.00
"	22.3	780.5	433.5	347.0	7.35	277.35
"	22.1	838.5	444.0	394.0	8.35	285.70
"	21.9	851.0	449.0	402.0	8.53	294.23
"	21.8	805.0	461.0	344.0	7.29	301.52
"	21.7	852.5	474.0	378.0	8.00	309.52
"	21.6	829.5	486.0	343.0	7.27	316.79
"	21.6	879.0	500.0	379.0	8.04	324.83
"	21.4	850.0	514.0	336.0	7.13	331.96
"	21.4	855.0	521.0	334.0	7.10	339.06
"	21.2	900.0	531.0	369.0	7.82	346.88
"	21.2	910.0	544.0	366.0	7.77	354.65
"	21.1	917.0	557.0	360.0	7.64	362.29
"	21.0	922.0	570.0	352.0	7.47	369.76
"	21.0	932.0	582.0	350.0	7.44	377.20
"	20.9	906.0	595.0	316.0	6.72	383.92
"	20.9	906.0	606.0	300.0	6.38	390.30
"	20.8	960.0	618.0	342.0	6.26	396.56
"	20.7	915.0	622.0	293.0	6.24	402.80
"	20.7	906.0	630.0	276.0	5.88	408.69
"	20.7	939.0	640.0	299.0	6.36	415.04
"	20.6	944.0	651.0	293.0	6.23	421.27
"	20.6	933.0	662.0	271.0	5.77	427.04
"	20.6	932.0	666.0	266.0	5.66	432.70
"	20.5	947.0	674.0	273.0	5.80	438.50
"	20.5	953.0	683.0	270.0	5.75	444.25
"	20.5	936.0	691.0	245.0	5.21	449.46
"	20.4	1005.0	699.0	306.0	6.52	455.98
"	20.4	923.0	711.0	212.0	4.52	460.50
"	20.3	973.0	718.0	255.0	5.44	465.94
17.125	20.2	990.0	727.0	263.0	5.60	471.54

DATE 1/19/57 RUN NO. 13

Vt (N_2) ml	Burette Temp. C	Px mm	Pr mm	P=Px-Pr mm	Vt (N_2) ml (stp)	Vt (N_2) ml (stp)
17.125	20.1	1,004.0	736.0	268.0	5.71	477.25
"	20.1	1,000.0	744.5	256.0	5.45	482.70
"	20.05	965.0	744.5	221.0	4.71	487.41
"	20.0	1,000.0	750.0	250.0	5.34	492.75
"	20.0	1,001.0	757.0	244.0	5.20	497.95
"	19.9	982.0	764.0	218.0	4.65	502.60
"	19.9	978.0	771.0	207.0	4.41	507.01
"	19.9	957.0	776.0	181.0	3.86	510.87
"	19.9	968.0	780.0	188.0	4.01	514.88
"	19.9	971.0	784.0	187.0	3.99	518.87
"	19.8	975.0	788.0	187.0	3.99	522.86
"	19.8	965.0	791.0	174.0	3.72	526.58
"	19.8	986.0	794.0	192.0	4.08	530.66
"	19.7	1,003.0	796.0	207.0	4.41	535.07
"	19.7	983.0	798.0	185.0	3.95	539.02
"	19.6	982.0	800.0	182.0	3.89	542.91
"	19.6	995.0	801.0	194.0	4.14	547.05
"	19.6	994.0	803.0	191.0	4.07	551.12
17.125	19.6	1,001.0	804.0	197.0	4.20	555.32
34.125	19.5	962.0	804.0	158.0	6.70	562.02
"	19.5	976.0	804.0	172.0	7.30	569.32
"	19.5	981.0	807.0	174.0	7.38	576.70
"	19.5	984.0	808.0	176.0	7.46	584.16
34.125	19.5	983.5	798.0	185.0	7.84	592.00

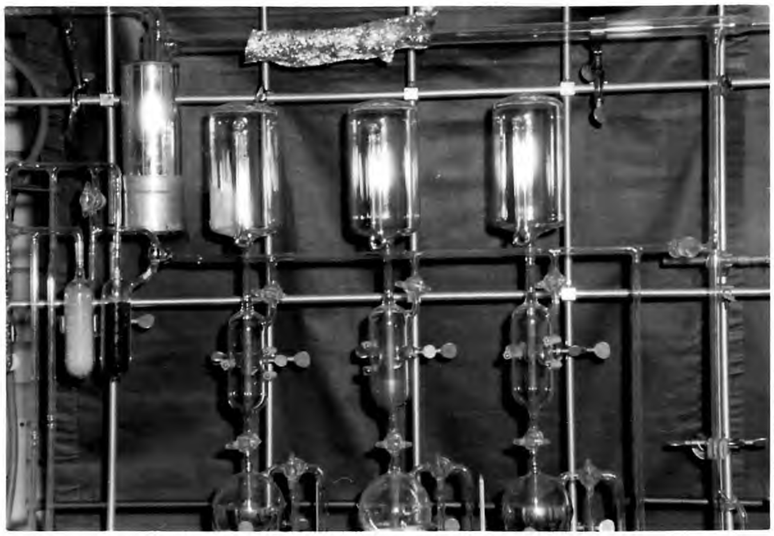
END OF RUN

DATE 1/19/57 RUN NO. 13

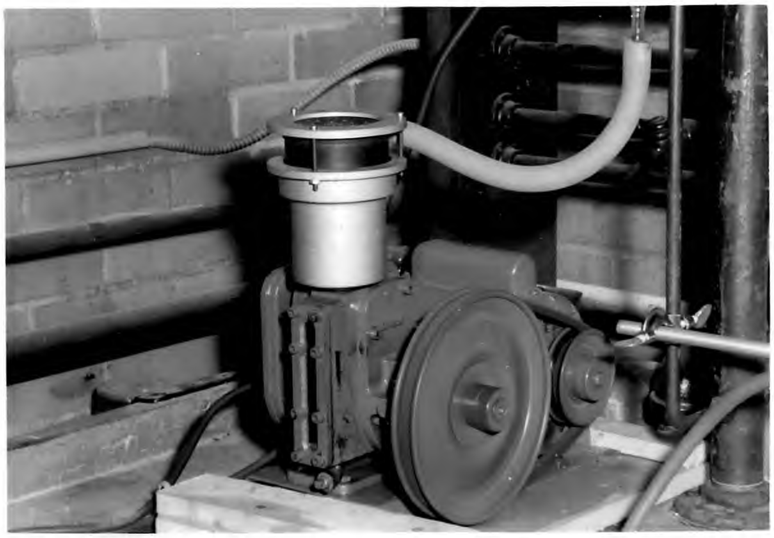
Vr (N_2) ml (stp)	Va (N_2) ml (stp)	Vads ml (stp)	Pr/Po	Po-Pr	Vads/g ml/g	$\frac{Pr}{Vads(Po-Pr)}$ x 10^3
-	-	-	-	819.0	-	-
0.186	5.27	10.57	0.011	810.0	0.486	1.05
0.372	10.56	14.34	0.022		0.660	
0.629	17.90	18.53	0.037		0.853	
0.949	27.00	17.03	0.056		0.784	
1.290	36.60	15.83	0.075		0.730	
1.610	45.60	14.01	0.095	741.0	0.645	7.50
1.840	52.10	17.14	0.108		0.789	
2.160	61.60	14.55	0.128		0.670	
2.410	68.40	15.95	0.142		0.734	
2.710	77.00	14.60	0.160	688.0	0.672	13.00
2.960	84.10	14.77	0.175		0.680	
3.240	92.00	12.63	0.192		0.582	
3.460	98.40	11.59	0.205		0.533	
3.660	103.80	11.99	0.216		0.552	
3.900	110.00	12.65	0.229	631.0	0.582	23.58
4.140	117.20	12.11	0.244		0.557	
4.410	125.00	9.09	0.260		0.417	
4.480	126.80	13.29	0.264		0.611	
4.640	131.60	15.03	0.274		0.691	
4.880	138.00	14.99	0.287	583.0	0.689	26.95
5.190	144.30	14.49	0.300		0.666	
5.410	151.00	13.14	0.314		0.605	
5.630	156.50	13.04	0.326	552.0	0.600	37.10
5.730	159.50	16.05	0.332		0.737	
5.920	164.80	17.39	0.343		0.800	
6.210	172.40	19.30	0.358	525.0	0.887	29.00
6.500	181.00	21.21	0.376		0.976	
6.940	191.80	20.97	0.378		0.964	
7.330	203.00	21.68	0.422		0.997	
7.750	215.00	19.31	0.448		0.888	
8.140	225.00	17.77	0.469		0.816	
8.420	232.00	20.16	0.483		0.926	
8.860	244.60	16.54	0.510		0.760	
9.200	254.00	14.15	0.530		0.650	
9.450	260.00	16.25	0.543	375.0	0.747	
9.500	263.00	21.73	0.549		1.000	
9.800	270.00	21.72	0.562		1.000	
10.100	277.70	21.72	0.580		1.000	
10.340	285.00	21.45	0.595		0.987	
10.620	293.00	21.21	0.611	319.0	0.976	
10.910	301.00	20.05	0.628		0.922	
11.100	306.00	21.96	0.637		1.010	

