



TABLE OF CONTENTS

	PAGE
I. INTRODUCTION	1
II. LITERATURE REVIEW	4
A. Early History of Adsorption	4
B. Theories of Adsorption	4
C. Industrial Adsorbents	9
D. Factors Affecting Adsorption by Carbonaceous Materials	10
E. Adsorptive Properties of Coal	13
F. Use of Coal as an Adsorbent	15
III. EXPERIMENTAL	16
A. Purpose of the Investigation	16
B. Plan of Investigation	16
C. Materials	24
D. Apparatus	29
E. Method of Procedure	39
F. Data and Results	49
G. Macrophotographs	80
H. Drawings of Equipment	85
IV. DISCUSSION	89
A. Discussion of Results	89
B. Recommendations	103
V. CONCLUSIONS	106
VI. SUMMARY	108
VII. BIBLIOGRAPHY	110
VIII. ACKNOWLEDGMENTS	116

## I. INTRODUCTION

Adsorption, as defined by Mantell, (30) is a unit operation in the chemical engineering field which deals with forces of attraction on surfaces and the concentration of a substance on a solid surface as a result of these attraction forces. The operation is generally a physical action and not a chemical one. However, the material being adsorbed may sometimes react chemically with the solid.

There are many theories on the mechanism of adsorption. Langmuir (26) proposed a theory that adsorption is a type of chemical union and that the adsorbed layer is unimolecular in thickness. Polanyi (43) stated that adsorption is a physical process, electrical in nature, and that the adsorbed substance is many layers thick. Magnus (28) proposed a theory of unimolecular adsorption assuming that the forces of attraction between the surface of the adsorbent and a gas are electrostatic in nature. DeBoer and Zwikker (15) suggested the polarization theory. Induced polarization of a gas by the surface of the adsorbent causes the concentration of the gas on the adsorbent.

Despite the fundamental work done, the efficiency of a certain adsorbent, when used in a given process, cannot be predicted. The selection of an adsorbent for a process is made by trial alone.

Little is known as to why one material is a good adsorbent for one process and not for another, or why another material of about the same composition is not a good adsorbent at all.

The industrial uses of adsorbents may be classified by the physical states of the substances treated. Gases are extracted from gases and liquids; liquids are separated from liquids; dissolved solids are extracted from liquid solutions; and colloidal suspensions are removed from liquids.

The adsorption of colloidal suspensions in the purification of water is accomplished by various adsorbents. One of the materials used is coal, but not all coals have the same adsorption properties. Hassler (20) states that the extent of the surface area influences adsorption. Jones and Buller, (22) in writing about coal as a filter medium, believe that the angularity of the particles adds a great deal to the effectiveness of the coal as an adsorbent.

Tannin is extracted from chestnut chips with steam by the Mead Corporation, Lynchburg, Virginia. The tannin solution obtained contains 0.08 percent chips of colloidal dimensions and certain non-desirable non-tannins. These "Insoluble Solids" must be allowed to settle out of suspension, a long and involved process. It is thought that possibly the "Insoluble Solids" may be adsorbed by percolating the solution through a bed of coal.

The purpose of this investigation is to correlate the adsorption of colloidal particles from tannin extract solution obtained

from the Mead Corporation of Lynchburg, Virginia, by coal, varying in rank from anthracite to lignite, with surface area and roughness of surface.

## II. REVIEW OF LITERATURE

### A. Early History of Adsorption

During the thirteenth century, Marco Polo observed, in Un-guen, the use of ashes of certain woods to refine sugar. (36) About the first scientific work done on the phenomena, adsorption, was in 1773 by C. W. Scheele. He investigated the "uptake" of gases by charcoal as did the Abbe F. Fontana in 1777. In 1785 T. Lowitz discovered that charcoal would remove the coloring matter from certain solutions (38). In 1794 an English sugar refinery used charcoal to remove color from sugar solutions. Bone char was introduced into the refining of sugar in 1812, and it is still used by the bulk of the sugar manufacturers (54).

### B. Theories of Adsorption

Evaluation of Adsorption. The first attempt to evaluate adsorption by means of an equation theoretically derived was made by Gibbs (19) in 1874. This equation is interpreted qualitatively to mean that a substance, which will lower the surface tension at the interface, will be positively adsorbed at the interface.

A system in which adsorption occurs is usually evaluated by means of the adsorption isotherm <sup>(35)</sup>. The isotherm is a plot of volume adsorbed against pressure of the system with the temperature constant. For most adsorbents the isotherms of gases at temperatures close to their condensation points are divided into two regions. At low pressures the curve is concave toward the pressure axis; while at high pressures, it is convex. Some investigators <sup>(35)</sup> believe that the convex portion is due to condensation in the capillaries of the adsorbent. Others <sup>(35)</sup> think that this portion is due to the formation of multimolecular layers.

Exponential Empirical Equation. Attempts have been made to apply the mass law to adsorption. The most valuable of these is the exponential equation of Freundlich <sup>(17)</sup>.

$$a = x c^{1/n}$$

where  $a$  = amount of substance adsorbed per gram of adsorbent

$c$  = amount of substance left in solution

$1/n$  = a constant usually between 0.1 and 0.7

$x$  = a constant.

He found that, as a rule, the quantity adsorbed decreased with rising temperature; while the constant  $1/n$  increased, approaching unity. Mantell <sup>(33)</sup> states that the equation holds fairly well over narrow ranges in many cases.

Unimolecular Theories of Adsorption. Langmuir <sup>(26)</sup> stated that adsorption is a direct consequence of the time lag between the condensation and evaporation of molecules on a surface. It is a

strong chemical union due to primary valences in the case of active adsorbents and a weak chemical union due to secondary valences in the case of inert adsorbents. He derived the equation:

$$\theta_1 = \frac{a u}{v_1 + a u}$$

in which  $\theta_1$  = amount of gas adsorbed

$u$  = number of gram molecules of gas striking each sq. cm. of surface per second

$v_1$  = rate of evaporation if the surface is completely covered

$a$  = constant, very close to unity.

Mantell <sup>(34)</sup> writes that the equation is true for nonporous surfaces where the gas molecules have free access and for adsorbents in which the attraction centers are equal and uniformly distributed over the surface. The equation does not give close agreement between experimental and calculated values at high pressures.

Benton <sup>(5)</sup> expanded on Langmuir's primary and secondary valences. Primary adsorption is selective and irreversible and is determined by chemical affinity in its usual sense. This type of adsorption increases and then decreases with rising temperature. It increases rapidly with pressure, at low pressures, but soon reaches a saturation value beyond which the quantity adsorbed is independent of further increases in pressure. In secondary adsorption the valence forces are weaker and less specific. The tendency for different gases to be held is measured by their relative boiling or melting points.

The tendency for different adsorbents to hold a gas by this type of adsorption may be measured by their melting points or related properties. Secondary adsorption decreases rapidly with increasing temperatures. The gas adsorbed increases gradually with increasing pressure, and the surface does not become saturated until relatively high pressures are reached.

Taylor (50) believes that the only essential distinction between primary and secondary adsorption is that secondary adsorption or reversible adsorption processes are accompanied by small heats of adsorption and small activation energies, while primary or irreversible adsorption processes are accompanied by moderate or large energies of activation, and, frequently, high heats of adsorption.

Magnus (28) proposed a theory of unimolecular adsorption assuming that the attraction of a gas to a solid surface is due to electrical forces on the surface of the solid of an opposite charge from the dipole charges of the gas. The dipole charges may be induced or natural. (Dipoles, according to Mantell (32), are neutral molecules which have a positive and negative part and are free to align themselves so as to satisfy the charges.) Magnus stated (29) that the adsorbed layer may be multimolecular for natural dipoles at comparatively low pressures.

Capillary - Condensation Theory. Zsigmondy (55) advanced the capillary-condensation theory to explain the adsorption of water vapor by silica gel. When a gas below its critical temperature is

brought in contact with a porous solid, the gas should condense in and fill the pores of the solid. Mantell (35) wrote that "capillary-condensation in the liquid state is probably an important factor if the adsorbent is porous" as the gas pressure approaches the saturation pressure. At lower pressures, 20 to 50 percent of saturation pressure, the theory does not apply.

Multimolecular Theories. Polanyi (43) proposed that adsorption is a physical phenomena, electrical in nature. The process is due to a deformation of molecules to bring opposite charges as close together as possible. At low temperatures the adsorption system is like an ideal concentrated solution. As the temperature increases, the behavior of the system gradually approaches that of a dilute solution. The adsorbed layer is highly compressed by molecular forces and is many molecules thick.

DeBoer and Zwikker (15) advanced the polarization theory. The surface molecules of an adsorbent induce dipoles in the first layer of molecules of the substance adsorbed, if the molecules are non-polar. These dipoles, in turn, attract other molecules of the adsorbate by inducing dipoles. Thus, by electrical attractive forces, several layers are built up. If the adsorbate is made of molecules of natural dipoles, the attractive forces are much stronger.

Brunauer, Emmett, and Teller (8) proposed that the adsorption energy due to attraction of dipoles induced into a non-polar gas is insufficient to account for the major portion of the binding energy

between the adsorbed layers. The same forces that produce condensation are also responsible for multimolecular adsorption.

### C. Industrial Adsorbents

The following is a list of the commercially important solid adsorbents, in the order of tonnage consumed (3/):

Fuller's earth - Refining of petroleum fractions vegetable and animal oils and fats, and waxes.

Bauxite - Percolation treatment of petroleum fractions; dehydration of gases.

Acid-treated clays - Contact filtration of petroleum fractions.

Bone char or bone black - Sugar refining; ash removal from solutions.

Decolorizing carbons and water carbons - Sugar refining; refining of vegetable and animal oils and fats and of waxes, miscellaneous decolorizing of inorganic and organic substances; water purification; purification of dry cleaning fluids; purification of food products.

Gas adsorbent carbon - Solvent recovery; recovery of gasoline from natural gas; elimination of industrial odors; purification of carbon dioxide and industrial gases; gas masks.

Alumina - Dehydration of air, gases, and liquids.

Silica gel - Dehydration and purification of air and industrial gases; air conditioning; refining of petroleum distillates; gas masks.

Base-exchange silicates - Water treatment.

Magnesia - Treatment of gasoline and regeneration of dry cleaning solvents.

Medicinal carbons - Elimination of bacteria and toxic poisons; an addition to animal foods.

Metal-adsorbent chars - Recovery of precious metals.

Synthetic Resinous Exchangers - Recovery of precious metals.<sup>(56)</sup>

Another commercial adsorbent <sup>(51)</sup> that is used, but has obtained little publicity, is "Anthrafilt". This is a cleaned and sized anthracite that is used as a substitute for sand in water filtration to remove oil, bacteria, and colloidal particles. To a lesser extent it removes tastes and odors from water. It is used to filter acetic acid, sulfuric acid and caustic soda solutions and can clarify other chemical solutions.

#### D. Factors Affecting Adsorption by Carbonaceous Materials

Nature of Carbon in Adsorbent. Chaney <sup>(10)</sup> proposed that, in the ordinary process of distillation of carbonaceous materials at low temperatures, active carbon is formed by the thermal decomposition of unstable hydrocarbons. This active carbon adsorbs a quantity of

hydrocarbons which are retained under conditions of temperature and pressure above that ordinarily needed for elimination. This is one reason for varying degrees of adsorption by activated carbons. He believed that anthracite could be regarded as a native carbon possessing an active carbon base saturated with adsorbed hydrocarbons.

Bituminous coal is the same, but has a greater adsorbed hydrocarbon content. Schoon <sup>(46)</sup>, on the basis of x-ray investigations of coal, stated that coals consist of layers of carbon atoms in a graphitic face lattice arrangement with adsorbed organic compounds present.

Chaney, Ray, and St. John <sup>(11)</sup> later amplified the above theory. Activated carbons contain a mixture of alpha and beta carbons. The alpha carbon is the active carbon and can be distinguished from the beta carbon by its formation of pseudo-graphite when heated to graphitization temperatures. The pseudo-graphite is crystalline but has a modified x-ray pattern as compared to graphite. They believed it possible that coals contain a certain percent alpha carbon mixed with beta carbon.

Ruff, Schmidt, and Olbrich <sup>(45)</sup> substantiated Chaney's theory on active carbons, but stated that the active carbon is amorphous carbon. By interpreting x-ray spectra, they reasoned that amorphous carbon is an independent form combined with graphite by means of paracrystalline intermediate forms. Later, Ruff <sup>(44)</sup> went further into the fundamentals of the problem and advanced the theory that the centers of adsorption are the unsaturated, unoriented atoms

or atom complexes present in all amorphous carbons together with saturated atoms and mineral residue. When the unsaturated groups are present on the surface, the carbon is active and adsorption can be explained by Langmuir's theory. When the unsaturated atoms are covered by a skin of saturated carbon, adsorption is explained by Polanyi's theory. But Berl (6) stated that activity cannot be ascribed to the presence of amorphous carbon. It probably depends on the development of the surface of graphite, the active carbon, and on the presence of active centers.

Density of Adsorbent. Chaney, Ray, and St. John (11) stated that there is a definite critical density or porosity of an adsorbent which corresponds to its maximum capacity per unit volume. If the porosity is increased or decreased around this critical point, the adsorptive capacity decreases. All highly active carbons of -8 +10 mesh size have an apparent density less than 0.4. The adsorptive power of carbon for colloidal particles is a function of the ratio of its activity (adsorptive power for iodine, in percent) to its density, fineness being constant.

Surface Area of Adsorbent. Hassler (20) and McMinn (21) stated that surface area influences adsorption considerably, but there are limits to which surface area can be increased by reducing particle size. If fine, active carbon is ground still more, its adsorptive power at first increases, then decreases. This suggests that long grinding destroys the capillaries in the carbon in which much of the adsorptive power resides.

Temperature of the Process. Hassler (20) stated that the influence of temperature is irregular. Substances having high vapor pressures are adsorbed better at low temperatures. Colors, gums, and similar adsorbates may be adsorbed better at high temperatures.

Effect of Electrical Charge. Chaney, Ray, and St. John (11) wrote that a given carbon is most effective when it carries an electrical charge opposite in sign to that carried by the particles to be adsorbed. Mantell (37) and Hassler (20) stated that the pH of the solution containing the adsorbate will affect the degree of adsorption.

#### E. Adsorptive Properties of Coal

Ash from the combustion of powdered coal (4) is used in some cases instead of activated earths or other adsorbents in clarification, neutralization, coagulation, and decolorization processes.

Ermolenko and Ginzburg (16) studied the adsorption of solutions of iodine in potassium iodide by coals varying in rank from peat to anthracite. They found that the process conformed with Freundlich's exponential equation.

Land (25) made some tests on the adsorption of solutions of lead salts by Illinois, No. 6 and Pocahontas No. 3 bituminous coals. The degrees of adsorption were checked by means of x-ray photographs of the coals. He found that the lower rank coal, Illinois No. 6, had the greater adsorptive properties. Swietoslowski (49) found that the adsorption of methyl blue from solutions was greatest for lignite.

The adsorption decreased as the rank of the coal increased until it became a minimum in low volatile bituminous coal. It then increased again in anthracite, but did not approach the value for sub-bituminous coal and lignite. King and Wilkins' (24) work on porosity of coal explains this variation in adsorptive properties. They found that the lowest rank coals had the highest porosity. A minimum porosity was obtained with coals containing 20 percent volatile matter. Porosity increased again in anthracite coals. Porosities were calculated from true and apparent specific gravities of the dust-free coals. Pentegov (40) found that the adsorptive properties of the younger coals were greatest. He attributed this to the presence of unsaturated, nonoriented carbon atoms in the younger coals. But Sinkinson and Turner (47), in studying the adsorption of carbon dioxide by coal, wrote that coals of high rank (anthracite) had greater adsorptive powers than lower ranked coals (bituminous and cannel). The greater adsorptive power appeared to be in the character of the carbon present rather than the quantity. Coppens (12) found the same trends in the adsorption of methane by coals. From these experiments he concluded that coal has a structure of dried colloidal gels and activated charcoals. (13)

Jones and Buller (22), in writing about the use of anthracite to clarify water, stated that a good filter medium for water must contain less than ten percent ash. As the ash increases the number of angular particles is reduced. They believed that the angularity of the particles of coal adds a great deal to the effectiveness of the

filter medium.

F. Use of Coal as an Adsorbent

In 1890, experiments were conducted in Massachusetts on the filtration of water using anthracite (22). This work was later continued at State College, Pennsylvania. One of the first filters using coal was built by James H. Fuertes about 1896 at Harrisburg, Pennsylvania (52). Dredged river coal was used as a filter medium because no local sand was available. Later, Fuertes built similar plants at Dallas, Texas, Cumberland, Maryland, and Denver, Colorado. In 1912, Weldert and Reichle (53) reported the use of Bohemian lignite in the preparation of a coal paste for the clarification of sewage. Another source (3) reported the use of a coal prefilter to adsorb constituents, from water, which caused the incrustation of sand in filters. At the present time there are about eleven hundred filter plants of all kinds using anthracite ("anthrafilt") as the filtering medium. (54)

### III. EXPERIMENTAL

#### Purpose of the Investigation

The purpose of this study is to compare the adsorption of colloidal particles from a tannin extract solution obtained from the Mead Corporation of Lynchburg, Virginia by coal, varying in rank from anthracite to lignite, with surface area and roughness of surface.

#### Plan of Investigation

##### A. Preliminary Investigation.

###### 1. Review of the Literature.

##### B. Comparison of Adsorption Properties of Coal by Rank with Proximate Analysis, Surface Area, and Roughness of Surface.

###### 1. The coal samples to be tested for their adsorption of colloidal particles will be -8 -16 U. S. Standard Screen Mesh.

###### The ranks to be tested will be:

- a. Anthracite.
- b. Semi-anthracite.
- c. Low Volatile Bituminous.
- d. Medium Volatile Bituminous.
- e. High Volatile Bituminous.
- f. Sub-bituminous.

g. Lignite.

2. Preparation of Coal Samples.

a. Size reduction and screening.

