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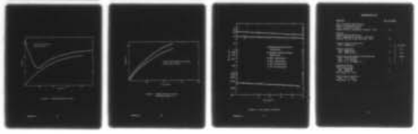

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CONTRACTOR REPORT ARCSL–CR–77027

GAS ADSORPTION BY ACTIVATED AND IMPREGNATED CARBONS

Quarterly Progress Report

January 1977 to April 1977

by

P. J. Reucroft
G. B. Freeman

July 1977

UNIVERSITY OF KENTUCKY
Lexington, Kentucky 40506
Contract DAAA15-74-C-0163

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ASC whetlerite
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Superactivated carbon 2
Carbon 2
Adsorption
Desorption
Carbon tetrachloride
Water vapor
Hydrogen cyanide
Binary mixture
Dubinin-Polanyi equation
Chemisorption
Hysteresis
Physisorption

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Carbon tetrachloride isotherms were measured for Amoco GX-32, a pelletized form of carbon 2. k and W_0 values were comparable to other forms of carbon 2. Mixed isotherms of H_2O and HCN were measured showing the almost complete domination of the adsorption process by HCN, the solubility of HCN in the adsorbed phase of H_2O, and the very low solubility of H_2O in the adsorbed phase of HCN. Feasibility data were gathered for the examination of ultrapure carbons.

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PREFACE

The work described in this report was authorized under Contract No. DAAA 15-74-C-0163, Task 1W762710A09501, Gas Adsorption by Activated and Impregnated Carbons. The work described covers the period from 9 January 1977 to 8 April 1977.

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CONTENTS

GAS ADSORPTION BY ACTIVATED AND IMPREGNATED CARBONS

RESEARCH PROGRESS DURING THE PERIOD

I. INTRODUCTION

The experimental apparatus has been modified by the addition of a three liter gas storage flask. The new gas handling system, in conjunction with the existing five liter gas storage flask, has allowed the measurement of several binary vapor isotherm points in succession in an experimental run rather than just one single point.[1] This apparatus change and the Baratron gauge addition (which took place October 1976) is shown in an updated diagram in Figure 1. CCl_4 isotherms were obtained on a new granular charcoal sample Amoco GX-32, (reported BET surface area of 3330 m^2/g). This is essentially the same material as PX-21 (Carbons 2, 4, 6 of previous measurements)[2,3] in a pelletized form (1/16 inch in diameter). The pertinent adsorption parameters were recorded and compared with the same parameters measured for the powdered form of this carbon (carbon 2).

An ultrapure (low surface area) carbon was examined in order to test the feasibility of studying such carbons in our apparatus. Such carbons contain very little metal impurity and, hence, can provide background adsorption data which reflects the carbon-adsorbate interaction uninfluenced by impregnant effects. Two ultrapure carbons, SP-2 and SP-3, were obtained from Union Carbide and carbon tetrachloride isotherms were measured to determine an estimate of the surface area and to compare their behavior with other carbons previously studied.

Whenever possible, k and W_0 values have been determined using a linear regression least squares fit program from Texas Instruments.[4] This program also gives the coefficient of determination, which provides a confidence level for the suitability of the data fit to the straight line defined by the slope and intercept calculated by the program.

II. ADSORPTION OF CARBON TETRACHLORIDE BY GX-32

The Amoco GX-32 carbon samples were outgassed at the same temperature, 100°C, as the carbon 2 samples previously investigated.[2] The carbon sample was heated at the outgassing temperature for several hours at a pressure on the order of 10^{-6} torr, until a stable minimum sample mass was obtained. The sample was allowed to return to ambient temperature under vacuum and then adsorbate vapor was introduced into the system and allowed to equilibrate with the carbon sample. After stabiliztion of both sample mass, gaseous adsorbate pressure, and the ambient

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[1] P. J. Reucroft and G. B. Freeman, Eleventh Quarterly Progress Report, October 1976 to January 1977, Edgewood Arsenal Contract DAAA15-74-C-0163.

[2] P. J. Reucroft and C. P. Madhusudhan, Eighth Quarterly Progress Report January 1976 to April 1976, Edgewood Arsenal Contract DAAA15-74-C-0163.

[3] P. J. Reucroft and C. P. Madhusudhan, Ninth Quarterly Progress Report April 1976 to July 1976, Edgewood Arsenal Contract DAAA15-74-C-0163.

[4] Program Manual ST1, Statistics Library, Program ST1-08, Texas Instruments Incorporated, Dallas, Texas 1975.

temperature, sample mass and the adsorbate pressure were recorded. Subsequent data points were measured by either increasing or decreasing the adsorbate vapor pressure. After the series of data points comprising the isotherms were recorded, the sample was again heated to the outgassing temperature and the system vacuum was pumped to about 10^{-6} torr for several hours. The sample mass was compared to the original sample mass under similar conditions and any increase in sample mass was considered to be the result of chemisorption.

III. ADSORPTION OF HCN AND H_2O VAPOR MIXTURES BY BPL ACTIVATED AND ASC WHETLERITE CARBONS.

Several procedures were followed in order to obtain four different types of isotherms. The first type of procedure involved the adsorption of HCN in increasing amounts by carbons already in equilibrium with H_2O vapor. In order to achieve this situation, the carbon samples were prepared in the standard manner described in the previous section, with an outgassing temperature of 400°C for BPL activated carbon samples and 150°C for ASC whetlerite impregnated carbon samples, and exposed to H_2O vapor. This permitted the measurement of a dry minimum mass for the carbon sample and an initial "loading" of water vapor W_{H_2O}. The relative water vapor pressure was about 0.8 and W_{H_2O} on the order of 0.37 cm^3g^{-1}. The water vapor was present in the five liter gas storage flask, the glass manifold enclosed by valves V8, V9, V12 and V3 (see Fig. 1 for this discussion) and in the portion of the system containing the microbalance. HCN was stored in the three liter storage flask and confined by valve V12. Valve V6 was closed and the water vapor cold trapped in the five liter storage flask at liquid nitrogen temperatures and confined by valve V7. A suitable amount of HCN was allowed to enter the now empty (<.01 torr) manifold between valves V7 and V5 and V12 was re-closed. Valve V7 was opened allowing the H_2O and HCN to mix in a gaseous state and then valve V5 opened. This procedure maintained the amount of water vapor in equilibrium with the carbon sample at a constant level and allowed variation of the HCN level. After equilibration had been reached, ambient temperature, sample mass and adsorbate pressure were recorded and the above procedure repeated in order to increase the HCN partial pressure in the system. Care was taken to complete these isotherms during the course of one day in order to minimize the problems associated with hysteresis effects accompanying any temperature changes in the carbon sample.