DATE 1/19/57 RUN NO. 13

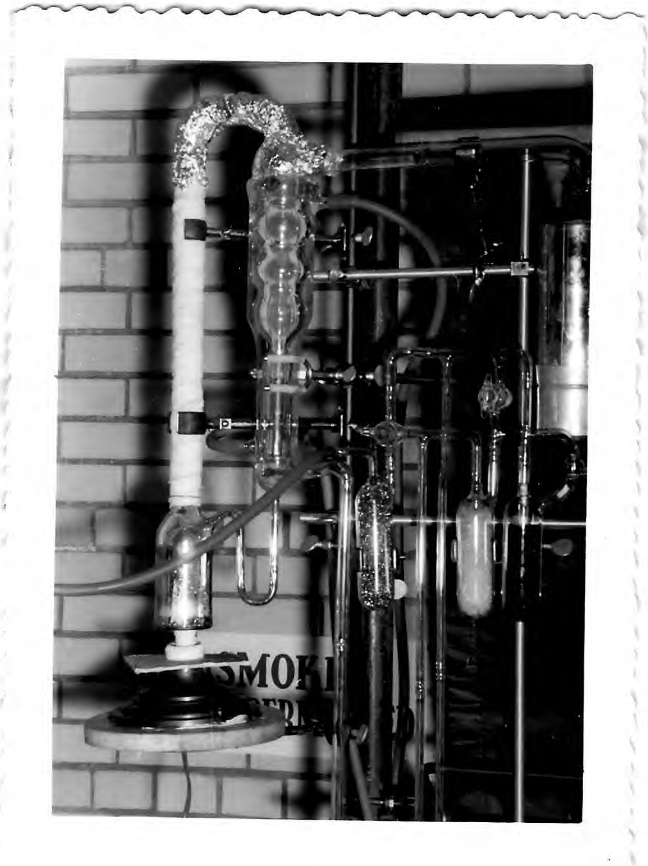
Vr (N_2) ml (stp)	Va (N_2) ml (stp)	Vads ml (stp)	Pr/Po	Po-Pr mm	Vads/g ml/g	$\frac{Pr}{Vads(Po-Pr)}$ x 10^3
11.30	311.70	23.88	0.650		1.100	
11.60	318.30	24.75	0.665		1.138	
11.87	326.40	24.02	0.681		1.108	
12.14	334.00	23.62	0.696		1.088	
12.40	341.70	23.10	0.710		1.062	
12.70	348.40	22.82	0.728		1.050	
12.93	356.00	21.37	0.741		0.985	
13.18	362.00	21.38	0.755		0.985	
13.25	364.40	25.15	0.761		1.156	
13.40	370.00	25.29	0.770		1.163	
13.60	375.00	26.44	0.783		1.218	
13.80	382.00	25.47	0.796		1.173	
14.16	388.00	24.88	0.810		1.145	
14.20	391.00	27.50	0.815		1.265	
14.35	395.00	29.15	0.824		1.341	
14.60	400.10	29.55	0.834		1.362	
14.78	405.00	29.68	0.845		1.365	
14.94	410.00	31.04	0.854		1.430	
15.20	416.00	29.30	0.869		1.348	
15.38	421.00	29.56	0.877		1.362	
15.54	426.00	30.02	0.890		1.380	
15.76	431.00	30.49	0.900		1.402	
15.90	436.00	30.80	0.909		1.417	
16.20	436.00	35.21	0.909		1.620	
16.05	440.00	36.70	0.916		1.690	
16.20	444.00	37.75	0.925		1.740	
16.40	448.00	38.20	0.933		1.766	
16.54	452.00	38.47	0.943		1.770	
16.62	455.00	39.25	0.949		1.810	
16.70	457.00	41.18	0.953		1.895	
16.78	460.00	42.09	0.957		1.940	
16.90	462.00	43.98	0.964		2.020	
16.95	464.00	45.63	0.968		2.100	
17.00	465.00	48.66	0.970		2.240	
17.07	467.00	51.00	0.973		2.340	
17.13	468.00	53.89	0.975		2.480	
17.16	469.00	56.75	0.977		2.630	
17.20	470.00	58.85	0.979		2.700	
17.17	470.50	63.45	0.981		2.920	
17.20	471.00	67.12	0.982		3.080	
17.20	471.00	73.82	0.982		3.390	
17.18	471.00	81.14	0.982		3.730	
17.22	473.00	85.48	0.988		3.930	
17.22	474.00	92.94	0.989		4.270	
17.10	468.00	106.90	0.975		4.920	



Gas Reservoirs

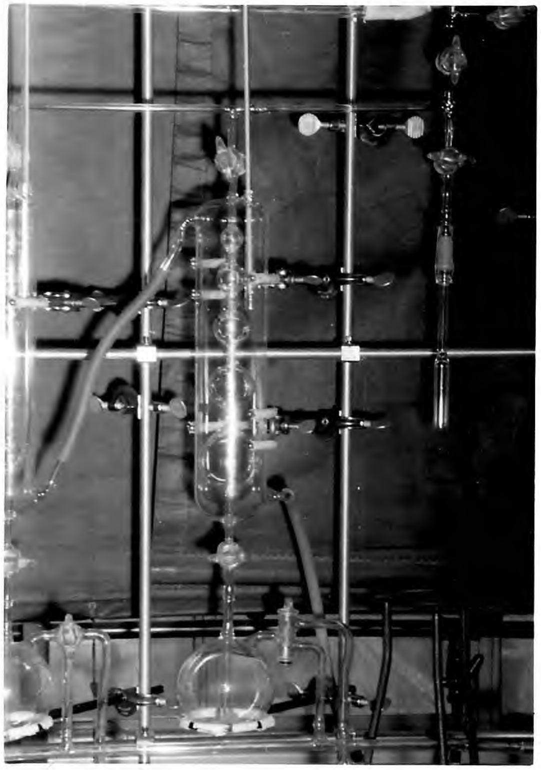


Mechanical Vacuum Pump



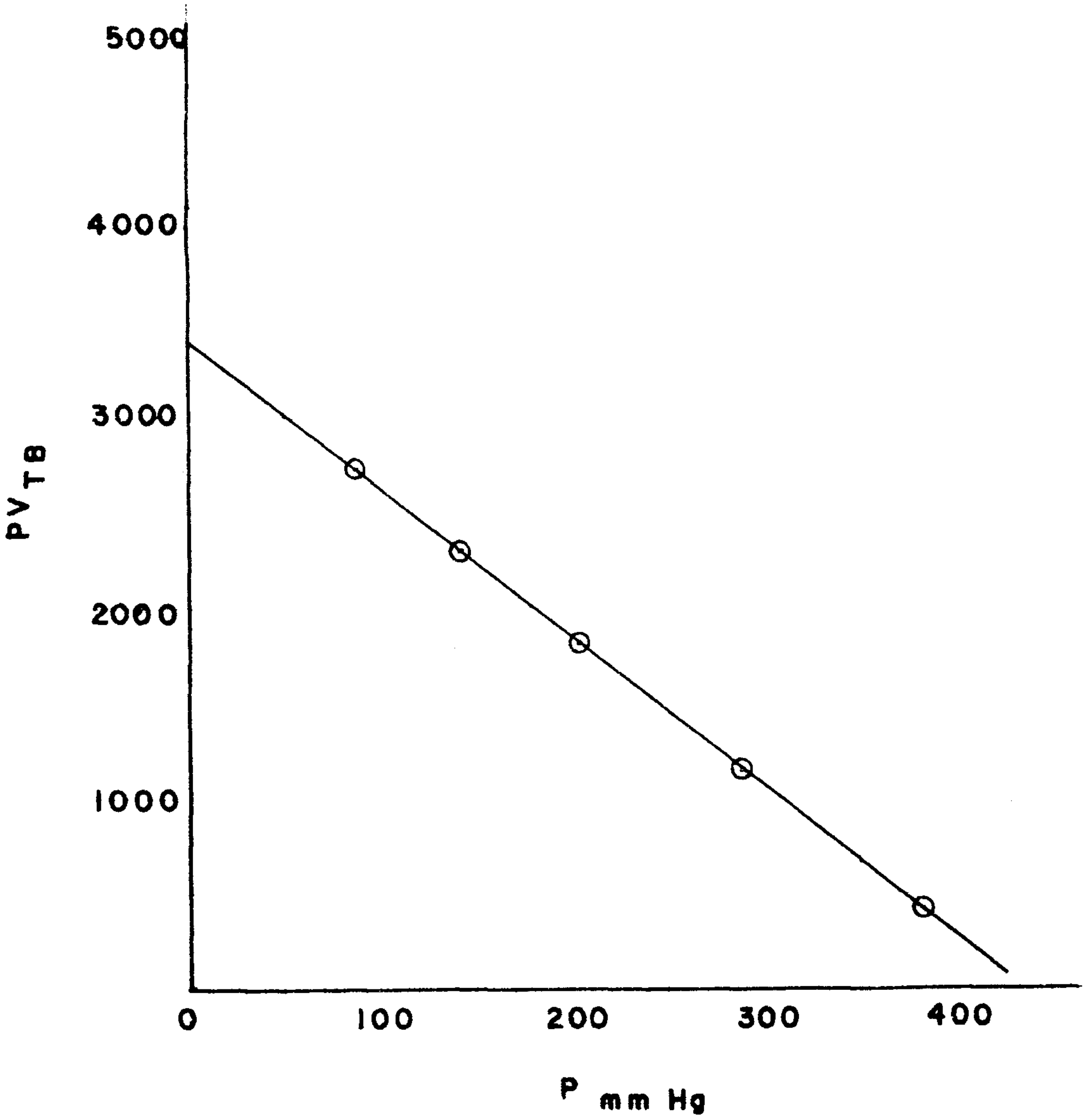
Mercury Diffusion
Pump

McLeod Gauge



Gas Burette,Water Jacketed

CALIBRATION OF FREE SPACE
He gas 20°C



DATA SHEET

Date 11/17/56 Run No. 9

Barometric pressure (mm)	764.7
Room temp. (°C)	22.0
Vapor pressure (P_o, mm)	802
Sample designation	Darco S-51
Weight of sample (g)	0.0413
Vacuum in system (mm)	1.8×10^{-6}
Volume of Helium (V_t, ml)	259.125
Initial Helium pressure (P, mm)	257.0
Residual Helium pressure (P_1, mm)	199.0
Burette temperature (°C)	21.7

$V_t(N_2)$ ml	Burette temp. °C	$P_x(N_2)$ mm	$P_r(N_2)$ mm
54.125	23.2	204	0
44.125	23.2	61	52
37.125	23.2	70	56
32.125	23.2	70	61
17.125	23.2	118	64
12.125	23.2	113	73
8.125	23.2	118	77
54.125	23.4	240	83
44.125	23.4	181	145
37.125	23.4	191	161
32.125	23.4	194	165
17.125	23.4	324	173
12.125	23.4	284	200
8.125	23.4	317	214

DATA SHEET

Date 12/1/56 Run No. 11

Barometric pressure (mm)	756.6
Room temp. (°C)	19.5
Vapor pressure (P_o, mm)	830
Sample Designation	Filter Cell C-46585
Weight of samples (g)	2.6809
Vacuum in system (mm)	9.6×10^{-6}
Volume of He (V_t, ml)	259.125
Initial He pressure (P, mm)	434.0
Residual He pressure (P_1, mm)	349.0
Burette temperature (°C)	23.0