- (1) A two hundred pound sample of each coal will be passed through a Universal Jaw Crusher, with corrugated jaws 6" x 9", with the jaw setting at  $\frac{1}{2}$ ".
- (2) The product from the jaw crusher will be screened through the following U. S. Standard Screens:

4 mesh

8 mesh

16 mesh

40 mesh.

The -40 mesh coal will be discarded.

- (3) The +4 mesh coal will be passed through the jaw crusher with the jaw setting at  $\frac{1}{4}$ ".
- (4) The product from the second crushing will be screened as before.

b. Chemical treatment of the coal samples.

- (1) Each coal sample will be leached with a 10% hydrochloric acid solution at 90°C. for one hour. Five grams of the sample will then be washed with water at 100°C., 5% sodium carbonate solution at 100°C., then with water at 100°C. until the sample is neutral to methyl orange indicator. This 5 gram sample will

then be tested for the presence of iron using tannin extract solution as the testing solution.

- (2) The procedure in (1) above will be repeated until the coal sample is free of iron.
- (3) The complete sample will be washed with water at 100°C., 5% sodium carbonate solution at 100°C., then with water at 100°C. until the coal is neutral to methyl orange indicator.
- (4) The coal sample will be air dried at 25°C.

### 3. Testing of Coal for Adsorption of Colloidal Particles.

- a. The colloidal solution that will be used to test the adsorption properties of the coal samples will be obtained from the Mead Corporation, Lynchburg, Virginia. The solution will be "Leach House Liquor". The solution is made by the extraction of tannins from chestnut chips with steam. It will contain 2.92% "Soluble Solids" and 0.08% "Insoluble Solids".
- b. A 390 cubic centimeters sample of coal will be placed in two glass cylinders in series. The tannin solution will be filtered through the coal in the cylinders from a constant head at 20 cubic centimeters per minute per unit surface area of sample. The unit surface area to be used will be determined by tests. A sample of 250 cubic centimeters of the liquid, after filtration, will be taken initially and every 500 cubic centimeters thereafter until

the coal has passed maximum adsorption as indicated by a plot of "Insoluble Solids" remaining in the solution versus cubic centimeters of tannin solution filtered.

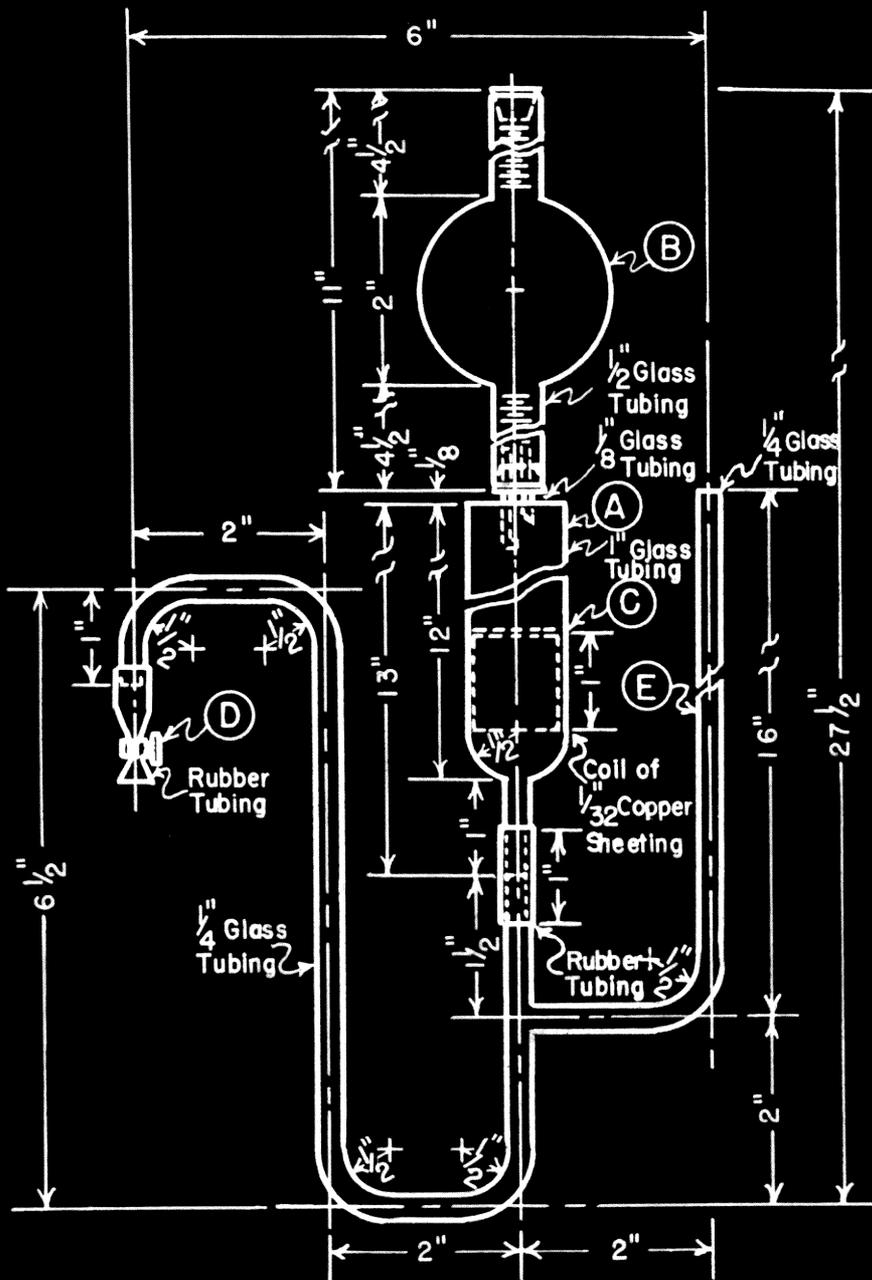
- c. The "Insoluble" and "Soluble Solids" of the samples of tannin solution will be determined by the Mead Corporation, Lynchburg, Virginia.

4. Determination of Proximate Analysis, Sulfur Content, and Density of the Coal Samples.

- a. A representative five pound sample of each coal tested will be placed in a 9-3/4" x 8-3/4" outside diameter Abbe Ball Mill and ground to pass a 60 mesh screen (U. S. Standard). This sample will be quartered down to 100 grams.
- b. The moisture, volatile matter, fixed carbon, ash, sulfur, and true density of the 100 gram samples of coal will be determined in accordance with American Standards of Testing Materials procedures on coal. (1)(2)

5. Determination of Surface Areas (9) of the Coal Samples.

- a. Tube A will be half filled with distilled water. (See Drawing No. 1) A representative 10 gram sample of coal will be poured into A. Then tube A will be filled up to the level shown with distilled water. Tube B will be clamped into position. The level of water in E will be noted. Clamp D will be opened, and 80 to 100 cubic centi-



- A Container for Sample Bed  
 B Water Reservoir  
 C Sample Support, Copper Plate  $\frac{1}{32}$ " Thick, Perforated With  $\frac{1}{32}$ " Holes  $\frac{1}{16}$ " Apart  
 D Clamp  
 E Manometer

DEPARTMENT OF CHEMICAL ENGINEERING  
 VIRGINIA POLYTECHNIC INSTITUTE  
 BLACKSBURG, VIRGINIA

APPARATUS FOR THE DETERMINATION  
 OF SURFACE AREAS OF  
 SMALL PARTICLES OF COAL

Drawn By: *P.N.P.*

Date *6-1-46*

Scale:  $\frac{1}{2}$ " = 1"

Checked By: *Fwb*

Date *6-3-46*

Date *6-3-46*

Drawing No. 1

Approved By: *Fwb*

meters of water will be allowed to drain out over a period of one minute. During this time, the drop in the level of the water in E will be noted. The temperature of the distilled water used will be recorded.

- b. The data obtained will be used in the following equation to obtain the specific surface of the sample:

$$S_0 = 14 \sqrt{\frac{Ah}{Qlv} \cdot \frac{E^3}{(1-E)^2}}$$

$S_0$  - Specific surface of particles in  $\text{cm}^2/\text{cm}^3$ .

A - Cross sectional area of sample bed in  $\text{cm}^2$ .

h - Difference in readings of water in E in cm.

Q - Volume of water drained in  $\text{cm}^3/\text{sec}$ .

L - Thickness of sample bed in cm.

v - Kinematic viscosity of the water in stokes  
( $\text{cm}^2/\text{sec}$ ).

E - Porosity or fractional free volume, i.e. volume of porespace per unit volume of bed.

6. Determination of Roughness of Surface Areas of Samples of Coal.

- a. A representative one gram sample of each coal will be examined under a microscope, piece by piece. A comparison of the roughness of the surfaces of the samples will be made in this manner.

- b. A representative 200 gram sample of each coal will be subjected to a 7500Å wave length beam of light. The angle of incidence and angle of reflection will be 60°. The reflected beam of light will be focused on a photoelectric cell connected to an amplifier system. The current produced will be proportional to the light reflected. The light reflected should be inversely proportional to the roughness of the surface areas of the coals.
- c. A representative 200 gram sample of each coal will be subjected to a beam of supersonic sound. The angle of incidence and the angle of reflection will be 60°. The intensity of the reflection measured should be inversely proportional to the roughness of the surface of the coal samples.

C. Correlation of Adsorption Properties with Surface Conditions and Surface Areas of Coal.

1. Preparation of Coal Samples.

- a. The three coal samples from the preceding tests, that show the greatest adsorbent properties, will be treated as follows:
  - (1) A portion of the samples treated in B, 2, a, will be separated, by screening through U. S. Standard Screens, as follows:

- 4 +8 mesh
- 8 +10 mesh
- 10 +16 mesh
- 16 +20 mesh
- 20 +30 mesh
- 30 +40 mesh

(2) The samples will be treated as in B, 2, b.

- b. A portion of the -4 +8 mesh fraction of the three samples selected will be placed in an Abbe Ball Mill, 12 $\frac{1}{2}$ " inside diameter, 17" long. The mill will be run until the particles of coal are smooth and rounded as evidenced by microscopic examination. The -16 mesh fines produced will be screened out and discarded.

2. Testing of Coal for Adsorption of Colloidal Particles.

- a. The coal samples will be tested for adsorption of colloidal particles following the procedure previously outlined.

3. Determination of Surface Areas.

- a. Determinations of surface areas will be made following the procedure previously given.

4. Determination of Roughness of Surfaces.

- b. Determinations of roughness of surfaces will be made following the procedures previously given.

D. Correlation of Adsorption Properties with Usage of Coals as Adsorbents.

1. Coal Samples Used.

- a. The -8 +16 mesh fraction of the three coals, that had shown the greatest adsorbent properties, will be used in the tests following.

2. First Procedure in Testing Samples.

- a. Each sample of coal will be tested for adsorption of colloidal particles six times.
- b. Between each test, the sample will be backwashed with tap water until no further cloudiness appears in the wash water. The sample will be allowed to air dry, and determinations of surface area and roughness of surface will be made.

3. Second Procedure in Testing Samples.

- a. If the results of the tests in D, 2, above, justify further investigation, duplicate tests will be made on other portions of the three coal samples, but 5 lbs./in<sup>2</sup>. of steam will be used to back wash the samples.

C. Materials

The following materials were required for the investigation of adsorption of colloidal particles from a tannin extract solution by coal:

Acid, Hydrochloric, C. P., Lot No. 32546C: (Six pounds)  
Manufactured by the J. T. Baker Chemical Company, Phillipsburg, New

Jersey. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute, Department of Chemical Engineering, Blacksburg, Virginia. Used to leach iron, affecting tannins, from coal.

Coals: Used to test their adsorption properties. Sources and descriptions are given in Table I, page<sup>26</sup>.

Carbonate, Sodium, Light, 58%: (One pound) Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute, Department of Chemical Engineering, Blacksburg, Virginia. Used to neutralize hydrochloric acid in leached coal.

Tannin Extract Solution: (Twenty gallons) "Leach house" liquor obtained from the Mead Corporation, Lynchburg, Virginia. Used to test adsorption properties of coal.

The following materials were required for the investigation of adsorption of colloidal particles from a viscose spinning bath by coal:

Acid, Sulfuric, C. P., 95.5%, Lot No. 72645: (Thirty-six pounds) Manufactured by the J. T. Baker Chemical Company, Phillipsburg, New Jersey. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Department of Chemical Engineering, Blacksburg, Virginia. Used in making spinning baths.

Chloride, Barium, C. P.: (Twenty grams) Manufactured by the J. T. Baker Chemical Company, Phillipsburg, New Jersey. Obtained from the Chemical Engineering Stock Room, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to test for sulphates in the analy-

TABLE I

Coals Used in Adsorption Tests

Sample No.	Quantity (lbs)	Rank	Size Received	Seam	Source
1	200	Anthracite	3 $\frac{1}{4}$ " x 3/16"	-	Lehigh Navigation Coal Co., Lansford, Pennsylvania
2	200	Low Volatile Bituminous	3" x 1 $\frac{1}{4}$ "	Pocahontas No. 3	Turkey Gap Coal and Coke Co., Dott, West Virginia
3	300	High Volatile Bituminous C	8" Run	Castlegate A	Independent Coal and Coke Co., Kenilworth, Utah
4	200	Semi-anthracite	Stove	Merrimac	Great Valley Coal Co., McCoy, Virginia
5	200	Medium Volatile Bituminous	-	Jewell (Raven)	Jewell Ridge Coal, Co., Jewell Ridge, Virginia
6	100	Anthracite	Screen Analysis † 8 Mesh 1% † 10 " 12.0% † 14 " 45.5% † 20 " 31.0% † 24 " 6.5% † 28 " 4.0%	-	Hudson Coal, Co., Scranton, Pennsylvania
7	200	High Volatile Bituminous A	2" x 4"	Taggart	Stonega Coke and Coal Co., Big Stone Gap, Virginia
8	25	High Volatile Bituminous C	-	Thacker	Pond Creek Colliery, Williamson, West Virginia (Sample prepared by H. R. Couper)
9	25	High Volatile Bituminous B	-	Freeport	Pond Creek Colliery, Williamson, West Virginia (Sample prepared by H. R. Couper)

sis of viscose spinning bath samples.

Coals: Used to test their adsorption properties. Sources and descriptions are given in Table I, page 26.

"Dexin", (Analysis: 75% Dextrin, 24% Maltose, 0.25% Mineral Ash, 0.75% Moisture): (One half pound) Manufactured by Burroughs Wellcome and Co., Inc., New York, New York. Obtained from Tech. Drug Company, Blacksburg, Virginia. Used in making spinning baths.

Dextrose, Technical, Syrup, (Glucose): (Two pounds) Manufactured by the J. T. Baker Chemical Company, Phillipsburg, New Jersey. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Chemistry Department, Blacksburg, Virginia. Used in making spinning baths.

Dextrose, U. S. P.: (Five pounds) Manufactured by the Mallinckrodt Chemical Works, Philadelphia, Pennsylvania. Obtained from Tech. Drug Company, Blacksburg, Virginia. Used in making spinning baths.

Disulfide, Carbon, Technical, Lot No. 42146: (Two pounds) Manufactured by Merck and Company, Inc., Rahway, New Jersey. Obtained from the Chemical Stock Room, Chemistry Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used in making viscose.

Hydroxide, Sodium, C. P., Pellets, Lot No. 71346: (One half pound) Manufactured by the J. T. Baker Chemical Company, Phillipsburg, New Jersey. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Department of Chemical Engineering,

Blacksburg, Virginia. Used in determining the acidity of spinning baths.

Hydroxide, Sodium, Flake: (Eight pounds) Manufactured by the Diamond Alkali Company, Pittsburgh, Pennsylvania. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Department of Chemical Engineering, Blacksburg, Virginia. Used in making viscose.

Matting, Pulp, Wood, Sulphite, Spruce, High Alpha: (Four pounds) Obtained from the Rayon Laboratory, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to make viscose.

Phenolphthalein, 1% in 50% Ethyl Alcohol: (Two cubic centimeters) Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Department of Chemical Engineering, Blacksburg, Virginia. Used as indicator in obtaining acidity of spinning baths.

Sulphate, Sodium, (Glauber Salt): (Ninety pounds) From Phipps and Bird, Inc., Richmond, Virginia. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Department of Chemical Engineering, Blacksburg, Virginia. Used in making spinning baths.

Sulphate, Zinc, C. P., Lot No. 71740: (One pound) Manufactured by the J. T. Baker Chemical Company, Phillipsburg, New Jersey. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Chemistry Department, Blacksburg, Virginia. Used in making spinning baths.

Sulphate, Zinc, Reagent, Lot No. 42794: (One pound) Manufactured by the J. T. Baker Chemical Company, Phillipeburg, New Jersey. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute, Chemistry Department, Blacksburg, Virginia. Used in making spinning baths.

Sulphate, Zinc, U. S. P., Granular: (Four pounds) Manufactured by Merck and Company, Inc., Rahway, New Jersey. Obtained from Tech. Drug Company, Blacksburg, Virginia. Used in making spinning baths.

Sulphide, Sodium, Crystal: (One pound) From Phipps and Bird, Inc., Richmond, Virginia. Obtained from the Chemical Stock Room of the Virginia Polytechnic Institute Department of Chemical Engineering, Blacksburg, Virginia. Used in making viscose.

#### D. Apparatus

The following apparatus used in crushing and sizing the coal samples is located in the Unit Operations Laboratory of the Department of Chemical Engineering, Virginia Polytechnic Institute, Blacksburg, Virginia:

Crusher, Jaw, Blake type: (6 inch corrugated jaws) Manufactured by the Universal Crusher Company, Cedar Rapids, Iowa. Used for crushing coal.

Drier, tray, atmospheric, Serial No. H 8122: Manufactured by Proctor and Schwartz, Inc., Philadelphia, Pennsylvania. Used to dry wet coal samples before crushing.

Mill, Ball: (12½" inside diameter, 17" long) Manufactured by Paul O. Abbe, Inc., Little Falls, New Jersey. Used to "round off" the corners of a portion of -4 +8 Mesh coal sample number 1.

Riddle, Combs Gyratory: Manufactured by Great Western Manufacturing Company, Leavenworth, Kansas. Used to vibrate screens.