Isotherms describing the adsorption of water vapor by BPL activated and ASC whetlerite impregnated carbons already in equilibrium with HCN were also determined. The procedure followed here was identical with that described for the adsorption of HCN by carbons in equilibrium with H_2O, with the roles of HCN and H_2O reversed. The relative vapor pressure of HCN was about 0.15-0.20 and the initial loading of HCN, W_{HCN}, equal to about 0.3 cm^3g^{-1}.

In a third type of experiment, a mixture of HCN and H_2O (HCN relative partial pressure = 0.15 and H_2O relative partial pressure = 0.82) was collected in the five liter gas storage flask. The isotherm was taken in the normal manner with the HCN/H_2O mixture serving as the adsorbate and a BPL activated carbon sample as the adsorbent. The relative amounts of HCN and H_2O were kept constant throughout the experiment but the total (binary) vapor pressure increased.

Finally, a BPL activated carbon sample prepared in the standard manner, was pre-exposed to water vapor at a relative pressure of 0.73 and initial loading 0.321 g g^{-1}. This water was desorbed and collected in the five liter flask and the BPL sample heated to 400°C under a vacuum of 10^{-6} torr. A minimum of water vapor was lost during this process. Valve V7 confined the water vapor and valve V5 isolated the sample from the glass manifold. A suitable amount of HCN was released to the manifold and allowed to combine with the H_2O in the five liter flask. This combination was then allowed access to the sample through valve V5 and equilibration reached. After recording the usual parameters, the total process including desorption was repeated and the amount of HCN in the system was increased. This allowed measurement of the adsorptive properties of an essentially fresh carbon exposed simultaneously to HCN and H_2O with the H_2O relative pressure held constant at a high value and the HCN relative pressure as a variable.

IV. ADSORPTION OF CARBON TETRACHLORIDE BY ULTRAPURE CARBONS

The two ultrapure carbons SP-2 and SP-3 were exposed to carbon tetrachloride in the usual manner utilizing a conservative outgassing temperature of 400°C. Prior to an experimental run, the glass boat to be used in that experiment was used as the adsorbent in a 'blank' isotherm run. The glass boat was treated as if it were a carbon sample and outgassed at 400°C after which a carbon tetrachloride isotherm was measured in the usual manner.

A summary of the impurity levels in these carbons has been provided by Union Carbide and guarantees less than 6 ppm metallic impurities.

V. RESULTS AND DISCUSSION

The results of the work on GX-32 are displayed in Figure 2 in a plot of W vs. ε^2 according to the Dubinin-Polanyi equation:

$$\log W = \log W_0 - k\varepsilon^2$$

where W, W_0, k and ε have their usual meanings.[5] A least squares fit gives a value of $k = 4.24 \times 10^{-8}$ $\text{cal.}^{-2}\text{mole}^2$ and $W = 1.305$ $\text{cm.}^3\text{g}^{-1}$. The previous values reported for the different forms of this carbon(PX-21) are given in Table 10. No hysteresis was detected within relative pressure limits of 0.0001 to 0.260, but significant chemisorption ranged from 0.0026 to 0.0067 $\text{cm.}^3\text{g}^{-1}$. No other unusual features for this carbon were noted.

Isotherms reflecting HCN adsorption by carbons pre-exposed to and in equilibrium with H_2O are displayed in both the Polanyi (Fig. 3) and conventional forms (Fig. 4). The parameters associated with these isotherms do not seem to be obviously related to the adsorption of HCN by the carbons involved but rather indicate a dependence on the solubility of HCN in the adsorbed H_2O phase. HCN is known to be highly

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[5] P. J. Reucroft, W. H. Simpson, and L. A. Jonas, J. Phys. Chem., 75, 3526 (1971).

soluble in water, so it would seem that the adsorption of HCN by the carbons in equilibrium with high levels of H_2O loading is largely dependent on the state of the H_2O phase. The data does not exhibit any particular consistency, however, between data runs and more work must be done before this situation can be fully understood. The HCN/H_2O mixture produced no significant chemisorption on BPL and 0.083 $cm.^3g^{-1}$ on ASC whetlerite, i.e., slightly higher than the normal amount retained for single HCN vapor chemisorption.

The results of experiments dealing with H_2O adsorption by carbons in equilibrium with HCN are shown in a Dubinin-Polanyi plot (Fig. 5). Very little water was adsorbed by the system and the adsorption noted is not unambiguously due to the H_2O. The temperature fluctuations that took place during the experiment were typically a degree or two. This is large enough to cause fluctuations in the amount of HCN adsorbed. The numbers reported for the H_2O adsorption are thus uncertain in view of their small values. Adsorption parameters are reported in Table 10.

The two experiments involving the simultaneous adsorption of the two vapors provided interesting results. Since there was no way of determining the relative proportions of the two vapors adsorbed during the determination of a single isotherm point, the isotherms were depicted in terms of grams of adsorbate adsorbed per gram of adsorbent. The total amount adsorbed was plotted as a function of HCN relative pressure. In Figure 6, the amount of HCN in the adsorbate mixture is varied while the amount of H_2O is kept constant. Upon the introduction of a very small relative pressure of HCN, the total mass of adsorption decreases. This continues with increasing HCN relative pressure until an adsorption minimum **is reached.** Subsequent to this point (~0.5 relative HCN pressure), the adsorption curve closely parallels the single vapor HCN adsorption curve.[6] Apparently, the HCN adsorption dominates and discourages the adsorption of H_2O even in the presence of relatively large vapor pressures of H_2O. There is some apparent adsorption of H_2O, but this appears to be a small fraction of the total amount adsorbed. This diagnosis is supported by Figure 7, which is a plot of mass of adsorbate per gram of adsorbent as a function of relative HCN pressure for the adsorption of an HCN/H_2O mixture maintained in essentially constant ratio. Again the adsorption curve follows the single vapor HCN curve in character with a small increase apparently due to H_2O adsorption. It is interesting to note that these two curves cross at (approximately) a relative water vapor pressure of 0.73 and a relative HCN pressure of 0.11. At this point both curves show an adsorption of about $0.2g.g^{-1}$. No significant chemisorption was found in these studies

The data strongly implies that a) the adsorption of HCN by carbons which are already in equilibrium with high levels of water vapor is significant and a function of the solubility of HCN in the adsorption phase of H_2O; b) the adsorption of HCN strongly dominates in any process involving the simultaneous adsorption of H_2O and HCN from the vapor phase; c) HCN discourages additional adsorption of water vapor.