V_t N_2 ml	Burette temp. °C	$P_x(N_2)$ mm	$P_r(N_2)$ mm
12.125	19.5	192	1.5
12.125	19.5	253	10.0
12.125	19.5	349	46
12.125	19.5	245	72.5
12.125	19.5	354	108
12.125	19.5	378	142.5
12.125	19.5	286	161.5
12.125	19.5	393	193
12.125	19.5	421	222
12.125	19.5	413	247
12.125	19.5	445	273
12.125	19.5	427	294
12.125	19.5	456	316
12.125	19.5	412	327
12.125	19.5	405	338
12.125	19.5	505	361
12.125	19.5	555	386
12.125	19.5	562	

DATA SHEET

Date 12/8/56 | Run No. 12

Barometric pressure (mm)	765
Room temp. (°C)	23.2
Vapor pressure (P_o, mm)	830
Sample designation	Filter Cell C-46585
Weight of sample (g)	13.7180
Vacuum in system (mm)	1.92×10^{-4}
Volume of He (V_t, ml)	233.125
Initial He Pressure (P, mm)	440.5
Residual He pressure (P_1, mm)	230.0
Burette temp. He (°C)	23.2

V_t (N_2) ml	Burette temp. °C	P_x(N_2) mm	P_r(N_2) mm
8.125	23.2	521	0.75
8.125	23.2	426.5	0.75
8.125	23.2	566	2.0
8.125	23.2	490	3.5
8.125	23.2	563.2	7.5
8.125	23.3	559	14.0
8.125	23.2	568	28
8.125	23.1	360	35.5
8.125	23.1	385	44
8.125	23.3	418	53
9.125	24.0	481	66
9.125	24.5	540	81
14.125	24.3	503	100.5
14.125	24.5	523	120
17.125	24.4	552	146
22.125	24.3	520	174
22.125	24.2	655	210
22.125	24.1	532	235
29.125	24.1	533	263
29.125	24.1	442	279
29.125	24.1	446	328

DATA SHEET

Date 2/2/57 Run No. 14

Barometric pressure (mm)	766.1
Room temp. (°C)	23.9
Vapor pressure (P_o, mm)	805.0
Sample designation	Celite 403 (C-46738)
Weight of samples (g)	2.89
Vacuum in system (mm)	9.8×10^{-6}
Volume of He (V_t, ml)	259.125
Initial He pressure (P, mm)	304
Final He pressure (P_1, mm)	243
Burette temp. He (°C)	25.2

V_t (N_2) ml	Burette temp	P_x(N_2) mm	P_r(N_2) mm
24.125	24.5	449	53
24.125	24.3	467	148
24.125	24.2	464	225
24.125	24.0	384	260
24.125	23.8	498	315
24.125	23.6	481	350
24.125	23.5	473	378
24.125	23.4	563	419
24.125	23.2	612	461
24.125	23.1	732	519
24.125	23.0	812	577
24.125	22.8	813	620
24.125	22.7	982	670
24.125	22.6	946	701
24.125	22.4	976	724
24.125	22.3	948	736
24.125	22.3	947	748
24.125	22.1	989	757
24.125	22.1	1000	763

DATA SHEET

Date 2/16/57 Run No. 15

Barometric pressure (mm)	758.7 - 753.7
Room temp. (°C)	26.8
Vapor pressure (P_o, mm)	605.0
Sample designation	300 Perlite Filter Aid
Weight of sample (g)	21.70
Vacuum in system (mm)	2.88×10^{-5}
Volume of He (V_t, ml)	259.125
Initial He pressure (P, mm)	253
Final He pressure (P_1, mm)	92
Burette temp. (He) (°C)	21.25

V_t (N_2) ml	Burette temp. °C	P_x(N_2) mm	P_r(N_2) mm
34.125	24.5	418	19
34.125	24.8	459	48
34.125	25.0	459	74
34.125	25.0	455	99
34.125	25.1	512	125
34.125	25.2	414	144
34.125	25.1	431	164
34.125	25.2	491	185
34.125	25.1	431	200
34.125	25.1	500	220
34.125	25.1	582	243
34.125	25.1	528	262
34.125	25.1	650	287
34.125	25.1	627	309
34.125	25.1	674	332
34.125	25.0	635	352
34.125	24.9	680	372
34.125	24.8	765	398
34.125	24.7	846	427
34.125	24.6	897	457
34.125	24.6	919	486
34.125	24.4	937	514
34.125	24.3	943	541
34.125	24.3	940	565
34.125	24.3	996	592
34.125	24.2	994	615

DATA SHEET

Date 2/16/57

Run No. 16

Barometric pressure (mm)	753.3
Room temp. (°C)	25.4
Vapor pressure (P_o, mm)	810
Sample designation	#100 Perlite Filter Aid
Weight of sample (g)	4.456
Vacuum in system (mm)	3.2×10^{-6}
Volume of Helium (V_t, ml)	259.125
Initial Helium pressure (P, mm)	418
Residual Helium pressure (P_1, mm)	231
Burette temperature (°C)	24.5

V_t (N_2) ml	Burette temp. °C	$P_x(N_2)$ mm	$P_r(N_2)$ mm
24.125	25.0	468	44
24.125	24.9	414	82
24.125	24.8	441.5	119
24.125	24.7	443	152
24.125	24.6	545	190.5
24.125	24.5	485	220.5
24.125	24.4	657.5	264
24.125	24.3	769	315.5
24.125	24.2	768	363
24.125	24.2	837	410.5
24.125	24.1	790	447
24.125	23.9	978	500
24.125	23.9	997	549
24.125	23.8	965	589
24.125	23.7	975	625
24.125	23.7	993	660
24.125	23.6	945	686
24.125	23.6	983.5	716
24.125	23.6	992	738
24.125	23.5	978	761
24.125	23.4	969	779
24.125	23.4	985	792
24.125	23.3	973.5	799
24.125	23.2	963.5	801.5
24.125	23.2	988	802
24.125	23.1	979	802.5
24.125	23.0	802.5	802.5

End of Run

DATA SHEET

Date 2/22/57 Run No. 17

Barometric pressure (mm)	769.5
Room temp. (°C)	21.7
Vapor pressure (P, mm)	810.0
Sample designation	#100 Perlite Filter Aid
Weight of sample (g)	4.456
Vacuum in system (mm)	5×10^{-4}
Volume of Helium (V_t, ml)	259.125
Initial Helium pressure (P, mm)	405.0
Residual Helium pressure (P_1, mm)	224.0
Burette temperature (°C)	20.8

$V_t(N_2)$ ml	Burette temp. °C	$P_x(N_2)$ mm	$P_r(N_2)$ mm
24.125	21.6	426	-
24.125	21.6	536	41.0
24.125	21.6	475	91.5
24.125	21.6	496.5	130
24.125	21.6	483	168
24.125	21.5	561	200.3
24.125	21.6	657	237.5
24.125	21.6	693.5	279.5
24.125	21.6	709.5	322
24.125	21.6	678.5	361
24.125	21.7	823	392.5

End of Run

DATA SHEET

Date 2/22/57

Run No. 18

Barometric pressure (mm)	769.5
Room temp. (°C)	21.2
Vapor pressure (P_o, mm)	800
Sample designation	Silex Sand C-46739
Weight of sample (g)	8.200
Vacuum in system (mm)	2.0×10^{-5}
Volume of Helium (V_t, ml)	259.125
Initial Helium pressure (P, mm)	435
Residual Helium pressure (P_1, mm)	360
Burette temperature (°C)	21.6

V_t (N_2) ml	Burette temp. °C	P_x(N_2) mm	P_r(N_2) mm
44.125	21.6	376	-
44.125	21.6	377	27.5
44.125	21.6	502	127
44.125	21.6	539	250
44.125	21.5	584	340
44.125	21.5	593	415
44.125	21.5	660	467
44.125	21.5	713	522
44.125	21.5	819	573
44.125	21.5	893	632
44.125	21.5	933	676
44.125	21.5	974	716
44.125	21.5	998	741
44.125	21.5	993	763
44.125	21.5	975	776
44.125	21.5	971	786
44.125	21.5	991	791

End of Run

DATA SHEET

Date 2/24/57 Run No. 19

Barometric pressure (mm)	770
Room temp. (°C)	15.8
Vapor pressure (P, mm)	828
Sample designation	#100 Perlite Filter Aid
Weight of sample (g)	11.700
Vacuum in system (mm)	2.0×10^{-5}
Volume of Helium (V_t, ml)	259.125
Initial Helium pressure (P, mm)	353.0
Residual Helium pressure (P, mm)	132.0
Burette temperature (°C)	14.3

V_t (N_2) ml	Burette temp. °C	P_x(N_2) mm	P_r(N_2) mm
24.125	15.3	455.0	-
24.125	15.7	511.0	19.0
24.125	15.8	528.0	45.0
24.125	15.9	552.0	69.0
24.125	16.0	574.0	94.0
24.125	16.1	708.0	119.0
24.125	16.2	714.0	148.0
24.125	16.4	756.0	178.0
24.125	16.5	770.0	208.0
24.125	16.6	826.0	238.0
24.125	16.7	868.0	267.0
24.125	16.8	911.0	297.0
24.125	16.9	959.0	326.0