Screens, U. S. Standard: (8 inch) Manufactured by Precision Scientific Company, Chicago, Illinois. Nest of seven, meshes 4, 8, 10, 16, 20, 30, and 40. Used to size coal samples.

The apparatus used to leach and wash coal samples for tests using tannin extract solution from the Stock Room of the Department of Chemical Engineering, Virginia Polytechnic Institute, Blacksburg, Virginia and is listed as follows:

Bucket, galvanized: (One gallon) Used as heating bath for leaching coal.

Flask, round bottom: (5 liter) Used to contain coal samples while leaching.

Hot Plate, electric, round, three heat: Used to furnish heat for leaching and neutralizing coal.

Stand, iron ring, with necessary clamps: Used to support flask in heating bath.

Tube, Nessler: (50 cc.) Two used in testing completeness of leaching of coal.

The equipment used in coal analyses is located in the coal laboratory of the Mining Engineering Department, Virginia Polytechnic

Institute, Blacksburg, Virginia and is listed as follows:

Balance, analytical: Manufactured by Wm. Ainsworth and Sons, Inc., Denver, Colorado. Obtained from Fisher Scientific Company, Pittsburgh, Pennsylvania. Used for weight determinations.

Capacitrol, Wheelco electronic: (Graduated from 0 to 2000°F) Manufactured by the Wheelco Instrument Company, Chicago, Illinois. Obtained from Burrell Technical Supply Company, Pittsburgh, Pennsylvania. Used to control furnace temperatures.

Crucibles, platinum, high form: Four used in making volatile matter determinations.

Crucibles, porcelain, Coors: (30 cc. capacity) Eighteen required in making ash and moisture determinations.

Furnace, Hoskins, electric, type FH 204C, Serial No. 21713: (Volts 44, Amperes 114) Manufactured by the Hoskins Manufacturing Company, Detroit, Michigan. Obtained from Burrell Technical Supply Company, Pittsburgh, Pennsylvania. Used for ash determinations.

Furnace, Hoskins, electric, tube, type FA 120, Serial No. 21677: (Volts 110, Amperes 3.27) Manufactured by the Hoskins Manufacturing Company, Detroit, Michigan. Obtained from Fisher Scientific Company, Pittsburgh, Pennsylvania. Used for volatile matter determinations.

Mill, Ball: (9" diameter) Manufactured by Paul O. Abbe, Inc., Little Falls, New Jersey. Obtained from Burrell Technical Supply Company, Pittsburgh, Pennsylvania. Used to grind samples of coal for analysis.

Oven, drying, allied, Serial No. A 64-630: (Volts 110, Watts 660, Amperes 6) Obtained from Phipps and Bird, Inc., Richmond, Virginia. Used in moisture determinations.

The following apparatus used in determining surface areas of the coal samples was obtained from the Chemical Engineering Stock Room, Virginia Polytechnic Institute, Blacksburg, Virginia:

Balance, analytical, chainomatic, dampened, Serial No. G-77: Manufactured by Seederer Kohlbusck, Inc., Jersey City, New Jersey. Sold by Phipps and Bird, Inc., Richmond, Virginia. Used for weight determinations.

Bottles, specific gravity, Hogarth, A.S.T.M., unadjusted: Obtained from Phipps and Bird, Inc., Richmond, Virginia. Two used to determine specific gravity of coal samples.

Bottles, specific gravity, Walker, unadjusted: Used to determine specific gravity of coal samples.

Thermometer, centigrade, Precision, set of seven: (No. 1 used, scale  $-11.6^{\circ}\text{C}$  to  $+60^{\circ}\text{C}$ ) Sold by Phipps and Bird, Inc., Richmond, Virginia. Used to calibrate test thermometer.

Thermometer, chemical engraved stem, mercury, centigrade: (Scale  $-10$  to  $+110^{\circ}\text{C}$ ) Used to determine temperature of testing water.

Timer, Precision, "Time-it": (Volts 110, 60 cycles) Manufactured by Precision Scientific Company, Chicago, Illinois. Used to time water flow in surface area apparatus.

Surface Area Apparatus: The apparatus used in determining surface area of coal samples was constructed as shown in Drawing No. 2, page 85. The materials of construction are listed as follows:

<u>Material</u>	<u>Size</u>	<u>Requirements</u>
Board, wood	1" x 4" x 20"	2
Bolts, Stove	3/16" x 1½"	4
Condenser jacket, Liebig	1½" x 18"	1
Nuts, Stove	3/16"	4
Screen, copper	16 Mesh, 1½" diameter	1
Screen, copper	16 Mesh 1" x 5"	1
Stand, ring and necessary clamps		1
Stopcock, Burette		1
Stopper, cork	½"	1
Stopper, rubber, solid	No. 4	1
Stopper, rubber, two hole	No. 4	1
Strapping, iron	1/8" x 1"	3 ft.
Tubing, glass	1" x 32"	1
Tubing, glass	¼"	6 ft.
Tubing, rubber	¼"	3 ft.
Wire, copper	No. 14	1 ft.

The following apparatus was used in preparing viscose spinning baths:

Compressor, Nash-Hytor, Size MD-2L, Test No. 2007: (R.P.M. 3500) Manufactured by the Nash Engineering Company, South Norwalk, Connecticut. Obtained from the Department of Chemical Engineering,

Virginia Polytechnic Institute, Blacksburg, Virginia. Used to furnish pressure in spinning viscose.

Grinder, water cooled, Serial No. P3354: Manufactured by Werner-Pfleiderer. Located in the Rayon Laboratory of the Chemistry Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to shred soda pulp before aging.

Jars, glass: (One gallon) Obtained from the Chemical Engineering Stock Room, Virginia Polytechnic Institute, Blacksburg, Virginia. Four were used as steeping, xanthating, and aging chambers in making viscose.

Press, hydraulic, Serial No. 4905-20: (Pressure gauge calibrated to 14,000 lbs./sq. in.) Manufactured by Fred S. Carver Company, New York, New York. Obtained from the Chemical Engineering Stock Room, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to press sodium hydroxide from soda pulp.

Thermometer, chemical engraved stem, mercury, centigrade: (Scale -10 to +110°C) Obtained from the Chemical Engineering Stock Room, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to determine temperatures in aging of viscose.

Spinning Equipment: The apparatus used to spin viscose was constructed as shown in Drawing No. 5, page 88. The materials of construction were obtained from the Chemical Engineering Stock Room, Virginia Polytechnic Institute, Blacksburg, Virginia and are listed as follows:

<u>Material</u>	<u>Size</u>	<u>Requirements</u>
Adaptors, (copper tubing to pipe)	$\frac{1}{2}$ "	2
Bolts, machine	$\frac{5}{8}$ " x 3"	4
Cap, black iron	$\frac{1}{4}$ "	1
Couplings, black iron	$\frac{1}{4}$ "	4
Couplings, black iron	$\frac{1}{2}$ "	2
Crock, earthenware	10 gallon	1
Drum, oil	45 gallon	1
Elbows, black iron	$\frac{1}{4}$ "	6
Elbows, black iron	$\frac{1}{2}$ "	3
Elbows, galvanized	$\frac{1}{2}$ "	4
Flange	4"	1
Flange, blind	4"	1
Gauge, Pressure	$3\frac{1}{2}$ " Connection $\frac{1}{4}$ "	1
Nipples, close, black iron	$\frac{1}{4}$ "	6
Nipples, close, black iron	$\frac{1}{2}$ "	3
Nuts, hexagonal	$\frac{5}{8}$ "	4
Petcock, brass	$\frac{1}{4}$ "	1
Plate, iron	1' x 1'	1
Pipe, black iron	$\frac{1}{4}$ "	5 ft.
Pipe, black iron	$\frac{1}{2}$ "	6 ft.
Pipe, black iron	4"	3 ft.
Pipe, galvanized	$\frac{1}{2}$ "	6 ft.
Steam trap, Sargeant, No. 87	$\frac{1}{2}$ "	1
Tee, black iron	$\frac{1}{4}$ "	1

<u>Material</u>	<u>Size</u>	<u>Requirements</u>
Tubing, copper	$\frac{1}{2}$ "	24 ft.
Unions, black iron	$\frac{1}{4}$ "	3
Valve, gate, bronze	$\frac{1}{4}$ "	1
Valves, globe, bronze	$\frac{1}{2}$ "	3
Valve, needle, bronze	$\frac{1}{4}$ "	1

The apparatus used in the adsorption tests of coal with viscose spinning bath and tannin extract solutions was constructed as shown in Drawing No. 3, page 86, and Drawing No. 4, page 87. The materials of construction were obtained from the Chemical Engineering and Chemistry Stock Rooms, Virginia Polytechnic Institute, Blacksburg, Virginia and are listed below.

<u>Material</u>	<u>Size</u>	<u>Requirements</u>
Bottle, glass	2 liter	1
Bottle, glass, aspirator	6 liter	1
Bottles, sample, screw top	250 cc.	24
Clamps, extension	large size	5
Clamp holders, castaloy		5
Clamps, screw		5
Condenser, jacket, Liebig	$1\frac{1}{4}$ " x 12"	8
Cylinders, graduated	500 cc.	4
Feldspar	-4 48 mesh	1 lb.
Stand, bar, $\frac{1}{2}$ " iron	3' x 3'	1
Stoppers, cork	$\frac{1}{2}$ "	8

<u>Material</u>	<u>Size</u>	<u>Requirements</u>
Stopper, rubber, one hole	1 $\frac{1}{4}$ "	1
Stopper, rubber, three hole	1 $\frac{1}{4}$ "	1
Tubing, glass	1/8"	5 ft.
Tubing, glass	$\frac{1}{4}$ "	5 ft.
Tubing, glass	1 $\frac{1}{4}$ "	1 ft.
Tubing, rubber	$\frac{1}{4}$ "	5 ft.

The analysis of the tannin extract and viscose spinning bath solutions required the following laboratory equipment which was obtained from the Chemical Engineering and Chemistry Stock Rooms, Virginia Polytechnic Institute, Blacksburg, Virginia:

Asbestos, acid washed: (One pound) Required to make filters in determining solids in viscose spinning bath samples.

Balance, analytical, chainomatic, dampened, Serial No. G-77: Manufactured by Seederer Kohlbusck, Inc., Jersey City, New Jersey. Sold by Phipps and Bird, Inc., Richmond, Virginia. Used in weight determinations.

Miscellaneous Apparatus: Beakers, 1000cc., 10 required; Gooch crucibles, 20 cc, 10 required; suction flasks, 500 cc; 10 required; evaporating dishes, 50 cc, 12 required; Burette, 50 cc, 1 required; pipettes, 50 cc. and 5 cc, 1 of each required; rubber policemen; stirring rods; and test tubes.

Oven, electric, drying, Serial No. 180B: (Volts 115 AC, Watts 400, Maximum Temperature 180°C) Obtained from Phipps and Bird,

Inc., Richmond, Virginia. Used for drying samples of tannin extract solution and solids from viscose spinning both samples.

Pump, vacuum, Cenco Hyvac: Manufactured by Central Scientific Company, Chicago, Illinois. Used to furnish suction in filtering viscose spinning bath solutions.

The following apparatus was used to make macrophotographs of coal samples:

Camera: 4 x 5 inch film holder and ground glass plate, adjustable bellows, 2 x lens. Obtained from the Mining Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to take macrophotographs of coal samples.

Equipment, Dark Room: Available in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to develop macrophotographs of coal samples.

Film, Photographic: (Ten required) Commercial Ortho, 4 x 5 inch. Manufactured by Agfa Ansco, Binghamton, New York. Obtained from the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used in taking macrophotographs of coal samples.

Paper, Photographic: (Fifty sheets required) Kodabrome, F - 3, 4 x 5 inch. Manufactured by Eastman Kodak Company, Rochester, New York. Obtained from the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used in printing macrophotographs of coal samples.

### E. Method of Procedure

The following procedures were used in conducting adsorption tests on coal using tannin extract solution and viscose spinning bath solution as testing liquids:

Crushing and Sizing of Coal Samples: The following is the procedure used in crushing and sizing the coal samples:

1. One hundred pounds of each sample, with the exception of sample number 6 (anthracite, Hudson Coal Company, Scranton, Pennsylvania) was passed through the Blake type jaw crusher with the jaw setting at  $\frac{1}{4}$ ".
2. The crushed coal was then screened by means of screens 4, 8, 16 and 40 mesh (U. S. Standard) and the gyratory ridde. The plus 4 and minus 40 mesh samples were discarded.
3. One half of sample number 1 (anthracite, Lehigh Navigation Coal Company, Lansford, Pennsylvania) was further sized with screens 10, 16, 20, and 30 mesh (U. S. Standard) to obtain samples of the following sizes: -4 +8, -8 +10, -10 +16, -16 +20, -20 +30, and -30 +40 mesh.
4. An attempt was made to produce one sample of coal with round shaped particles. This was accomplished by placing one half of the -4 +8 mesh coal sample number 1 in the empty Abbe ball mill and running the mill for seventy-nine hours at 54 revolutions per minute. This sample

was then screened to eliminate the -16 mesh fines.

5. Coal sample number 6 was neither crushed nor screened but tested as received.

#### Preparation of Coal Samples for Tannin Extract Solution

Adsorption Tests: In order to eliminate any iron in the coal samples that might react with tannin extract solution to precipitate iron tannates during adsorption tests, the following procedure was used to leach the coal samples:

1. Six hundred grams of the -8 +16 mesh, (U. S. Standard screen) fraction of each coal sample were leached for one hour at  $90 \pm 5^{\circ}\text{C}$  with one liter of 10% HCl in a five liter round bottom flask.
2. The coal was tested for completeness of leaching by washing a five gram sample with tap water, then with 250 ml. of 5%  $\text{Na}_2\text{CO}_3$  at  $90 \pm 5^{\circ}\text{C}$ , and finally with distilled water at  $90 \pm 5^{\circ}\text{C}$  until the wash water showed no alkalinity, using phenolphthalein as an indicator. The five gram sample was then placed in 50 ml. of tannin extract solution for twenty-four hours. Twenty milliliters of this tannin extract solution were diluted to 50 ml. with distilled water in a 50 ml. Nessler tube. The appearance of this sample was compared with a sample of untreated tannin extract solution diluted in the same manner. When the two samples had the same appearance,

leaching was complete.

3. When complete leaching had been attained as indicated by 2, above, the six hundred gram sample of coal was washed with a constant flow of tap water for thirty minutes, followed by one liter of distilled water at  $90 \pm 5^{\circ}\text{C}$ , then with 750 ml. of 5%  $\text{Na}_2\text{CO}_3$  at  $90 \pm 5^{\circ}\text{C}$ , and finally with distilled water at  $90 \pm 5^{\circ}\text{C}$  until the wash water was neutral to phenolphthaline indicator.
4. The coal was then spread to a thickness of approximately  $\frac{1}{4}$ " on a sheet of paper and dried for one hour in the drying oven (serial number 180B) at  $105 \pm 5^{\circ}\text{C}$ .

Coal Analyses: The procedure used in the coal analyses is as follows:

1. The -8 +16 mesh (U. S. Standard Screen) fractions of the coal samples were analyzed according to the procedure adopted by the American Society of Testing Materials. ( / ). The work was carried out in the Coal Laboratory of the Mining Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.
2. A representative sample of each -8 +16 mesh (U. S. Standard Screen) fraction of the coal samples was ground in a 9-3/4" Abbe ball mill to a minus 60 mesh (U. S. Standard Screen) size. A representative fifty gram portion of each of these samples was used in the coal analyses.

3. Percent moisture was obtained by determining the weight loss of a one gram sample of coal on heating in drying oven (Allied, serial number A 64-630) for one hour at  $105 \pm 5^{\circ}\text{C}$ .
4. The weight loss on burning the one gram sample to a constant weight in the Hoskins electric furnace (serial number 21713) at  $750 \pm 25^{\circ}\text{C}$  gave the percent ash.
5. Percent volatile matter was obtained by determining the weight loss of a one gram sample of coal in a covered platinum crucible on heating in the Hoskins electric tube furnace (serial number 21677) at  $950 \pm 20^{\circ}\text{C}$  for seven minutes, weight loss due to moisture being subtracted from this determination.
6. The percent fixed carbon was obtained by subtracting the above percentages from one hundred.

Surface Area Determinations: The following procedure (9) was used in determining surface areas of all coal samples prior to conducting adsorption tests:

1. An accurately weighed  $150 \pm 0.1$  gm. sample of coal was boiled in distilled water until all occluded air was removed from the coal.
2. Tube A (see Drawing No. 2, page 85) was half filled with distilled water ( $20 - 25^{\circ}\text{C}$ ) and the wet coal poured in.

3. Tube A was then filled to the level shown with distilled water (20 - 25°C).
4. Tube B, filled with distilled water (20 - 25°C), was clamped into position.
5. The depth of the coal bed was measured (accuracy  $\pm 0.1$  cm.) at three points around tube A.
6. The level of water in E was recorded.
7. Stopcock D was opened, and 180 to 190 ml. of water were allowed to drain over a period of two to four minutes.
8. During this time, the drop in the level of the water in E was recorded.
9. The temperature of the distilled water used was recorded.
10. The density of each coal sample was determined according to the procedure for determining the density of coke adopted by the American Society of Testing Materials (2).

Adsorption Tests Using Tannin Extract Solution: The procedure used in adsorption tests on the coal samples using Tannin Extract Solution is as follows:

1. A -8  $\pm$ 16 mesh, U. S. Standard Screen, two hundred and fifty gram sample of coal was placed in glass cylinders D and E (see Drawing No. 3, page 86) on copper screen supports. A  $\frac{1}{2}$ " layer of glass wool was placed on top

of the coal beds to prevent carry over of the coal particles by the flow of tannin extract solution.

