AN ABSTRACT OF THE THESIS OF

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Title Test on Various Activated Carbons

Abstract Approved: [redacted]
(Major Professor)

A new product called "Activated Carbon" was brought into existance during the world war. It originated as a means of defense against toxic gases in warfare. Since then, increasing applications of gas adsorbent carbon are found in many industries, such as in the recovery of volatile liquids, the extraction of gasoline from natural, gas, and the purification of fermentation gases.

There are several methods to determine the activity of activated carbon, but some are incomplete and some have no fundamental basis. Recently, the California Natural Gasoline Association collected and correlated data on the different methods to provide a standard procedure which embodies the best current practice.

The writer has followed closely this standard method to test the activity of Columbia Activated Carbon, which is considered to be one of the best activated carbons produced in this country. The following tests have been conducted.

1. Adsorption Test

Portland city gas was passed through a tube filled with Columbia Activated Carbon, and the gases were adsorbed and heat was envolved. A thermometer was inserted into the tube from the top, and the temperature indicated rose to a maximum then lowered as the heat was led away by the gas stream. When the carbon was saturated with the lighter gas, the next heavier gas will replace the lighter gas and the temperature rose again due to the difference of the heat of condensation. By this means, any desired constituent could be obtained with the help of a curve.

2. Carbon Tetrachloride Humidity Test

Thsi test is designed to determine the activity of the carbon. The principle is to pass air which was saturated with carbon tetrachloride through a 10 mm I.D. x 21 cm long glass tube, The time required to saturate the carbon times 3 gives so called "chloropicrine time".

3. Carbon Tetrachloride Humidity Test

This test is to show the rate of adsorption of carbon tetrachloride by the activated carbon. Air saturated with carbon tetrachloride was passed through a carbon tube which is equipped with a wet bulb thermometer and a dry bulb thermometer so that the percentage of saturation of the carbon versus the volume of air passed through could be calculated.

ABSTRACT

4. Reactivation

The used activated carbon was washed with dry steam for 24 hours, and then followed by superheated steam at 1000 °F. The extent of reactivation was then measured.

5. Activation

Chars of apricot pits, watnut shells and filbert shells were activated in the above described manner and their activities were tested.

TESTS
ON
VARIOUS ACTIVATED CARBONS

by

SZE YEN PO

A THESIS

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ACKNOWLEDGMENT

My interest in this problem was aroused by Professor G. W. Gleeson. In this study, his suggestion and his constant help have been of utmost value and I here wish to express my most sincere thanks.

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INTRODUCTION

In industrial applications, the study of activated carbon is a very wide and very specialized subject. The ramifications of active carbon are many and its forms are numerous. The technology of the application is young and its development is rapid.

The literature concerned with tests of adsorbent carbon is widely scattered and there is not a standard one to follow. In some phases, tests have been imcomplete and biased, in others there are many variations which have no fundamental basis and only tend to increase the personal element in the test. The confusion resulting from such variations in testing procedure is particularly distressing in determining the gasoline content of gas for royalty proration of gasoline plant production.

In the response to the demand for a standard procedure, the California Natural Gasoline Association collected and correlated data on the different methods in order to provide a standard procedure which embodies the best current practice.

The writer of this thesis has followed closely

the standard procedure issued by the organization mentioned above.

The writer tried to activate carbon from the chars of fruit pits and nut shells by steaming for a shorter period than usual, and tested the degree of activation produced. On account of the lack of time, further study on this branch was limited.

LITERATURE REVIEW

History

The fact that wood charcoal could be used to remove undesired odor has been known for many years. In the form of wood ashes, charcoal has been employed for centuries to eliminate odors. Shenstone(10) in 1854 invented a respirator filled with powdered wood charcoal to be worn in a hospital. It was used to adsorb and destroy germs of cholera and other disease. More than a half century ago, Hunter (8) showed that many different gases and vapors are adsorbed by charcoal to a marked extent. Dewar(11) measured the adsorption of argon, helium and some more common gases on cocoanut char at low temperature. The volume of gases adsorbed were calculated back to 0 degree Centigrade and 760 mm pressure.

It was the world war that brought into existence, as an article of commerce, a new product known as "Activated Carbon". It orginated as a mean of defense against toxic gases in warfare. The use of adsorbent carbon was extended very rapidly both in commercial and experimental work, the extent and novelty of which are as yet not generally appreciated.

Uses

Carbon, in its elementary and allotropic forms,

aside from its use as a fuel, plays a very important part in our everyday experience.

The diamond has ranked as the leading precious stone for centuries, and its important roll played in the modern world is too obvious.

Graphite, an allotropic form of carbon, makes the world roll on more smoothly, less noisely and less wearingly, for graphite is widely used as lubricating material. All of our electrolytic industries depend upon graphite electrodes for their very existence. Practically no other material is suitable.

In the form of bone black and decolorizing chars, carbon acts as a purifying and decolorizing agent in the manufacture of many materials which the civilized world consumes.

As metal adsorbent chars, carbon recovers gold and silver from their metallurgical solutions to increase the wealth of the world.

In medicine, carbon aids us to fight against such disease as cholera; carbon saves our lives by the adsorption of toxic gases, a use which no other chemical substances could economically replace. Its value in time of war against poisonous gas cannot be extimated. In peace times, increasing application of gas adsorbent carbon is found in industry, such as in the recovery of volatile liquids, the extraction of gasoline from natural gas,

and the purification of fermentation gases.

Theory of The Formation of Activated Carbon

The only theory of the formation of activated carbon which satisfies the known facts is given by Dr. N.K. Chaney (4). According to his theory, elementary carbon (other than diamond and graphite) exists in two modifications, "Active" and "Inactive" or "alpha" and "beta".

The amorphous form of carbon resulting from the adsorption of hydrocarbon by active carbon is called "primary" carbon.

Active and inactive carbon differ in two particulars, i.e.

(1) Temperature of formation.

(2) Chemical activity or susceptibility to oxidation.

The active modification is formed whenever the carbon is deposited at low temperatures* (generally 600-700 degree Centigrade)

Inactive carbon is obtained by similiar decomposition at relatively higher temperatures (generally above 700 degree Centigrate). To quote the original paper of Dr. Chaney's: "It woulb be premature to assert that these two forms of carbon (active and inactive) are

* Regarding this temperature range the writer fails to find the same values in other literature.

true allotropic modifications. It is not yet established that both are amorphous..... The two forms are characteristically distinct and easily differentiated both by their properties and conditions of formation." Professor Henry Briggs (15) expressed the same idea by refering to the active form as probably less highly "polymerized and has interstices of molecular dimension."

As an adsorbent, the active carbon is either highly dispersed or is traversed by numerous canals or capillaries which enable gas or vapor molecules to reach an enormous surface so that they are adsorbed and condensed on the surface.

It is desirable that an adsorbent carbon have the maximum adsorptive capacity per unit volume rather than per unit weight. The carbon has a critical density for maximum adsorptivity, that is, if the carbon is too dense its permeability is destroyed. If its density falls below the critical value its adsorptive capacity decreases.

Manufacturing Methods

From the foregoing, it is seen that the adsorptive power or activity of an activated carbon is determined by the amount of the available active form. It has been shown that no one type of activated carbon could be universally used with high efficiency. Carbon which is entirely suitable for one purpose is not necessarily satisfactory under different circumstances.

The raw materials available for the manufacture of activated carbon are various vegetable substances, chars of nut shells and fruit pits, low temperature coke, hard wood, and many other organic materials. No matter what raw material is used the temperature of formation of activated carbon must be below 600-700 degree Centigrade, and adsorption of hydrocarbons must be prevented by removing them as they are formed..

The processes by which various types of activated carbon can be made may be classified as follows.

(1) The Action of Chemicals.

By this process, many inorganic compounds (bases, acids, and salts) are mixed with carbon bearing material and the calcination is in general conducted around 900 degree Centigrade, The chemical residue is then removed by extraction with acids or bases. (20)

(2) The Action of Solvent.

According to the theory of formation of activated carbon, suggestion has been made that the hydrocarbon with which the active carbon is saturated could be removed by solvents such as used for resins, tars and pitches. Several processes belonging to this group have been patented.*

* U.S. Pat. 1,423,231 (1922)

(3) Long Calcination at Elevated Temperature.

It is claimed that primary carbon can be activated by prolonged heating in a neutral atmosphere at about 850 degree Centigrade. The difficulty with this process is that at this temperature the hydrocarbon is cracked and adsorbed by the active form.

(4) Selective Oxidation.

The final and superior method for the preparation of highly activated carbons was developed by Dr. Chaney (4) and those under his direction.

