THE DENSITY OF CHARCOAL

BY

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

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ENTITLED The Density of Charcoal

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Bachelor of Science in Chemical Engineering.

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INDEX

THE DENSITY OF CHARCOAL

INTRODUCTION

The density of any one of the charcoals used by the United States army in the great conflict just terminated is of little value in itself except as a characteristic of the material. Moreover, as it is a very porous material, it is exceedingly difficult to determine its exact specific gravity. In fact, if we determine its density by evacuating it at a high temperature to remove all the gases, and then immerse it in a liquid, we find that the density is different depending on the liquid used. The variation is considerable, and the increase from the lowest to the highest value is about one hundred percent. From this we may say that the charcoal has not one but a number of densities.

The object of this investigation was to determine the density of one charcoal using several liquids and necessarily to perfect an apparatus and procedure for this purpose. With this physical property determined for a number of different liquids, we would have data that would be of great value in studying the phenomenon of adsorption. This knowledge would enable us to better speculate as to the physical condition of the adsorbent and perhaps

also increase our knowledge about liquids.

An examination of the published literature resulted in the finding of a very meager quantity of material on this subject. Several authors gave a figure which was to represent the density of the charcoal they used for their investigations, but none of them told in what manner this was obtained, and so a comparison of their results would be without profit. A few of the densities published by some of the workers on adsorption will illustrate this. Gerhard C. Schmidt and Bernhard Hinteler in their article, Uber Adsorption, in the Zeitschrift für Physicalishe Chemie, volume ninety-one gave the specific gravity of Kahlbaum's cocoanut charcoal as 1.47. Arthur B. Lamb, Robert E. Wilson, and N. K. Chancy in their article on, Gas Mask Absorbents, in the Journal of Industrial and Engineering Chemistry, Volume Eleven 1919 gave the apparent density of the cocoanut charcoal used by the United States in 1917 as 0.60 and that used in 1918 as 0.51. Alexander Titoff gave a figure of 1.860 as the specific gravity of the cocoanut shell charcoal he used, in his article, Die Adsorption von Gas durch Kohle, in the Zeitschrift für Physicalishe Chemie, Volume Seventy-four. He explains that the charcoal was evacuated at four hundred degrees Centigrade to a pressure of three thousandths of a millimeter of mercury for this determination. It is thus apparent that an investigation based on the varying density of charcoal is in an entirely new field.

APPARATUS

The apparatus used for this investigation consisted of an outgassing battery surrounded by an electric furnace. A high vacuum pump system produced the low pressure which was measured with a McLeod gage. The entire set-up is shown in the photograph, plate 3 and the diagrams, plates 1 and 2.

Plate 1 shows the outgassing battery with all the necessary equipment. The Pyrex glass tubes (A) in which the charcoal was placed were connected by means of a ground connection to the stop cocks (B) which in turn were connected to the tubes from the main line of the outgassing apparatus. The tubes held about fifteen grams of charcoal and were surrounded by an electric furnace, which was supplied by a one-hundred-ten volt direct current and had a rheostat in series to regulate the temperature. The furnace was suspended by two cords which passed over pulleys to counterweights which enabled it to be readily raised and lowered. When in place a stool was slid under it to support it. An asbestos board was placed on the top of the furnace and another one held in place just below the first ground joints, three or four inches above the first board. An electric fan created a current of air between these heat insulators to keep the upper part of the apparatus cool so that the grease in the stop cocks and ground joints would not soften and cause leaks. A pyrometer (L) consisting of a copper-constantin thermo couple, incased in a Pyrex glass tube, and a millivoltmeter served to determine the temperature in the furnace. The stop cocks (B), which were connected to the vacuum line above and the Pyrex tubes below, permitted the tubes to