2. Feed jar A was filled with tannin extract solution. A 250 ml. sample of the feed solution was taken for analysis to be used in comparison with the treated samples.
3. Screw clamp  $F_1$  was opened, allowing the constant head weir B to fill and overflow into the overflow receiver C.
4. Screw clamp  $F_2$  was opened and adjusted to permit the tannin extract solution to flow through adsorption chambers D and E at a rate of coverage of  $1000 \pm 100$   $\text{cm}^2/\text{min}$ . This rate amounted to 11 - 17 ml./min. depending on the coal sample tested. Rate of flow was determined by counting the drops per minute and checking the time required to collect 250 ml.
5. A sample of 250 ml. of the liquid (after filtration through the coal beds) was taken initially and every 500 ml. thereafter until a total of five treated samples were obtained.
6. After a sample of the -8  $\pm$ 16 mesh (U. S. Standard Screen) fractions of each different coal was tested, a sample of fraction -16  $\pm$ 40 mesh (U. S. Standard Screen) of coal number 4 (semi-anthracite, Great Valley Coal Company, McCoy, Virginia) was tested in a similar manner.

This sample was wet with tap water when placed in chambers D and E. It was washed with two liters of tap water while in the chambers. Nine hundred and seventy-five milliliters of tannin extract solution were passed through the coal to displace the water before the first treated solution sample was taken. The velocity of flow of the tannin extract solution was 16 ml./min.

7. A sample of fraction -8 #16 mesh (U. S. Standard Screen) of coal number 2 (low volatile bituminous, Turkey Gap Coal and Coke Company, Dott, West Virginia) was tested with a solution rate of coverage of  $500 \pm 50$  cm<sup>2</sup>/min. This coal sample had not been leached with 10% HCl, but had been washed with tap water to remove the dust in it.

Preparation of Viscose Spinning Baths: The preparation of viscose spinning bath solution was carried out as follows:

1. Two hundred grams of spruce wood pulp were cut into 2" x 4" strips, placed in a one gallon glass jar, and covered with a liter and a half of 18% NaOH. The pulp was allowed to soak at  $18 \pm 1^{\circ}\text{C}$  for two hours.
2. Excess 18% NaOH was drained off and the pulp pressed in hydraulic press (serial number 4905-20) to a weight of  $582 \pm 3$  gms.
3. The pressed pulp was shredded in the water-cooled

grinder (serial number P3354) for thirty minutes.

4. The shredded pulp was aged for  $45 \pm 5$  hours at 21 to  $23^{\circ}\text{C}$  in a sealed one gallon glass jar.
5. Sixty-four grams of carbon disulphide were added and the jar sealed and shaken for one hour at  $20 - 30^{\circ}\text{C}$ .
6. Thirteen hundred and fifty-four milliliters of tap water, 303 ml. of 17.8% NaOH, and 37.6 ml. of 12%  $\text{Na}_2\text{SO}_3$  were stirred into the pulp.
7. The freshly prepared viscose, was aged eighty hours at  $24 - 26^{\circ}\text{C}$ .
8. One liter of tap water was added to decrease the viscosity of the viscose.
9. A bath of the following composition was prepared in earthenware crock A (see Drawing No. 5, page 88):  
975 ml. Conc.  $\text{H}_2\text{SO}_4$  (95.5%), 286 gms.  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ,  
410 gms. Dextrose (USP), 25 gms. "Dexin" (75% Dextrins,  
24% Maltose), 4740 gms.  $\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$ , and 7500 ml.  
of tap water.
10. Water bath B was filled with water by opening valve C, allowing the water to flow in. Valve C was then closed. The water bath was heated by steam, controlled by valve D, until the spinning bath in A was at a temperature of  $45 \pm 5^{\circ}\text{C}$ .

11. The viscose was poured into pressure chamber E. The top of the chamber was bolted in place and the air line connected to it.
12. The Nash-Hytor was started and valve F was opened, producing 30 lbs./in<sup>2</sup> air pressure in E.
13. The viscose was forced out into the agitated spinning bath through spinneret G by the air pressure.
14. The rayon produced was removed by hand as fast as it was formed.

Adsorption Tests Using Viscose Spinning Bath Solution: The adsorption tests using viscose spinning bath solution were accomplished as follows:

1. An accurately weighed 150 ± 0.1 gm. sample of -8 + 16 mesh (U. S. Standard Screen) coal was boiled in tap water until all occluded air was removed from the coal.
2. The coal was washed with tap water by decantation until it was free from fines.
3. A wet coal sample was placed in each adsorption chamber C (see Drawing No. 4, page 87) on an one inch bed of -4 +8 mesh (U. S. Standard Screen) feldspar, the coal sample being covered with tap water.
4. Feed jar A was filled with viscose spinning bath solution (filtered through glass wool to eliminate extraneous material). A one liter sample of the feed

solution was taken for analysis to be used in comparison with the treated samples.

5. Screw clamp  $D_1$  was opened allowing the constant head weir B to fill and overflow into the overflow receiver F.
6. Screw clamps  $D_2$  were opened and adjusted to permit the viscose spinning bath solution to flow through adsorption chambers C at a rate of coverage of  $1750 \pm 100$   $\text{cm}^2/\text{min}$ . This rate amounted to 19 - 30 ml./min. depending on the coal sample tested. Rate of flow was determined by the rise of the liquid level in graduated cylinders E per minute.
7. Eight hundred and fifty milliliters of liquid were allowed to flow through each coal bed to displace the water before one liter, for each coal bed, was collected as a treated sample.
8. Similar tests were made on samples of sizes -4 +8 "Rounded" (see item 4, page 39) -4 +8, -8 +10, -10 +16, -16 +20, -20 +30, and -30 + 40 mesh (U. S. Standard Screen) of coal number 1 (anthracite, Lehigh Navigation Coal Company, Lansford, Pennsylvania) at  $42 \pm 2$  ml./min.

Analysis of Tannin Extract Solutions: The analysis procedure used was that adopted by the American Leather Chemists Association (39). The weight of an evaporated and dried 50 ml. sample of

solution was calculated to a percent basis to give total solids.

Analysis of Viscose Spinning Bath Solutions: The samples of viscose spinning bath solution were analyzed as follows:

1. A one liter sample of solution was filtered through a weighed Gooch crucible (with asbestos filter cake) under suction.
2. The precipitate in the crucible was washed with hot distilled water (below 85°C) until there were no sulfates in the wash water as indicated by 10% BaCl<sub>2</sub> in a boiling sample.
3. The crucible and its contents were dried to a constant weight in drying oven, (serial number 180B) at 70 ± 5°C.
4. The gain in weight of the crucible gave the amount of solids in a one liter sample.

#### F. Data and Results

The data obtained in the investigation of the adsorption of colloidal particles from tannin extract and viscose spinning bath solutions by coal is given in the form of tables, grouped as follows:

1. The data used to compare proximate analyses of the coals with their adsorption properties is given in:

TABLE II - Data for Proximate Analyses of Coal Samples. Page 53.

2. In order to obtain the porosity of the coal beds for correlation with adsorption properties, it was necessary to determine the specific gravity of each coal sample and the cross sectional area of each adsorption chamber. This data was also needed in surface area determinations. The data obtained is given in the following tables:

TABLE III - Data for Obtaining the Volume of Specific Gravity Bottles. Page 54.

TABLE IV - Data for Determining Specific Gravity of Coal Samples (Leached with 10% HCl). Page 55.

TABLE V - Data for Determining Specific Gravity of Coal Samples (Washed with Tap Water). Page 56.

TABLE VI - Data for Determination of Cross Sectional Area of Adsorption Chambers. Page 57.

3. The data necessary for comparison of the surface areas of the coals with their adsorption properties is given as follows:

TABLE VII - Data for Determination of Cross Sectional Area of Container for Sample Bed in Apparatus for the Determination of Surface Areas of Small Particles of Coal. Page 58.

TABLE VIII- Data for Resistance to Flow of Distilled Water by Empty Container for Sample Bed in

Apparatus Used for the Determination of  
Surface Areas of Small Particles of Coal.  
Page 59.

TABLE IX - Data for Obtaining Surface Area of Coal  
Samples (Size: -8 +16 U. S. Standard  
Screen Mesh). Page 60.

TABLE X - Data for Obtaining Surface Area of Differ-  
ent Sizes of Coal Sample No. 1. Page 61.

4. The following data was obtained in order to determine  
the adsorbing properties of the coals to colloidal  
particles in tannin extract solution:

TABLE XI - Data Recorded During Adsorption Tests of  
Coal Samples (Size: -8 +16 Mesh, U. S.  
Standard Screen) Using Tannin Extract  
Solution as the Testing Liquid. Page 62.

TABLE XII - Data for Analysis of Total Solids in Tannin  
Extract Solution Samples. Page 63.

5. Determinations of the adsorbing properties of the coals  
to colloidal particles in viscose spinning bath solution  
were made from the following data:

TABLE XIII- Data Recorded During Adsorption Tests of  
Coal Samples Using Viscose Spinning Bath  
Solution as the Testing Liquid. Page 64.

TABLE XIV - Data for Analysis of Solids in One Liter  
Viscose Spinning Bath Solution Samples.

Page 65.

TABLE XV - Data for Analysis of Solids in Viscose  
Spinning Bath Solution Samples from  
One Spinning Bath. Check on Reproducibility of Analyses. Page 66.

TABLE II

Data for Proximate Analyses of Coal Samples

Coal Sample No. (Size -8 +16 Mesh)	Moisture and Ash					Volatile Matter		
	Crucible No.	Wgt. of Crucible (gms.)	Wgt. of Crucible and Coal Sample (gms.)	Wgt. of Crucible and Coal sample After Drying (gms.)	Wgt. of Crucible and Coal Sample After Ignition (gms.)	Wgt. of Crucible (gms.)	Wgt. of Crucible and Coal Sample (gms.)	Wgt. of Crucible and Coal Sample after Heating (7 min. at) (950 ± 20°C) (gms.)
1	130	13.6689	14.6689	14.6360	13.7469	12.1875	13.1875	13.1182
	102	13.7659	14.7659	14.7326	13.8445	12.1082	13.1082	13.0383
2	114	12.2732	13.2732	13.2684	12.3864	11.9227	12.9227	12.7658
	104	14.0841	15.0841	15.0799	14.1969	11.8780	12.8780	12.7208
3	103	13.4159	14.4159	14.3901	13.4645	12.1875	13.1875	12.7190
	128	12.5256	13.5256	13.4995	12.5748	12.1082	13.1082	12.6378
4	131	13.5720	14.5720	14.5673	13.8204	11.8778	12.8778	12.7498
	108	14.1970	15.1970	15.1921	14.4484	11.9223	12.9223	12.7944
5	124	13.2161	14.2161	14.2120	13.2398	12.1878	13.1878	12.9473
	100	13.7986	14.7986	14.7968	13.8230	12.1085	13.1085	12.8695
6*	111	14.3968	15.3968	15.3784	14.4866	11.8780	12.8780	12.7953
	101	13.7408	14.7408	14.7214	13.8312	11.9225	12.9225	12.8417
7	106	14.3839	15.3839	15.3734	14.4117	12.1875	13.1875	12.8160
	118	12.5511	13.5511	13.5396	12.5767	12.1083	13.1083	12.7355
8	126	13.2537	14.2537	14.2387	13.2960	11.8780	12.8780	12.4438
	122	12.1978	13.1978	13.1824	12.2394	11.9227	12.9227	12.4896
9	135	13.0758	14.0758	14.0527	13.1025	12.1877	13.1877	12.7892
	115	12.8540	13.8540	13.8305	12.8811	12.1083	13.1083	12.7085

\* See TABLE I, page 26, for the size of this coal.

TABLE III

Data for Obtaining the Volume of Specific  
Gravity Bottles

Type of Specific Gravity Bottle	Specific Gravity Bottle No.	Wgt. of Specific Gravity Bottle (gms.)	Wgt. of Specific Gravity Bottle and Distilled Water (gms.)	Observed Temperature of Distilled Water (°C)	Observed Temperature of Air (°C)
Walker	1	37.4922	80.5419	27.0	26.3
		37.4922	80.5864	22.8	23.1
	2	35.3265	80.0780	26.7	26.3
		35.3265	80.1165	22.8	23.1
Hogarth	1	69.8880	175.6701	23.2	26.5
	2	65.7237	170.6398	23.1	26.5

TABLE IV

Data for Determining Specific Gravity of Coal Samples  
(Leached with 10% HCl)

Coal Sample No. (Size -8 +16 Mesh)	Wgt. of Evaporating Dish (gms.)	Wgt. of Evaporating Dish and Coal Sample (gms.)	Wgt. of Specific Gravity Bottle, Coal Sample and Distilled Water (gms.)	Observed Temperature of Distilled Water (°C)
1	34.6408	44.6484	83.6807	28.6
	34.6412	44.6406	83.6790	27.0
2	34.6412	44.7337	83.4969	23.6
	34.6412	44.6489	83.0015	25.6
3	34.6414	44.6467	82.9617	23.7
	34.6415	44.6430	82.4746	25.3
4	34.6416	44.6662	83.7899	26.3
	34.6415	44.6479	83.7302	26.0
5	34.6421	44.6481	82.6440	21.8
	34.6421	44.6469	82.5212	22.7
6*	33.9374	43.9725	83.9312	29.0
	33.9378	43.9643	83.8178	29.6
7	38.4665	48.4696	82.4840	24.5
	38.4666	48.4769	82.3692	25.0
8	38.4647	48.4650	177.9667	24.1
	38.4647	48.4647	172.9000	25.0
9	38.4648	48.4648	177.9885	25.0
	38.4648	48.4653	172.9678	24.0

\* See TABLE I, page 26, for the size of this coal.

TABLE V

Data for Determining Specific Gravity of Coal Samples  
(Washed with Tap Water)

Coal Sample No. (Size -8 +16 Mesh)	Wgt. of Evaporating Dish (gms.)	Evaporating Dish and Coal Sample (gms.)	Wgt. of Specific Gravity Bottle Coal Sample, and Distilled Water (gms.)	Observed Temperature of Distilled Water (°C)
1	38.4653	48.4653	84.6329	23.0
2	38.4643	48.4643	173.5085	22.3
3	38.4639	48.4639	82.5516	23.6
4	38.4649	48.4649	84.3878	22.0
5	38.4649	48.4649	172.9750	21.9
6#	38.4649	48.4649	83.8523	21.9
7	38.4651	48.4651	82.8813	25.3
8	38.4651	48.4656	172.9326	24.5
9	38.4651	48.4651	82.5090	26.0
1 (-4 +8 Mesh)* "Rounded"	34.2159	44.2327	84.0785	23.0
1 (-4 +8 Mesh)* "Rounded"	34.2159	44.2154	174.5961	22.0

\* See Item 4, Page 39.

# See TABLE I, page 26, for the size of this coal.

TABLE VI

Data for Determination of Cross Sectional Area  
of Adsorption Chambers

Adsorption Chamber No.	Distilled Water Level in Chamber (0.05 in.)	Observed Temperature of Distilled Water (°C)	Wgt. of Weighing Bottle (gms.)	Wgt. of Weighing Bottle and Distilled Water (gms.)
1	20	-	-	-
	60.4	25.9	37.8236	78.2818
	101.5	25.8	37.8264	79.0182
	142.5	26.0	37.7912	78.7617
	183.5	26.2	37.8316	79.1318
	20	-	-	-
	60.3	26.1	37.8615	78.0104
	100.5	26.2	37.8111	78.0180
	142.5	26.3	37.7680	79.6216
	184.6	26.2	37.7368	80.1881
2	20	-	-	-
	63.5	23.5	37.1493	79.3470
	107.8	23.7	37.0725	79.1143
	149.0	23.8	37.0516	76.6590
	190.0	24.0	37.0479	76.2432
	20	-	-	-
	60.6	24.2	36.9950	76.2848
	102.5	24.1	36.9917	77.0736
	144.5	24.2	37.0148	77.2077
	186.8	24.0	37.0021	77.3459
3	20	-	-	-
	58.4	27.2	37.4531	79.0213
	96.0	27.0	37.4269	78.9806
	134.5	27.2	37.4789	79.3076
	171.0	27.3	37.4399	78.6965
	185.8	27.4	37.5014	52.9550
	20	-	-	-
	59	27.2	37.5148	78.7576
	96.8	27.2	37.4459	77.8904
	134.5	27.3	37.4369	77.7369
172.0	27.4	37.3897	75.6299	
186.0	27.3	37.4135	54.5525	
4	20	-	-	-
	43.8	25.7	35.1697	60.4530
	67.6	25.9	35.4563	60.3765
	92.0	25.8	35.4176	61.1367
	116.6	25.7	35.4325	61.3275
	140.8	25.8	35.4545	61.1105
	160.2	25.8	35.4402	61.2025
	188.0	25.8	35.3875	59.0264
	30.0	-	-	-
	55.7	24.7	35.2262	61.5249
80.8	24.7	35.4017	61.1436	
107.0	24.8	35.4618	61.6678	
131.6	24.9	35.4666	61.2327	
157.0	24.9	35.4500	61.6883	
183.6	25.0	35.4542	62.8663	
190.0	25.0	35.4808	41.6508	
5	20	-	-	-
	61.0	25.8	37.5046	79.1474
	101.5	26.0	37.3907	78.3055
	142.5	26.2	37.3852	79.3767
	185.0	26.3	37.3448	80.0700
	20.0	-	-	-
	61.0	26.0	37.3957	79.3271
	102.4	26.0	37.3986	79.2397
	143.0	26.3	37.4026	78.5351
	183.5	26.3	37.3569	78.3723
6	20	-	-	-
	61.5	26.7	37.3270	78.3557
	103.0	26.6	37.3862	78.5521
	144.5	26.6	37.4765	78.5422
	186.0	26.6	37.3144	78.8300
	20.0	-	-	-
	61.0	26.7	37.4104	78.2606
	103.0	26.4	37.4188	78.9603
	145.0	26.7	37.3775	78.7324
	186.7	26.7	37.4309	78.9730
7	20	-	-	-
	60.5	21.4	37.3887	78.9334
	100.5	21.4	37.3438	78.1811
	140.5	21.4	37.3928	78.4364
	180.6	21.4	37.3695	78.2968
	20.0	-	-	-
	61.5	21.8	37.4190	78.7375
	102.0	22.0	37.4914	77.8678
	143.5	22.0	37.4978	78.5410
	182.2	22.0	37.5435	76.3241
8	20.0	-	-	-
	62.5	22.1	37.3598	78.5994
	104.5	22.1	37.3935	77.8034
	146.0	22.2	37.5647	78.4035
	183.7	22.2	37.4664	77.7259
	20.0	-	-	-
	63.0	20.2	37.4444	79.0748
	104.5	20.8	37.4885	77.9521
	147.5	20.8	37.4637	78.8883
	180.0	20.4	37.4340	78.0235