The primary carbon as defined by Dr. Charey includes all chars, cokes and carbon materials formed below 600-700 degree Centigrade. When a gaseous oxidizing agent is employed, such gases as air, carbon dioxide, and/or steam may be used. Air activated carbon could be produced at low temperature (around 350 degree Centigrade), but the change in the oxidation potential with temperature is very rapid and the process is extremely difficult to control. Employing steam or carbon dioxide, the temperature should be around 900 degree Centigrade. These reagents excerise a selective oxidizing effect and the reaction is easy to control. It appears that the removal of the hydrocarbons with minium cracking and deposition of inactive carbon can be easily accomplished only when the concentration of the oxidizing agent is high. This

means that steam is introduced in excess so that the reaction products are effectively led away. The great advantage of the Chaney process is that by employing proper steam rates for different periods at selected temperatures, the characteristics of the activated product may be widely varied within limits depending upon the character of the primary carbon employed.

Theory of Adsorption

(1) Equation of Adsorption.

The earliest mathematical expression of adsorption is given by the Freundlich equation.

$$(X/m)^{n} = Kp$$

Where X is the amount of gas adsorbed by the amount of adsorbing solid m, p is the pressure and K and n are constants which vary with temperature. Hinteler (17) found that this equation holds for vapors of hexane, ethyl alcohol, methyl alcohol, and water when adsorbed by charcoal.

In 1932 there were two formulae proposed concerning adsorption of various gases by active carbon, namely, The isothermal (9):

$$\log V = a - b.\log p$$

Where V is the volume of gas adsorbed in cubic centimeters at one atmosphere, p is the gas pressure, a and b are two empirical constants which are functions

of temperature, pressure, and the gas which is adsorbed. The Adiabatic. (9)

$$\log V/V_s = b.\log p - E.\log p_s$$

Where p_s is the vapor pressure of the substance being adsorbed at the experimental temperature T, V_s is the volume of the gas or vapor at its boiling point, V and p are the volume and pressure.

Variables of Adsorption

The variables which control adsorption are temperature, pressure, moisture content and the bulk density of the carbon. The results of some experiments can be summarized as follow.

(1) Temperature

The adsorption of gases is greatly increased by lowering the temperature of the carbon (12).

(2) Pressure.

The adsorption of gases is increased by the increasing the pressure (18).

(3) Moisture.

Moisture is strongly adsorbed at ordinary temperatures, but carbon holds this water very feebly losing all but one or two percent when exposed to dry air for some time. This adsorbed moisture will diminish somewhat the adsorption capacity of the carbon for other gases except those which react with water.

PART I

ADSORPTION TESTS

Theory

When a gas is passed through a tube of activated carbon, it is immediately adsorbed with the liberation of heat until the carbon is saturated. Various gases are adsorbed to different extent. In a natural gas, where the gases are of homologous series, the gases of higher molecular weight are more strongly adsorbed than are the lighter gases and the heavier gases posses the ability to displace the lighter gases from the carbon. All of the gas is adsorbed when it is first passed through the carbon and a thermometer placed in the outlet of a tube of carbon indicates a sharp temperature rise.

In the case of a gaseous mixture, only the carbon in the bottom portion of the tube will have adsorbed any specific constituent of the gas at the first temperature rise. Since the top portion is rapidly saturated with the more easily adsorbed constituent, the temperature will drop due to the heat being carried off in the stream of gas. This drop in temperature will continue until the carbon is contacted by a gas of higher molecular weight. The next rise of temperature indicates the replacement of the lighter constituent, by the next

homologous member, this rise is due to the difference in latent heat of vaporization: the first constituent is evaporated and the replacing gas is condensed.

Similarly, a third and fourth rise will occur when displacement takes place.

The height of the rise in each case will depend principally upon the composition of the gas and the velocity of the gas flow through the tube.

Equipment

Figure 1 illustrates the equipment used for the previously described experiment. It consists of a charcoal tube, two thermometers, two water bubblers and a gas meter. All connections must be tight. The tubes are of standard galvanized iron pipe. The connections are all of rubber tubing. The meter is a Sargent wet test meter, reading to 0.001 cubic-foot.

Experimental Method.

Before the carbon is charged into the tube, gas is passed through the whole apparatus for a time sufficient to purge the branch pipe lines. The gas is then drawn directly from the ground line until the difference between thermometers "C" and "E" is constant. The rate valve is then closed and the carbon is charged into the tube to such a height that the thermometer bulb is entirely buried. Gas is admitted by opening valve "B" to the

desired constant rate. The lower thermometer "C" shows slight variation, but the upper thermometer "E" varies so rapidly, particularly at the beginning, that it must be watched closely. The readings of these two thermometer and the volume of gas passed through are recorded.

In these tests Columbia Activated carbon of 8-14 mesh was used, and the Portland City Gas which has an approximate composition of 1.2% CO_2, 0.1% C_6H_6, 3.0% C_nH_{2n} 0.5% O_2, 7.7% CO, 54.2% H_2, 30.1% CH_4, and 2.4% N_2 was emp loyed.

At the end of the experiment, the carbon was removed to be reactivated.

Results

The data obtained are included in the Appendix A, division A , and a representative curve is drawn as Figure 2.

Discussion

There are three peaks on the above curve. The first peak is undoubtly due to the adsorption of hydrogen. The second peak is probably due to the adsorption of carbon monoxide, and the third, to methane. The quantities of the other constituents are too small to affect the shape of the curve.

Effect of Gas Rate

Tn order to determine the effect of rate of gas

flow upon adsorption, three tests were run. The first was run with a gas rate of 0.045 cu.ft./min. at an average inlet temperature of 20.50 degree Centigrade, the second at a gas rate of 0.10 cu.ft./min. at an inlet temperature of 20.04 degree Centigrade, and the third, a test of the carbon removed after the first run after reactivation, at a gas rate of 0.075 cu.ft./min. at an average inlet temperature of 21.00 degree Centigrade.

All runs were conducted at a pressure of one atmosphere.

Data and Results.

The data obtained are shown in Appendix Part I, division B,C, and D, and three curves are given as figure 3. The comparative results are evident from this figure. Curve I represents the results of reactivated Columbia Activated Carbon, Curve II represents the results at the gas rate of 0.10 cu.ft./min. and Curve III is that at the rate of 0.045 cu.ft./min.

Discussion.

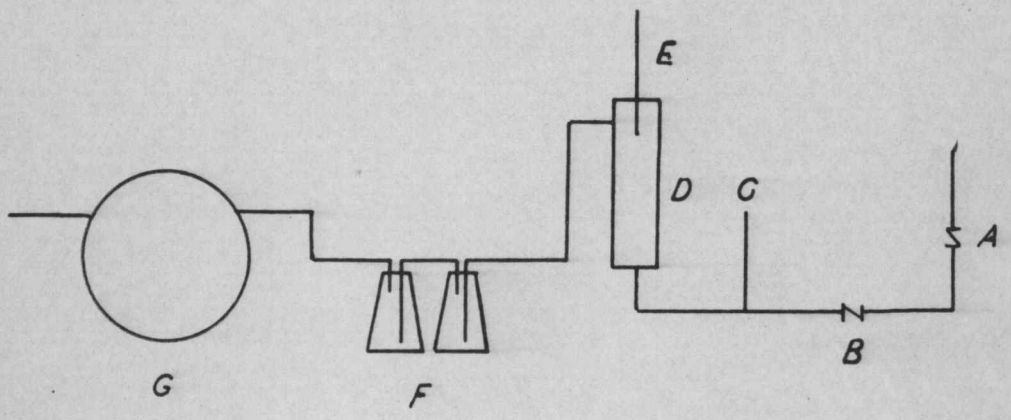
The peaks of the curves are shifted very little to the right by an increase in the rate of flow. It seems that a slight change of gas rate has very little effect on the extent of adsorption. Part of the adsorption heat is lost through the mechanism of radiation to the surroundings, and some heat is carried off by the gas. The

loss becomes more rapid when the gas rate is increased. This, very likely, is the reason that the peaks are shifted to the right.

When we study Curve III of Figure 3 we noticed that the downward slope from each peak is less steep than that in Curve I and Curve II. It is likely that there is still some adsorbed gas remaining from the previous run. The activity of the carbon can be restored to its original degree only by very prolonged treatment.

The room temperature at which these experiments were conducted averaged 25 degree Centigrade. The temperature gradient was sufficient to cause a difference between the upper and the lower thermometers. Although the carbon tubes were carefully lagged with 85%-magnesia, approximately a two-degree Centigrade difference could not be eliminated.

The temperature of the inlet gas was not strictly constant, showing a variation of one degree Centigrade. This, however, causes no particular difficulty.



