

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

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QUARTERLY REPORT NO. 2

PROJECT NO. 228-197

STANDARDIZATION OF SURFACE PROPERTIES
OF FINE PARTICLES

By

J. M. DALLAVALLE, CLYDE ORR, JR.,
H. G. BLOCKER and D. JANE BARRETT

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CONTRACT NO. DA-36-039-sc-42588

DEPARTMENT OF THE ARMY PROJECT: 3-99-15-022
SIGNAL CORPS PROJECT: 152B

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OCTOBER 30, 1953

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TABLE OF CONTENTS

	Page
I. ABSTRACT	1
II. PURPOSE	1
III. EXPERIMENTAL WORK.	2
A. Preparation of Finely Divided Nickel	2
B. Heat of Adsorption	3
1. Direct Measurement	3
2. Calculation from Measurement of Adsorption	4
a. Method	4
b. Apparatus.	7
c. Adsorption Procedure	7
IV. DISCUSSION OF RESULTS.	12
A. Finely Divided Nickel.	12
B. Heat of Adsorption	12
1. Direct Measurement	12
2. Calculation from Measurement of Adsorption	12
V. FUTURE WORK.	13
VI. PERSONNEL.	15
VII. APPENDIX	16

LIST OF TABLES

I. CALCULATED HEATS OF ADSORPTION OF NITROGEN BY CHARCOAL AND POWDERED NICKEL.	11
II. HEAT OF ADSORPTION DATA.	17
III. EXPERIMENTAL NITROGEN GAS ADSORPTION DATA.	19

Quarterly Report No. 2, Project No. 228-197

LIST OF FIGURES

	Page
1. Heat of Adsorption as a Function of the Surface of Adsorbent Covered	5
2. Schematic Diagram of Gas Adsorption Apparatus	8
3. Adsorption of Nitrogen by Charcoal at Various Temperatures. . . .	14

I. ABSTRACT

This investigation has as its general purpose an evaluation of the surface properties of finely divided solids; its immediate objective is a study of heat of adsorption, i.e., the heat released upon the adsorption of a gas by a solid.

Two completely different apparatus utilizing different principles were employed. Heat of adsorption measurements for the adsorption of nitrogen gas on charcoal at rather low temperatures indicated general agreement between the two methods. Heats of adsorption of nitrogen gas on a specially prepared nickel powder at low temperatures were also determined and are believed to be of the correct order of magnitude. A spreading of the experimental data, the source of which is not believed to be entirely experimental error, was evident, however. Additional work will be directed toward eliminating this source of error.

II. PURPOSE

The purpose of this investigation is an evaluation of the surface properties of finely divided solid materials so that the standardization of these properties may eventually be attained. In previous investigations under other contracts the evaluation of particle size and of specific surface area has been considered. The heat of adsorption, i.e., the heat liberated when a gas is adsorbed on the surface of a powdered solid, is the property of primary concern in this investigation.

An elucidation of the heat of adsorption phenomena is of great theoretical as well as practical importance, since the result is indicative of the energy states of atoms in surface layers. Much, then, is to be learned from heat of adsorption measurements about the structure of solid surfaces--why some

Quarterly Report No. 2, Project No. 228-197

solids act as catalysts for particular reactions under certain conditions, about chemisorption, about physical adsorption and about other properties.

III. EXPERIMENTAL WORK

A. Preparation of Finely Divided Nickel

Nickel of colloidal dimensions was prepared using high-surface sodium.¹ The apparatus employed was designed so that this sodium could be prepared and then used to produce the nickel in a single reaction vessel. A three-necked flask was used as the vessel. Through the center opening entered a pivoted semicircular glass stirring blade connected to a motor by a glass rod. A mercury-seal bearing prevented gas leaks. One of the side openings of the three-necked flask was used for the introduction of reactants. The third opening in the reaction vessel was equipped with a gas lock and was used for the addition of sodium. A rubber hose attached to this opening was opened and closed with a pinchclamp, which allowed the sodium to be kept in an inert atmosphere before and during addition into the reaction vessel. The reaction vessel was encased by a heating mantle and was provided with a thermocouple with which its temperature was determined.

The nickel preparation was accomplished as follows: the air in the reaction vessel was displaced by nitrogen, and this inert atmosphere was then maintained throughout the process by continuously admitting and venting more of the gas. Since sodium chloride was employed as the inert carrier for the high-surface sodium, 114 grams of dry sodium chloride was placed in the reaction flask through the addition tube. The temperature of the flask was raised to 150°-200° C., and approximately 2.5 grams of sodium was added. The sodium

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1 U. S. Industrial Chemicals Co., High Surface Sodium on Inert Solids. New York, 1953.

Quarterly Report No. 2, Project No. 228-197

was dispersed on the surface of the salt by vigorous mixing of the sodium and the salt. After the sodium was dispersed and while the stirring continued, 10 grams of dried and finely powdered nickel chloride was added slowly. The temperature of the reaction vessel was then raised slowly to 250° C. and its contents were stirred at this temperature for one hour.

The product, consisting of sodium chloride, sodium and nickel metal, was cooled, and methanol was added to decompose the excess sodium. The remaining product was washed with water and decanted several times to remove the salt and was then filtered through a fritted glass disc. Methanol was again added to dissolve the sodium soap. The finely divided nickel was then dried in a vacuum oven. The specific surface area of a product compounded by mixing the products of a number of preparations was measured by low-temperature nitrogen gas adsorption and found to be 25.7 m²/g.

B. Heat of Adsorption

1. Direct Measurement

Only one minor change has been made in the heat of adsorption apparatus which has been described previously.² A 9-mm.-O.D. tube leading directly to the high-vacuum manifold at the top of the apparatus was sealed onto the sample tube. Thus it became possible to evacuate the sample in less time than was formerly required because the gas removed during the evacuation period did not have to pass through the long, coiled tubing surrounding the sample tube.