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[6] P. J. Reucroft and C. T. Chiou, Seventh Quarterly Progress Report, October 1975 to January 1976, Edgewood Arsenal Contract DAAA15-74-C-0163.

The two ultrapure carbons examined were similar in behavior and isotherm data was obtained for SP-3 which appeared to have the greater surface area ($\leq 10 m.^2 g^{-1}$). In view of the very small amounts of carbon tetrachloride adsorbed by the SP-3 carbon samples, it was decided to measure background isotherms involving the glass boats used in the experiments. Adsorption by the glass was found to vary several orders of magnitude, depending on the glass boat examined, and reached saturation at very low relative vapor pressure ($<.01$). It was approximately the same order of magnitude as the SP-3 carbon adsorption in the low pressure region. The ultrapure carbon data in this report has been corrected for the adsorption of CCl_4 by the glass boat. Figure 8 shows three Polanyi isotherms for the adsorption of CCl_4 by BPL activated carbon,[2] super-activated carbon 2,[7] and the SP-3 carbon. The adsorption parameters are included in Table 10. An increasing k value is indicative of decreasing affinity and appears to correlate with increasing purity. If it is assumed that the ultrapure carbon data provides a k value which reflects the pure carbon-adsorbate interaction, the lower k values observed in the case of the other two carbons can most likely be attributed to the effect of metal ion impurities in the carbons. A more detailed study of this situation, beginning with a further examination of ultrapure carbons, would help to clarify the role played by the non-carbon elements present in activated carbon samples.

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[7] P. J. Reucroft, C. P. Madhusudhan, G. B. Freeman, Tenth Quarterly Progress Report, July 1976 to October 1976, Edgewood Arsenal Contract DAAA15-74-C-0163.

APPENDIX A
TABLES

TABLE 1. ADSORPTION AND DESORPTION DATA

Adsorbate: Carbon Tetrachloride ρ(liquid) = 1.594

Adsorbent: GX-32

Sample I

P/P_0	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})	W (cm.^{3}g^{-1})	P/P_0	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})	W (cm.^{3}g^{-1})
0.0001	33.315	0.0535	0.260	0.627	1.1127
0.0009	16.919	0.2546	0.119	1.560	1.0659
0.0087	7.755	0.6275	0.0691	2.461	0.9952
0.0283	4.372	0.8358	0.0399	3.576	0.8969
0.0577	2.798	0.9649	0.0180	5.566	0.7547
0.0781	2.239	1.0148	0.0057	9.241	0.5450
0.136	1.370	1.0769	0.0017	14.018	0.3143
0.260	0.627	1.1127	0.0004	21.060	0.1528

TABLE 1. CONTD.

Adsorbate: Carbon Tetrachloride ρ(liquid) = 1.594

Adsorbent: GX-32

Sample II

P/P_0	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})	W (cm.^{3}g^{-1})	P/P_0	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})	W (cm.^{3}g^{-1})
0.0001	30.482	0.0316	0.0574	2.810	0.9528
0.0011	16.146	0.2314	0.0436	3.407	0.8969
0.0083	7.946	0.5862	0.0337	3.993	0.8477
0.0359	3.828	0.8575	0.0173	5.718	0.7282
0.0435	3.402	0.8941	0.0082	8.000	0.5934
0.0547	2.920	0.9383	0.0036	10.892	0.4210
0.0574	2.810	0.9528	0.0023	12.680	0.3506
			0.0008	17.580	0.1999
			0.0004	20.856	0.1603

TABLE 2. ADSORPTION DATA, I.

Adsorbate: Hydrogen Cyanide ρ(liquid) = 0.687

Adsorbent: BPL Activated Carbon with $W_{H_2O} = 0.358 (g.g^{-1})$

P/P_0 (HCN)	W $(g.g^{-1})$	$\varepsilon^2 \times 10^{-6}$ $(cal.^2 mole^{-2})$
0.0057	0.033	9.27
0.0088	0.057	7.80
0.026	0.071	4.63
0.051	0.092	3.07
0.093	0.103	1.97

TABLE 3. ADSORPTION DATA, II.

Adsorbate: Hydrogen Cyanide ρ(liquid) = 0.687

Adsorbent: BPL Activated Carbon with W_{H_2O} = 0.373(g.g^{-1})

P/P_0(HCN)	W (g.g^{-1})	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})
0.029	0.040	4.34
0.060	0.055	2.74
0.089	0.068	2.04
0.128	0.086	1.47
0.160	0.097	1.17
0.197	0.112	0.92
0.231	0.125	0.75

TABLE 4: ADSORPTION DATA, III.

Adsorbate: Hydrogen Cyanide ρ(liquid) = 0.687

Adsorbent: ASC Whetlerite with W_{H_2O} = 0.368(g.g^{-1})

P/P_0(HCN)	W (g.g^{-1})	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})
0.0025	0.025	12.48
0.015	0.058	6.04
0.088	0.086	2.06
0.147	0.102	1.27

TABLE 5. ADSORPTION DATA, IV.

Adsorbate: Water $\rho(\text{liquid}) = 0.998$

Adsorbent: BPL Activated Carbon Pre-exposed to $W_{HCN} = 0.33$ cm.^{3}g^{-1}

P/P_0	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})	W (cm.^{3}g^{-1})
0.0348	3.88	0.0028
0.0807	2.18	0.0059
0.146	1.28	0.0067
0.507	0.16	0.0135
0.578	0.10	0.0136
0.836	0.11	0.0177

TABLE 6. ADSORPTION DATA, V.

Adsorbate: Water ρ(liquid) = 0.998

Adsorbent: ASC Whetlerite Pre-exposed to $W_{HCN} = 0.26\ cm.^3 g^{-1}$

P/P_0	$\varepsilon^2 \times 10^{-6}$ $(cal.^2 mole^{-2})$	W $(cm.^3 g^{-1})$
0.0561	2.90	0.00018
0.127	1.49	0.00054
0.335	0.42	0.0031
0.698	0.46	0.0071
0.946	0.0011	0.0104

TABLE 7. ADSORPTION DATA, VI.