A--1/4" brass globe valve
B--1/4" brass needle valve
C--Thermometer
D--Carbon Tube
E--Thermometer
F--Water bubblers
G--Sargent wet gas meter

Figure I - Equipment for Adsorption Test

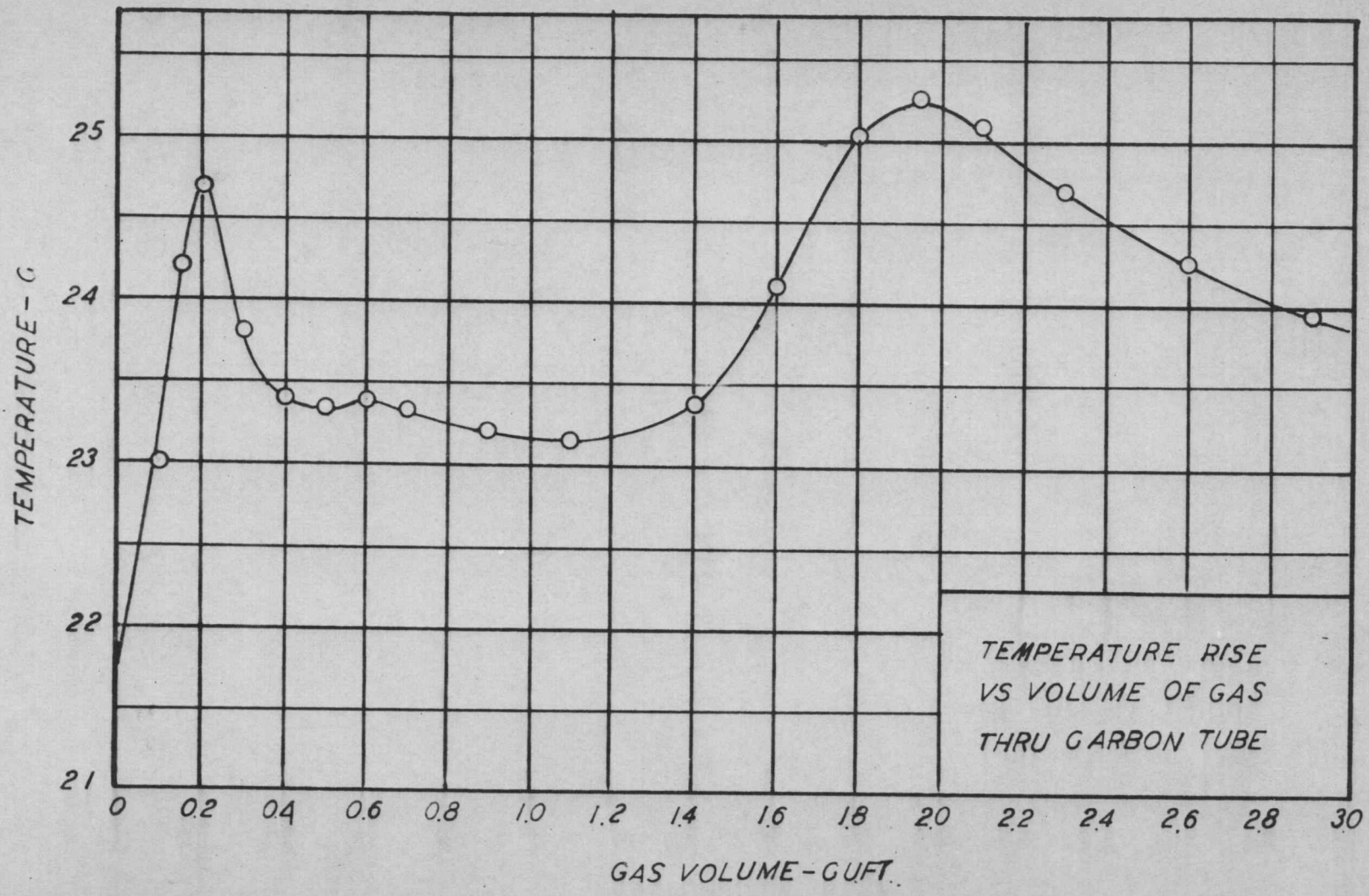

TEMPERATURE - C
25
24
23
22
21
0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
GAS VOLUME - CUFT.
TEMPERATURE RISE
VS VOLUME OF GAS
THRU CARBON TUBE

FIG. 2

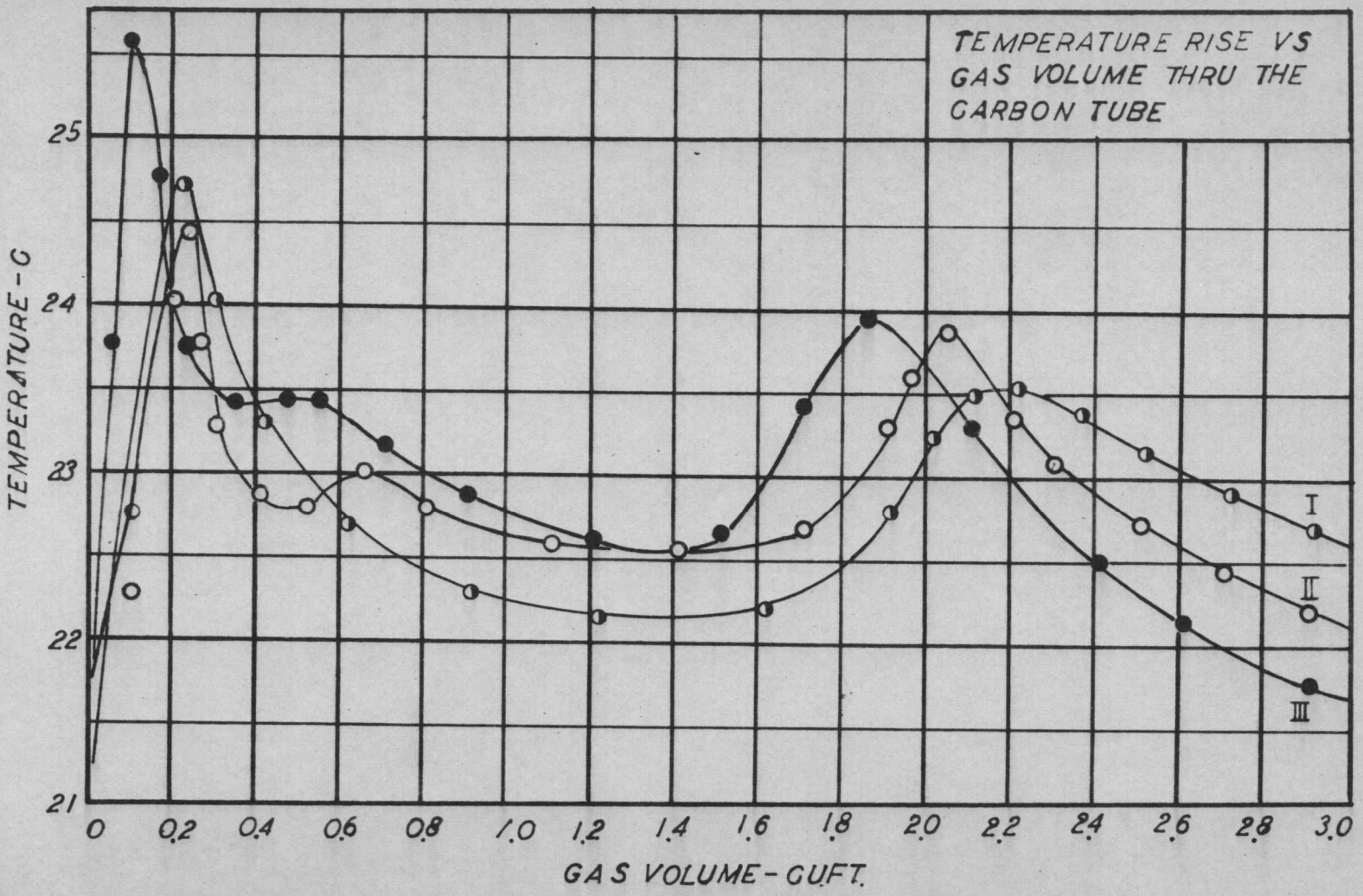

TEMPERATURE RISE VS
GAS VOLUME THRU THE
CARBON TUBE
TEMPERATURE - C
25
24
23
22
21
0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
GAS VOLUME - CU.FT.
I
II
III

FIG. 3

PART II

CARBON TETRACHLORIDE

MODIFICATION OF THE CHLOROPICRINE TEST

Purpose.