Additional measurements were made of the heat of adsorption of nitrogen gas on the coconut charcoal also described previously.³ The data resulting

² DallaValle, J. M., Orr, Clyde, Jr., and Blocker, H. G., Research on Surface Properties of Fine Particles. Final Report, Project No. 181-119, Contract No. DA-36-039-sc-5411, Georgia Institute of Technology, Atlanta, April 30, 1953.

³ Ibid.

from these measurements are presented in the appendix in Table II. The results calculated from them and from previous determinations are presented in Figure 1. Since the heat of adsorption measured by this method is the average heat liberated during one increment of gas admitted to the sample, the data were plotted as short lines whose length was estimated by the amount of gas adsorbed in each increment.

2. Calculation from Adsorption

a. Method. It is possible to calculate differential heats of adsorption by means of an equation developed by Clapeyron and later extended by Clausius. The form of this equation used by Hansen⁴ is

$$\left(\frac{d \ln p}{dT} \right)_V = \frac{H_d}{RT^2} \quad (1)$$

where p is the equilibrium pressure, V is the volume adsorbed at the temperature T and the pressure p , H_d is the differential heat of adsorption, and R is the gas constant. Substituting dp/p for $d \ln p$ and solving for H_d , the heat of adsorption is

$$H_d = \frac{RT^2}{p} \left(\frac{dp}{dT} \right)_V \quad (2)$$

Since the derivative must be evaluated with the volume adsorbed constant, it is necessary to obtain an adsorption isostere for each condition at which the heat of adsorption is to be determined. It was decided to determine these isosteres from several isotherms run at different temperatures.

Two procedures were employed in the treatment of the data. In the first, several adsorption isotherms for different temperatures were plotted (V as ordinate versus p as abscissa) on coordinate paper. The pressure on each of

4 Hansen, R. S., "Calculations of Heats of Adsorption from Adsorption Isosteres." J. Phys. Chem. 54, 411-15 (1950).

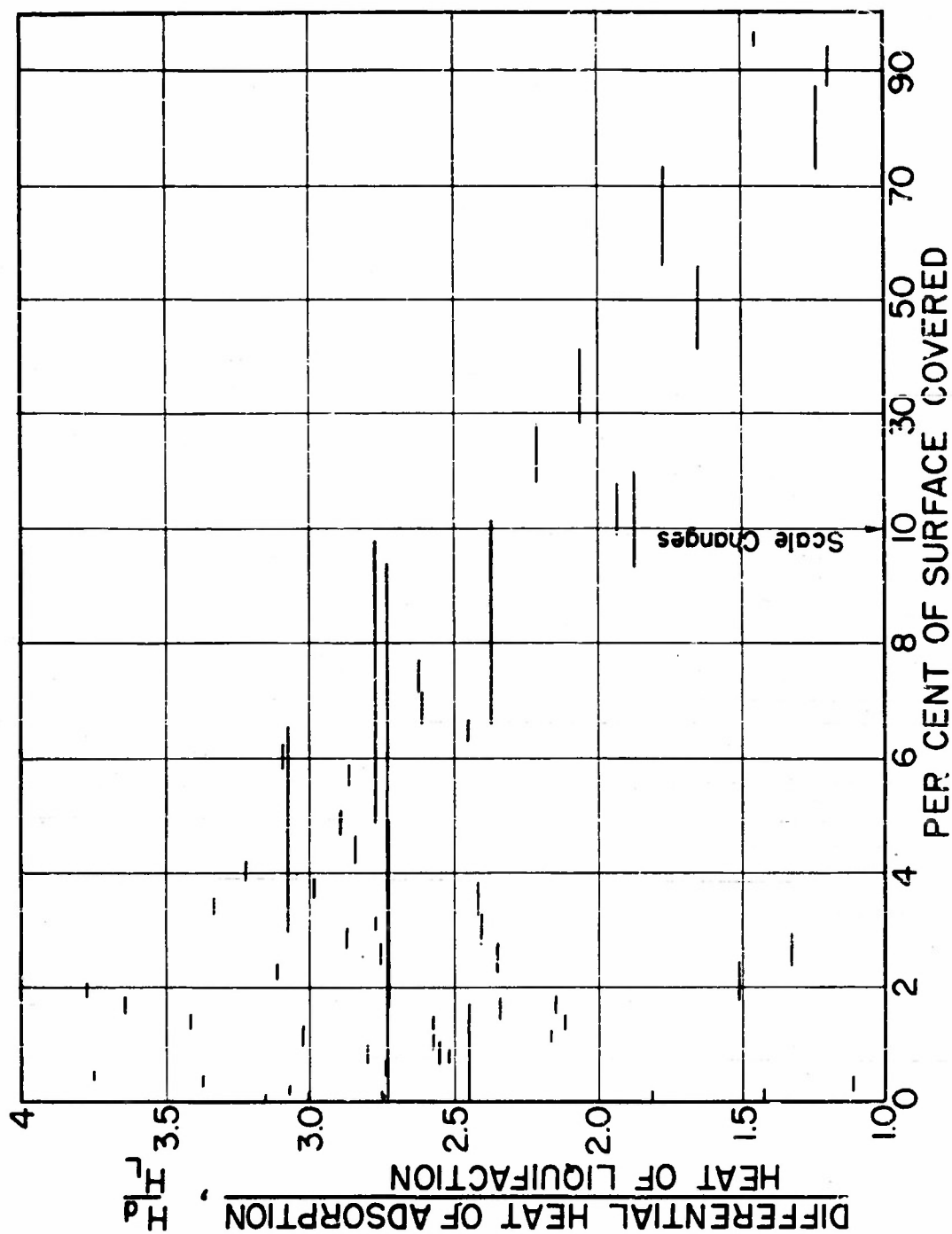


Figure 1.. Heat of Adsorption as a Function of the Surface of Adsorbent Covered.

these isotherms was then determined for particular volumes of adsorbed gas, and these pressures were plotted against the corresponding temperatures of the isotherms. The resulting curve was an adsorption isostere from which the pressure, p , and the derivative $(dp/dT)_V$ were evaluated at particular temperatures. By means of equation 2, the differential heat of adsorption was then evaluated for a particular volume of gas adsorbed. The procedure was repeated at other volumes of adsorbed gas.