Adsorbate: H_2O/HCN Mixture ρ(liquid)HCN = 0.687

ρ(liquid)H_2O = 0.998

Adsorbent: BPL Activated Carbon

$P/P_0(H_2O)$	P/P_0(HCN)	W ($g.g^{-1}$)
0.006	0.0009	0.0131
0.025	0.0037	0.0214
0.065	0.0095	0.0374
0.160	0.023	0.0705
0.321	0.048	0.116
0.670	0.098	0.189
0.816	0.120	0.205

TABLE 8. ADSORPTION DATA, VII.

Adsorbate: H_2O/HCN Mixture

ρ(liquid)HCN = 0.687 ρ(liquid)H_2O = 0.998

Adsorbent: BPL Activated Carbon

P/P_0[*]	W $(g \cdot g^{-1})$
0.73[†]	0.321
0.0027	0.294
0.0273	0.157
0.0997	0.192
0.178	0.236
0.230	0.244

[*] P/P_0 ratio given as a ratio of HCN partial pressure to the saturation vapor pressure of HCN at that temperature unless otherwise noted.

[†] Relative water vapor pressure given as a ratio of the pressure of H_2O vapor (at zero HCN partial pressure) to the saturation vapor pressure of H_2O at that temperature.

TABLE 9. ADSORPTION AND DESORPTION DATA, II.

Adsorbate: Carbon Tetrachloride ρ(liquid) = 1.594

Adsorbent: SP-3 Ultrapure Carbon

Adsorption			Desorption		
P/P_0	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})	W (cm.^{3}g^{-1})	P/P_0	$\varepsilon^2 \times 10^{-6}$ (cal.^{2}mole^{-2})	W (cm.^{3}g^{-1})
0.0244	4.862	0.00251	0.263	0.631	0.00336
0.0311	4.248	0.00266	0.186	0.998	0.00325
0.0368	3.844	0.00275	0.136	1.408	0.00317
0.0527	3.056	0.00290	0.0797	2.261	0.00304
0.0873	2.100	0.00305	0.0428	3.507	0.00283
0.174	1.078	0.00323	0.0261	4.698	0.00257
0.263	0.631	0.00336	0.0191	5.521	0.00229

TABLE 10. SUMMARY OF ADSORPTION PARAMETERS

Adsorbent	Adsorbate	W_0*	k** x 10^8	r^2***
Amoco GX-32	CCl_4	1.31	4.24	0.818
Carbon 2†	CCl_4	1.40	3.41	--
Carbon 4†	CCl_4	1.30	3.47	--
Carbon 6†	CCl_4	1.38	3.50	--
Superactivated Carbon 2 (Lot E 70-90)	CCl_4	0.82	2.74	--
BPL	CCl_4	0.41	1.56	--
SP-3	CCl_4	0.00353	3.01	0.973
H_2O/BPL[a]	HCN	0.142	13.63	0.958
H_2O/BPL[b]	HCN	0.140	6.08	0.925
H_2O/ASC	HCN	0.116	5.30	0.996
HCN/BPL	H_2O	0.015	19.32	0.954
HCN/ASC	H_2O	0.021	81.03	0.788