This experiment is employed to test the activity of the carbon. In the determination of the sorptive capacity of activated carbon it is necessary to have some measure of its activity in order to keep the carbon in a state of activity sufficient to meet requirements or specifications.

Equipment.

The apparatus used is that recommended by the California Natural Gasoline Association (12). Activated carbon of 8-14 mesh which will show an activity of not less than 45 minutes by this method is considered satisfactory for gas adsorbent carbon.

The apparatus used and its arrangement are illustrated by Figure 4. It consists of a metal box with 10 feet of 1/8" copper tubing coiling along the wall. The end of the tubing is provided with a 3-way glass cock "D" which is connected to an acid trap bottle "E". Following "E" are two bottles containing concentrated sulfuric acid, "F" and "G", Bottle "H" is filled with glass wool to eliminate any acid the air brings over. Connected to "H" is a carbon

tetrachloride trap bottle "I" which is followed by four bottles "J. K. L. M." of carbon tetrachloride. "O" is a manometer to measure the rate of flow of air; "P" is the carbon tube filled with the tested carbon. The dimensions of the carbon tube is strictly required to be a glass tube of 10 mm. I.D. x 21 cm. long. "Q" is a glass Y in which fuel gas is mixed with the air and burned. A copper bead on a wire is fixed to the arm of the Y and is held in the flame. In the presence of any carbon tetrachloride vapor in the air coming from the apparatus, the copper bead imparts a blue coloration to the flame. A mercury bottle with sufficient depth of mercury to overcome the back pressure from the series of bottles acts as a pressure control.

Experimental Procedure.

The general procedure is to pass air saturated with carbon tetrachloride through a given sample of activated carbon at a rate of 766 ml. per minute, and at a temperature of 0 degree Centigrade. The box is filled with ice, and air is passed through the whole system preceding the test, so that the contents of the bottles are stirred and the temperature lowered to 0 degree Centigrade. The rate, indicated by the manometer "O" is adjusted accurately to 766 ml. per minute by the 3-way glass cock "D". The charcoal tube "P" which contains a sample of Columbia Activated Carbon is

put in place, and the burner "Q" is lighted. The end point of the run is indicated by a blue color developed in the flame. The elapsed time multiplied by 3 gives the chloropicrine time. The 3 is a factor number correlating this procedure with the chloropicrine test.

Data and Results

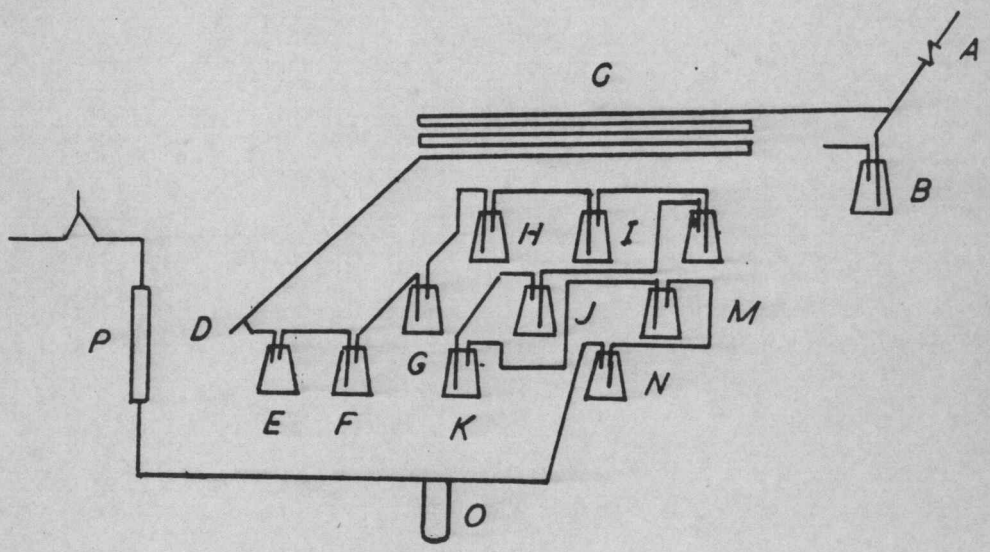
	1	2
Weight of Columbia Carbon	5.95	6.10
Time elapsed in minute	19.5	20.8
Average time elapsed		20.1
CCl_4 adsorbed in gram	3.35	3.50
Percent CCl_4 adsorbed	56.5	57.5
Average percentage		57.0

The equivalent chloropicrine time is therefore 20.1 x 3 or 60.3 minutes. This result compares with 45 minutes. This result compares with 45 minutes as specified in this procedure shows that the Columbia Activated Carbon is an excellent adsorptive carbon.

Discussion

In this test the temperature and rate of the air are very important, one degree variation in temperature causing a 3% variation in the final result. Fortunately the temperature is comparatively easy to keep constant. The air used was taken directly from the pipe line of the air compressor, the pressure of which fluctuated. Although the rate was

adjusted continuously, slight error introduced by this factor is to be expected.



A -- 1/4" brass needle valve
B -- Mercury regulator
C -- Copper coil - about 10 ft. long
D -- 3-way glass cock
E -- Acid trap
F -- Concentrated H_2SO_4 bottle
G -- Same as "F"
H -- Acid Scrubber
I -- Carbon tetrachloride trap
J -- Carbon tetrachloride trap
K -- Same as J
L -- Same as J
M -- Same as J
N -- Same as I
O -- Calibrated glass manometer
P -- Charcoal tube - glass tube 10 mm I.D. x 9.82 mm. long
Q -- Glass Y

Figure - IV , Equipment for Carbon Tetrachloride Test

PART III

CARBON TETRACHLORIDE HUMIDITY TEST

Equipment and Apparatus

The equipment in this test consists of a combination of portions of the apparatus used in the adsorption and in the carbon tetrachloride tests. (see Figure 5). The 3-way glass cock "D" of the carbon tetrachloride apparatus, The final bottle "N" was connected to the adsorption tube inlet. A dry bulb thermometer "S" and a wet bulb thermometer "T" are placed at the end of the carbon tube. From there the gas will pass through two water bubblers and into the wet gas meter.

Experimental Procedure:

By the 3-way glass cock, air is admitted to the carbon tube at a constant rate without passing through the series of bottles. The readings of the two thermometers are carefully recorded at given intervals (in term of air volume) until the wet bulb thermometer remains constant. As the carbon gradually becomes saturated, the emergent air will contain some carbon tetrachloride, and the wet bulb thermometer will gradually rise until the carbon becomes saturated. This condition is indicated by a new constant temperature of the wet bulb thermometer.

The readings of the dry and wet bulb thermometers,

the volume of air passed through and the adsorption heat of the carbon tube are taken. The carbon is then subjected to 150 degree Centigrade superheated steam for an hour, and the process repeated after the carbon is dried and cooled.

In the following tests the rate of air flow was 0.075 cu.ft./min., and a sample of 127.1 gram Columbia Activated Carbon was used. But the dried steamed carbon weighed 130.4 grams, that is the carbon gained 3.3 grams.

Data and Results

Weight of Activated Carbon = 127.1 grams
Weight of CCl_4 adsorbed = 49.2 grams
Per cent CCl_4 adsorbed = 38.8

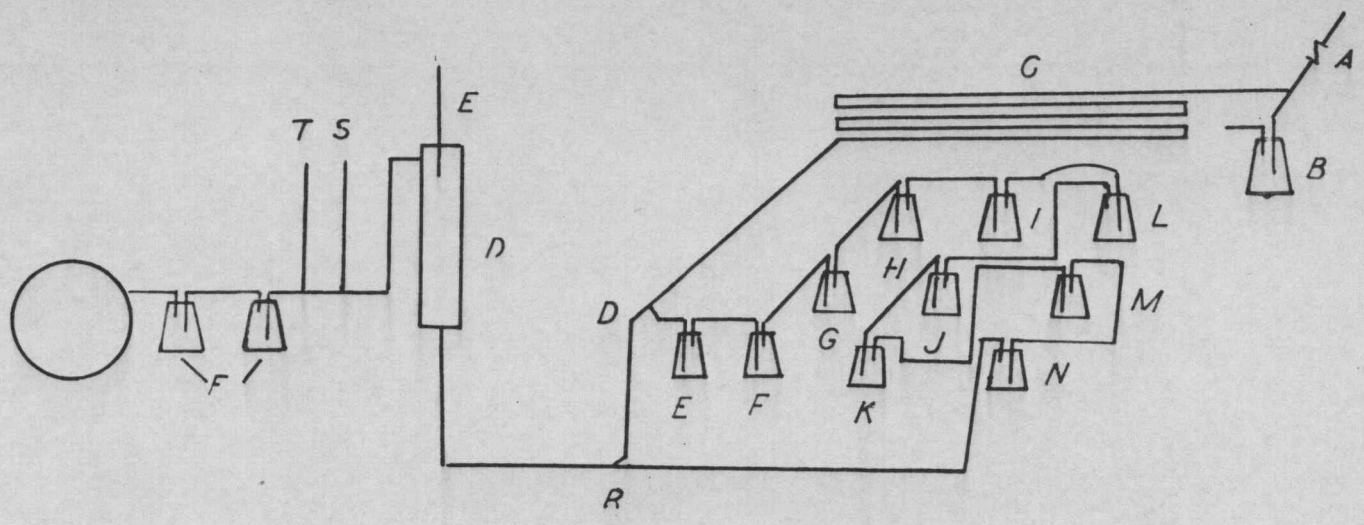
The data are shown in Appendix Part III, division A and B. Three curves are drawn in Figure 6. Curve I shows the result of the test on the original carbon and Curve II that of the steamed carbon. Curve III is the curve for adsorption heat. Based on the saturation weight as 100%, a curve of the percentage of saturation against the volume of gas passed through is drawn in Figure 7.