The second procedure required that the isostere be fitted by an equation derived from equation 2. This equation may be written

$$\frac{dp}{p} = \frac{H_d}{RT^2} dT \quad (3)$$

If the assumption is made that H_d is independent of T over the narrow limits of T employed in these measurements, equation 3 may be integrated giving

$$\ln p = C - \frac{H_d}{RT} \quad (4)$$

where C is a constant of integration. The value of H_d at each volume of adsorbed gas was determined by the method of least squares. The two simultaneous equations required to evaluate the constants C and H_d in equation 4 are

$$\begin{aligned} \sum \ln p &= Cn - \frac{H_d}{R} \sum \frac{1}{T} \\ \sum \frac{1}{T} \ln p &= C \sum \frac{1}{T} - \frac{H_d}{R} \sum \frac{1}{T^2} \end{aligned} \quad (5)$$

where n is the number of isotherms employed. Pressure and temperature measurements were taken directly from adsorption isotherms and equations 5 were solved for C and H_d/R , from which the differential heat of adsorption, H_d , was determined.

Quarterly Report No. 2, Project No. 228-197

b. Apparatus. In order to obtain additional adsorption isotherms without interfering with the experimental determination of heats of adsorption, it was decided to construct a second low-temperature adsorption apparatus. A schematic drawing of the new apparatus is given in Figure 2. Three sample tubes were provided, each with three-way stopcocks, so that two samples could be evacuated while adsorption measurements were being made on the other sample.

The new adsorption apparatus was constructed with Pyrex glass tubing and standard-taper stopcocks with interchangeable plugs. Some difficulty was encountered with leaking stopcocks during the first attempts to use the equipment. The stopcocks were then lapped in with No. 400 and with No. 600 silicon carbide grit. The difficulty was greatly reduced.

The volumes of the connective tubing and of the sample tubes were determined by calibration with helium gas. Baths of liquid nitrogen and liquid nitrogen and oxygen mixtures were employed to cool the samples. The temperature of each bath was measured with an argon-vapor-pressure thermometer while the temperature of the burette space was measured with a mercury-in-glass-type thermometer. Pressures less than 2 mm. Hg were measured with a McLeod gauge provided with two different-sized capillaries mounted end-to-end. Pressures as low as 0.001 micron and as high as 2,000 microns can be measured with the gauge. A mercury manometer was used for measuring pressures from 2 to 800 mm. Hg.

c. Adsorption Procedure. The volume of gas adsorbed from each increment of gas admitted to the adsorption space was determined from a material balance of the gas in the space before and after the adsorption had taken place. Thus, referring to Figure 2,

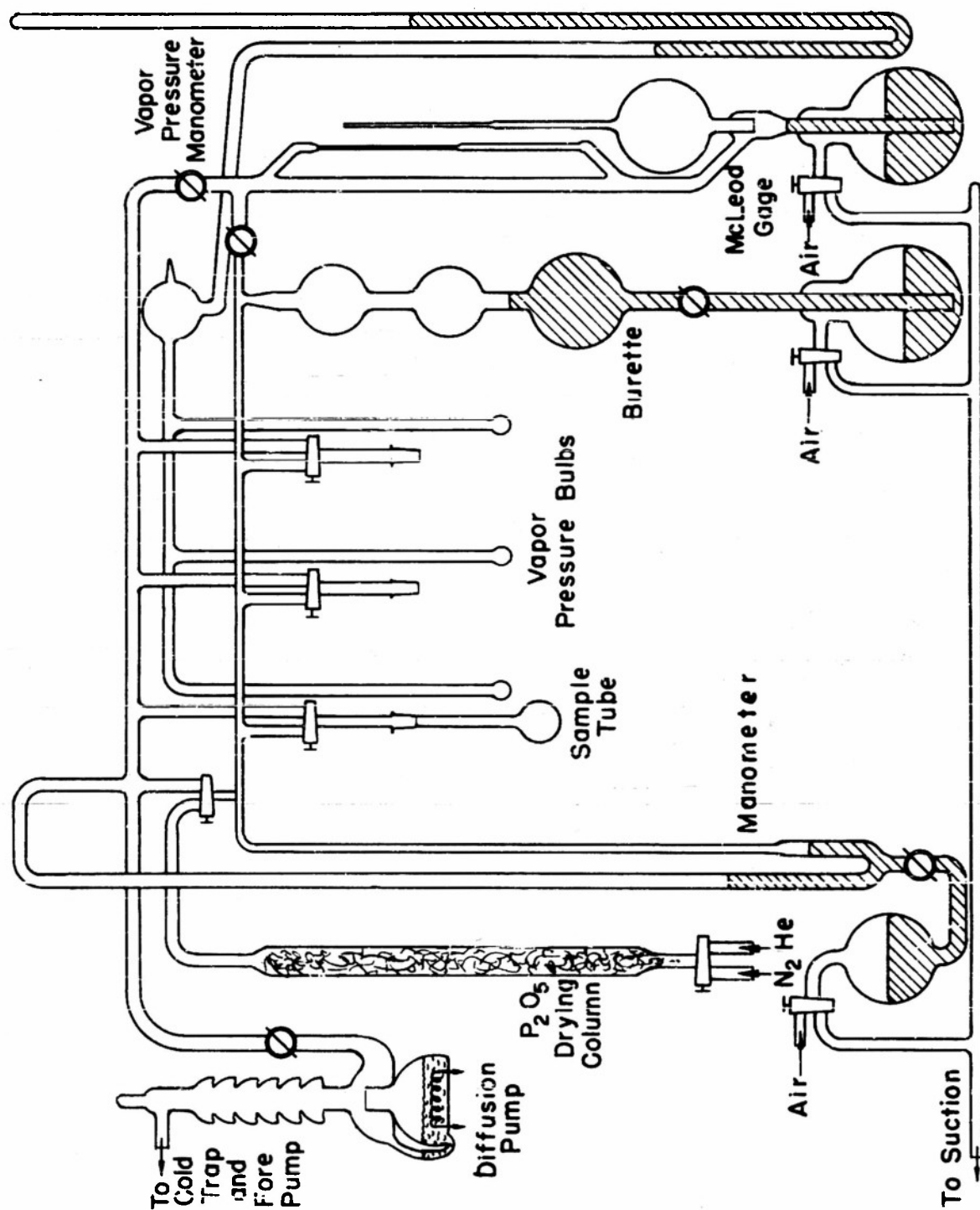


Figure 2. Schematic Diagram of Gas Apparatus.