be removed from the frame and the vacuum retained. They also served to make pycnometers out of the tubes. Just below the stop cocks (C), which enabled one or more of the tubes to be evacuated at a time and served to isolate the tubes from the system when liquids or gases were admitted, were the side tubes on which were the stop cocks (D). These tubes led off to the side of the apparatus and terminated in the capillaries (E). The lower extremities of these capillaries were sealed off and by breaking them under the surface of a liquid the tubes could be filled without the admission of air. From the main vacuum line to the supply line was the connecting tube on which was the large stop cock (F). The tube (G), which had a ground cap fitted to it and which connected to the vacuum supply line, had a glass boat containing phosphoric anhydrid as a drying agent inserted in it. The stop cock (H) on the side tube served as a means to relieve the vacuum in the apparatus. A small discharge tube (I) connected to an induction coil was of use in determining the rough vacuum without the use of the gage. It was designed to enable the spectrum of the enclosed gas to be taken. Several stop cocks (J) were on the line from the main line to the McLeod gage (K) which measured the pressure in the apparatus. On the reservoir side of the gage was a tube leading to a stop cock that connected a Cenco-Nelson two stage oil pump and a constricted tube open to the air, to the mercury reservoir. By use of this contrivance the mercury could be easily raised into the capillary and lowered from it.

A frame of wood supported the glass tubing. The glass was firmly fastened to the wood with copper wire and was protected from the wood and the copper with sheet rubber.