*Units for W_0: $cm.^3 g^{-1}$

**Units for k: $cal.^{-2} mole^2$

***Coefficient of determination for W_0 & k values obtained from linear regression program

†Data taken from reference 2

[a] 1st BPL sample

[b] 2nd BPL sample

APPENDIX B
FIGURES

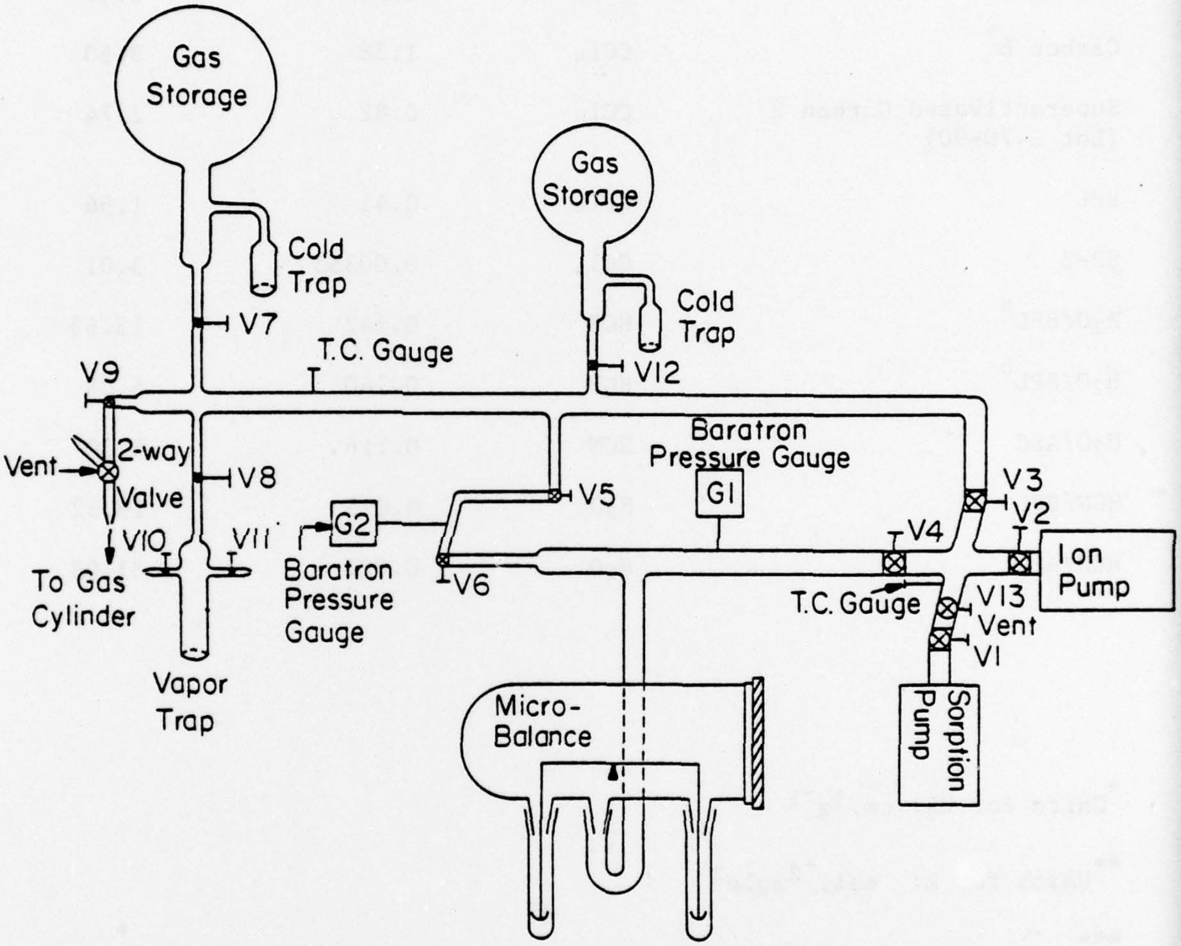

Figure 1. Adsorption Apparatus

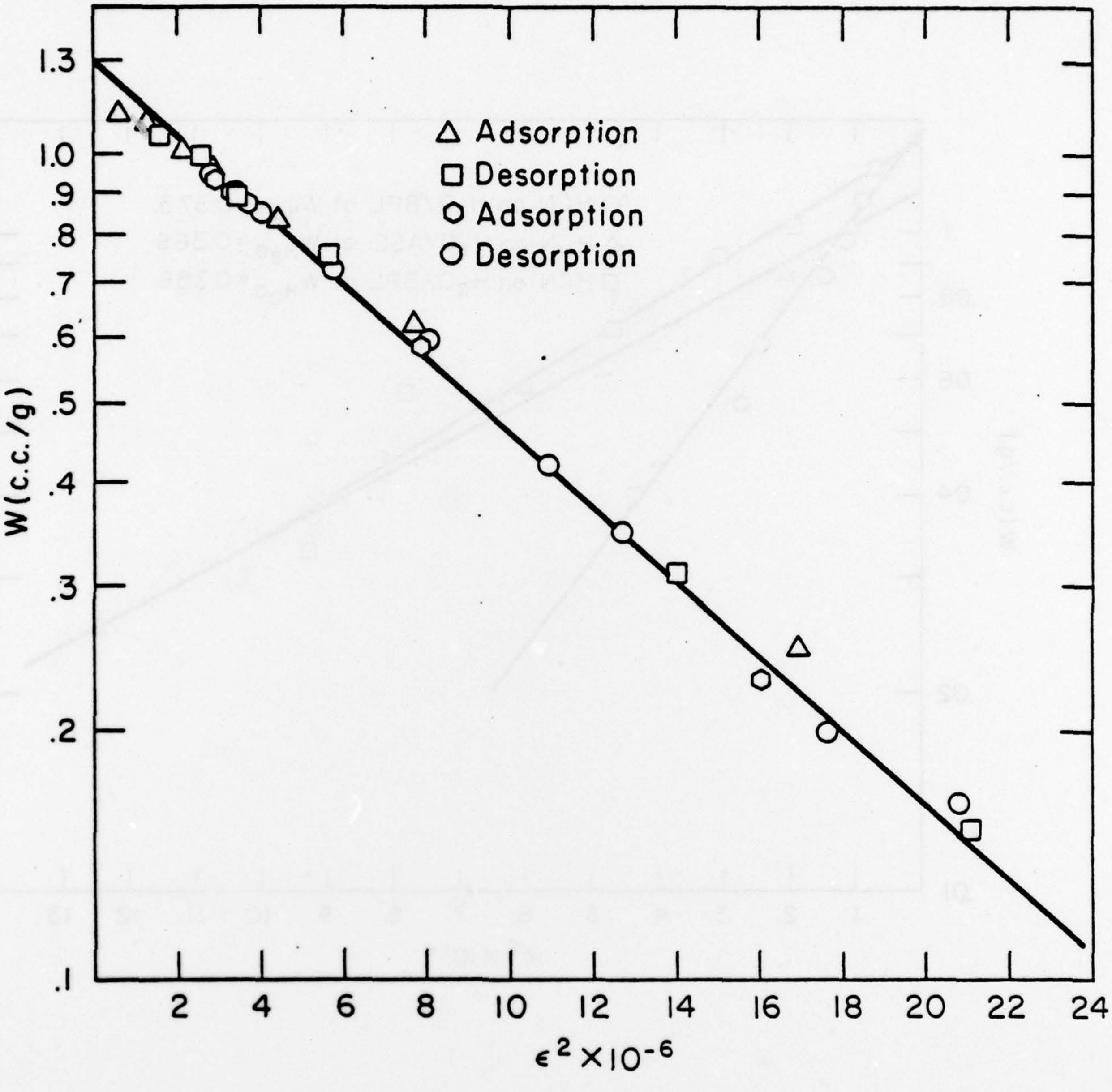

Figure 2. CCl_4 on GX-32 Carbon

APPENDIX B

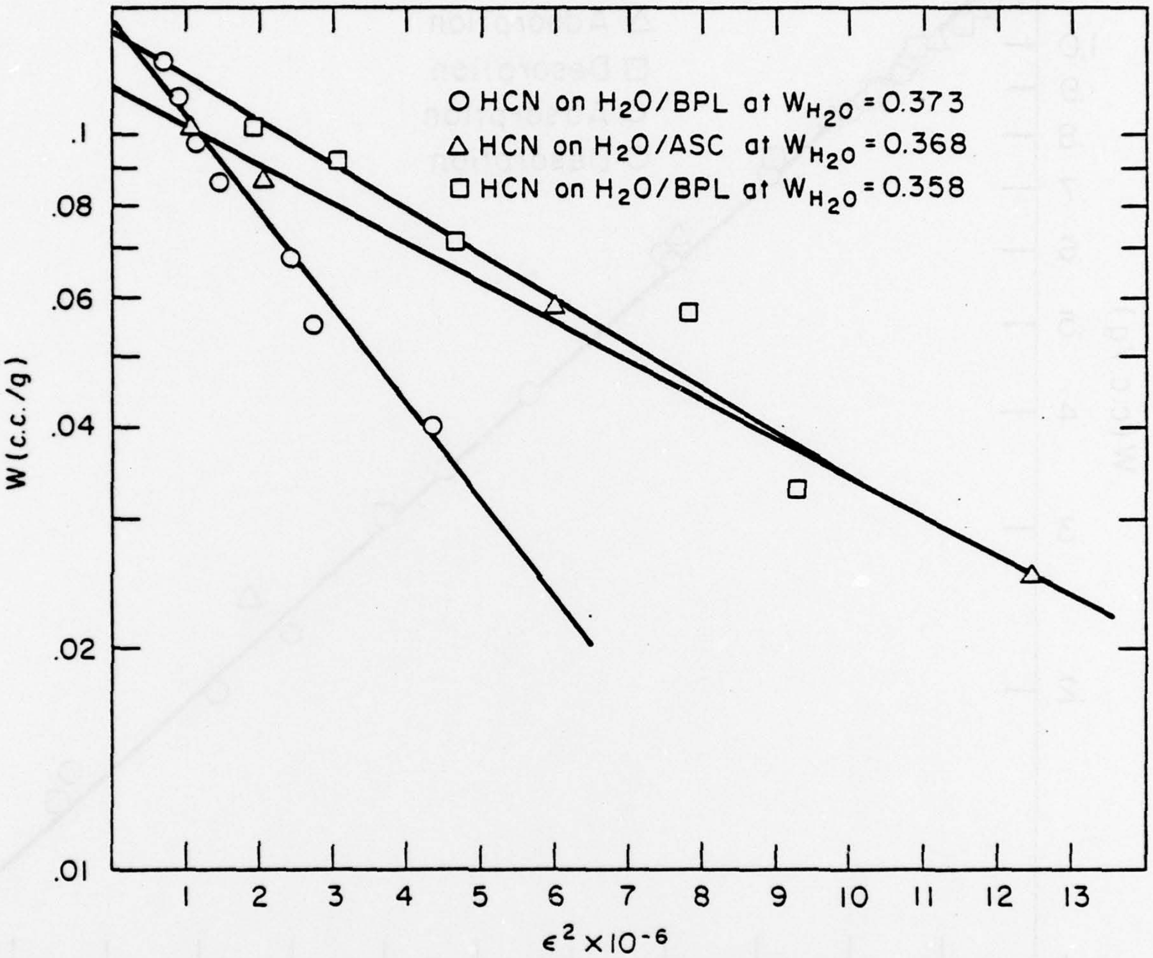

Figure 3. HCN on Carbons Preexposed to H_2O, I.