$$\frac{p_1 V_t}{T_b} + \frac{p_0 V_s (1 + \alpha p_0)}{T_s'} = \frac{p_2 V_t}{T_b} + \frac{p_2 V_s (1 + \alpha p_2)}{T_s'} + V_a \frac{760}{273} \quad (6)$$

where p_0 = initial pressure in sample tube, mm. Hg,

p_1 = initial pressure in burette space, mm. Hg,

p_2 = final pressure in burette space and sample tube, mm. Hg,

V_t = volume of burettes and connective tubing, ml.,

V_s = volume of sample tube, ml.,

V_a = volume of gas adsorbed, cc. at S.T.P.,

T_b = temperature of burettes, °K.,

T_s = temperature of liquid nitrogen bath, °K.,

T_s' = average temperature of sample tube, °K.,

α = correction for deviation of nitrogen from perfect gas

law = 6.6×10^{-5} (mm. Hg) $^{-1}$.

Solving for the volume adsorbed,

$$V_a = \frac{273}{760} \left[\frac{V_t}{T_b} (p_1 - p_2) - \frac{V_s}{T_s'} (p_2 - p_0) - \alpha \frac{V_s}{T_s'} (p_2^2 - p_0^2) \right] \quad (7)$$

Samples were prepared for adsorption in the usual manner, i.e., by drying at 110° C. and cooling in a desiccator before weighing. They were prepared for adsorption by heating at 250°-300° C. while being evacuated to pressures of the order of 0.1 micron. The volume of the sample tube was determined with helium with the sample tube at room temperature.

Since the glass tubing between the sample stopcock and the sample is neither at the temperature of the sample nor at that of the surroundings, it is necessary to establish an effective or average, temperature for the sample tube. This was done by placing the liquid nitrogen bath about the sample tube in its normal position and then removing the bath while the sample tube still

Quarterly Report No. 2, Project No. 228-197

contained the helium used in determining its volume. The average temperature was then found from material balances on the gas in the system when the bath was in place and after the bath had been removed, assuming helium to be not measurably adsorbed at liquid nitrogen temperature. This procedure was used exclusively in the case of the nickel powder.

There is some indication that helium may be adsorbed to an appreciable extent by some high-surface carbonaceous materials at the temperature of liquid nitrogen.⁵ Indeed, this seems to be the case with the charcoal used in the measurements reported herein, because temperatures lower than that of the liquid nitrogen bath are obtained by the procedure outlined above. For this reason, the average temperature of the tube in the case of charcoal was estimated from the temperatures obtained with nickel as the adsorbent.

Changes in the temperature at which adsorption measurements were made were brought about by changing the percentages of oxygen and nitrogen in the liquid bath. Commercial liquid nitrogen gave the lowest temperature employed. Progressively higher temperatures were obtained by bubbling oxygen into this nearly pure nitrogen until the desired temperature was attained.

Isotherms were determined for the adsorption of nitrogen by colloidal nickel prepared by the high-surface-sodium method and for the acid-washed coconut charcoal, the preparation of which was described in a previous report.⁶ These data are summarized in Table III in the appendix. Calculated heats of adsorption for various per cent coverages of the adsorbent surface are given in Table I.

⁵ Beebe, R. A., Millard, B., and Cynarski, J., "Heats of Adsorption of Nitrogen and Argon on Porous and non-Porous Carbon Adsorbents at -195°." J. Am. Chem. Soc. 75, 839-45 (1953).

⁶ DallaValle, et al., op. cit.

Quarterly Report No. 2, Project No. 228-197

TABLE I.

CALCULATED HEATS OF ADSORPTION OF NITROGEN BY
CHARCOAL AND POWDERED NICKEL

<u>Material</u>	<u>T</u> <u>(°K.)</u>	<u>Surface</u> <u>Coverage</u> <u>(Per Cent)</u>	<u>H_d/H_L</u>
Powdered Nickel	79	46.4	2.75
Powdered Nickel	79	55.7	3.10
Powdered Nickel	79	64.9	2.02
Powdered Nickel	79	74.2	1.71
Powdered Nickel	79	83.5	2.17
Powdered Nickel	79	92.8	1.86
Powdered Nickel	79	102.0	1.79
Powdered Nickel	79	111.3	1.95
Powdered Nickel	79	120.5	1.80
Powdered Nickel	79	129.9	1.63
Charcoal	77-83	11.8	2.98
Charcoal	77-83	23.7	2.32
Charcoal	77-83	35.5	2.80
Charcoal	77-83	47.3	2.97
Charcoal	77-83	59.2	3.77
Charcoal	77-83	71.0	3.08
Charcoal	77-83	82.8	2.05
Charcoal	77-83	88.8	0.95

IV. DISCUSSION OF RESULTS

A. Finely Divided Nickel

The specific surface area of the nickel produced by the sodium-reduction method proved not to be as great as had been hoped. This, of course, made the material less attractive as an adsorbent than it might have been, but the nickel has been used nevertheless. In some respects, the results obtained with nickel appear to be more consistent than those with charcoal.

B. Heat of Adsorption

1. Direct Measurement

Figure 1 permits visual evaluation of all of the heat of adsorption data for charcoal obtained by direct measurement. Considerable spread in the results is obvious; the reason for the spread is not as obvious. While some of the discrepancies are undoubtedly due to experimental error, it is believed that this is not the only source, because the experimental data, when plotted as shown in Figure 9 of a previous report,⁷ consistently gave very smooth curves. The temperature to which the adsorbent was heated, the duration of the heating and the pressure of the evacuation are all believed to affect the course of the adsorption. Sufficient data to determine the effect of each of these factors have not been obtained.