PLATE 1.
OUTGASSING BATTERY

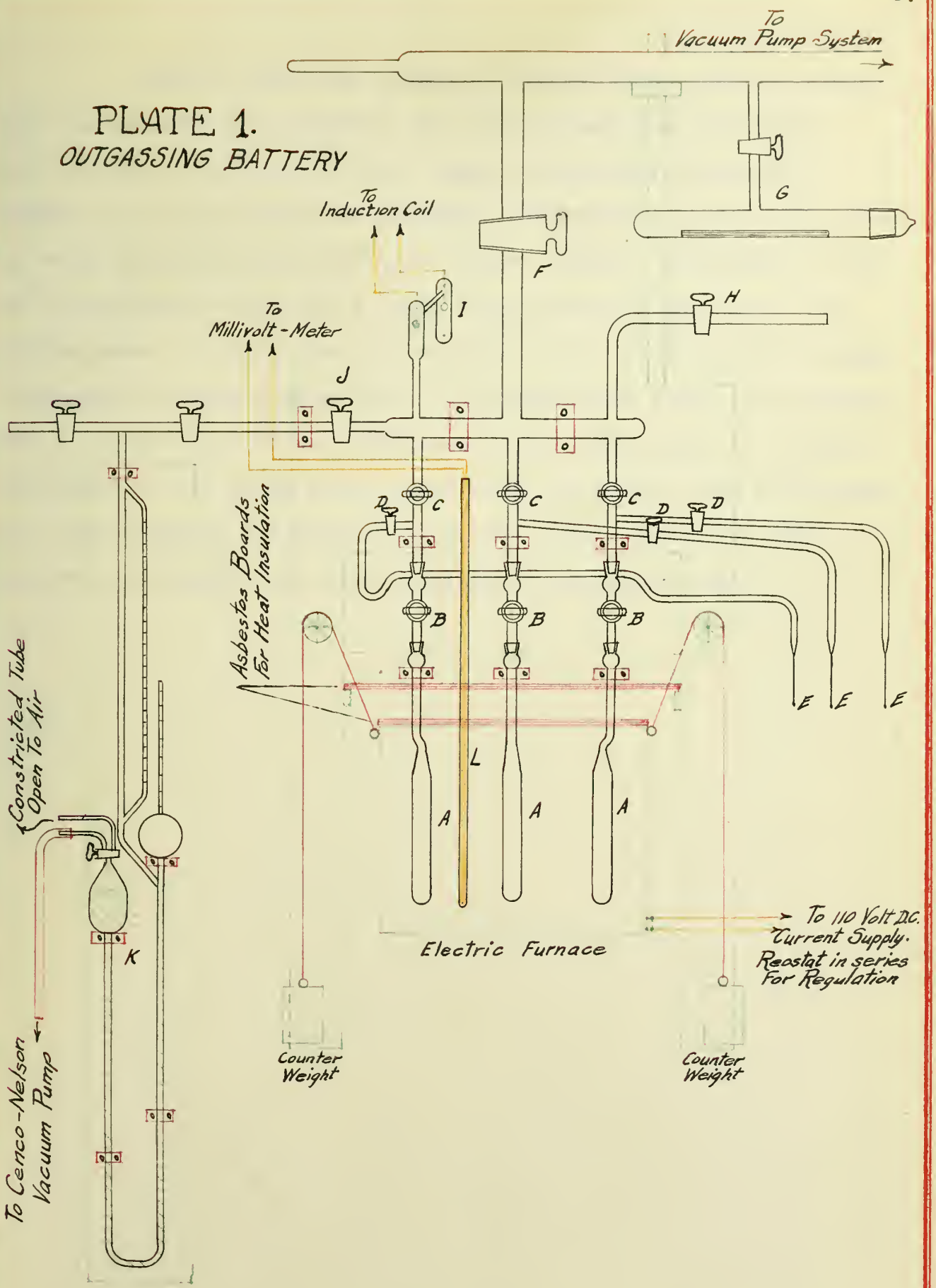