End of Run

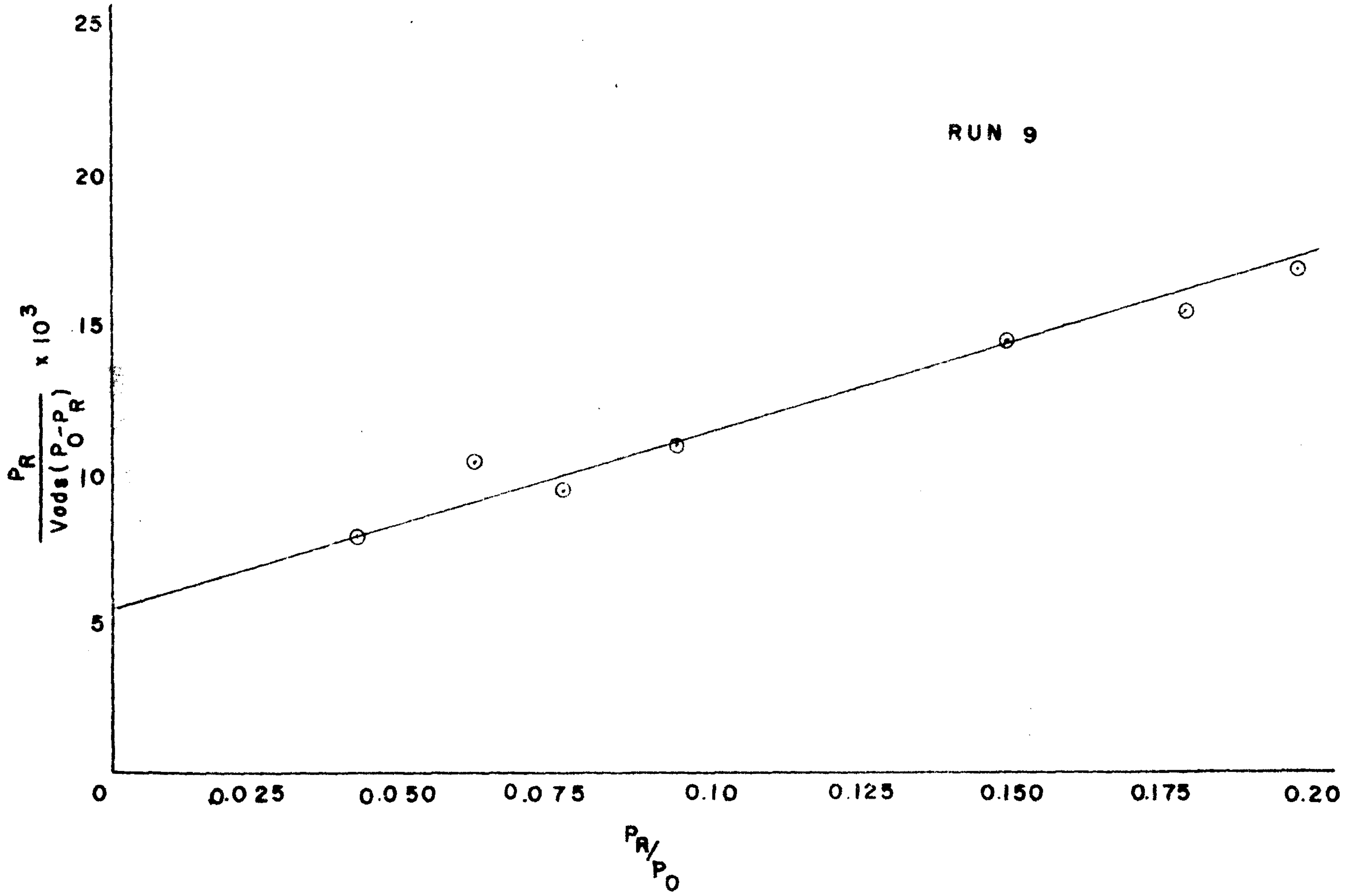
DATA SHEET

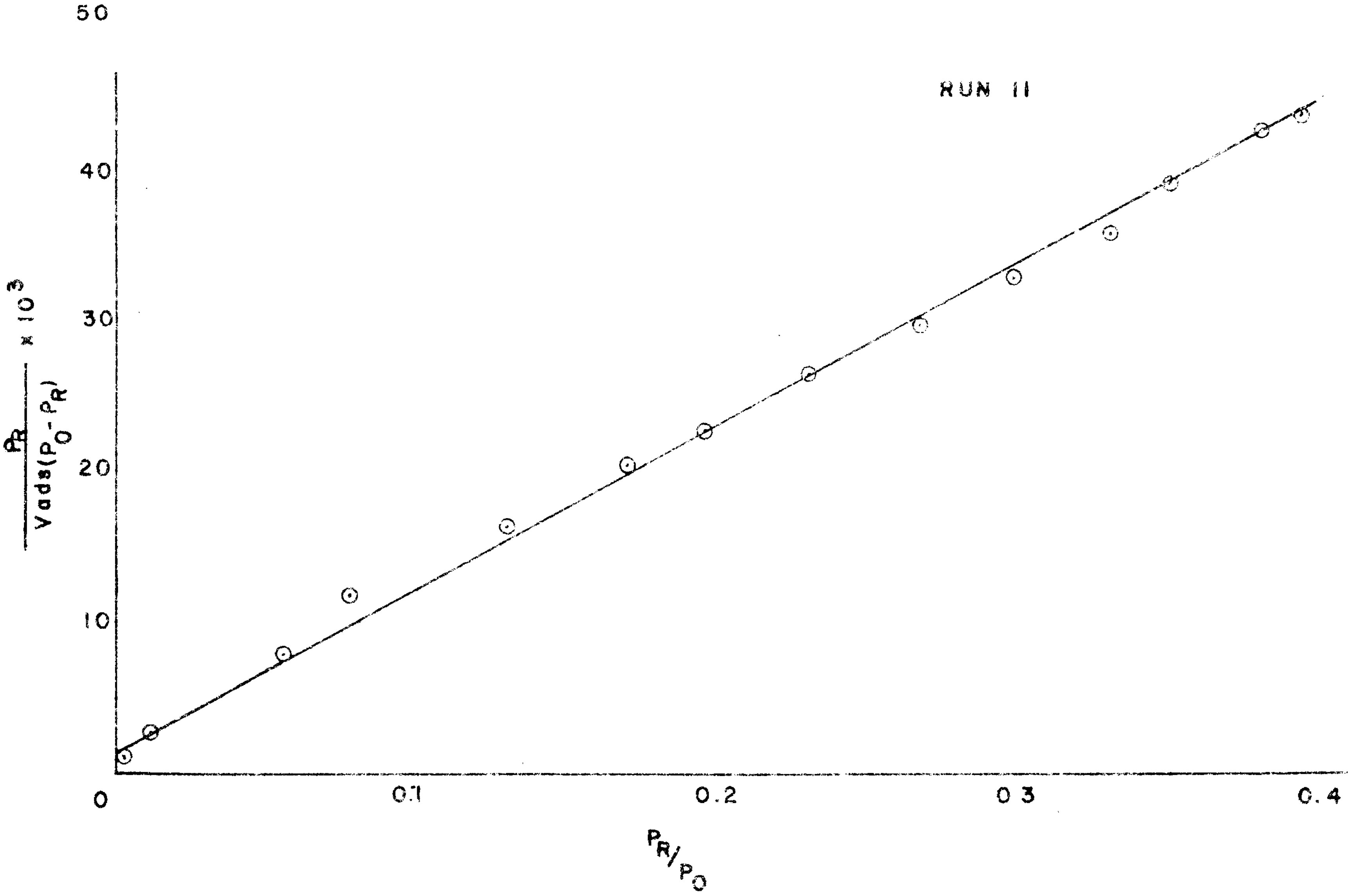
Date 2/24/57 Run No. 20

Barometric pressure (mm)	768.0-767.5
Room temp. (°C)	17.8- 17.9
Vapor pressure (P_o, mm)	825.0
Sample designation	Silex Sand C-46739
Weight of sample (g)	8.2
Vacuum in system (mm)	1.4×10^{-5}
Volume of Helium (V_t, ml)	259.125
Initial Helium pressure (P, mm)	299.0
Residual Helium pressure (P_1, mm)	246.0
Burette temperature (°C)	17.5

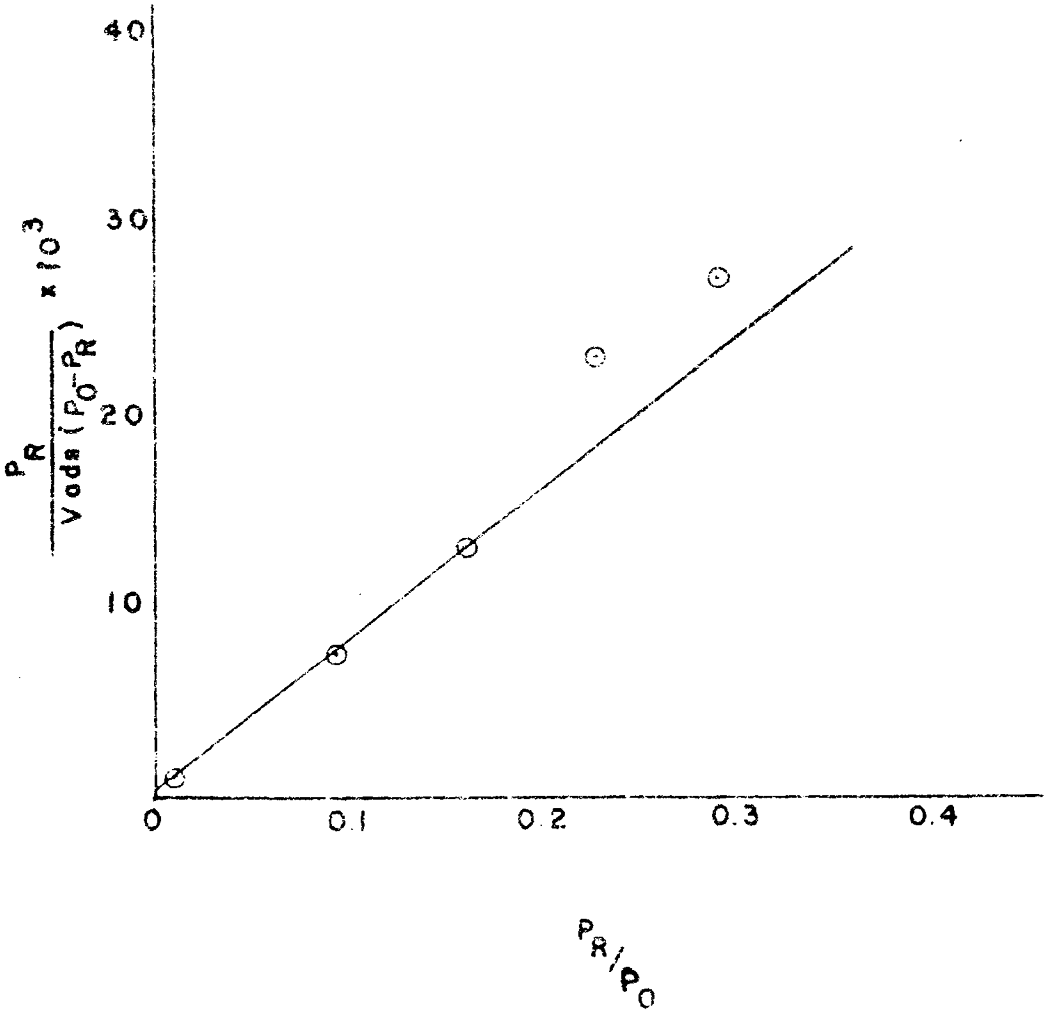
V_t (N_2) ml	Burette temp. °C	P_x(N_2) mm	P_r(N_2) mm
24.125	18.3	368.0	-
24.125	18.2	473.0	3.5
24.125	18.2	549.0	56.5
24.125	18.2	690.0	156.0
24.125	18.2	684.0	272.0
24.125	18.2	792.0	360.0