2. Calculation from Measurement of Adsorption

The heats of adsorption obtained from adsorption isotherms as functions of the per cent of the adsorbent surface covered are presented in Table I. In general, the results for charcoal agree with those obtained by direct measurement, and those for nickel are of the order of magnitude expected.

⁷ DallaValle, et al., op. cit.

Quarterly Report No. 2, Project No. 228-197

The charcoal results were obtained by the least-squares method outlined above; the nickel results were obtained by the simpler graphical analysis.

It was necessary to apply the mathematical analysis to the data for charcoal because of the scatter of the isosteric data. These data were scattered because they originated from differences in the adsorption isotherms which were quite small. As in the case of direct measurement of heat of adsorption, it is believed that the conditions of evacuation have some effect on heats of adsorption. It is possible that equilibrium pressures were not reached for some points, since the pressure change with time becomes quite low for charcoal. These and other minor inaccuracies, while entirely negligible for most purposes, may prove to be quite important in these measurements. The adsorption data did show small irregularities, but a plot of these data in accordance with the BET equation,⁸ Figure 3, showed them to be quite consistent by previous standards.

V. FUTURE WORK

Contemplated studies will be concerned with the effects of heating and evacuating conditions on heats of adsorption. This problem will be approached by exposing several samples to as nearly identical conditions as possible and then measuring their adsorption characteristics at different temperatures. In general, experiments will be designed to eliminate as many variables as possible in order to obtain consistent data. Larger temperature differences will be employed in obtaining adsorption isotherms intended for use with the Clapeyron equation. Attempts will be continued to obtain heat of adsorption

⁸ Brunauer, S., Emmett, P.H., and Teller, E., "Adsorption of Gases in Multimolecular Layers." J. Am. Chem. Soc. 60, 309-19 (1938).

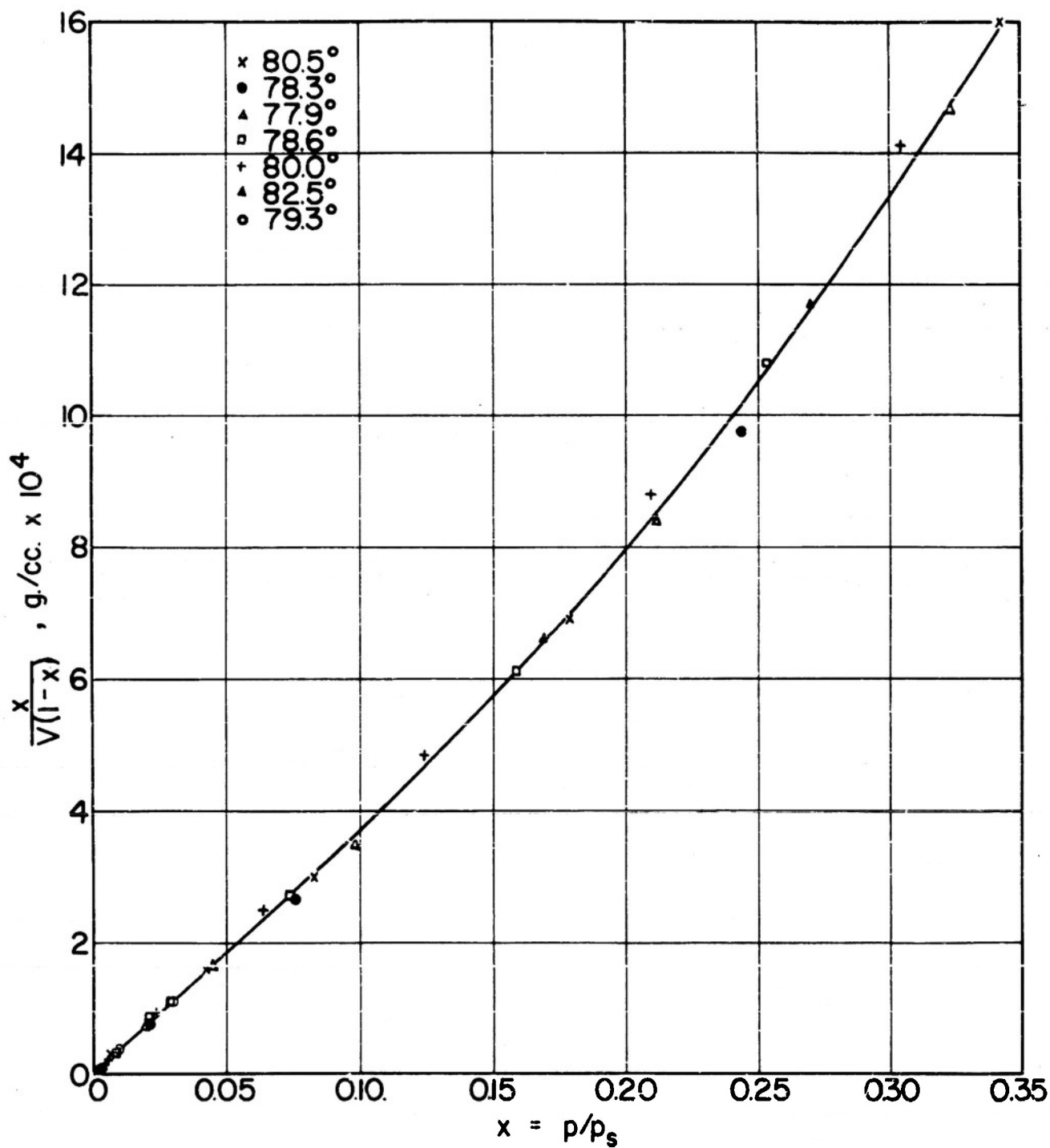


Figure 3. Adsorption of Nitrogen by Charcoal at Various Temperatures.

Quarterly Report No. 2, Project No. 228-197

data when very small fractions of the adsorbent's surface are covered with adsorbed molecules.