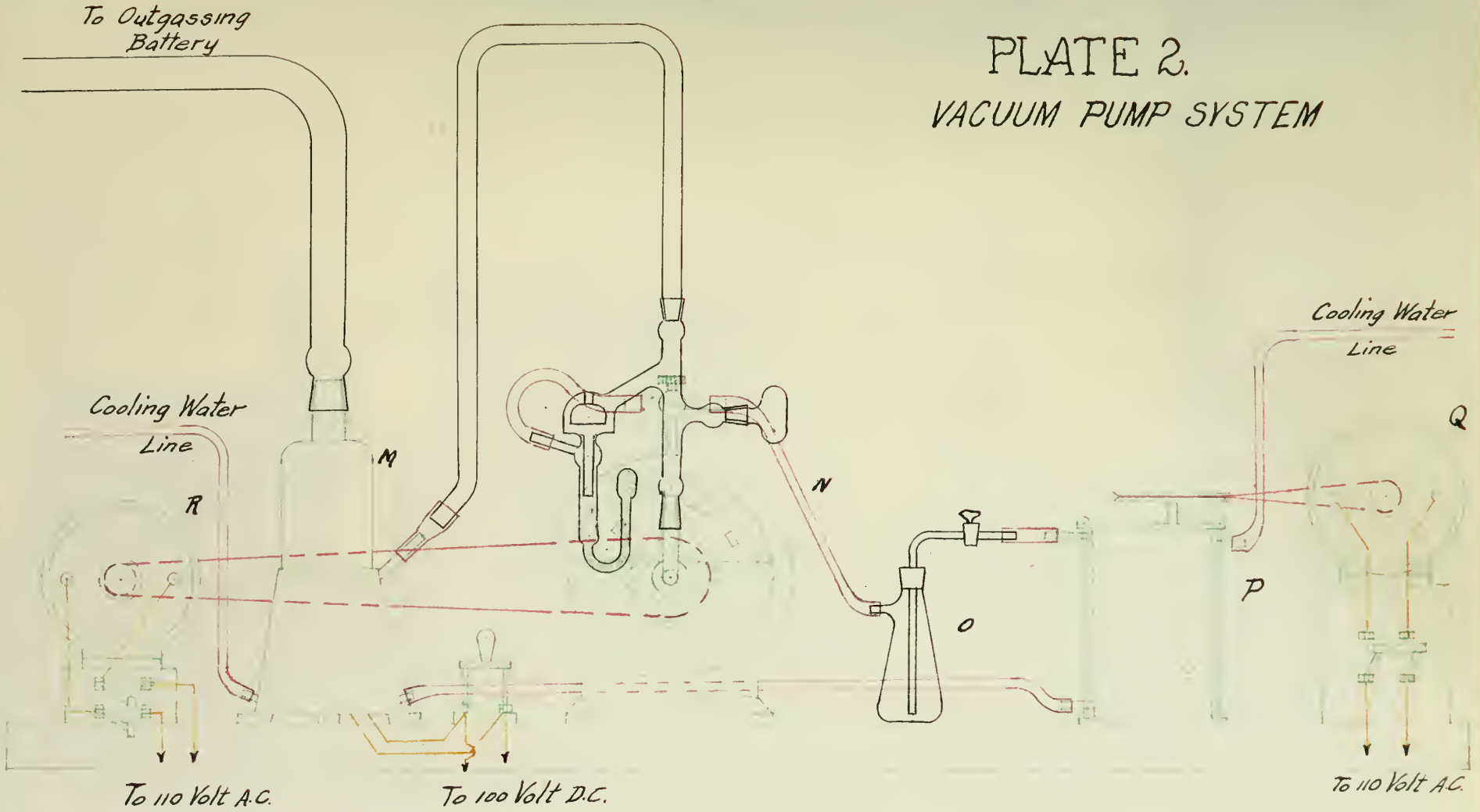