TABLE VII

Data for Determination of Cross Sectional Area of  
Container for Sample Bed in Apparatus for the  
Determination of Surface Areas of Small  
Particles of Coal

Distilled Water Level in Chambers (0.05 in.)	Observed Temperature of Distilled Water (°C)	Wgt. of Weighing Bottle (gms.)	Wgt. of Weighing Bottle and Dis- tilled Water (gms.)
0	-	-	-
40.3	26.7	37.3248	77.8418
80.0	26.6	37.4340	77.7493
119.7	26.7	37.5328	77.7658
159.8	26.7	37.4956	77.8888
199.0	26.7	37.4167	76.2424
0	-	-	-
40.7	26.7	37.3808	78.5037
81.0	26.7	37.3858	78.5035
121.5	26.8	37.3788	78.0990
162.8	26.8	37.4221	78.3093
199.0	26.9	37.3914	74.0434

TABLE VIII

Data for Resistance to Flow of Distilled Water by  
Empty Container for Sample Bed in Apparatus Used  
for the Determination of Surface Areas of Small  
Particles of Coal

Run No.	1	2	3
Manometer Reading before Run (0.0358 in.)	47.5	43.5	46.0
Manometer Reading During Flow of Distilled Water (0.0358 in.)	49.5	45.5	48.0
Observed Temperature of Distilled Water (°C)	25.5	25.0	24.7
Volume of Distilled Water Flowing (ml.)	195.2	191.5	194.5
Time of Flow of Distilled Water (sec.)	184	180.2	172.2

TABLE IX

Data for Obtaining Surface Area of Coal Samples  
(Size -8 +16 U. S. Standard Screen Mesh)

Coal Sample No.	1				2				3				4				5				6*				7				8				9			
Wgt. of Container (gms.)	67.5137				67.5109				67.5140				67.5117				67.5105				67.5102				67.5099				67.5090				67.5092			
Wgt. of Container and Coal Sample (gms.)	236.7203				215.0569				202.5675				229.5556				203.7330				233.5092				203.0165				203.0246				203.5248			
Depth of Coal Bed (cm.)	27.27	27.27	27.27	27.27	25.0	25.0	25.0	25.0	25.0	25.37	25.53	25.53	26.2	26.2	26.2	26.2	25.15	25.15	25.15	25.15	26.93	26.93	26.93	26.9	26.93	26.97	27.0	27.0	26.3	26.3	26.3	26.3	27.03	27.03	27.03	27.03
Manometer Reading before Run (0.0358 in.)	37.5	36.5	36.5	36	43.0	41.0	36.5	38.0	38.0	37.5	37.0	38.0	43.0	41.5	40.0	38.0	39.0	35.0	41.0	40.5	37.0	40.0	42.5	38.0	36.5	36.0	40.0	42.0	38.0	37.0	40.0	38.0	36.5	34.0	38.0	35.0
Manometer Reading During Run (0.0358 in.)	59.0	53.0	54.0	51.5	61.0	59.0	54.0	56.0	56.0	53.0	54.0	55.0	65.5	64.0	62.0	56.5	58.0	51.0	57.0	57.0	65.0	67.0	67.0	66.0	50.5	50.0	54.0	58.0	56.0	54.0	57.0	56.0	56.0	52.0	54.0	54.0
Observed Temperature of Distilled Water (°C)	26.6	26.8	27.0	27.0	26.2	25.7	24.8	23.4	23.8	24.2	24.3	24.6	25.6	25.6	25.7	25.7	25.5	25.5	25.7	25.7	25.7	26.1	26.1	26.3	26.7	27.1	26.7	26.6	21.8	22.7	23.0	23.0	24.4	25.0	24.7	24.6
Volume of Distilled Water Flowing During Run (ml.)	190.4	185.0	192.9	188.4	191.2	198.0	194.5	189.8	199.5	192.6	193.1	184.1	195.9	189.5	190.2	189.5	191.0	191.5	190.1	185.0	193.9	190.4	189.2	190.4	184.3	187.6	189.5	188.0	187.5	186.8	192.7	191.7	190.3	190.3	186.7	194.2
Time of Run (sec.)	150.9	178.4	174.9	175.5	173.0	200.8	186.2	170.1	185.8	175.1	182.9	177.3	187.7	184.6	182.3	195.8	160.8	185.5	184.7	187.3	192.1	191.4	203.6	183.8	177.5	180.4	187.2	168.3	188.6	185.1	189.1	180.5	179.6	180.9	193.1	171.7

\* See TABLE I, page 26, for size of this coal.

TABLE X

Data for Obtaining Surface Area of Different  
Sizes of Coal Sample No. 1

Size (U. S. Standard Screen)	-4 +8				-4 +8 "Rounded" (See Item 4, Page 39)				-8 +16				-10 +16				-16 +20				-20 +30				-30 +40			
Wgt. of Container (gms.)	67.5119				67.5096				67.5115				67.5122				67.5123				67.5114				67.5091			
Wgt. of Container and Coal Sample (gms.)	228.5361				259.7734				228.1122				228.2855				225.0728				222.2679				220.2717			
Depth of Coal Bed (cm.)	25.03	25.03	25.03	25.03	25.3	25.3	25.3	25.3	26.7	26.7	26.7	26.7	26.87	26.87	26.87	26.87	27.17	27.07	27.07	27.07	27.0	27.0	27.0	27.0	27.9	27.9	27.9	27.9
Manometer Reading before Run (0.0358 in.)	44.0	42.5	45.5	41.5	38.5	37.5	43	42	36.0	37.0	31.0	35.0	32.0	34.5	36.0	41.0	41.0	41.0	38.5	41.5	38.0	32.5	34.0	38.5	31.0	32.0	34.0	34.0
Manometer Reading During Run (0.0358 in.)	48.0	48.0	50.5	46.5	46.5	45.0	53.0	50.0	47.0	48.0	41.5	45.0	49.0	52.5	54.5	56.5	65.0	72.0	64.5	69.5	90.0	85.5	78.0	85.5	104.0	100.0	115.0	114.0
Observed Temperature Distilled Water (°C)	24.9	25	25	24.7	26.7	27.0	27.0	26.4	25.0	25.0	25.0	25.3	25.0	25.0	25.2	25.4	26.4	26.7	26.4	26.4	25.6	25.9	26.0	26.0	27.8	27.8	27.8	28.0
Volume of Distilled Water (ml)	187.4	177.3	181.2	189.1	189.5	186.0	184.4	186.8	185.2	180.8	187.3	188.9	193.0	187.2	189.9	187.3	185.0	195.5	190.3	183.8	191.3	185.1	190.5	191.2	191.5	192.5	188.8	193.5
Time of Run (sec.)	184.7	142.9	163.5	150.0	166.5	172.1	134.2	172.5	158.1	159.7	175.5	177.2	205.1	186.2	175.0	219.6	238.8	206.0	231.5	221.6	206.2	208.6	246.9	230.7	271.4	294.8	241.5	249.9

TABLE XI

Data Recorded During Adsorption Tests of Coal Samples (Size -8 to 6 Mesh, U. S. Standard Screen)  
Using Tannin Extract Solution as the Testing Liquid

Coal Sample No.		1		2		3		4		5		6		7		8		9		4*		1#	
Adsorption Chamber No.		2	4	1	3	5	6	7	8	2	4	5	6	1	3	7	8	2	4	5	6	1	3
Depth of Coal Bed (cm.)	Before Run	20.4	18.15	22.7	22.7	23.0	23.5	24.1	23.9	24.0	23.9	23.3	23.6	22.5	22.3	22.8	23.6	23.3	22.3	22.5	23.8	22.2	20.8
	After Run	20.6	18.15	23.1	22.9	23.2	23.5	24.1	23.9	24.0	24.0	23.6	24.0	22.5	22.3	22.9	23.6	23.3	22.3	22.5	23.8	22.2	20.8
Clock Time of Run	Begin	3:07 PM		7:28 PM		7:52 PM		9:25 AM		9:23 AM		9:06 AM		7:05 AM		7:14 PM		7:16 PM		10:35 AM		9:52 AM	
	End	4:38 AM		10:35 PM		11:37 PM		1:33 PM		12:45 PM		4:42 PM		1:42 PM		10:48 PM		11:07 PM		1:54 PM		4:30 PM	
Velocity of Tannin Extract Solution (ml./min.)		36		17		15		13		16		11		16		15		14		16		8	
Room Temperature (°C)	Before	25.6		25.2		25.2		25.4		25.4		26.4		26.4		22.0		22.0		21.0		21.0	
	After	24.3		26.0		24.2		27.5		26.2		26.3		25.2		24.0		24.2		26.6		24.8	
Temperature of Tannin Extract Solution (°C)	Before	25.6		24.5		24.5		24.5		24.5		23.8		23.8		20.5		20.5		21.4		21.4	
	After	23.8		24.0		26.0		27.4		27.5		26.0		25.3		24.0		23.8		25.7		24.0	
Volume of Tannin Extract Solution Through Coal Sample (ml.)		3250		3250		3250		3250		3250		3250		3250		3250		3250		4225		3250	

\* This sample of coal sample No. 4 was -16 +40 Mesh. It was pretreated as indicated in item 6, page 44.

# This sample of coal sample No. 2 was pretreated as indicated in item 7, page 45.

\$ See TABLE I, page 26, for the size of this coal sample.

TABLE XII

Data for Analysis of Total Solids in Tannin Extract  
Solution Samples

Coal Sample No. (Size 8 #16 Mesh)	Tannin Extract Solution No.	Evaporating Dish No.	Wgt. of Evaporating Dish (gms.)	Wgt. of Evaporating Dish and Tannin Extract Solution (gms.)	Wgt. of Evaporating Dish and Tannin Extract Solution After Evaporating and Drying (gms.)
1	Feed	Blank	85.0372	135.5036	86.8518
	1A <sub>1</sub>	1	86.1949	136.6718	88.0180
	1A <sub>2</sub>	2	85.8250	136.3182	87.6490
	1A <sub>3</sub>	4	82.1850	132.6929	84.0191
	1A <sub>4</sub>	6	86.0221	136.4294	87.8644
	1A <sub>5</sub>	12	85.1115	135.6390	86.9435
2	Feed	Blank	85.0365	135.4700	86.8667
	2A <sub>1</sub>	1	86.1932	136.7369	88.0328
	2A <sub>2</sub>	2	85.8238	136.3485	87.6518
	2A <sub>3</sub>	4	82.1840	132.6879	84.0309
	2A <sub>4</sub>	6	86.0212	136.5563	87.8709
	2A <sub>5</sub>	12	85.1106	135.6812	86.9622
3	Feed	B	86.9171	137.4316	88.7719
	3A <sub>1</sub>	1	86.4950	136.8800	88.3320
	3A <sub>2</sub>	3	84.6430	135.1520	86.4806
	3A <sub>3</sub>	5	82.6530	133.1804	84.4959
	3A <sub>4</sub>	7	85.9926	136.5188	87.8445
	3A <sub>5</sub>	9	85.8975	136.3746	87.7530
4	Feed	B <sub>1</sub>	86.9171	137.4591	88.7349
	4A <sub>1</sub>	1	86.4940	137.0602	88.3402
	4A <sub>2</sub>	3	84.6422	135.2025	86.4594
	4A <sub>3</sub>	5	82.6517	133.2035	84.4895
	4A <sub>4</sub>	7	85.9914	136.5286	87.8351
	4A <sub>5</sub>	9	85.8965	138.4319	87.7388
5	Feed	B <sub>2</sub>	85.0336	135.5367	86.8902
	5A <sub>1</sub>	1	86.1926	136.7352	88.0508
	5A <sub>2</sub>	2	85.8232	136.3657	87.6666
	5A <sub>3</sub>	4	82.1830	132.6870	84.0324
	5A <sub>4</sub>	6	86.0200	136.5539	87.8917
	5A <sub>5</sub>	12	85.1098	135.6533	86.9708
6§	Feed	B <sub>1</sub>	86.4888	136.9588	88.3405
	6A <sub>1</sub>	1	86.1925	136.7331	88.1024
	6A <sub>2</sub>	3	84.6424	135.1494	86.4866
	6A <sub>3</sub>	5	82.6519	133.1495	84.5105
	6A <sub>4</sub>	7	85.9921	136.4846	87.8543
	6A <sub>5</sub>	9	85.8974	136.3716	87.7385
7	Feed	9	86.9169	137.5050	88.7641
	7A <sub>1</sub>	0	86.9146	137.3693	88.7934
	7A <sub>2</sub>	2	85.8217	136.3084	87.6806
	7A <sub>3</sub>	4	82.1821	132.6602	84.0484
	7A <sub>4</sub>	6	86.0202	136.5139	87.8929
	7A <sub>5</sub>	12	85.1096	135.6152	86.9869
8	Feed	B <sub>1</sub>	86.4911	137.0094	88.3450
	8A <sub>1</sub>	1	86.1891	136.7410	88.0541
	8A <sub>2</sub>	3	84.6397	135.1433	86.4865
	8A <sub>3</sub>	5	82.6508	133.1680	84.5050
	8A <sub>4</sub>	7	85.9909	136.5267	87.8589
	8A <sub>5</sub>	9	85.8970	136.4395	87.7605
9	Feed	B <sub>2</sub>	85.0364	135.5146	86.9254
	9A <sub>1</sub>	0	86.9176	137.4505	88.7965
	9A <sub>2</sub>	2	85.8224	136.3384	87.6792
	9A <sub>3</sub>	4	82.1826	132.6978	84.0511
	9A <sub>4</sub>	6	86.0205	136.5373	87.8901
	9A <sub>5</sub>	12	85.1099	135.6780	86.9754
4*	Feed	B <sub>2</sub>	85.0373	135.6014	86.9465
	11A <sub>1</sub>	0	86.9180	137.4443	88.8062
	11A <sub>2</sub>	2	85.8234	136.8467	87.6992
	11A <sub>3</sub>	4	82.1835	132.6904	84.0740
	11A <sub>4</sub>	6	86.0211	136.5471	87.9157
	11A <sub>5</sub>	12	85.1108	135.6888	86.9937
2#	Feed	B <sub>1</sub>	86.4948	137.0035	88.3817
	10A <sub>1</sub>	1	86.1927	136.7294	88.0815
	10A <sub>2</sub>	3	84.6423	135.1690	86.5240
	10A <sub>3</sub>	5	82.6530	133.1896	84.5325
	10A <sub>4</sub>	7	85.9922	136.4839	87.8721
	10A <sub>5</sub>	9	85.8984	136.3874	87.7866

\* See item 6, page 44.

# See item 7, page 45.

§ See TABLE I, page 26, for the size of this coal.

TABLE XIII

Data Recorded During Adsorption Tests of Coal Samples  
Using Spinning Bath Solution As the Testing Liquid

Coal Sample No.	1	2	3	4	5	6	7	8	9	1	1	1	1	1	1	1	
Size (U. S. Standard Mesh)	-8 #16	-8 #16	-8 #16	-8 #16	-8 #16	*	-8 #16	-8 #16	-8 #16	-4 #8 Rounded#	-4 #8	-8 #10	-10 #16	-16 #20	-20 #30	-30 #40	
Adsorption Chamber No.	7	1	7	5	6	1	7	5	6	6	1	5	5	6	8	7	
Wgt. of Container (gms.)	67.5177	67.5103	67.5103	67.5103	67.5103	67.5103	67.5103	67.5141	67.5138	67.5177	67.5177	67.5177	67.5177	67.577	67.5177	67.5177	
Wgt. of Container and Coal (gms.)	220.5236	196.5173	188.5123	214.0367	187.5177	213.5270	186.5013	188.0623	189.0688	222.6668	218.5323	217.6214	217.5299	214.504	212.5653	209.5708	
Velocity of Spinning Bath Solution (ml./min.)	24	30	26	23	28	19	26	26	24	41	41	41	41	41	41	41	
Depth of Coal Bed (cm.)	Before Run	24.5	22.0	23.0	23.0	22.7	24.0	23.1	23.0	24.5	20.5	23.6	24.0	23.7	25.0	26.0	24.8
	After Run	24.5	22.0	23.6	23.6	22.7	24.0	23.1	23.0	24.5	20.5	23.6	24.0	23.7	25.0	26.0	24.8
Clock Time of Run	Before	1:58 AM	11:43 PM	11:31 PM	11:29 PM	11:37 PM	2:54 PM	3:09 AM	3:11 AM	3:05 AM	5:03 AM	2:06 AM	5:00 AM	2:08 AM	2:11 AM	4:54 AM	4:57 AM
	End	3:16 AM	12:46 AM	12:42 AM	12:51 AM	12:44 AM	4:32 AM	4:19 AM	4:24 AM	4:24 AM	5:48 AM	2:51 AM	5:45 AM	2:53 AM	2:56 AM	5:37 AM	5:39 AM
Room Temperature (°C)	Before	24.6	25.4	25.4	25.4	25.4	25.5	25.5	25.5	25.5	24.0	24.6	24.0	24.6	24.6	24.0	24.0
	After	26.8	27.0	27.0	27.0	27.0	24.6	24.6	24.6	24.6	25.6	26.2	25.6	26.2	26.2	25.6	25.6
Temperature of Spinning Bath Solu- tion (°C)	Before	35.0	41.0	41.0	41.0	41.0	36.8	36.8	36.8	36.8	37.3	35.0	37.3	35.0	35.0	37.3	37.3
	After	29.5	33.4	33.4	33.4	33.4	29.0	29.0	29.0	29.0	29.5	31.0	29.5	31.0	31.0	29.5	29.5
Volume of Spinning Bath Solution Through Coal Sample (ml.)	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850	1850

\* See TABLE I, page 26, for size of this coal sample.