Discussion

Curve I and Curve II of Figure 6 show that the steamed carbon becomes saturated more quickly and abruptly than the original sample. By the fact that the steamed carbon after drying at 110 degree Centigrade showed 4%

increase in weight, we may conclude that a portion of the carbon tetrachloride remains in the carbon in some combination stable to 105 degree Centigrade Steam.

As noticed from the results of the carbon tetrachloride modification test, the carbon became saturated when it had adsorbed 57.0% of its own weight as carbon tetrachloride. (see page 19) In this test, however, the saturated weight of carbon tetrachloride adsorbed is only 38.8. This discrepency is due to the effect of the increase in temperature of the carbon tube as noticed in Curve III in Figure 6.



R -- A 3-way glass cock
S -- Dry bulb thermometer
T -- Wet bulb thermometer
Other notations are the same as in Gigure (I) and (IV)

Figure V - Equipment for The Carbon Tetrachloride Humidity Test

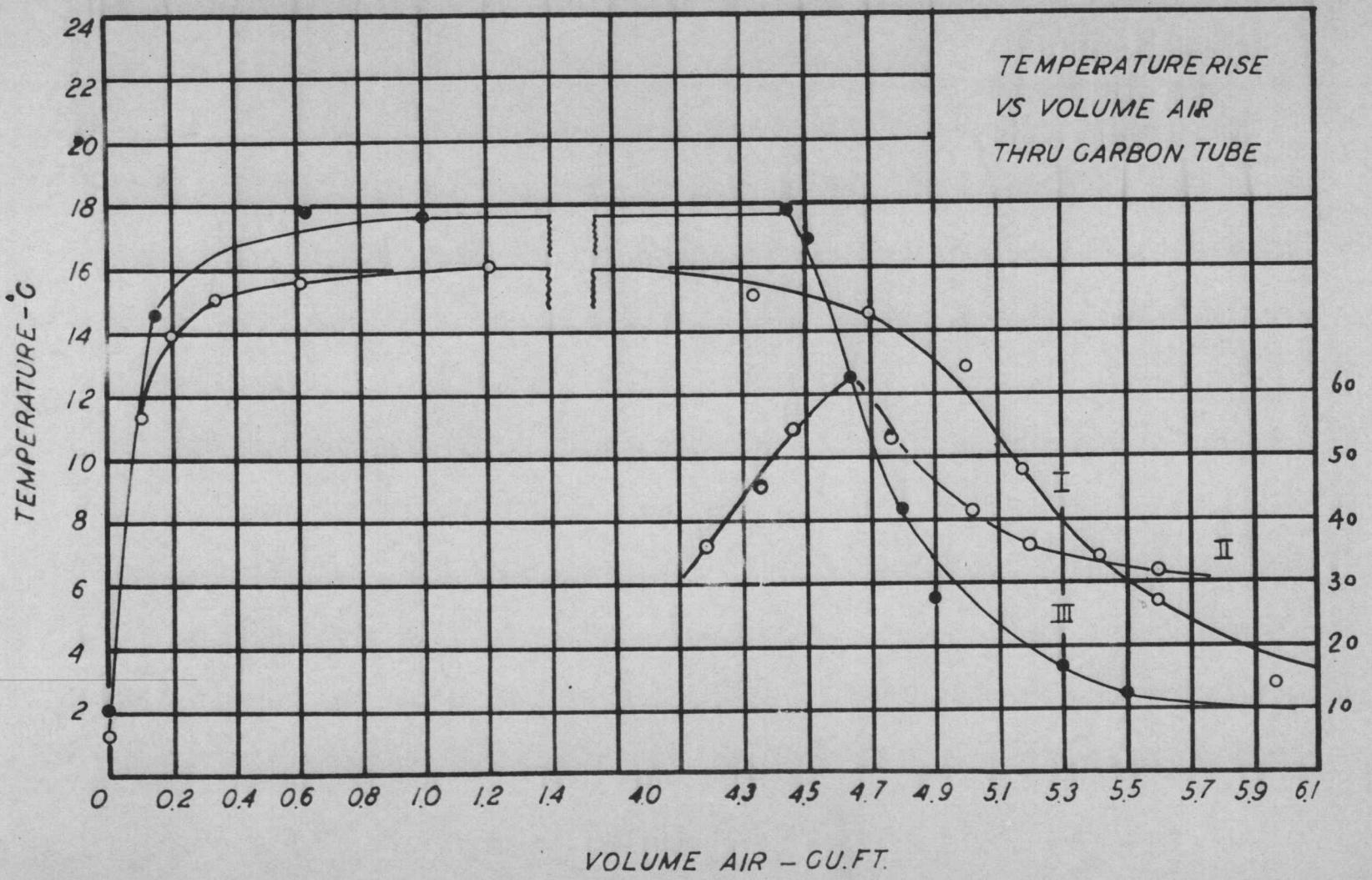

TEMPERATURE RISE
VS VOLUME AIR
THRU CARBON TUBE
TEMPERATURE - °C
VOLUME AIR - CU.FT.
24
22
20
18
16
14
12
10
8
6
4
2
60
50
40
30
20
10
0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
4.0
4.3
4.5
4.7
4.9
5.1
5.3
5.5
5.7
5.9
6.1
I
II
III