VI. PERSONNEL

The technical personnel who worked on this project and the percentage of working time each devoted to the project during the period covered by this report are shown below:

<u>Name</u>	<u>Title</u>	<u>Percentage of Working Time Devoted to Project</u>
J. M. DallaValle	Project Director (Until October 15, 1953)	36
Clyde Orr, Jr.	Research Engineer (Until October 15, 1953)	39
H. G. Blocker	Research Engineer	94
Miss Jane Barrett	Research Assistant	44

Respectfully submitted:

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D. Jane Barrett
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Approved:

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Herschel H. Cudd, Director
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Quarterly Report No. 2, Project No. 228-197

VII. APPENDIX

Quarterly Report No. 2, Project No. 228-197

TABLE II
HEAT OF ADSORPTION DATA

P_1 (mm. Hg)	p (mm. Hg)	V (cc./g.)	H_d $\left(\frac{\text{Cal.}}{\text{g.}}\right)$	T_b (°K)	T_s (°K)	T_e (°K)	Constants
<u>Charcoal:*</u>							
29.8	0.0	0.273	---	303	78.50	287	$w = 8.456 \text{ g.}$ $V_t = 65.2 \text{ cc.}$ $V_e = 3.0 \text{ cc.}$ $V_s = 68.0 \text{ cc.}$
26.0	0.0	0.511	0.165	303	78.50	287	
38.2	0.0	0.861	1.286	302	78.85	287	
47.3	0.0	1.295	1.067	302	78.85	287	
47.3	0.0	1.729	0.226	302	78.85	287	
67.7	0.0	2.348	0.165	303	78.93	287	
71.3	0.0	3.000	0.152	303	79.16	287	
64.0	0.0	3.585	0.155	303	79.16	287	
71.0	0.0	4.234	0.130	303	79.16	287	
62.9	0.0	4.809	0.155	303	79.16	287	
117.2	0.0	5.881	0.141	303	79.16	287	
205.8	0.0	7.763	0.091	303	79.3	287	
222.0	0.0	9.793	0.080	303	79.3	287	
<u>Coconut Charcoal:**</u>							
50.0	0.0	0.458	0.1892	302	77.9	286	$w = 8.456 \text{ g.}$ $V_t = 65.2 \text{ cc.}$ $V_e = 3 \text{ cc.}$ $V_s = 67.6 \text{ cc.}$
55.4	0.0	0.966	0.1846	302	77.9	286	
61.2	0.0	1.527	0.2023	302	77.9	286	
90.2	0.0	2.354	0.1648	302	77.9	286	
94.9	0.0	3.224	0.1685	302	77.9	286	
115.0	0.0	4.279	0.1815	302	77.9	286	
93.6	0.0	5.137	0.2048	302	78.0	286	
108.8	0.0	6.135	0.2187	302	78.0	286	
107.7	0.0	7.123	0.1667	302	78.0	286	
107.1	0.0	8.105	0.1870	302	78.0	286	
112.9	0.0	9.140	0.1660	302	78.0	286	
104.8	0.0	10.101	0.1722	302	78.0	286	
105.8	0.0	11.071	0.1665	302	78.0	286	
111.8	0.0	12.096	0.1999	302	78.1	286	
106.9	0.0	13.076	0.1793	302	78.1	286	
116.0	0.0	14.139	0.1935	302	78.1	286	
160.0	0.0	15.606	0.1708	302	78.1	286	
158.5	0.0	17.055	0.1735	302	78.1	286	
152.0	0.0	18.457	---	302	78.1	286	

(Continued)

TABLE II (Continued)

HEAT OF ADSORPTION DATA

P_1 (mm. Hg)	P (mm. Hg)	V (cc./g.)	H_d $\left(\frac{\text{Cal.}}{\text{g.}}\right)$	T_b (°K)	T_s (°K)	T_e (°K)	Constants
<u>Coconut Charcoal (Continued):</u>							
150.0	0.0	19.833	0.1718	302	78.1	286	
153.4	0.0	21.240	0.1859	302	78.1	286	
132.8	0.0	22.458	0.1475	302	78.1	286	
184.8	0.0	24.153	0.1571	302	78.1	286	
133.0	0.0	25.373	0.1576	302	78.1	286	
* Acid-washed, evacuated 24 hours at 450° C. to 0.05μ pressure.							
** Acid-washed, evacuated 48 hours at 400° C. to 0.01μ pressure.							

Quarterly Report No. 2, Project No. 228-197

TABLE III

EXPERIMENTAL NITROGEN GAS ADSORPTION DATA

P_1 (mm. Hg)	p (mm. Hg)	V (cc./g.)	Constants*
<u>1. Coconut Charcoal:**</u>			
143.2	0.00	8.95	w = 2.092 g.
197.1	0.00	21.27	T _b = 304.3° K
223.0	0.00	35.20	T _s = 79.3° K
270.3	0.00	52.09	T _s = 95.0° K
234.6	0.00	66.74	V _s = 9.9 cc.
309.5	0.00	86.08	V _t = 110.9 cc.,
324.3	0.00	106.34	135.8 cc.
415.7	0.00	132.31	
443.0	0.10	159.98	
318.2	0.35	184.32	
348.7	1.09	210.92	
360.9	3.3	233.24	
326.0	9.4	252.91	
433.9	28.7	277.88	
<u>2. Coconut Charcoal:</u>			
273.9	0.050	36.4	w = 2.092 g.
204.1	0.071	63.5	T _b = 304.5° K
208.8	0.095	91.3	T _s = 80.5° K
214.0	0.124	119.8	T _s = 95.0° K
234.8	0.197	151.5	V _s = 10.0 cc.
227.7	0.43	181.9	V _t = 235.8 cc.,
239.0	1.7	211.0	210.9 cc.
233.0	6.5	238.0	
250.0	22.1	264.9	
207.1	46.3	283.7	
240.9	90.2	300.9	
336.8	195.5	315.9	
471.3	373.0	324.6	

(Continued)