# See item 4, page 39.

TABLE XIV

Data for Analysis of Solids in One Liter  
Spinning Bath Solution Samples

Spinning Bath Solu- tion No.	Coal Bed Tested		Gooch Crucible No.	Wgt. of Dry Gooch Crucible (gms.)	Wgt. of Gooch Crucible and Solids from Solution After Drying (gms.)
	Sample No.	Size (U. S. Standard Mesh)			
1	Feed Sample No. 1		1	17.0498	17.0655
			1-5	17.0606	17.0727
	2	-8 +16	2	16.6291	16.6375
	3	-8 +16	3	17.1590	17.1688
	4	-8 +16	4	17.0106	17.0230
	5	-8 +16	5	17.1900	17.2014
	Feed Sample No. 2		10	18.5875	18.6266
	6	*	6	18.1599	18.1665
	7	-8 +16	7	17.6147	17.6244
	8	-8 +16	8	16.4062	16.4189
9	-8 +16	9	16.5635	16.5719	
2	Feed Sample No. 1		1	16.2032	16.2176
			1-7	18.3419	18.3595
	1	-8 +16	2	18.0804	18.0934
	1	-16+20	5	17.6480	17.6589
	1	-20+30	4	17.3060	17.3128
	1	-30+40	3	16.7162	16.7190
	Feed Sample No. 2		10	18.6385	18.6754
	1	-4 +8#	9	17.2585	17.2818
	1	-4 +8	8	16.6873	16.7139
	1	-8 +10	7	16.9719	16.9892
1	-10+16	6	18.0609	18.0772	

\* See TABLE I, page 26, for the size of this coal sample.

# See item 4, page 39.

TABLE XV

Data for Analysis of Solids in Spinning Bath  
Solution Samples from One Spinning Bath.  
Check on Reproducibility of Analyses

Quantity of Spinning Bath Solution (ml.)	Gooch Crucible No.	Wgt. of Dry Gooch Crucible (gms.)	Wgt. of Gooch Crucible and Solids from Solution After Drying (gms.)
1000	1	18.4867	18.5771
1000	2	17.0627	17.1507
820	3	17.6332	17.7072

The following calculations were used in converting data to results:

To obtain the specific surface of the coal sample the following equation (9) was used:

$$S_o = 14 \sqrt{\frac{Ah}{QLv} \cdot \frac{E^3}{(1-E)^2}}$$

$S_o$  = Specific surface of particles in  $\text{cm}^2/\text{cm}^3$ .

$A$  = Cross sectional area of sample bed in  $\text{cm}^2$ .

$h$  = Difference in readings of water in  $E$  (see Drawing No. 2, page 85) in cm.

$Q$  = Volume of water drained in  $\text{cm}^3/\text{sec}$ .

$L$  = Thickness of sample bed in cm.

$v$  = Kinematic viscosity of water in stokes ( $\text{cm}^2/\text{sec}$ ).

$E$  = Porosity or fractional free volume, i.e. volume of pore space per unit volume of bed.

The values in the above equation were solved for the specific surface of coal sample no. 2, using the data from test number one (TABLE IX, page 60), in the example below:

1. Cross sectional area of sample bed (A): From TABLE VII, page 58, the difference between the first and last reading of the distilled water level in the first determination was:

$$199 \text{ units (1 unit = 0.05 in.)}$$

$$199 \times .05 \times 2.54 = 25.273 \text{ cm.}$$

The average observed temperature of the distilled water was 26.7°C.

The total weight of water drained was 200.2742 gms.

From FIGURE 3, page 69, an observed temperature of 26.7°C = 26.9°C corrected temperature. One gram of water at 26.9°C occupies 1.00344 cm<sup>3</sup>. (4/)

$$\therefore 200.2742 \times 1.00344 = 200.9631 \text{ cm}^3 = \text{the volume of the empty sample bed container.}$$

$$\therefore \frac{200.9631}{25.273} = 7.952 \text{ cm}^2 = A.$$

2. Difference in readings of water level (h) in manometer (E) (see Drawing No. 2, page 85):

From TABLE IX, page 60, the difference between the initial and final manometer readings was 61 - 43 = 18 units (1 unit = 0.0358 in.).

From TABLE VIII, page 59, the resistance of the empty sample bed container was 49.5 - 47.5 = 2.0 units (1 unit = 0.0358 in.).

$$\therefore (18 - 2) \times .0358 \times 2.54 = 1.533 \text{ cm.} = h.$$

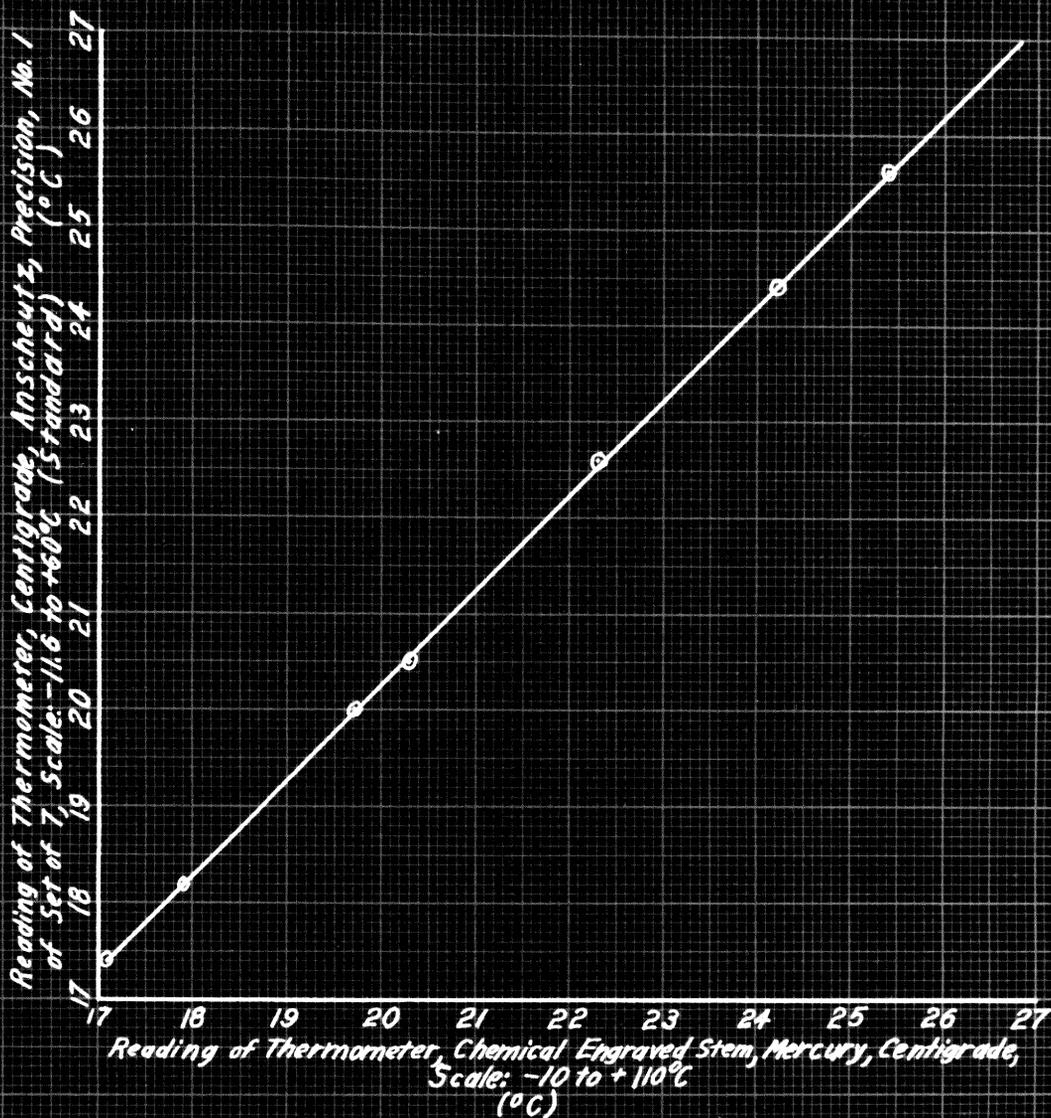


FIGURE 3

Plot of  
Testing Thermometer Reading (°C)  
Versus  
Standard Thermometer Reading (°C)

3. Volume of water drained (Q):

From TABLE IX, page 60, the volume of water flowing through the coal sample bed was  $191.2 \text{ cm}^3$ . The time of run was 172 sec.

$$i. \frac{191.2}{173} = 1.104 \text{ cm}^3/\text{sec.} = Q.$$

4. Thickness of sample bed (L):

From TABLE IX, page 60, the thickness of the sample bed was 25 cm. = L.

5. Kinematic viscosity of the water (v):

From TABLE IX, page 60, the observed temperature of the distilled water was  $26.2^\circ\text{C}$ .

From FIGURE 3, page 69, an observed temperature of  $26.2^\circ\text{C} = 26.4^\circ\text{C}$  corrected temperature.

From FIGURE 4, page 71, the kinematic viscosity of distilled water at  $26.4^\circ\text{C}$  is  $0.0087 \text{ cm}^2/\text{sec.} = v$ .

6. Porosity of fractional free volume (E):

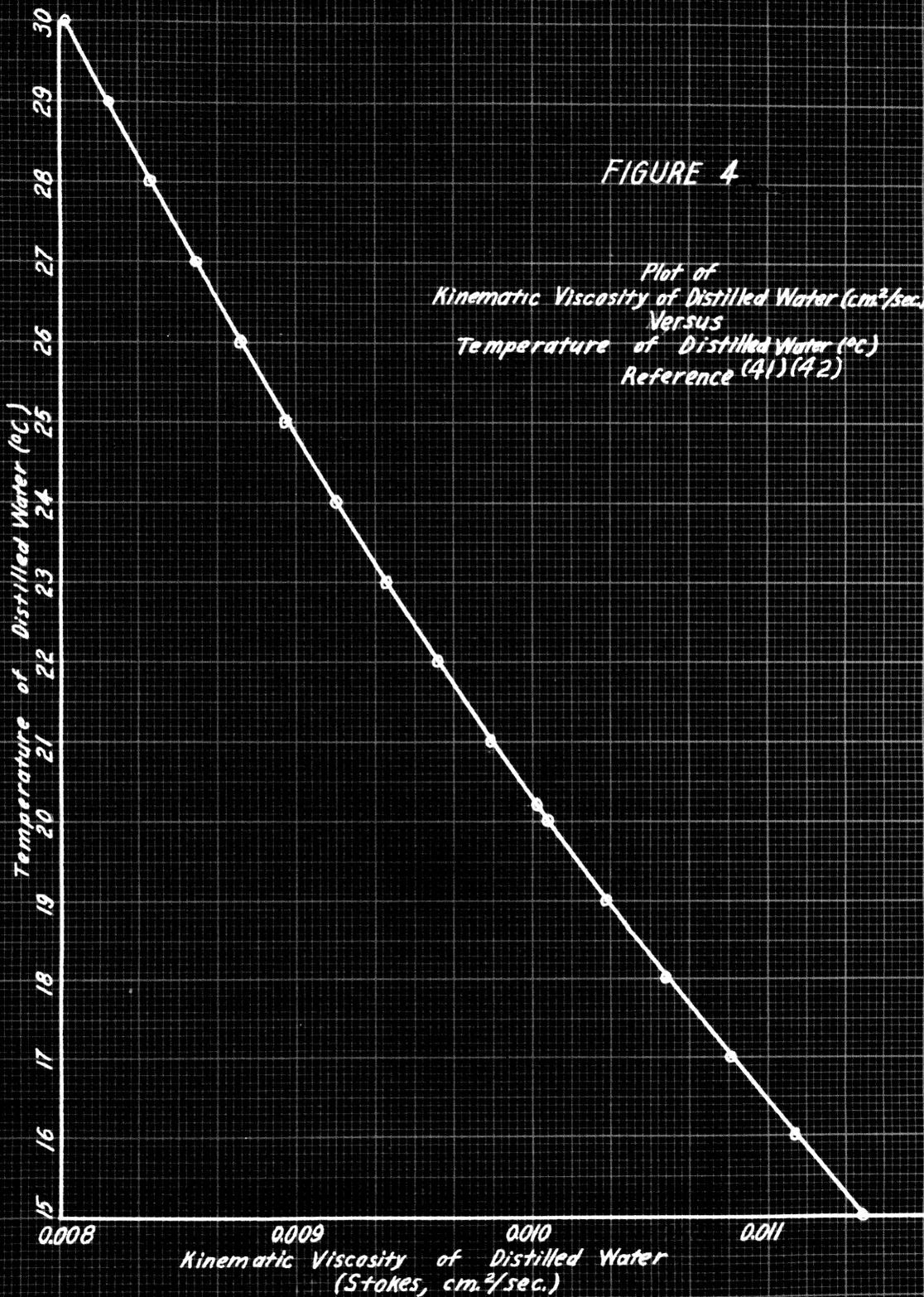
From 2, above,  $A = 7.952 \text{ cm}^2$ .

From 5, above,  $L = 25 \text{ cm}$ .

From TABLE IX, page 60, the weight of coal in the sample container was:

$$215.0569 - 67.5109 = 147.5460 \text{ gms.}$$

Using data from TABLE IV, the specific gravity of coal sample no. 2 was calculated (2) to be 1.408.



Since  $\frac{\text{Total Volume} - \text{Volume of Coal}}{\text{Total Volume}} = \text{Porosity.}$

$$\therefore \frac{(7.952 \times 25) - \frac{(147.5460)}{1.408}}{7.952 \times 25} = 0.461 = E.$$

7. Summarizing the above results:

$$A = 7.952 \text{ cm}^2.$$

$$h = 1.533 \text{ cm.}$$

$$Q = 1.104 \text{ cm.}^3/\text{sec.}$$

$$L = 25 \text{ cm.}$$

$$v = 0.0087 \text{ cm.}^2/\text{sec.}$$

$$E = 0.461$$

$$\therefore S_0 = 14 \frac{7.952 \times 1.533}{1.104 \times 25 \times 0.0087} \cdot \frac{0.461^3}{(1 - 0.461)^2}$$

$$S_0 = 58 \text{ cm.}^2/\text{cm.}^3 = \text{specific surface area of coal sample no. 2.}$$

The results obtained in the investigation of the adsorption of colloidal particles from tannin extract and viscose spinning bath solutions by coal are given in the following tables and graphs:

TABLE XVI - Percent Total Solids in Samples of Tannin Extract Solution Passed Through Coals Tested.

Page 74.

TABLE XVII - Comparison of Solids Removed from Viscose Spinning Bath Solution by Coal with Proximate Analysis of Coal. Page 75.

FIGURE 1 - Plot of Solids Removed from Spinning Bath Solution for Different Coals per 1000 cm.<sup>2</sup> of Surface Area of Coal (Per Cent) Versus Porosity of Coal Bed (Per Cent Volume). Page 77.

TABLE XLX -Comparison of Solids Removed from Viscose Spinning Bath Solution by Different Sizes of Coal Sample No. 1 with Total Surface Area of Coal. Page 78.

FIGURE 2 - Plot of Solids Removed from Spinning Bath Solution by Different Sizes of Coal Sample No. 1 Versus Total Surface Area of Coal. Page 79.

TABLE XVI

Per Cent Total Solids in Samples of Tannin Extract Solution  
Passed Through Coals Tested

Coal Sample No. (Size: -4 †8 Mesh)	Liquid Rate of Coverage of Coal (cm. <sup>2</sup> /min.)***	Total Solids in Tannin Extract Solution Samples (Per Cent)						Maximum Deviation from Feed (Per Cent)
		Feed	A <sub>1</sub>	A <sub>2</sub>	A <sub>3</sub>	A <sub>4</sub>	A <sub>5</sub>	
1	2600	3.60	3.61	3.61	3.63	3.66	3.63	+ 0.06
		*3.54	3.55	3.55	3.56	3.57	-	+ 0.03
2	1000	3.63	3.64	3.62	3.66	3.66	3.66	+ 0.03
3	1000	3.67	3.65	3.64	3.65	3.66	3.68	- 0.03
4	1000	3.60	3.65	3.59	3.64	3.65	3.65	+ 0.05
5	1000	3.68	3.68	3.65	3.66	3.70	3.68	- 0.03
6**	1000	3.67	3.78	3.65	3.68	3.69	3.65	+ 0.10
7	1000	3.67	3.72	3.68	3.70	3.71	3.72	+ 0.05
8	1000	3.67	3.69	3.66	3.67	3.70	3.69	+ 0.03
9	1000	3.74	3.72	3.68	3.69	3.70	3.69	- 0.06
4†	16 ml/min.	3.74	3.73	3.73	3.72	3.72	3.74	- 0.02
2††	500	3.76	3.74	3.74	3.74	3.74	3.73	- 0.03

\* Samples analyzed by Mead Corporation, Tannin Extract Division, Lynchburg, Virginia

\*\* For size, see TABLE I, page 26.

\*\*\* Liquid Rate of Coverage of Coal:

cm.<sup>3</sup> of liquid/min. × cm.<sup>2</sup> of surface/cm.<sup>3</sup> of volume of coal = cm.<sup>2</sup>/min.

† Size -16 †40 Mesh (U. S. Standard Screen). For treatment, see item 6, page 44.

†† This coal had not been leached with 10% HCl, but had been washed with tap water to remove dust in it.

TABLE XVII

Comparison of Solids Removed from Viscose Spinning Bath Solution by Coal  
with Proximate Analysis of Coal

Coal Sample No. (Size: -4 +8 Mesh)	Solids Removed from Spinning Bath Solution by Coal (p.p.m.)	Solids Removed from Spinning Bath Solution by Coal (Per Cent)	Total Surface Area of Coal in Bed (cm <sup>2</sup> )	Solids Removed from Spinning Bath Solution by Coal per 1000 cm. <sup>2</sup> of Surface Area (Per Cent)	Proximate Analysis of Coal Sample			
					Ash (Per Cent)	Volatile Matter (Per Cent)	Fixed Carbon (Per Cent)	Moisture (Per Cent)
4	26.7	68.3	7,090	9.6	24.99	12.32	62.21	0.48
6*	32.5	83.1	8,520	9.7	9.01	6.48	82.62	1.89
1	23.9	64.7	6,690	9.7	7.83	3.65	85.21	3.31
8	26.4	67.5	6,130	11.0	4.20	41.84	52.44	1.52
9	30.7	78.6	6,690	11.8	2.70	37.58	57.39	2.33
3	29.3	74.9	6,100	12.3	4.89	44.34	48.17	2.60
5	27.7	70.8	5,700	12.4	2.40	23.82	73.48	0.30
7	29.4	75.2	5,880	12.8	2.67	36.12	60.11	1.10
2	30.7	78.5	5,330	14.7	11.30	15.26	72.99	0.45

\* For size, see TABLE I, page 26.