FIG. 6

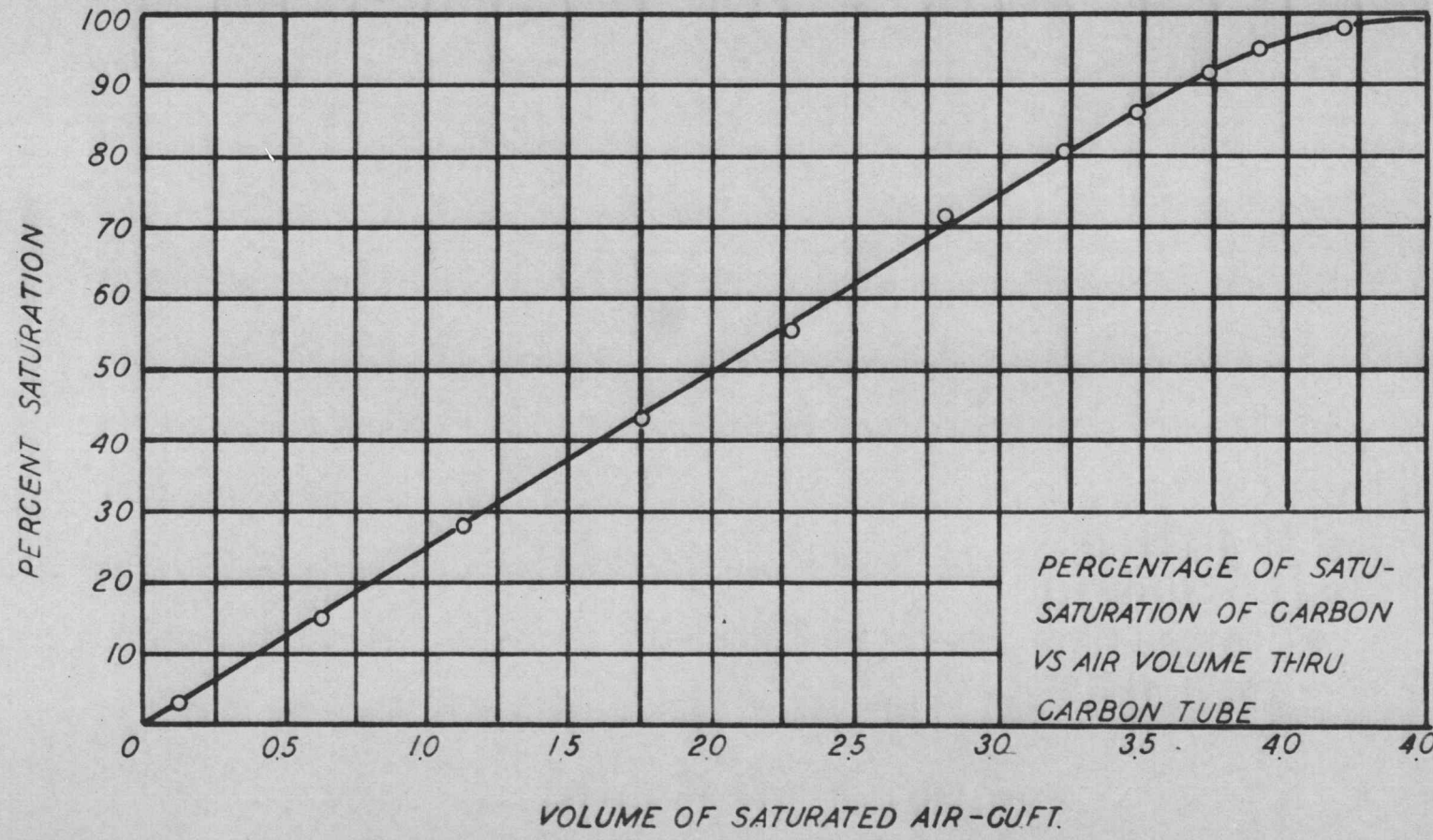

PERCENT SATURATION
100
90
80
70
60
50
40
30
20
10
0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.0
PERCENTAGE OF SATU-
SATURATION OF CARBON
VS AIR VOLUME THRU
CARBON TUBE
VOLUME OF SATURATED AIR-CU.FT.

FIG. 7

PART IV

IODINE TEST

The test gives an indication of the ability of the carbon to remove odors and flavors rather than color bodies. A good activated carbon will remove from 70-90% of the iodine in this test.

Procedure

A 0.5 gram sample of 100-mesh Columbia Activated carbon was placed in a 150 cc beaker, and the carbon wetted by 10 cc of 10% sulfuric acid. The mixture was then heated to boiling. 100 cc of 0.196 N iodine solution was added and the mixture stirred for 10 minutes. The mixture was then filtered through a No. 5 Whatman filter paper and the filtrate was titrated with N/100 sodium thiosulfate solution.

The percent iodine removed by carbon is calculated.

Result

99.6% of the iodine was removed.

PART V

PERMANGANATE TEST

This test gives an indication as to the ability of the carbon to remove colloids and colored bodies.

Procedure

A 0.40 gram sample of 100-mesh Columbia Activated Carbon was added to 25 cc of 0.519 N permanganate solution in a 150 cc beaker. The mixture was stirred vigorously for 30 seconds and allowed to digest at room temperature for mine and half minutes and then filtered through a Gooch crucible. The filtrate was titrated iodometrically for the amount of permanganate remain. The percent of permanganate adsorbed was calculated.

Result

40.5% of the permanganate was removed.

PART VI

REACTIVATION

Equipment

The apparatus employed in the process of reactivation is illustrated in the diagram of Figure 8. The tube bank is made of one foot length of 1/4" standard pipe. It was so joined by elbows that it forms 18 passes with a length of about half foot above the floor of the heating chamber. The burner was constructed by inserting five Bunsen burner tips in line in a single pipe. The row is placed under the tube sheet between the two tube walls; gas is used as fuel; compressed air is introduced to the burner from a T at the entering end.

The reactivator is constructed of a one and one-half foot length of one and one-third inch pipe, both ends of the chamber bring capped by reducing couplings and these also held in place a perforated plate screen. The upper end is joined at the reducer by an elbow connected to a long condenser. The reactivator is insulated by 85% magnesia lagging.

Procedure

The used Columbia Carbon is charged into the reactivator chamber from the top, and steamed with wet saturated steam for a period of 24 hours. This is followed by react-

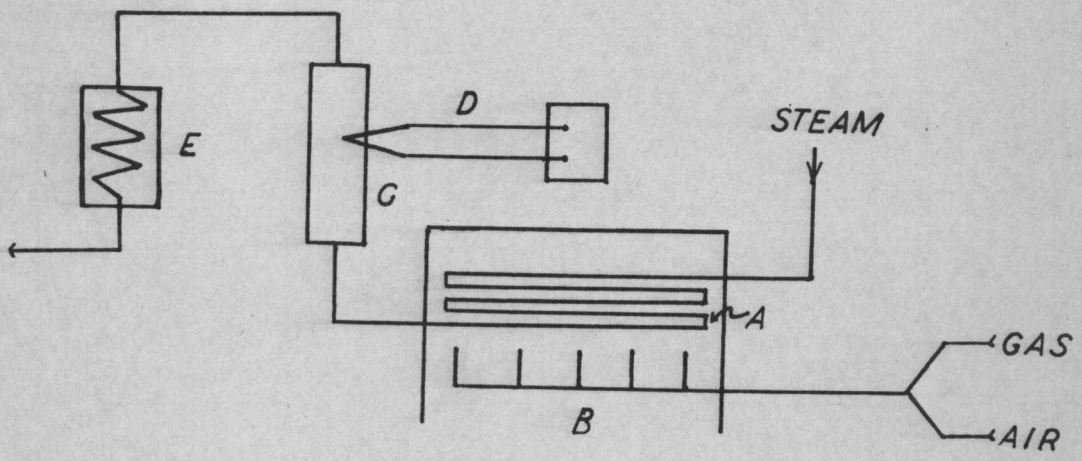
ivation with super-heated steam for 12 hours. The temperature is under control and ranges between 1000-1050 degree Fahrenhite. After the required time for superheating has elapsed, the fire is turned off and the steam allowed to pass through the carbon until the temperature has dropped to 800 degree Fahrenheit.

Result

The results on adsorption for the reactivated Columbia Activated Carbon is shown in Appendix Part I division D, and a representive Curve was shown by Curve I in Figure 2.

Discussion

The curve of heat of adsorption of the reactivated carbon failed to show the small peak which appears distinctly in the other two curves. This shows the great difficult to restore the efficiency to its original value.



A -- Tube sheet
B -- Burners
C--- Activating chamber
D -- Condenser

Figure VIII - Activator

PART VII

ACTIVATION OF CHARCOAL

Equipment

Same as the one used for reactivation.

Procedure

Same as the one used for reactivation.

Adsorption Test

An adsorption test on each of the charcoals before activation was conducted and thermal curves are drawn in Figure 9. The results of the adsorption test after activation are shown in Figure 10.

The specifications are listed as follow:

Before Activation:

	Appendix VII,A	Fig. 9, Curve
Apricot	a	I
Walnut	b	III
Filbert	c	II
Silica gell	d	IV

After Activation:

	Appendix VII,B	Gig. 10, Curve
Apricot	a	I
Walnut	b	III
Filbert	c	II

Carbon Tetrachloride Test

The equipment and procedure of this test are the same as former tests. No positive results were given to any of the activated charcoal. The time elapsed in the carbon tetrachloride modification test were a few seconds or zero, and the percentages of carbon tetrachloride adsorbed were not determined.

Iodine and Permanganate Tests

The procedures are the same as before; and the results are listed as follow:

Charcoal	Percent I_2 removed	Percent $KmnO_4$ removed
Walnut shell	55.5	9.75
Apricot pit	43.6	8.73
Filbert shell	46.3	7.68

Discussion

The results of the three tests show that, the charcoals were not activated enough. In order to activate these charcoals to be qualified as adsorption carbon, it seems to be necessary to prolong the heating by superheated steam.