Plate 2 shows the system of vacuum pumps used to produce and maintain the low pressure. The high vacuum pump (M) was a mercury vapor condensation pump made by the General Electric Company after the patent of Langmuir. It is water cooled and heated by means of one-hundred-ten volt direct current. Connected to this as a preliminary pump was a Gaede rotory mercury pump (N). The motive power for this pump was obtained from an Emerson one eighth horsepower induction motor (R). The preliminary pump of the system was a Cenco-Nelson three stage water cooled oil pump (P). It was connected to the Gaede with an oil trap (O) in the line to prevent oil from entering the mercury pump. The motive power for this pump was an Emerson one sixth horsepower induction motor.

PLATE 2.

VACUUM PUMP SYSTEM



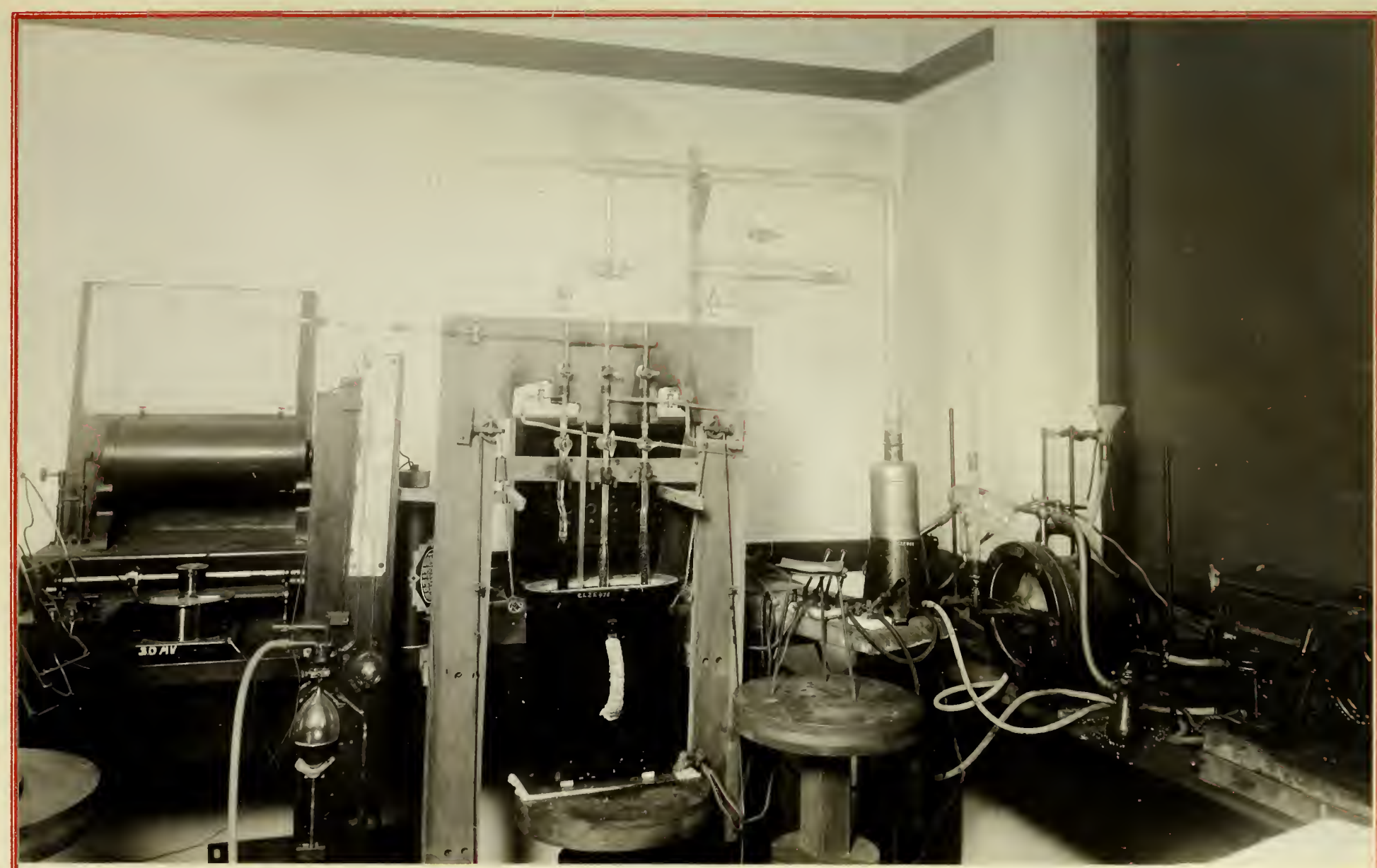


PLATE 3. *THE APPARATUS.*

MATERIALS USED

The charcoal used in this work was all obtained from the United States Chemical Warfare Service at the American University in Washington D. C. It was a cocoanut shell charcoal made for the use in gas masks by the United States and was designated as E-621. It was sized, and only that which passed a ten mesh sieve and remained on a fourteen was used.

The liquids used were four in number, mercury, water, benzene, and pseudocumene. The mercury was purified by distillation and then by passing it in a fine stream through a column of a mercurous nitrate solution. The water was distilled and then boiled just before use to remove dissolved gases. It was used while hot. The benzene was the pure material obtained from the laboratory store. The pseudocumene was obtained by the fractional distillation of the commericial 160 to 170 degree Centigrade pseudocumene. The fraction boiling between 168 and 170 degrees Centigrade was used.

METHOD OF MAKING
A DENSITY DETERMINATION

The following procedure was followed in making a density determination. The Pyrex tube (A) and the first stop cock (B) were cleaned, put together with vacuum grease, and weighed empty. The assembled tube was then put on the apparatus, and the pumps started. The Cenco-Nelson pump (P) was started first; and when the manometer on the Gaede showed about a centimeter of pressure, it was started. The heating coil of the Langmuir was turned on. The electric furnace was drawn up to surround the tube, and the current turned on. The stop cocks (F), (C), and (B) were opened. The temperature of the furnace was kept at 500 degrees Centigrade; and when the gage gave a reading of 0.01 mm. of mercury or less, the stop cocks (C) and (B) were closed, the furnace turned off and removed, the connection between these stop cocks broken, and when the tube was cool it was again weighed. The difference between these two weighings gave the weigh of the charcoal without any of the adsorbed air or moisture. There would be an appreciable error encountered if the charcoal were weighed before these were removed. The tube was connected to the apparatus again, the furnace replaced, and the evacuation continued, this time until the gage gave a reading of about 0.001 mm. of mercury pressure while the temperature stood at 500 degrees. The stop cock (C) was then closed, the furnace removed; and, when the tube was cool enough, the tip of the cappillary (E) was broken off under the surface of the liquid with which the tube was to be filled.

When the tube was full, it was removed from the apparatus,

put in a thermostat at 25 degrees Centigrade, and allowed to remain there until it assumed that temperature. It was then dried, stop cock (B) closed, and any of the liquid above it removed. The weight was taken, and the difference between this weight and the last one gave the weight of the liquid admitted.

The tube was then cleaned out, weighed, filled with mercury, and put in the thermostat at 25 degrees. On its removal stop cock (B) was closed, the excess liquid poured off, and the dried tube weighed again. The volume of the tube was then calculated from the amount of mercury it held. It was necessary to determine this after every run, because it changed appreciably. The density of the charcoal was then calculated, knowing its weight and volume. Its volume was the difference between the volume of the tube and that of the liquid admitted.

RESULTS OF THE DETERMINATIONS MADE

The density of the charcoal, as determined, expressed in grams per cubic centimeter, is as follows.