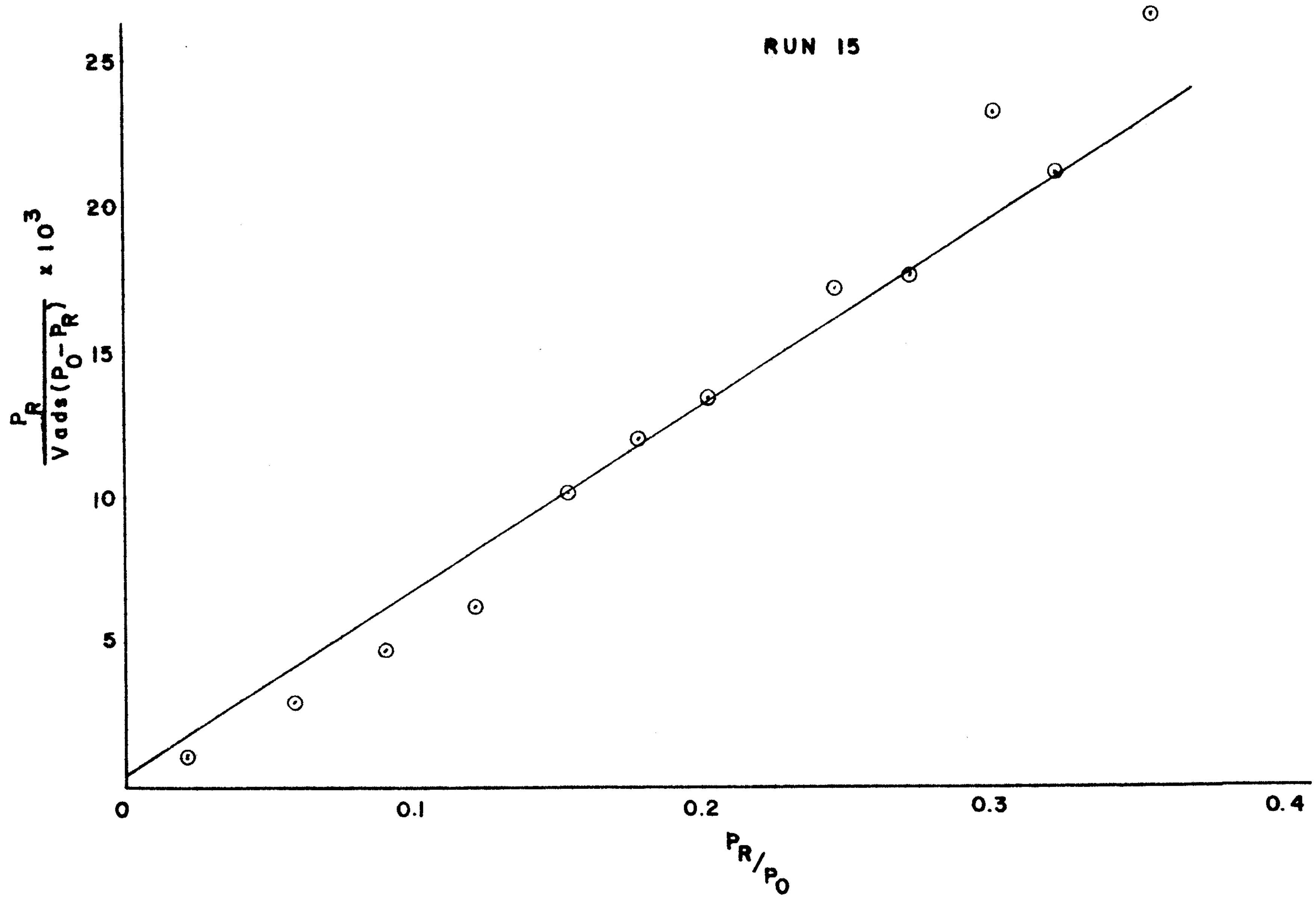
End of Run

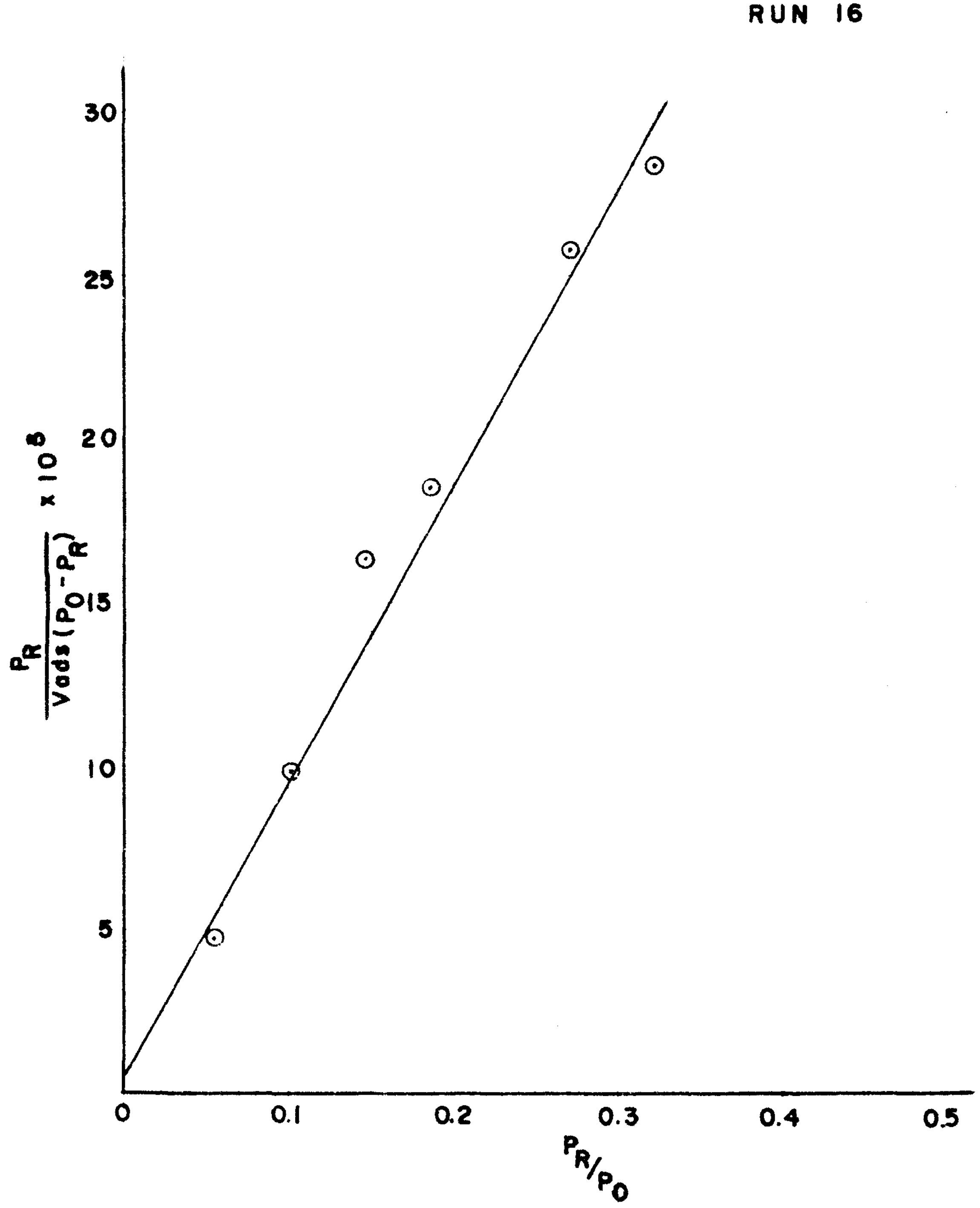

RUN 9
$\frac{P_R}{Vods(P_O - P_R)} \times 10^3$
25
20
15
10
5
0
0.025
0.050
0.075
0.10
0.125
0.150
0.175
0.20
P_R/P_O


RUN II
50
40
30
20
10
0
0.1
0.2
0.3
0.4
$\frac{P_R}{V_{ads}(P_0 - P_R)} \times 10^3$
P_R/P_0