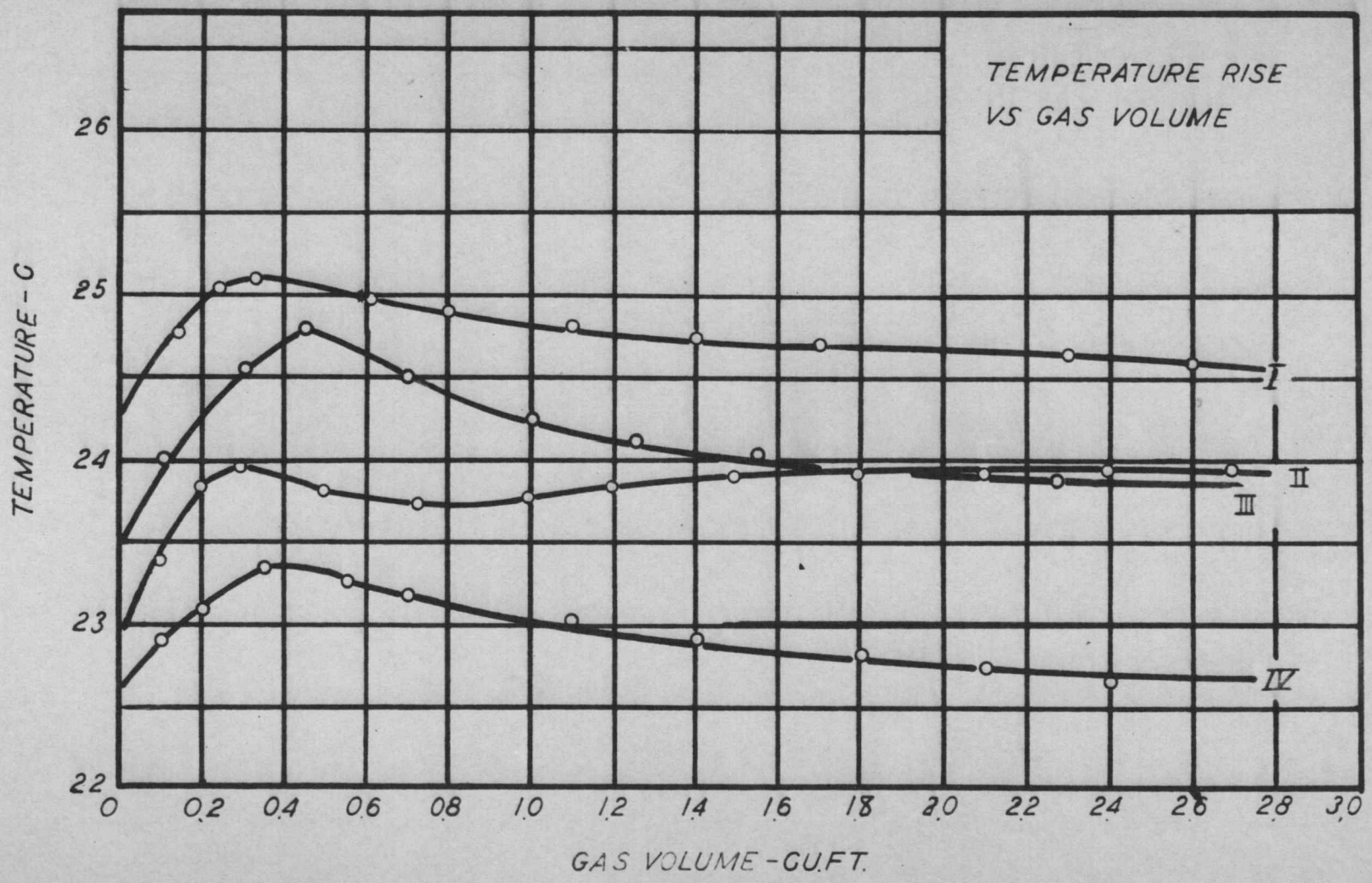

TEMPERATURE RISE
VS GAS VOLUME
TEMPERATURE - C
26
25
24
23
22
0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
GAS VOLUME - CU.FT.
I
II
III
IV

FIG. 9

28-A

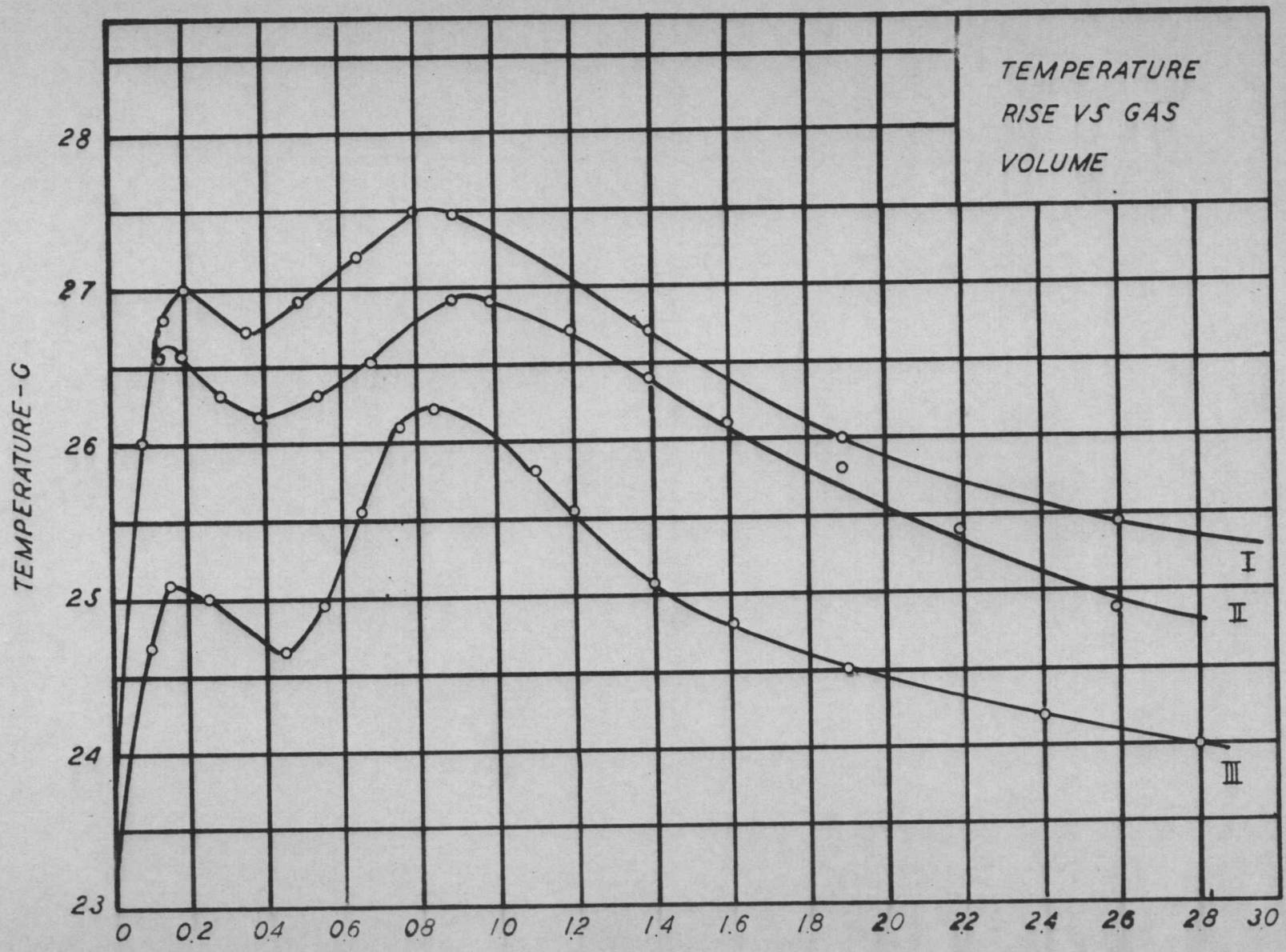

TEMPERATURE RISE VS GAS VOLUME
TEMPERATURE-C
28
27
26
25
24
23
0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
I
II
III
GAS VOLUME-CU.FT

FIG. 10

CONCLUSIONS

As the results of the research discribed in this writing, general conclusions can be drawn. For the sake of clearness they are numerated as follows:

(1) For the detection of constituents in a gas mixture, as shown by Curve I, in Figure 2, the correlation between temperature peaks and gas volume proves to be a satisfactory method.

(2) The results of carbon tetrachloride modification test on the carbon proves the specified time is a reliable indication of quality.

(3) A conclusion from the curve in Figure 7 on the carbon tetrachloride humidity test is that the relation between per cent stauration of the carbon and volume of air passed through were directly proportional up to 92% saturation.

(4) The result of iodine test of Columbia Activated Carbon indicates its value is beyond the usual specified value.

(5) The comparision of Curves I, II, and III, in Figure 3 shows the method of reactivation to be sound.

(6) The time for activation as reported in this writing is not enough.

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Part I

A Data on Adsorption Test

Sample: Columbia Activated Carbon

Gas: Portland city gas

Rate of gas flow: 0.10 cu.ft./min.