TABLE XVIII

Comparison of Solids Removed from Viscose Spinning Bath Solution by Coal  
Physical Properties of Coal

Coal Sample No. (Size: -4 +8 Mesh)	Solids Removed from Spinning Bath Solution by Coal (p.p.m.)	Solids Removed from Spinning Bath Solution by Coal (Per Cent)	Specific Surface Area of Coal (cm. <sup>2</sup> /cm. <sup>3</sup> )	Total Surface Area of Coal in Bed (cm. <sup>2</sup> )	Solids Removed from Spinning Bath Solution by Coal per 1000 cm. <sup>2</sup> of Surface Area (Per Cent)	Specific Gravity of Coal	Porosity of Coal Bed (Per Cent Volume)	Relative Roughness of Coal Surface (Arbitrary Units)**
4	26.7	68.3	77	7,090	9.6	1.596	51.4	3.5
6*	32.5	83.1	93	8,520	9.7	1.590	51.7	1
1	23.9	64.7	73	6,690	9.7	1.667	52.3	1
8	26.4	67.5	67	6,130	11.0	1.308	50.0	2
9	30.7	78.6	73	6,690	11.8	1.320	52.1	2
3	29.3	74.9	67	6,100	12.3	1.319	50.5	1.5
5	27.7	70.8	62	5,700	12.4	1.301	48.2	3
7	29.4	75.2	64	5,880	12.8	1.295	49.2	1.5
2	30.7	78.6	58	5,330	14.7	1.403	47.2	3

\* For size, see TABLE I, page 26.

\*\* Visual classification = 1 = smoothest surface.

FIGURE 1

Plot of  
 Porosity of Coal Sample Bed (Per Cent Volume)  
 Versus  
 Solids Removed from Spinning Bath Solution  
 by Different Coals per 1000 cm<sup>2</sup> of  
 Surface Area (Per Cent)

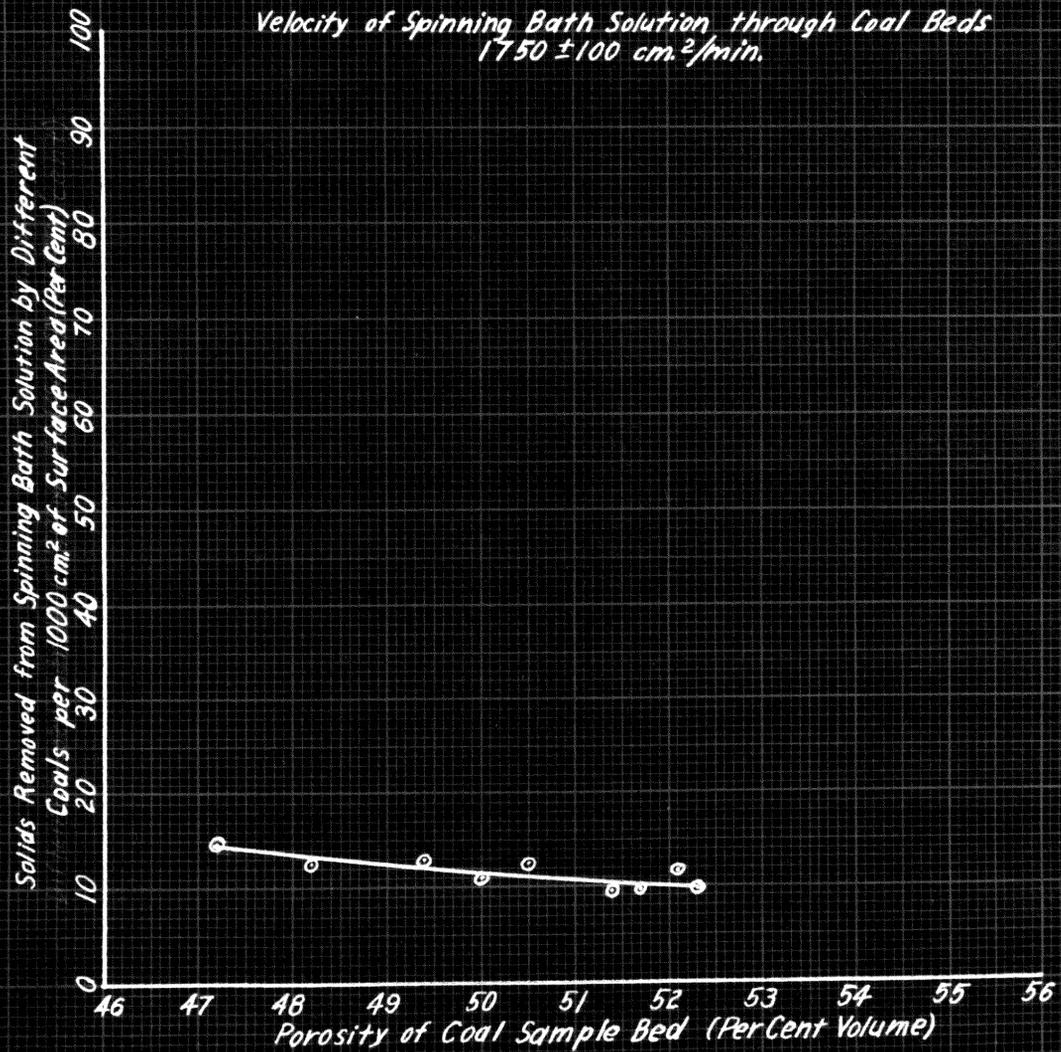


TABLE XIX

Comparison of Solids Removed from Viscose Spinning Bath Solution by Different Sizes of Coal Sample No. 1 with Total Surface Area of Coal

Size of Coal (U.S. Standard Screen Mesh)	Solids Re-Moved from Spinning Bath Solution by Coal (p.p.m.)	Solids Re-Moved from Spinning Bath Solution by Coal (Per Cent)	Total Surface Area of Coal in Bed (cm. <sup>2</sup> )	Porosity of Coal Bed (Per Cent Volume)
-4 +8*	13.6	36.9	2,670	41.7
-4 +8	10.3	27.9	2,740	51.4
-8 +10	19.6	53.1	5,480	53.1
-10 +16	20.6	55.8	7,740	52.6
-16 +20	26.0	70.4	11,340	55.0
-20 +30	30.1	81.6	15,630	56.2
-30 +40	34.1	92.4	22,940	56.2

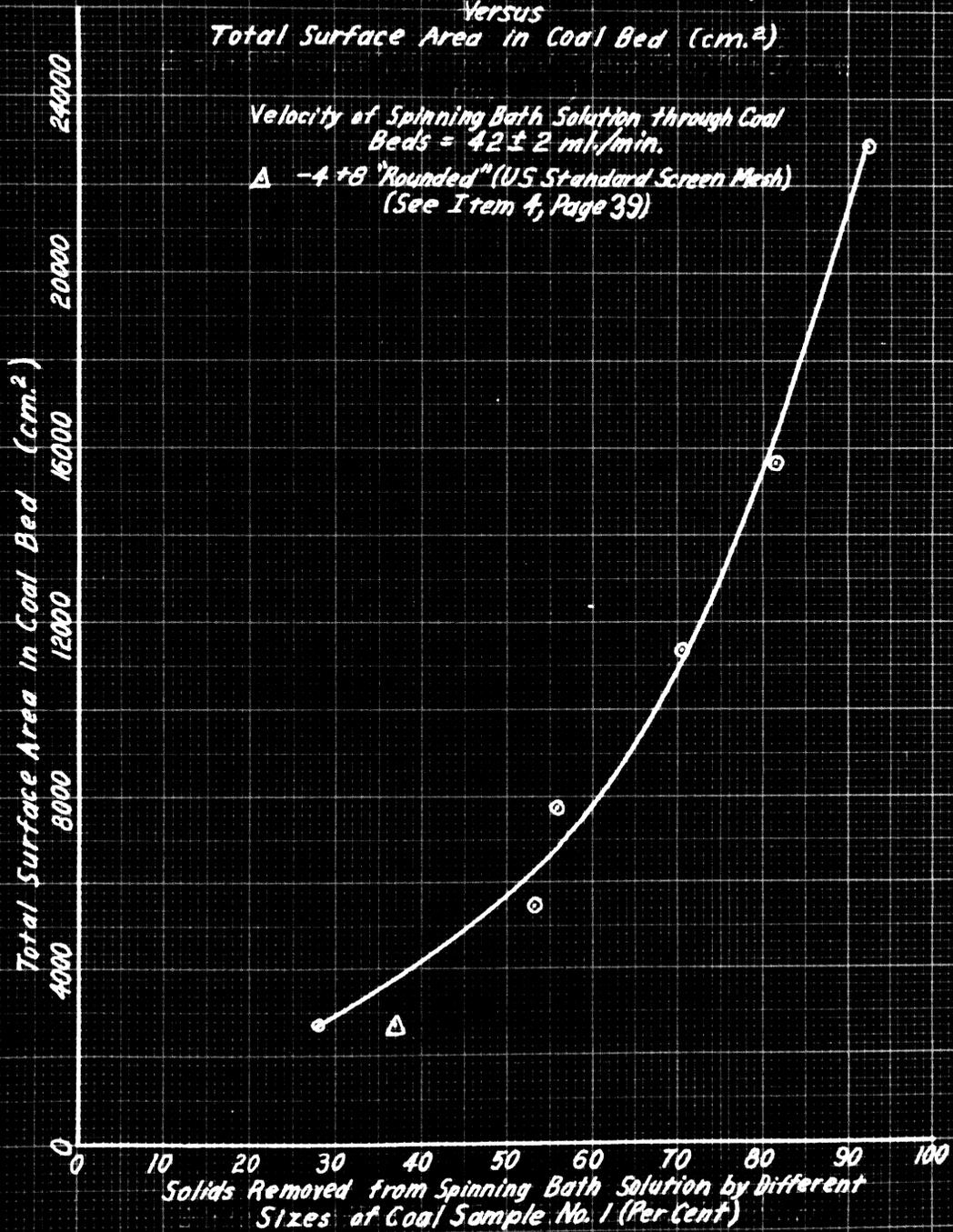
\* This sample was "rounded." See item 4, page 39.

## FIGURE 2

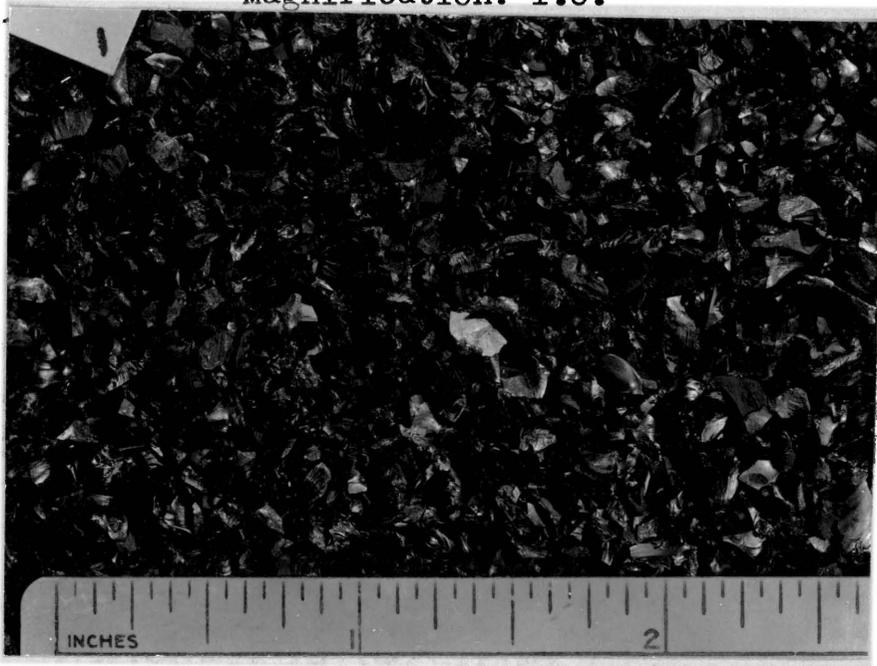
Plot of  
 Solids Removed from Spinning Bath Solution by Different  
 Sizes of Coal Sample No. 1 (Per Cent)  
 Versus  
 Total Surface Area in Coal Bed (cm.<sup>2</sup>)

Velocity of Spinning Bath Solution through Coal  
 Beds =  $42 \pm 2$  ml./min.

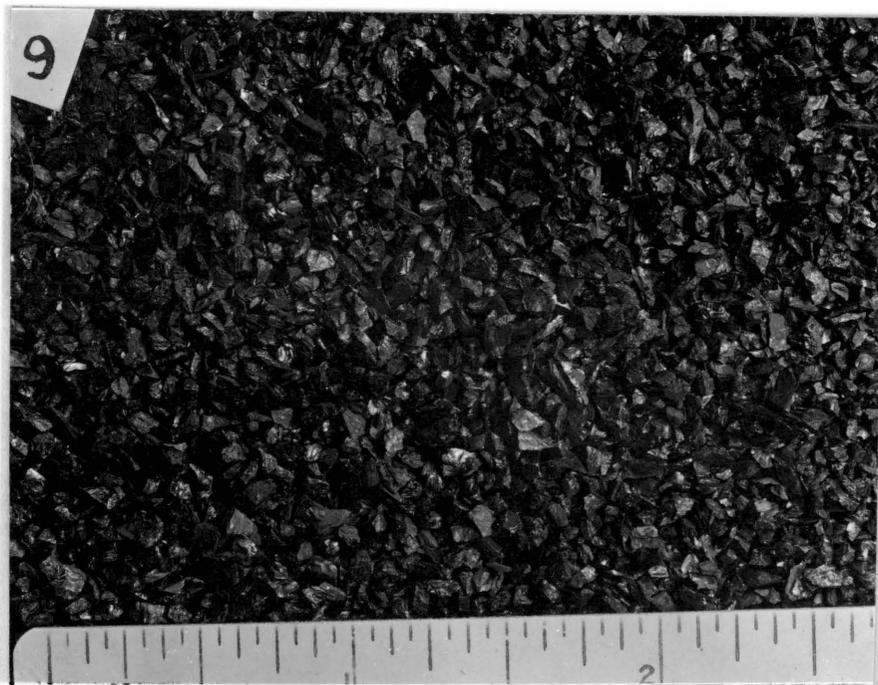
△ -4 +8 "Rounded" (US Standard Screen Mesh)  
 (See Item 4, Page 39)



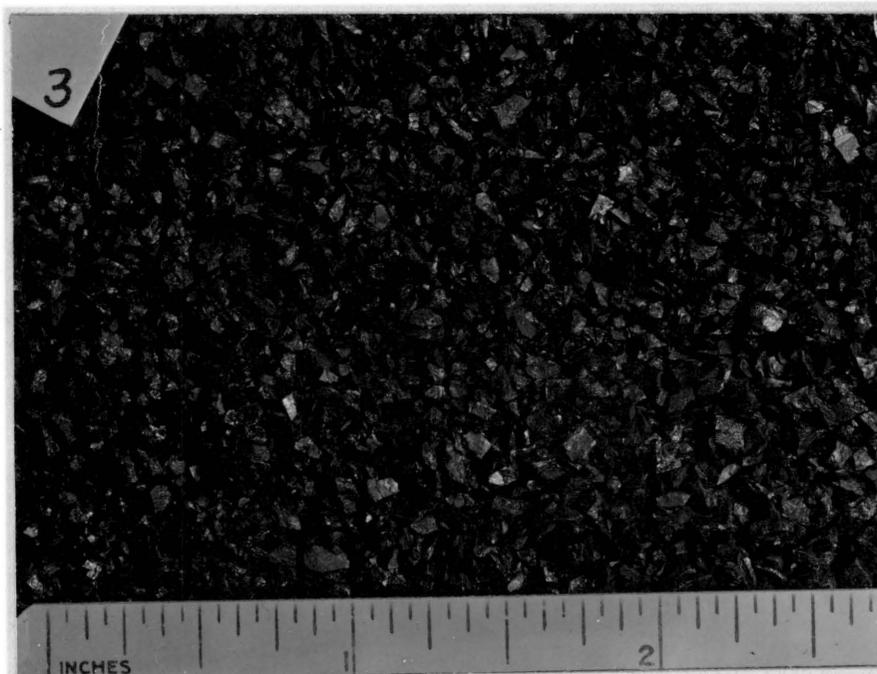
G. Macrophotographs  
Magnification: 1.6.



Coal Sample No. 1, Anthracite,  
Lehigh Navigation Coal Co., Lansford, Pennsylvania.



Coal Sample No. 9, High Volatile Bituminous B,  
Pond Creek Colliery, Williamson, West Virginia.



Coal Sample No. 3, High Volatile Bituminous C,  
Independent Coal and Coke Co., Kenilworth, Utah.