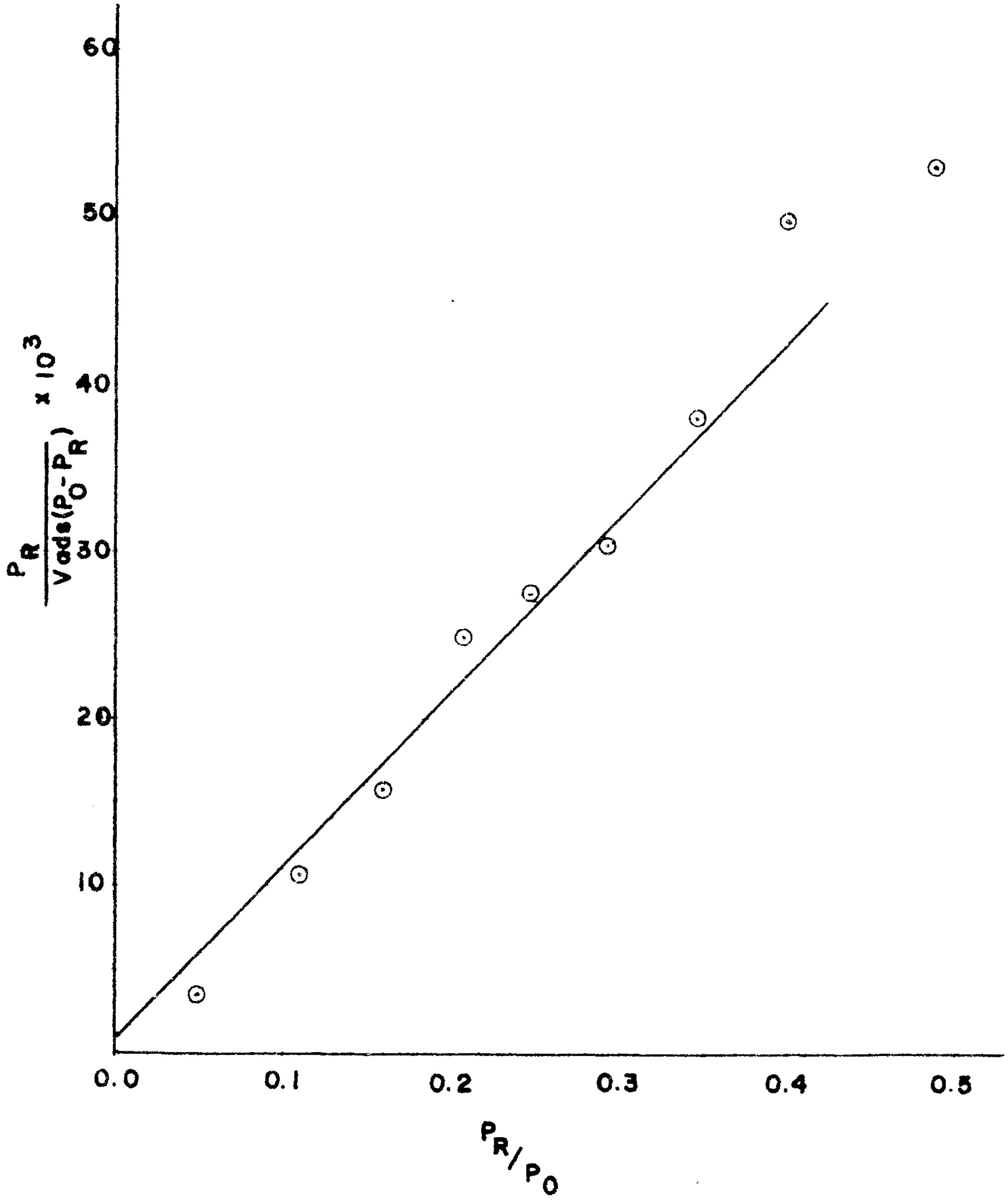
RUN 13

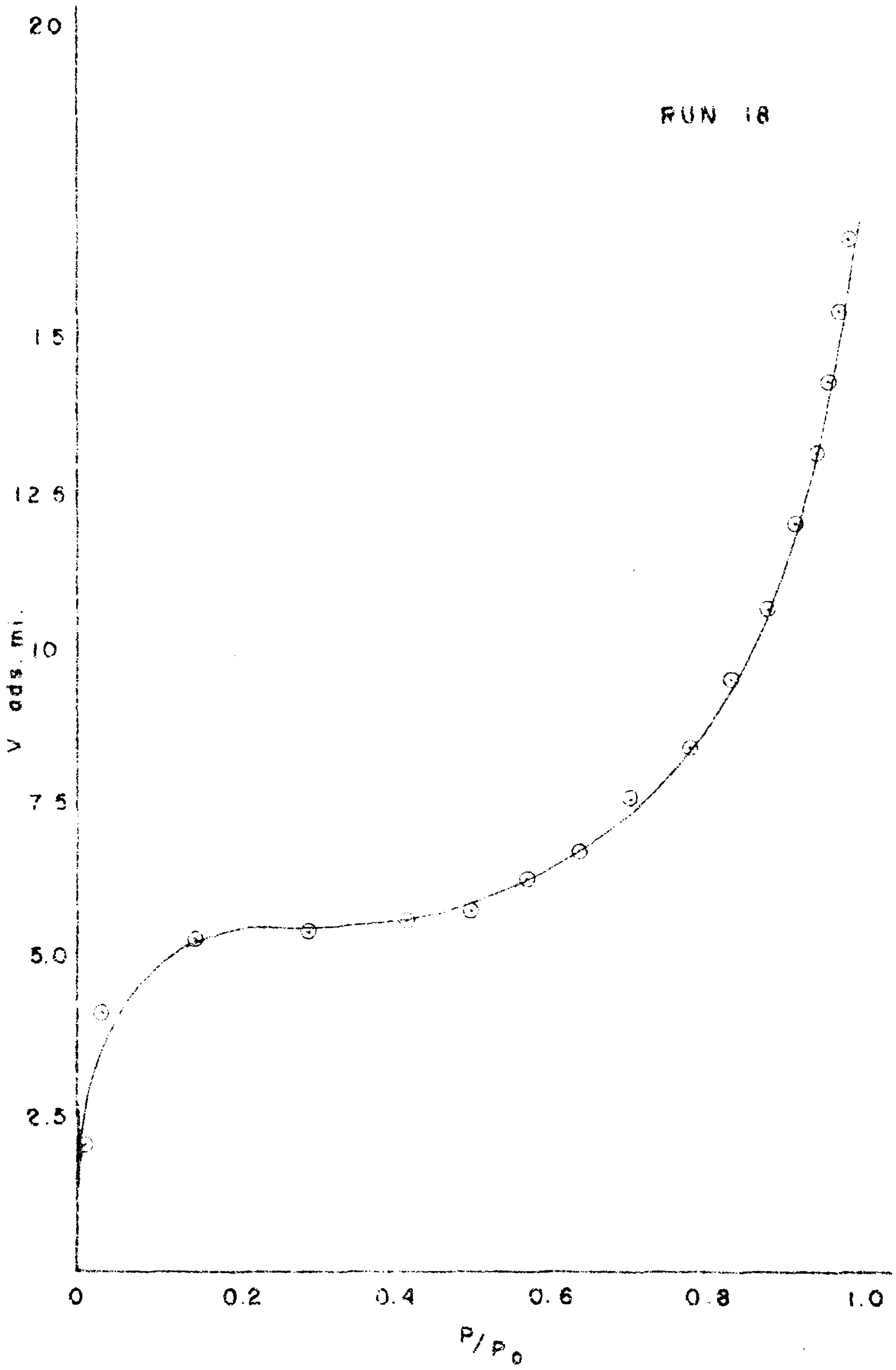


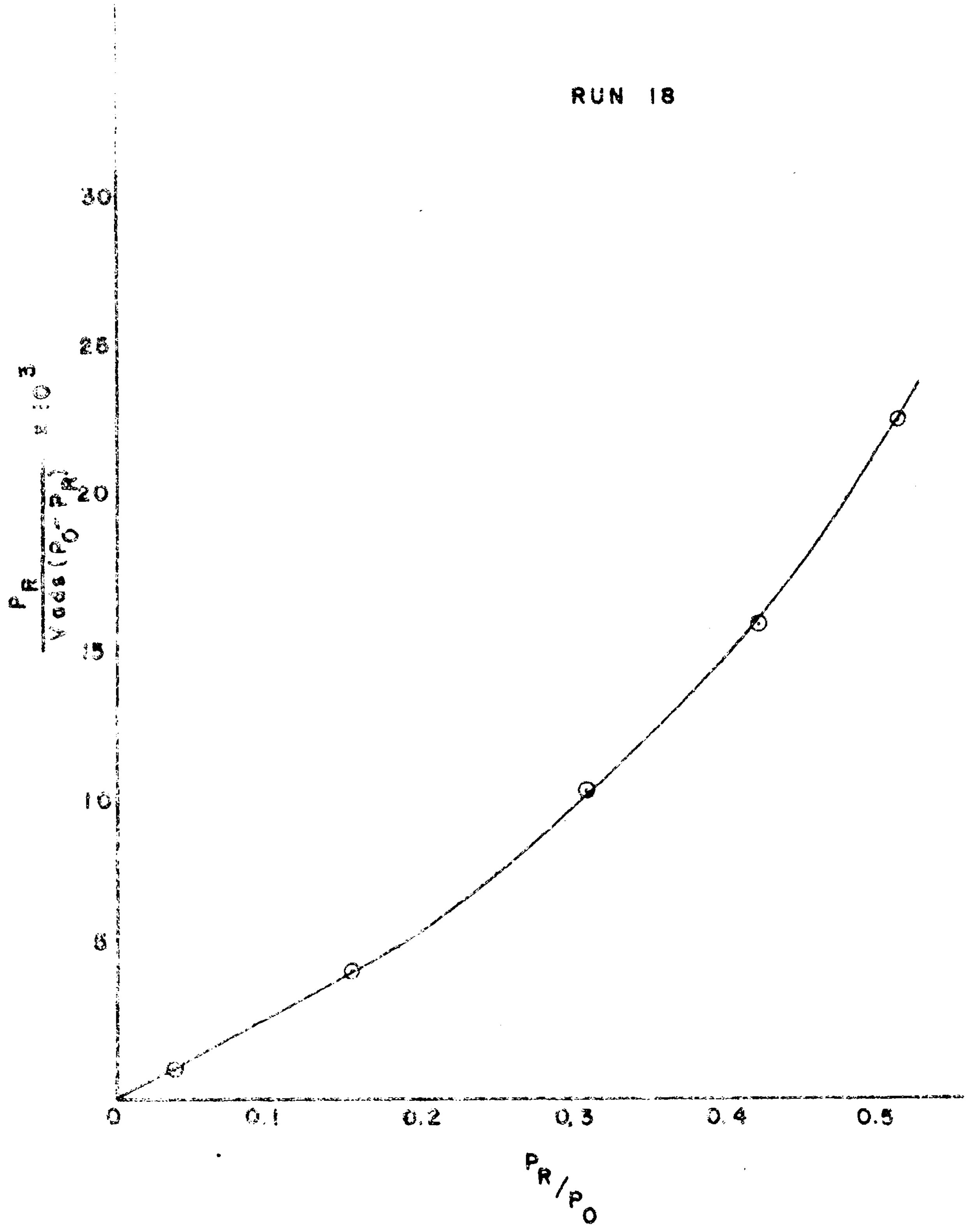

RUN 15
$\frac{P_R}{Vads(P_O-P_R)} \times 10^3$
25
20
15
10
5
0
0.1
0.2
0.3
0.4
P_R/P_O