The liquid admitted----------Mercury

0.99898

0.99817

1.00343

Mean 1.0002

The liquid admitted----------Water

1.8401

1.8205

1.8356

Mean 1.8321

The liquid admitted----------Pseudocumene

1.9023

1.8300

Mean 1.8661

The liquid admitted----------Benzene

1.9068

1.9068

1.9282

Mean 1.9139

The apparent density of the charcoal was deyermined by filling a pycnometer with it and pouring mercury in until the flask was full. A value of 0.8855 grams per cubic centimeter was obtained as the apparent density.

EXPERIMENTAL OBSERVATIONS AND DIFFICULTIES

In admitting benzene to the outgassed charcoal with a small capillary, the liquid evaporated at the upper end of the capillary with such rapidity that it solidified; and the admission proceeded only as rapidly as the evaporation took place from the solid state. At the same time the tube became appreciably warmer due to the heat of adsorption. It was not until this rapid adsorption ceased that the benzene went over as the liquid.

It was first attempted to carry out the evacuation at 550 degrees Centigrade, but with this charcoal it was found to be impossible. As the temperature rose above 500 degrees, the pressure also rose; and even after evacuating for eight hours at 550 degrees, the pressure would still be toohigh. By using 500 as the temperature to outgas the charcoal, a pressure of 0.001 mm. of mercury could be obtained in about eight hours.

Another difficulty encountered was that the heat from the furnace would soften the stop cock grease on the ground joints and stop cocks and cause them to leak. This was remedied by placing two parallel asbostos boards above the furnace and having an electric fan blow a current of air between them.

THEORETICAL CONSIDERATIONS

The charcoal made for adsorption purposes contains a large number of very small interstices. The carbon comprising the walls of these small holes is in an amorphous condition, which exerts very strong attractive forces on the molecules of gases and, according to the prevailing theories, will cause a layer of molecules one or two in thickness to adhere to its surface.

We may attempt to explain the differences in the densities, as obtained with the use of the different liquids, on two general lines. We may first assume the liquid to enter the interstices of decreasing size until the surface tension of the liquid is equal to the attraction of the charcoal and the pressure differences caused by the vacuum in the pores and the atmospheric pressure. The results show the density of the charcoal diminishing with increasing surface tension of the liquid.

We come into difficulties with this assumption, when we take into consideration the attractive force of the carbon for the molecules of the liquid. These would evaporate from the liquid surface, adhere to the carbon, and so fill up the entire pore space except for such pores that are smaller than the size of the nolecule. If this is the case, then liquids with smaller molecules would give larger figures for the density of the charcoal. That mercury, being adsorbed very little, would not fill the entire available space, does not seem incredible; but liquids which are very strongly adsorbed, as for example benzene, we would assume to fill up all interstices

If we consider that all liquids with the exception of

mercury entered the same amount of pore space, then we may explain the differences of the densities obtained, by considering a layer of molecules on the surface of the carbon to be held by a very strong attractive force and to thus be compressed to a greater density than is normal for the liquid. Under this assumption we would expect that liquids with either greater compressibility or having a greater attractive force for the charcoal or a combination of both would give higher values for the density of the charcoal. Apparently this is the case.

POSSIBILITIES OF THIS FORM OF INVESTIGATION

As has been mentioned before, a large number of results taken under similar conditions to enable them to be compared would perhaps shed light upon the matter of adsorption and the condition of the charcoal used for that purpose. By using liquids varying in properties, a comparison of results in any one line would be profitable. If we use liquids with as great and continuous a range of molecular size as is possible, we could investigate the variation in determined density with molecular size. It may be that the compressibility of the liquid is the important thing and that size of molecule and surface tension do not play important parts. An investigation along this line would point this out. To sum it all up this sort of experiment would determine which the important factor is, and a knowledge of this would help greatly in the study of the phenomenon of adsorption.

If we knew the manner in which the various physical properties of the liquids affected the density when determined in the manner explained in this thesis, we could, by standardizing one charcoal with liquids of known properties, investigate these for liquids for which they were unknown. This might afford a means of determining the size of molecules or other properties of liquids.