Top thermometer	Low thermometer	Volume of gas
21.70	20.10	0
23.00	"	0.172
26.90	"	0.203
23.80	"	0.284
23.40	20.15	0.400
23/30	"	0.440
23.40	"	0.600
23.25	"	o.700
23.20	20.12	0.880
23.14	20.10	1.100
23.43	20.10	1.400
24.10	"	1.600
25.07	"	1.800
25.25	"	1.940
25.12	"	2.010
24.73	"	2.280
24.30	"	2.600
23.92	"	2.900

B. Data on Adsorption Test

Sample: Columbia Activated Carbon

Gas: Portland City Gas

Rate of gas flow: 0.45 cu.ft./min.

Top thermometer	Low thermometer	Volume of gas
21.70	20.50	0
23.74	"	0.05
25.60	"	0.10
23.70	"	0.16
23.40	"	0.35
23.45	"	0.47
23.45	"	0.52
23.20	20.40	0.70
22.86	20.45	0.90
22.63	20.47	1.20
22.72	20.45	1.50
23.45	"	1.70
23.95	20.42	1.82
23.30	20.40	2.10
22.50	20.40	2.40
22.15	"	2.60
22.76	"	2.90

C. Data on Adsorption Test

Sample: Columbia Activated Carbon

Gas: Portland City Gas

Rate of gas flow: 0.10 cu.ft./min.

Top thermometer	Low thermometer	Volume of gas
21.75	21.04	0
22.75	"	0.10
24.45	"	0.22
23.75	"	0.28
23.25	"	0.31
22.80	"	0.40
22.77	"	0.51
23.00	"	0.65
22.77	"	0.80
22.60	"	1.10
22.55	"	1.40
22.70	"	1.71
23.30	"	1.91
23.60	"	1.96
23.85	"	2.05
23.35	"	2.20
23.10	"	2.30

D. Data on Adsorption Test

Sample: Columbia Activated Carbon - Reactivated

Gas: Portland City Gas

Rate of gas flow: .075 cu. ft./min.

Top thermometer	Low thermometer	Volume of gas
21.25	21.00	0
24.70	"	0.21
24.00	"	0.30
23.30	"	0.41
22.70	"	0.61
22.30	"	0.91
22.15	"	1.21
22.23	19.80	1.61
22.75	"	1.91
23.30	"	2.01
23.50	"	2.11
23.53	"	2.21
23.42	"	2.38
23.17	"	2.52
22.90	"	2.72
22.70	"	2.90

PART III

A. Data on Carbon Tetrachloride Humidity Test

Sample: Columbia Activated Carbon = 127.1 gm.

Rate of Air Flow: 0.075 cu.ft./min.

Wet Bulb thermometer	Dry Bulb thermometer	Tempera-ture diff.	Volume
22.0	23.2	1.2	0.000
11.7	23.0	11.3	0.112
8.0	23.0	15.0	0.336
7.4	22.9	15.5	0.610
6.8	22.8	16.0	1.210
5.8	21.4	15.6	4.030
6.0	21.3	15.3	4.185
6.1	21.1	15.0	4.345
6.2	21.0	14.8	4.455
6.4	21.0	14.6	4.640
7.5	21.0	13.5	5.000

B. Data on Carbon Tetrachloride Humidity Test

Sample: Steamed Used Columbia Activated Carbon = 130.4 grams.

Rate of Air Flow: 0.075 cu.ft./min.

Wet Bulb thermometer	Dry Bulb thermometer	Temperature difference	Vol.	Top therm.
24.0	26.0	2.0	0	

Wet Bulb thermometer	Dry Bulb thermometer	Temperature difference	Vol.	Top therm.
11.0	25.6	14.6	0.150	
6.9	24.5	17.6	1.102	
6.0	23.9	17.5	4.448	50.0
7.1	23.9	16.9	4.513	58.2
11.8	24.3	12.5	4.645	62.0
16.2	24.5	8.3	4.775	52.2
19.0	24.6	5.6	4.908	47.1
21.2	24.6	3.4	5.308	35.0
22.0	24.5	2.5	5.507	33.2

C. Data on Percent Saturation of Carbon

Sample: Columbia Activated Carbon - 127.1 gm.

Rate of Air Flow: 0.075 cu.ft./min.

Total Carbon Tetrachloride Adsorbed = 49.33 gm

Volume of Air Passed Through	Percentage of Saturation
0.131	3.25
0.5197	15.0
1.121	28.2
1.710	43.0
2.276	55.5
2.816	71.5

Volume of Air Passed Through	Percentage of Saturation
3.231	80.5
3.471	86.0
3.620	92.0
3.894	95.0
4.197	98.0
4.510	100.0

Part VII

Activation

A. Adsorption Test Before Activation

a. Sample: Apricot Pit Charcoal

Gas: Portland City Gas

Rate of Gas Flow: 0.084 cu.ft./min.

Volume of Gas	Top thermometer	Low thermometer
0.00	24.30	22.80
0.14	24.70	22.80
0.22	25.05	22.80
0.32	25.15	22.83
0.60	25.00	22.81
0.80	24.85	22.81
1.40	24.75	22.80
1.70	24.73	22.80
2.30	24.68	22.80
2.60	24.65	22.83

b, Sample: Walnut Shell Charcoal

Gas: Portland City Gas

Rate of Gas Flow: 0.075 cu.ft./min.

Volume of Gas	Top thermometer	Low thermometer
0.00	23.50	22.43
0.10	24.00	22.43
0.30	24.05	22.43
0.43	24.80	22.43
0.70	24.50	22.43
0.99	24.25	22.43
1.25	24.10	22.43
1.55	24.04	22.43
1.80	23.90	22.43
2.10	23.85	22.43
2.27	23.75	22.43

d. Sample: Filbert Shell Charcoal

Gas: Portland City Gas

Rate of Gas Flow: 0.084 cu.ft./min.

Volume of Gas	Top thermometer	Low thermometer
0.00	22.90	21.00
0.10	23.35	21.00
0.20	23.80	21.00
0.39	23.97	21.00
0.49	23.80	21.00

Volume of Gas	Top thermometer	Low thermometer
0.71	23.70	21.00
1.00	23.75	21.00
1.20	23.80	21.08
1.49	23.90	21.14
1.80	23.87	21.14
2.10	23.87	21.14
2.40	23.89	21.14
2.68	23.88	21.14

d. Sample: Silica Gel

Gas: Portland City Gas

Rate of Gas Flow: 0.092 cu.ft./min.

Volume of Gas	Top Thermometer	Low Thermometer
0.00	22.65	21.40
0.10	22.84	21.40
0.20	23.12	21.40
0.35	23.35	21.40
0.55	23.28	21.46
0.70	23.18	21.44
1.10	23.00	21.42
1.40	22.90	21.38
1.80	22.80	21.42

Volume of Gas	Top thermometer	Low thermometer
2.10	22.75	21.44
2.40	22.68	21.46

B. Adsorption Test After Activation

a. Sample: Apricot Pit Charcoal

Gas: Portland City Gas

Rate of Gas Flow: 0.073 cu.ft./min.

Volume of Gas	Top thermometer	Low Thermometer
0.00	23.75	21.60
0.08	26.00	21.60
0.12	26.80	21.60
0.20	27.00	21.60
0.33	26.75	21.60
0.48	26.90	21.60
0.62	27.20	21.58
0.80	27.50	21.58
0.88	27.48	21.63
1.40	26.70	21.63
1.88	26.00	21.63
2.60	25.48	21.63

b. Sample: Walnut Shell Charcoal

Gas: Portland City Gas

Rate of Gas Flow: 0.091 cu.ft./min.

Volume of Gas	Top thermometer	Low thermometer
0.00	23.20	21.00
0.10	24.70	21.00
0.15	25.13	21.00
0.25	25.00	21.00
0.44	24.65	21.08
0.55	24.96	21.10
0.65	25.56	21.06
0.73	26.12	21.05
0.84	26.34	21.02
1.10	25.77	21.20
1.20	25.54	21.20
1.40	25.07	21.08
1.60	24.75	21.10
1.90	24.50	21.12
2.40	24.20	21.12

c. Sample: Filbert Shell Charcoal

Gas: Portland City Gas

Rate of Gas Flow: 0.085 cu.ft./min.

Volume of Gas	Top thermometer	Low thermometer
0.00	24.00	22.10
0.08	26.04	22.10
0.14	26.56	22.12
0.20	26.57	22.09
0.28	26.32	22.09
0.39	26.20	22.09
0.54	26.35	22.09
0.68	26.50	22.09
0.88	26.90	22.09
0.99	26.87	22.09
1.20	26.70	22.09
1.40	26.40	22.09
1.60	26.13	22.09
1.89	25.78	22.09
2.20	25.43	22.09
2.60	24.83	22.09