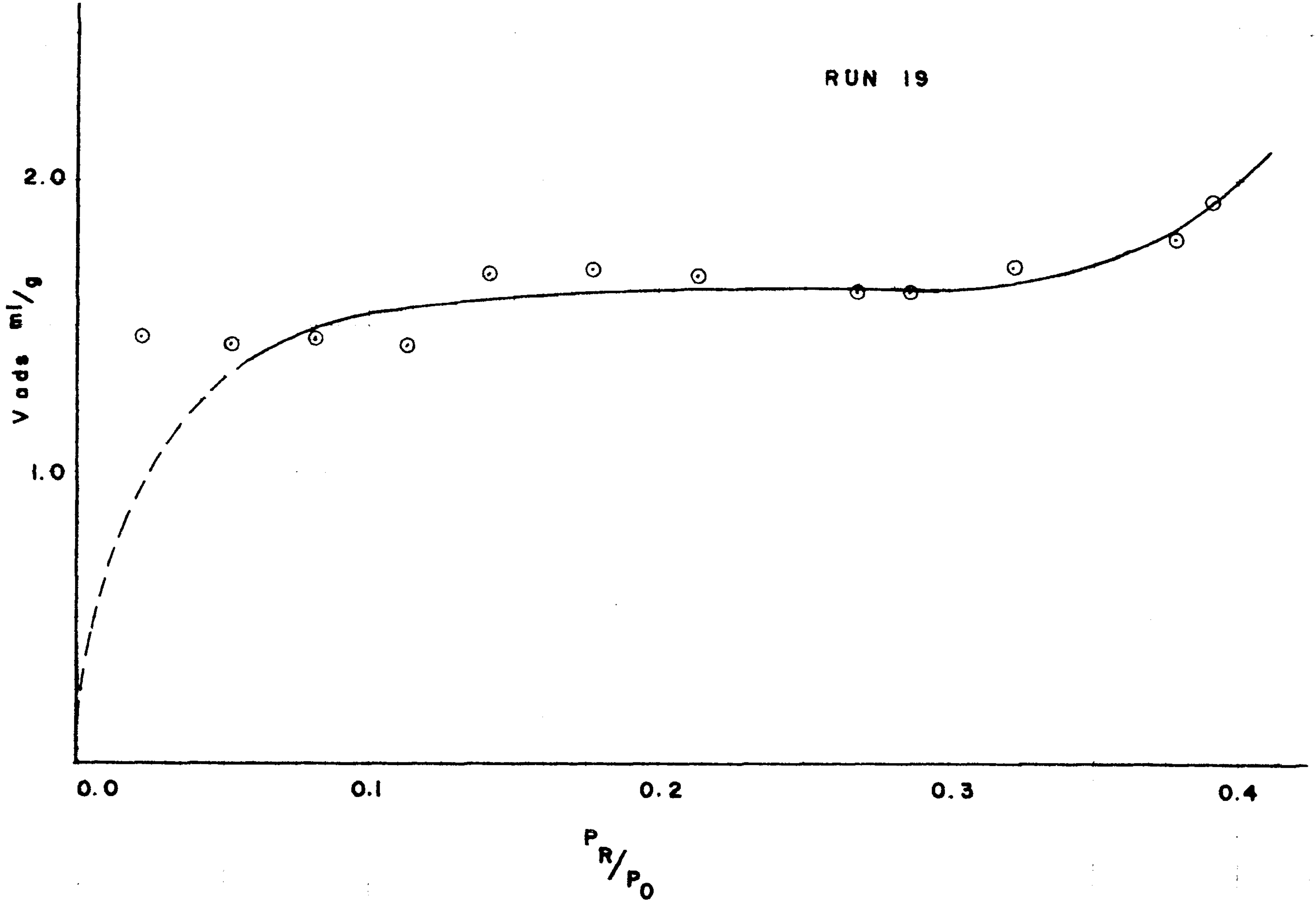

RUN 16
$\frac{P_R}{V_{ads}(P_0 - P_R)} \times 10^8$
30
25
20
15
10
5
0
0.1
0.2
0.3
0.4
0.5
P_R/P_0