TABLE III (Continued)
EXPERIMENTAL NITROGEN GAS ADSORPTION DATA

P_1 (mm. Hg)	p (mm. Hg)	V (cc./g.)	Constants*
<u>3. Coconut Charcoal:</u>			
208.2	0.0018	16.15	$w = 2.092 \text{ g.}$ $T_b = 301.0^\circ \text{ K}$ $T_s = 78.3^\circ \text{ K}$ $T_{s'} = 95.0^\circ \text{ K}$ $V_s = 10.0 \text{ cc.}$ $V_t = 235.9 \text{ cc.},$ 212.5 cc.
236.5	0.0052	47.98	
241.2	0.0130	80.45	
250.0	0.0238	114.20	
281.5	0.064	152.20	
346.0	0.034	198.87	
318.3	2.8	241.46	
314.6	18.5	277.10	
337.2	64.9	309.29	
398.9	209.8	329.58	
454.1	370.5	336.71	
<u>4. Coconut Charcoal:</u>			
206.1	0.0024	27.73	$w = 2.029 \text{ g.}$ $T_b = 302.5^\circ \text{ K}$ $T_s = 79.2^\circ \text{ K}$ $T_{s'} = 95.0^\circ \text{ K}$ $V_s = 10.0 \text{ cc.}$ $V_t = 235.8 \text{ cc.}$
211.2	0.0055	56.01	
226.7	0.0143	86.37	
237.0	0.032	118.10	
243.4	0.089	150.69	
249.4	0.280	184.04	
272.0	1.80	220.19	
<u>5. Coconut Charcoal:</u>			
201.4	0.0068	26.754	$w = 2.1259 \text{ g.}$ $T_b = 300^\circ \text{ K}$ $T_s = 78.6^\circ \text{ K}$ $T_{s'} = 95^\circ \text{ K}$ $V_s = 9.8 \text{ cc.}$ $V_t = 235.8 \text{ cc.},$ 210.9 cc.
202.8	0.0145	53.694	
211.4	0.0280	81.776	
218.4	0.0480	110.783	
222.2	0.0950	140.291	
230.0	0.270	170.802	
225.8	1.26	200.637	
259.2	7.0	234.037	
255.5	25.4	264.283	
273.2	66.1	291.083	
278.4	141.5	307.942	
290.0	226.0	314.941	

(Continued)

Quarterly Report No. 2, Project No. 226-197

TABLE III (Continued)

EXPERIMENTAL NITROGEN GAS ADSORPTION DATA

P_1 (mm. Hg)	P (mm. Hg)	V (cc./g.)	Constants*
<u>6. Coconut Charcoal:</u>			
246.0	0.0030	32.99	$w = 2.092 \text{ g.}$
248.9	0.0080	66.37	$T_b = 302^\circ \text{ K,}$
249.0	0.0180	99.76	301.5° K
262.7	0.0450	134.99	$T_s = 77.9^\circ \text{ K,}$
245.0	0.113	167.83	78.0° K
298.7	0.620	207.79	$T_{s'} = 95^\circ \text{ K}$
			$V_s = 10.0 \text{ cc.}$
301.6	6.0	247.33	$V_t = 235.8 \text{ cc.,}$
200.5	15.7	269.25	210.9 cc.
215.0	36.8	289.98	
241.8	80.2	308.55	
294.0	174.0	321.24	
325.8	266.3	326.67	
392.0	350.3	330.10	
<u>7. Coconut Charcoal:</u>			
205.5	0.0140	27.444	$w = 2.1253 \text{ g.}$
230.8	0.039	58.267	$T_b = 298.5^\circ \text{ K,}$
228.5	0.067	88.769	301° K
261.3	0.150	123.651	$T_s = 80.0^\circ \text{ K,}$
264.0	0.245	158.879	80.3° K
277.5	1.065	191.605	$T_{s'} = 95^\circ \text{ K}$
			$V_s = 10.1 \text{ cc.}$
279.9	5.9	223.974	$V_t = 235.8 \text{ cc.,}$
265.3	24.2	252.204	210.9 cc.
271.1	64.9	275.895	
269.9	125.0	291.967	
294.7	207.2	300.825	
368.0	307.0	306.200	

(Continued)

Quarterly Report No. 2, Project No. 228-197

TABLE III (Continued)

EXPERIMENTAL NITROGEN GAS ADSORPTION DATA

<u>P₁</u> (mm. Hg)	<u>P</u> (mm. Hg)	<u>V</u> (cc./g.)	<u>Constants*</u>
<u>8. Coconut Charcoal:</u>			
215.5	0.0026	28.64	w = 2.1292 g.
315.0	0.0171	70.56	T _b = 299° K,
342.5	0.070	116.14	298° K
375.0	0.425	166.04	T _g = 82.5° K
359.7	4.0	213.27	T _g ⁱ = 95° K
			V _g = 17.12 cc.
296.1	22.0	249.20	V _t = 235.8 cc.,
368.0	81.1	285.57	210.9 cc.
366.5	181.5	307.19	
388.4	289.6	316.99	
<u>9. Nickel Powder:***</u>			
54.9	0.042	1.190	w = 7.44 g.
90.7	2.7	3.037	T _b = 302.5° K
			T _g = 77.9° K
93.2	6.0	3.810	T _g ⁱ = 81.1° K
124.2	13.9	4.703	V _g = 38.1 cc.
159.0	31.2	5.552	V _t = 135.8 cc.,
193.9	63.4	6.085	60.9 cc.
241.2	97.5	6.701	
274.5	134.1	7.223	
316.2	171.5	7.764	
356.7	208.2	8.353	
378.0	243.2	8.856	
<u>10. Nickel Powder:</u>			
83.9	0.400	1.816	w = 7.44 g.
94.5	6.1	3.631	T _b = 300.3° K
			T _g = 80.0° K
107.1	12.7	4.421	T _g ⁱ = 83.0° K
125.0	25.6	5.131	V _g = 38.1 cc.
160.0	48.0	5.770	V _t = 135.8 cc.,
191.7	76.9	6.302	60.9 cc.
246.2	112.6	6.815	
302.0	154.1	7.464	
357.0	197.2	8.132	

(Continued)