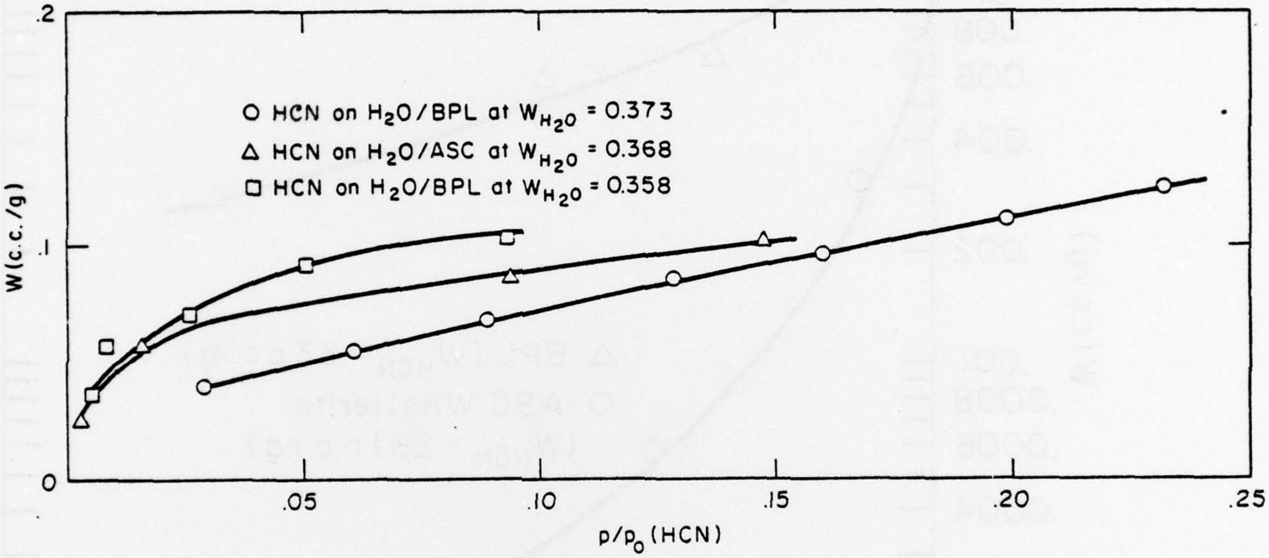

Figure 4. HCN on Carbons Preexposed to H_2O, II

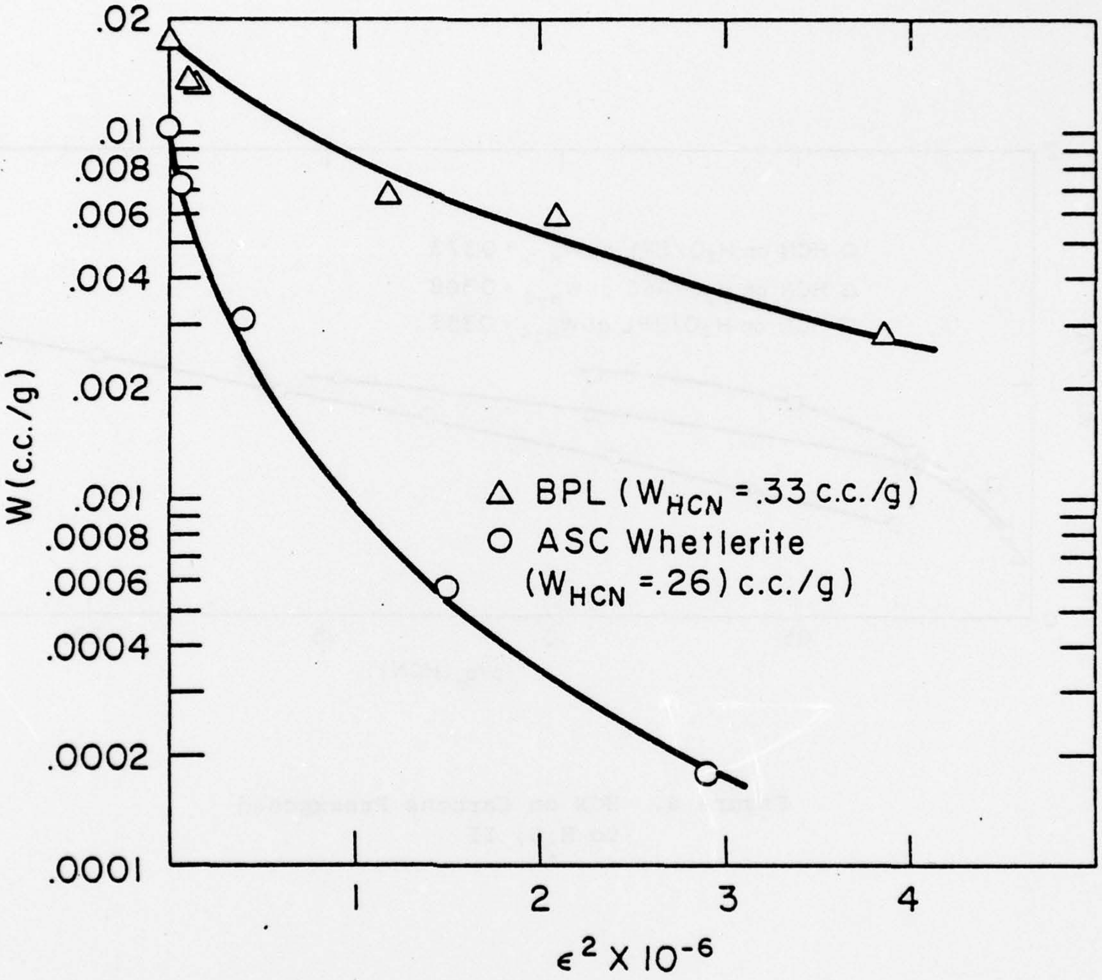

Figure 5. H_2O on Carbons Preexposed to HCN, III

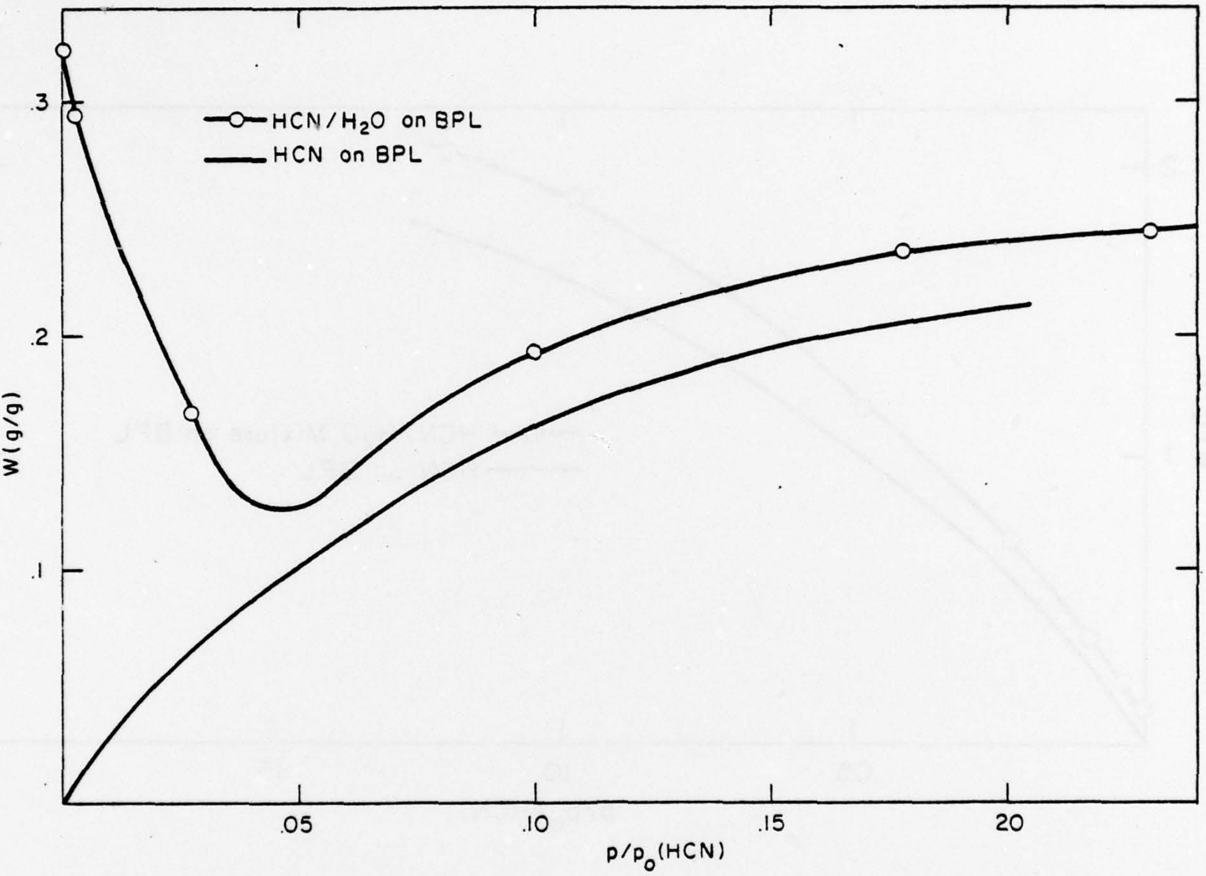

Figure 6. HCN/H_2O Mixtures on BPL