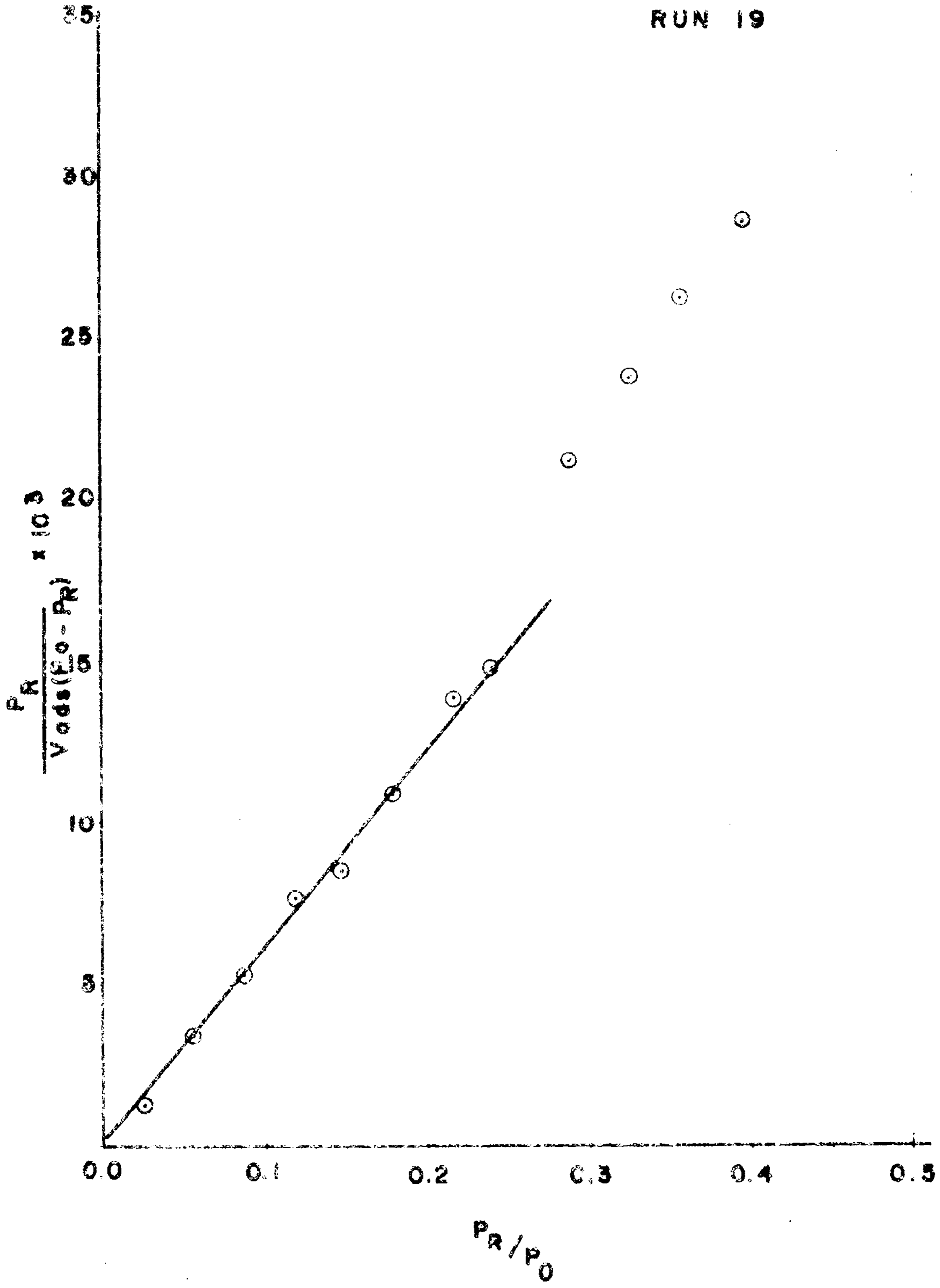
RUN 17

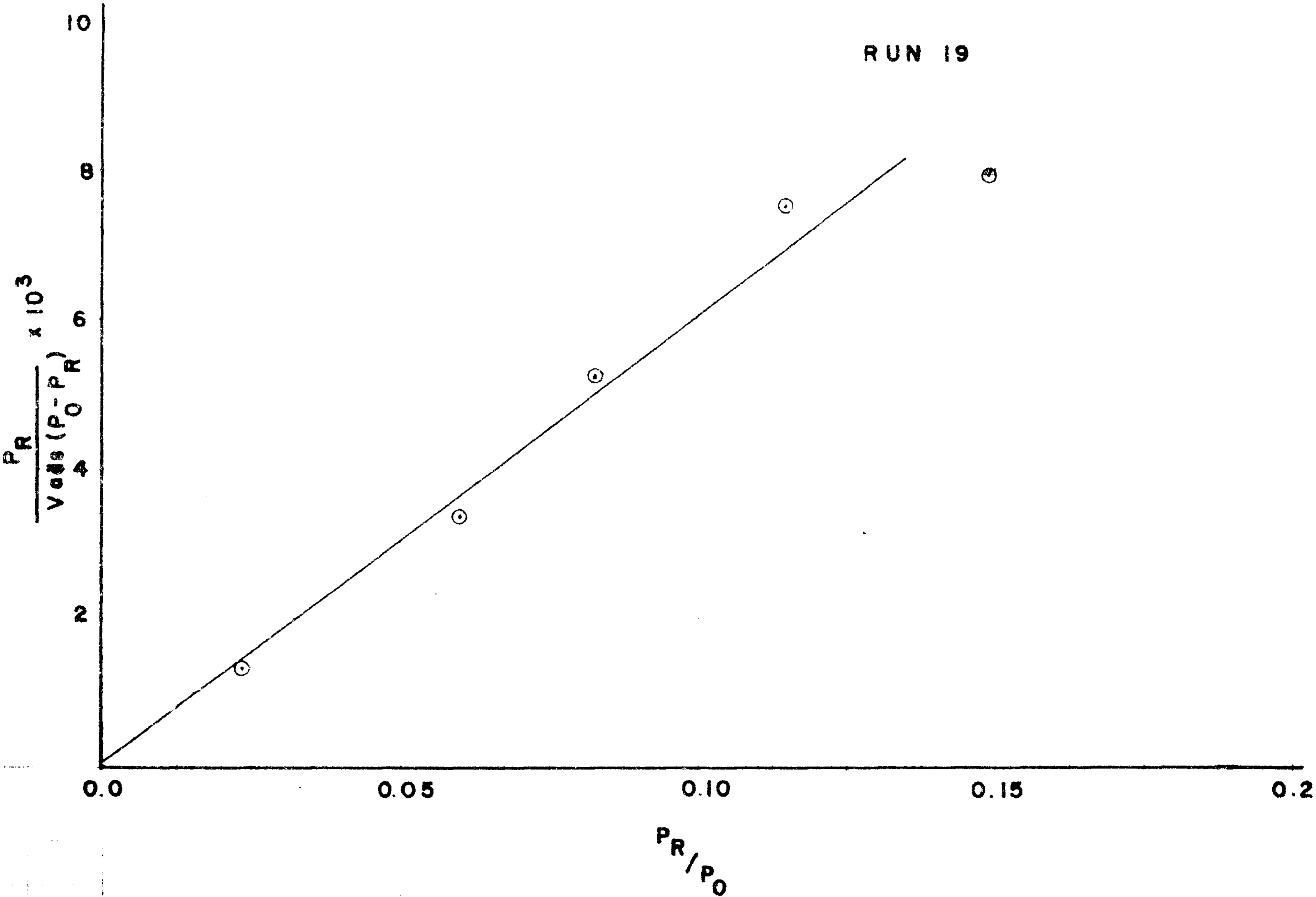


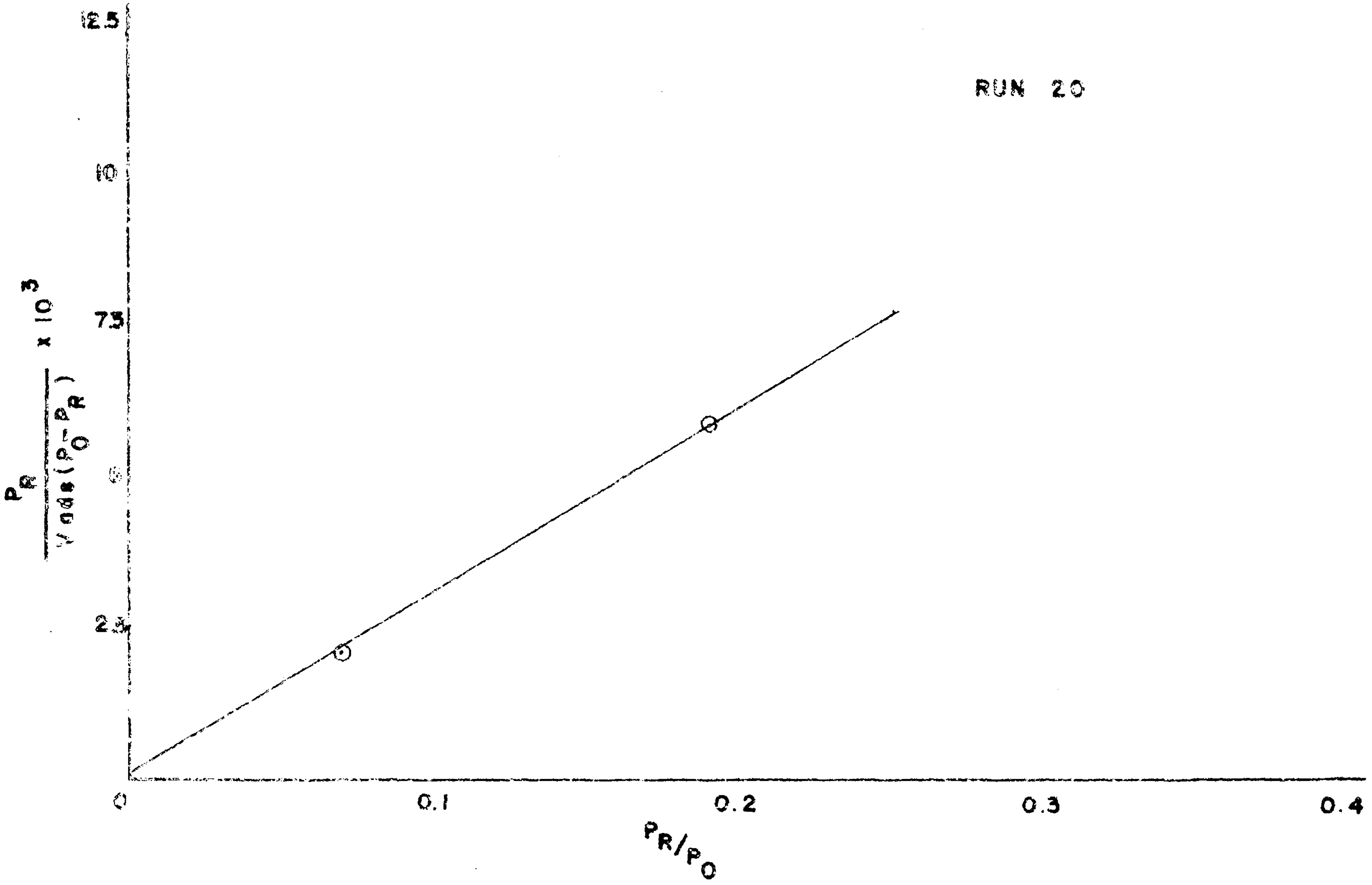

RUN 18
V ads. ml.
20
15
12.5
10
7.5
5.0
2.5
0
0.2
0.4
0.6
0.8
1.0
P/Po

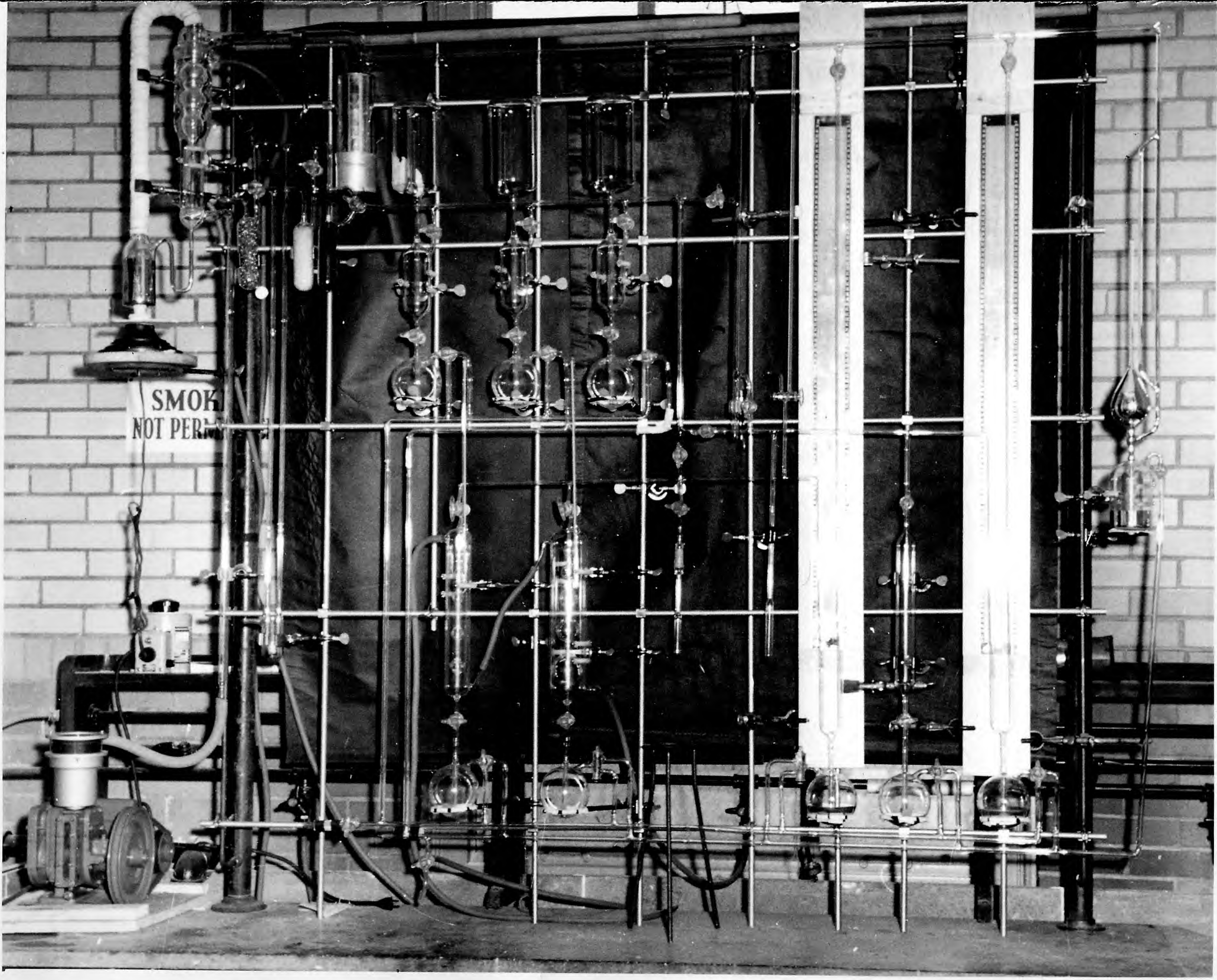

RUN 18
$\frac{P_R}{V_{ads}(P_0-P_R)} \times 10^3$
30
25
20
15
10
5
0
0.1
0.2
0.3
0.4
0.5
P_R/P_0


RUN 19
2.0
1.0
Vads ml/g
0.0
0.1
0.2
0.3
0.4
PR/P0


RUN 19
35
30
25
20
15
10
5
0.0
0.1
0.2
0.3
0.4
0.5
$\frac{P_R}{V_{ads}(P_o - P_R)} \times 10^3$
P_R / P_0


RUN 19
10
8
6
4
2
$\frac{P_R}{V_{ads}(P_0 - P_R)} \times 10^3$
0.0
0.05
0.10
0.15
0.2
P_R/P_0


RUN 20
12.5
10
7.5
5
2.5
0
0.1
0.2
0.3
0.4
P_R/P_O
$\frac{P_R}{V\,ads\,(P_O - P_R)} \times 10^3$


SMOK
NOT PER

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