Quarterly Report No. 2, Project No. 228-197

TABLE III (Continued)
EXPERIMENTAL NITROGEN GAS ADSORPTION DATA

P_1 (mm. Hg)	p (mm. Hg)	V (cc./g.)	Constants*
<u>11. Nickel Powder:</u>			
18.9	0.0105	0.407	$w = 7.440 \text{ g.}$ $T_b = 300^\circ \text{ K,}$ 301° K, 302° K $T_{s1} = 83.0^\circ \text{ K}$ $T_{s2} = 86.7^\circ \text{ K}$ $V_s = 38.2 \text{ cc.}$ $V_t = 135.8 \text{ cc.,}$ 60.9 cc. 60.9 cc.
27.0	0.0260	0.985	
34.9	2.2	1.269	
50.1	5.2	1.645	
72.0	11.5	2.104	
95.8	23.0	2.572	
111.0	39.1	2.933	
142.9	61.5	3.251	
174.0	88.5	3.505	
205.0	116.0	3.782	
230.7	147.9	3.900	
274.0	178.9	4.152	
330.2	218.0	4.391	
380.7	261.9	4.584	
455.1	312.5	4.855	
554.0	373.5	5.256	
<u>12. Nickel Powder:</u>			
23.1	0.0020	0.502	$w = 7.440 \text{ g.}$ $T_b = 302.0^\circ \text{ K}$ $T_{s1} = 78.0^\circ \text{ K}$ $T_{s2} = 79.5^\circ \text{ K}$ $V_s = 37.9 \text{ cc.}$ $V_t = 135.8 \text{ cc.,}$ 60.9 cc.
22.9	0.0110	0.999	
32.9	0.040	1.712	
28.0	0.370	2.305	
28.0	1.38	2.860	
55.9	3.0	3.338	
52.1	4.9	3.754	
69.9	8.3	4.275	
74.7	14.1	4.732	
97.1	25.0	5.184	
103.8	38.2	5.518	
142.7	58.1	5.881	
170.5	81.1	6.217	
219.4	109.7	6.619	
264.1	141.6	7.066	
302.9	175.3	7.518	

(Continued)

Quarterly Report No. 2, Project No. 228-197

TABLE III (Continued)

EXPERIMENTAL NITROGEN GAS ADSORPTION DATA

P_1 (mm. Hg)	P (mm. Hg)	V (cc./g.)	Constants*
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<u>13. Nickel Powder:</u>			
27.7	0.0076	0.602	$w = 7.440 \text{ g.}$ $T_b = 302.0^\circ \text{ K,}$ 303.0° K $T_s = 80.0^\circ \text{ K}$ $T_s' = 82.6^\circ \text{ K}$ $V_s = 37.9 \text{ cc.}$ $V_t = 135.8 \text{ cc.,}$ 60.9 cc.
32.2	0.050	1.300	
35.0	0.349	2.047	
36.3	1.65	2.772	
53.6	4.0	3.202	
71.1	7.1	3.736	
90.6	14.5	4.313	
111.8	28.1	4.824	
126.4	44.2	5.264	
138.2	63.8	5.549	
158.0	84.7	5.793	

<u>14. Nickel Powder:</u>			
29.3	0.040	0.643	$w = 7.440 \text{ g.}$ $T_b = 299^\circ \text{ K,}$ 300° K $T_s = 82.0^\circ \text{ K}$ $T_s' = 85.2^\circ \text{ K}$ $V_s = 32.2 \text{ cc.}$ $V_t = 135.8 \text{ cc.,}$ 60.9 cc.
32.2	0.735	1.333	
60.0	3.0	1.842	
64.9	7.9	2.295	
68.3	13.1	2.723	
83.4	23.9	3.070	
86.0	34.9	3.332	
106.5	49.9	3.560	
128.9	67.0	3.793	
152.3	88.0	3.963	
192.0	114.0	4.156	
211.2	139.0	4.313	
258.5	169.3	4.517	
309.0	203.1	4.805	
362.8	245.0	5.024	

(Continued)

TABLE III (Concluded)

EXPERIMENTAL NITROGEN GAS ADSORPTION DATA

P_1 (mm. Hg)	P (mm. Hg)	V (cc./g.)	Constants*
<u>15. Nickel Powder:</u>			
458.1	19.0	3.7344	$w = 7.440 \text{ g.}$
201.7	50.8	4.4951	$T_b = 303.6^\circ \text{ K}$
258.0	96.0	5.0632	$T_s = 80.8^\circ \text{ K}$
330.3	151.0	5.5740	$T_s^f = 84.2^\circ \text{ K}$
406.6	212.8	6.0629	$V_s = 38.2 \text{ cc.}$
462.5	272.4	6.5552	$V_t = 60.9 \text{ cc.}$
523.0	330.7	7.0882	
583.1	389.6	7.6091	
705.3	458.0	8.4202	

* When more than one value is given for a constant, the first value refers to the first group of data, the second to the second group, etc.

** Descriptions of coconut charcoals:

1. Evacuated 20 hrs. at 300° C. to 0.3μ pressure.
2. Evacuated 18 hrs. at 300° C. to 0.1μ pressure.
3. Evacuated 20 hrs. at 300° C. to 0.1μ pressure.
4. Evacuated 20 hrs. at 375° C. to 0.001μ pressure.
5. Evacuated 20 hrs. at 300° C. to 0.01μ pressure.
6. Evacuated 24 hrs. at 375° C. to 0.01μ pressure.
7. Evacuated 24 hrs. at 375° C. to 0.01μ pressure.
8. Evacuated 24 hrs. at 375° C. to 0.01μ pressure.

*** Descriptions of nickel powders:

9. Colloidal-HSS technique.
10. Colloidal-HSS technique.
11. Colloidal-HSS technique.
12. Colloidal, evacuated 20 hrs. at 300° C. to 0.001μ pressure.
13. Colloidal-HSS technique. Exposed to air, then evacuated 20 hrs. at 250° C. to 0.1μ pressure.
14. Colloidal-HSS technique.
15. Colloidal-HSS technique.