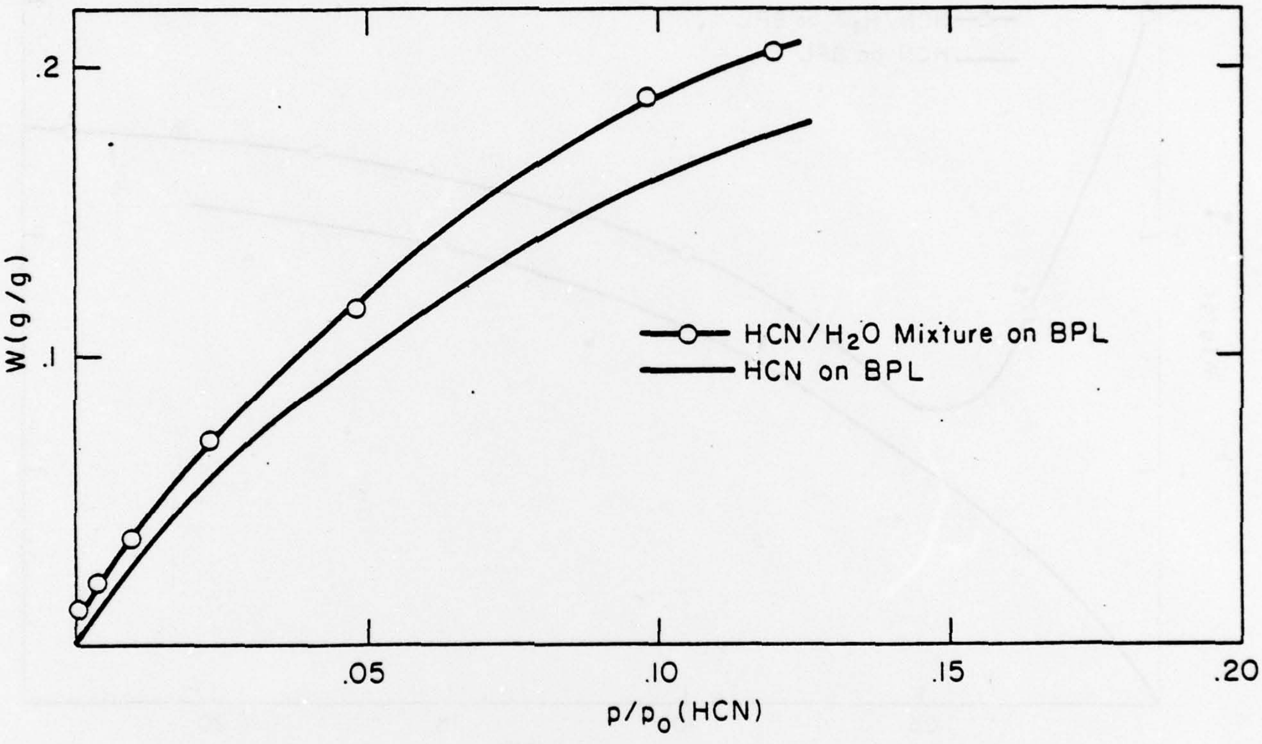

Figure 7. HCN/H_2O Mixtures on BPL
Activated Carbon, II

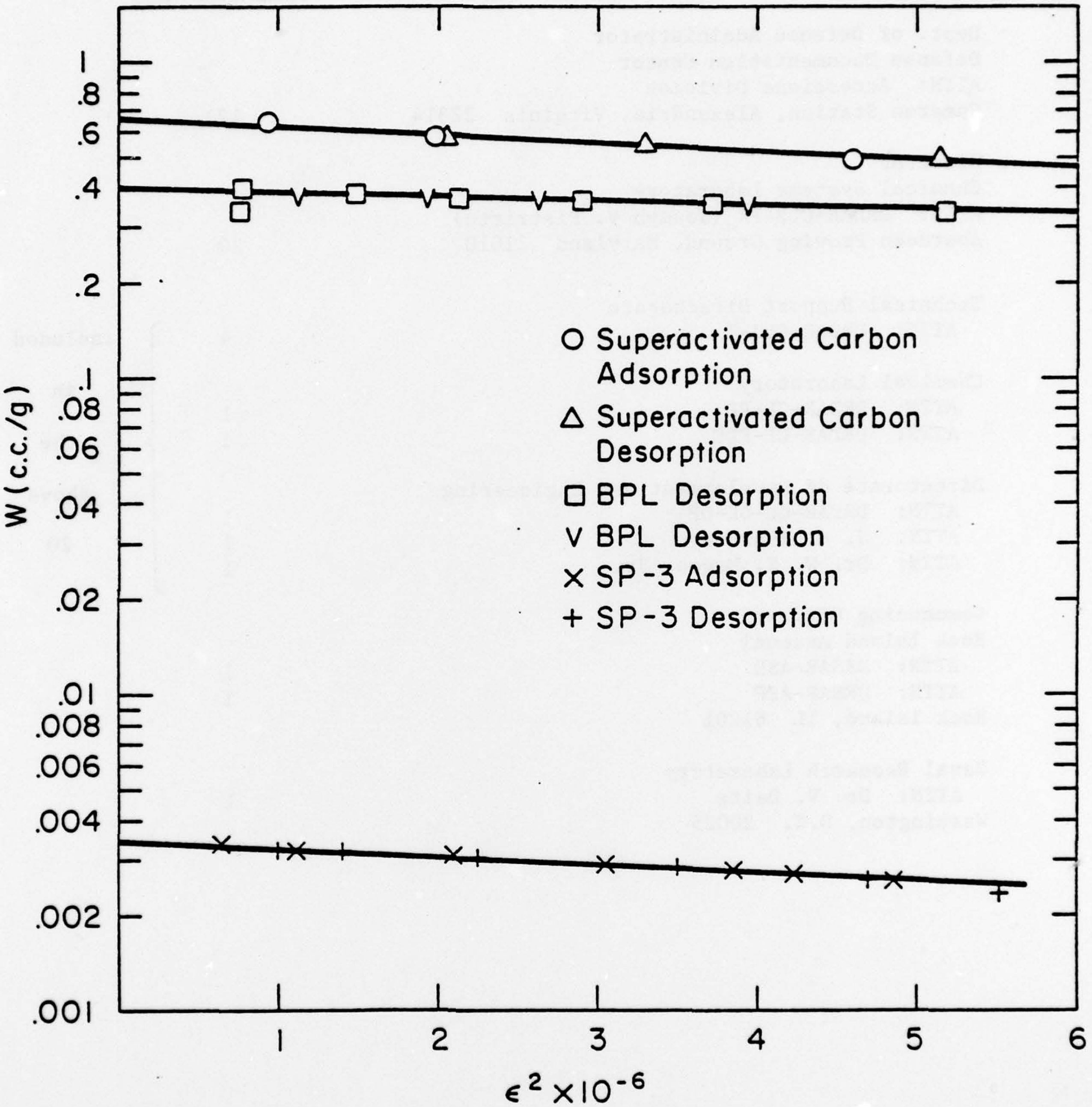

Figure 8. CCl_4 Polanyi Isotherms

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