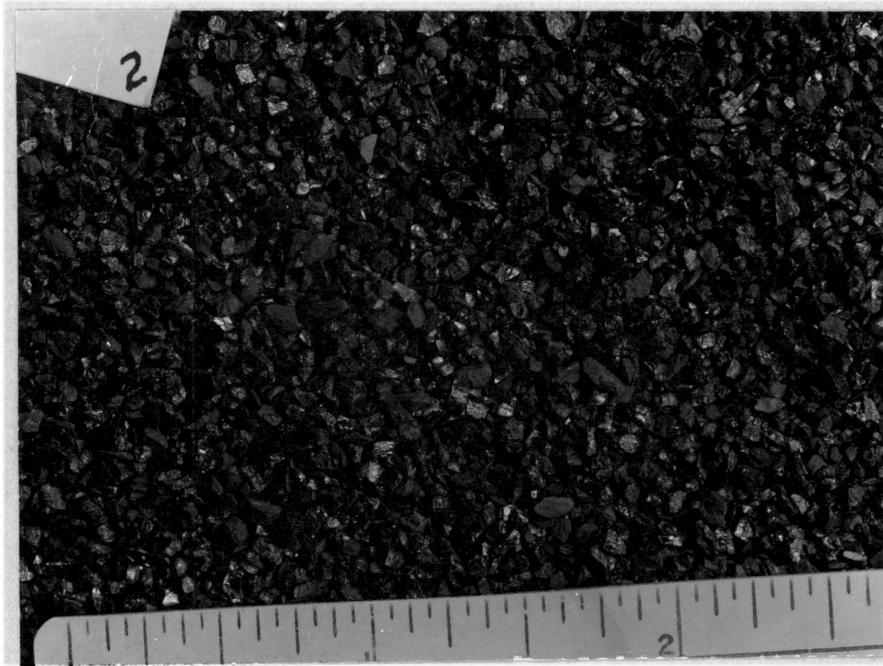
Coal Sample No. 8, High Volatile Bituminous C,  
Pond Creek Colliery, Williamson, West Virginia.



Coal Sample No. 5, Medium Volatile Bituminous,  
Jewell Ridge Coal Co., Jewell Ridge, Virginia.



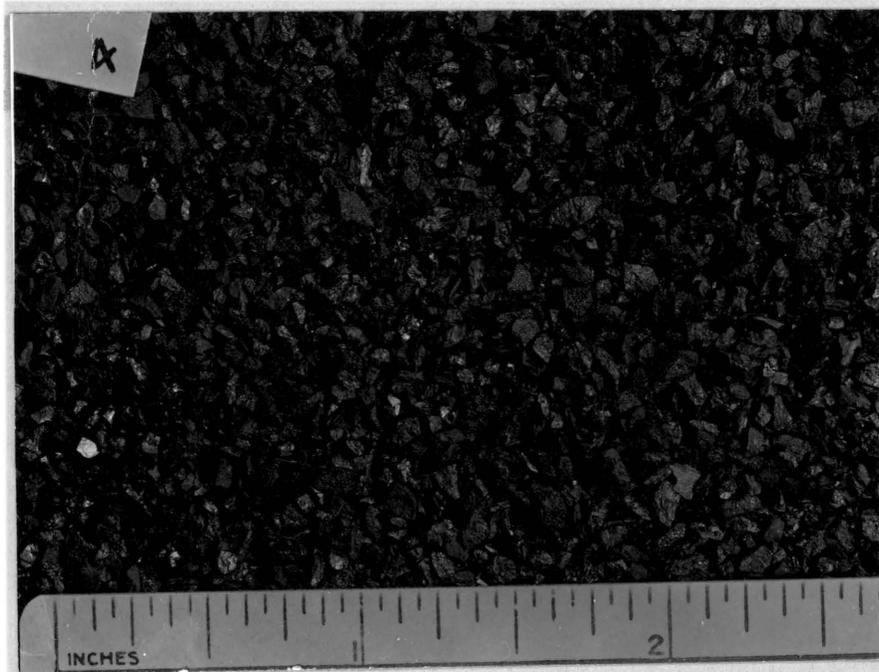
Coal Sample No. 7, High Volatile Bituminous A,  
Stonega Coke and Coal Co., Big Stone Gap, Virginia.



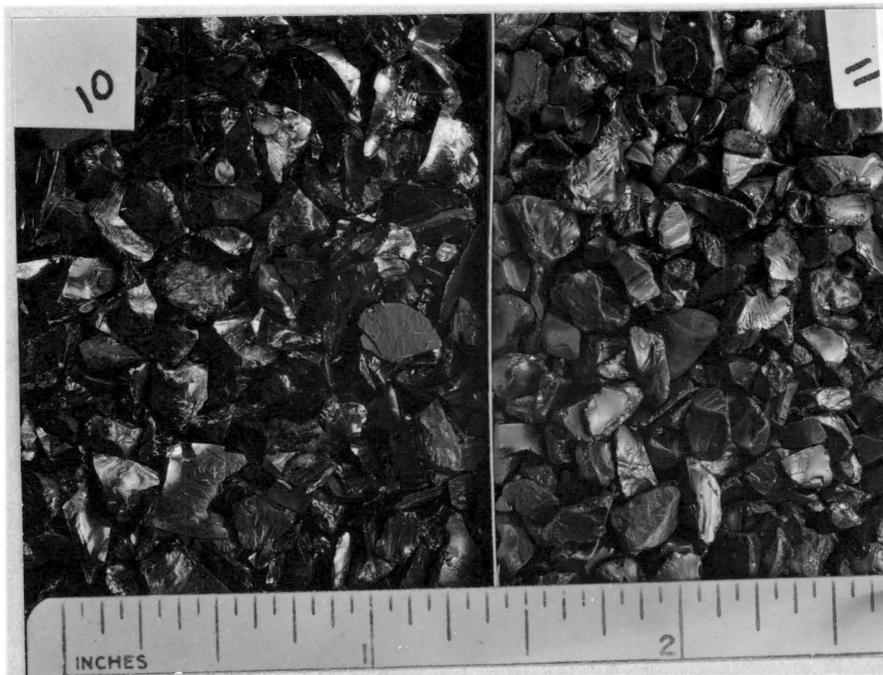
Coal Sample No. 2, Low Volatile Bituminous,  
Turkey Gap Coal and Coke Co., Dott, West Virginia.



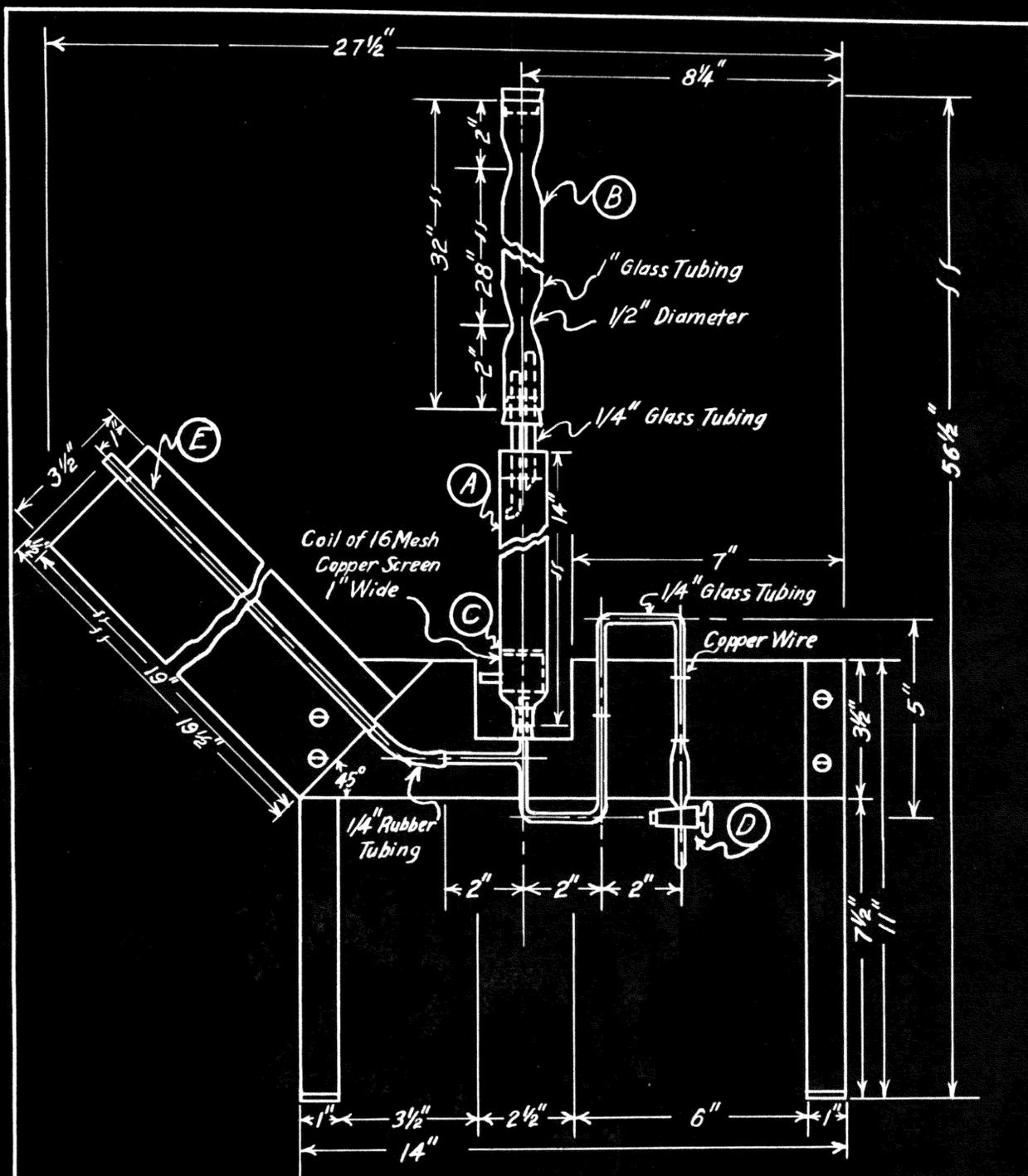
Coal Sample No. 6, Anthracite,  
Hudson Coal Co., Scranton, Pennsylvania.



Coal Sample No. 4, Semi-anthracite,  
Great Valley Coal Co., McCoy, Virginia.



Coal Sample No. 1, Area 10: -4+8 Mesh, Area 11: -4+8 Mesh  
"Rounded", Anthracite, Lehigh Navigation Coal Co.,  
Lansford, Pennsylvania.

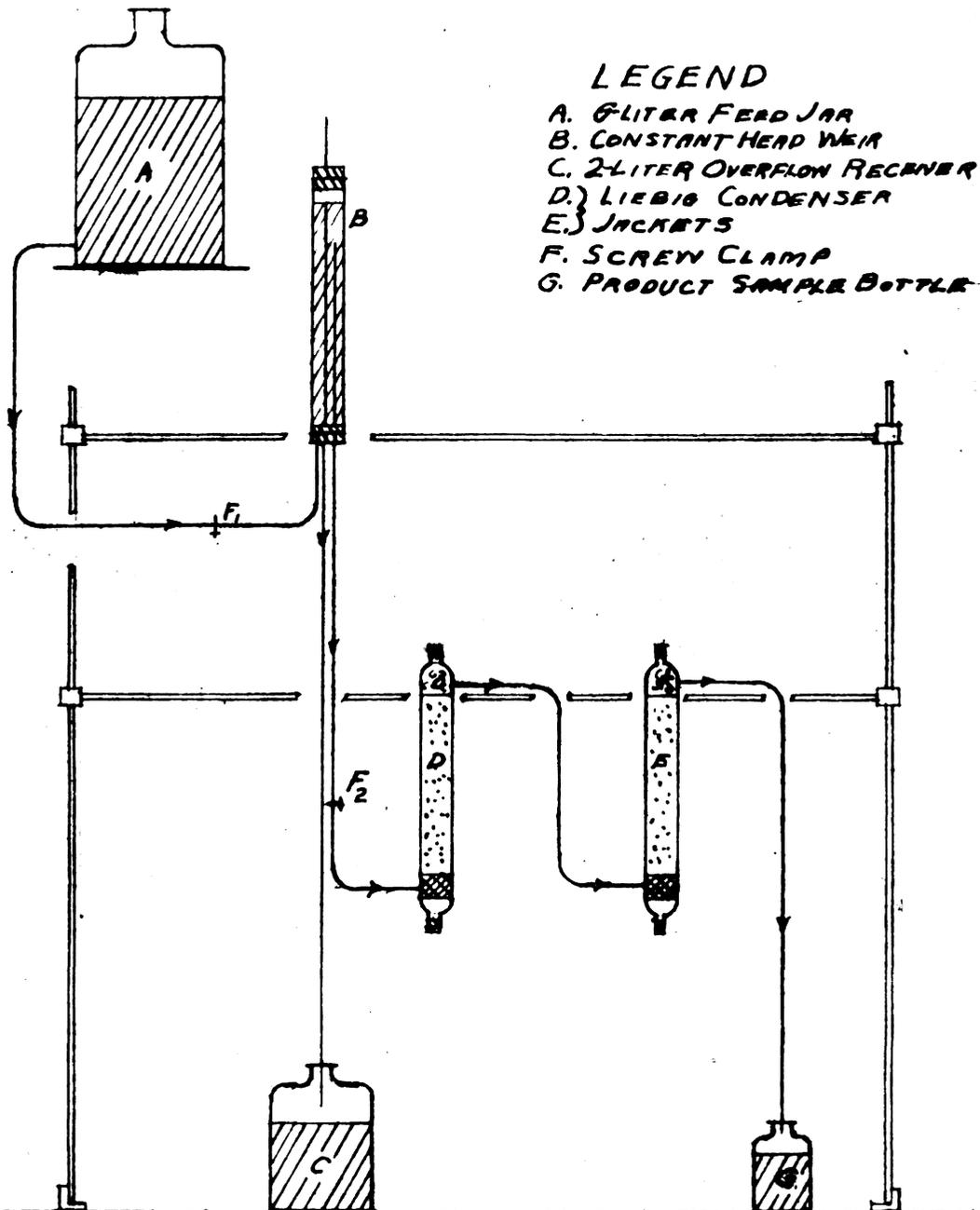


- A Container for Sample Bed,  
1 1/4" Liebig Condenser
- B Water Reservoir
- C Sample Support, Copper  
Screen, 16 Mesh,  
1 1/4" Diameter
- D Stopcock
- E Manometer

DEPARTMENT OF CHEMICAL ENGINEERING  
VIRGINIA POLYTECHNIC INSTITUTE  
BLACKSBURG, VIRGINIA

APPARATUS FOR THE DETERMINATION  
OF SURFACE AREAS OF  
SMALL PARTICLES OF COAL

Drawn By <i>P.H.P.</i>	Date 1/8/47	Scale; 1/2" = 1"
Checked By <i>P.H.P.</i>	Date 1/8/47	Drawing No.
Approved By <i>P.H.P.</i>	Date 1/11/47	2

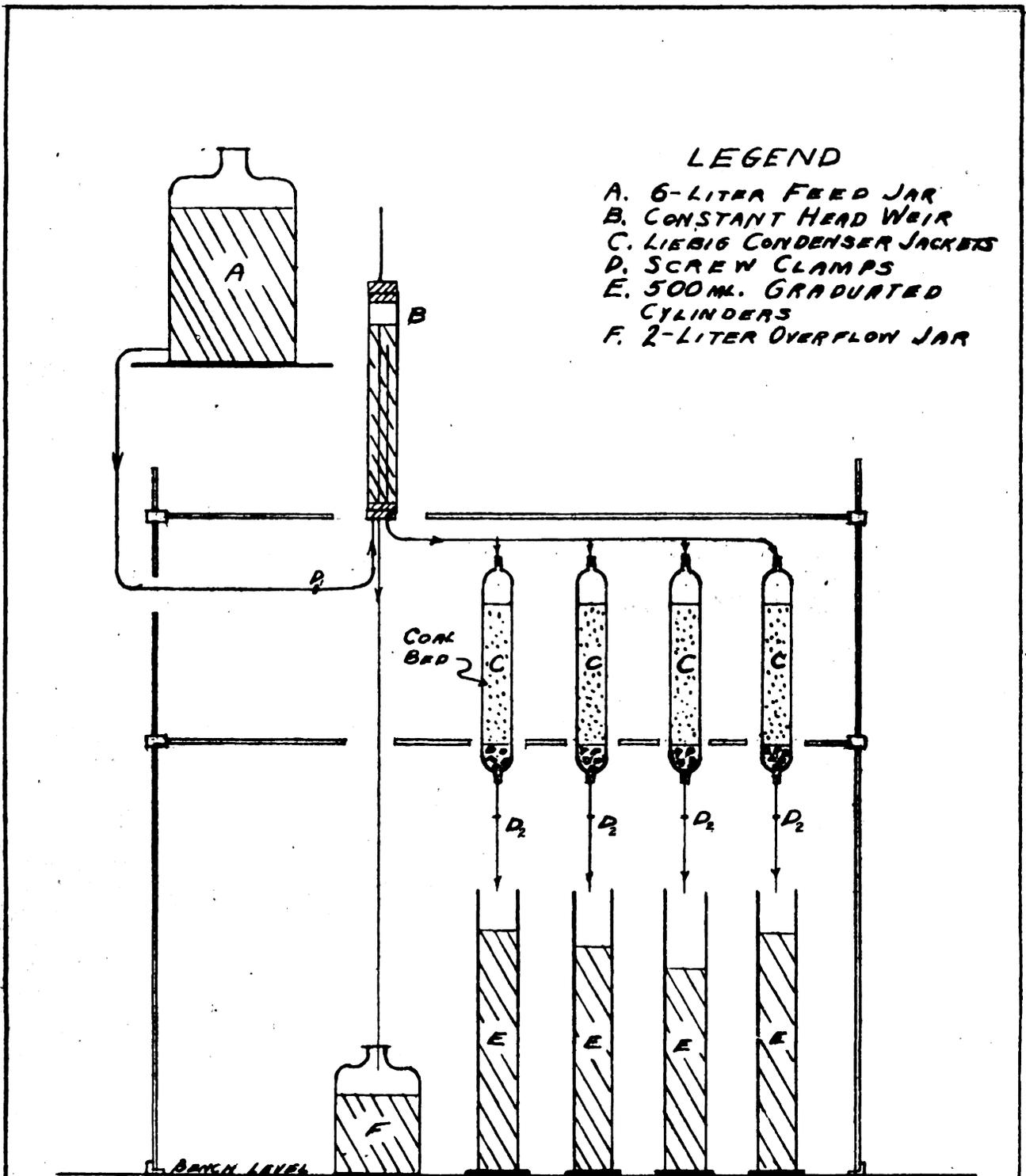


DEPARTMENT OF CHEMICAL ENGINEERING  
 VIRGINIA POLYTECHNIC INSTITUTE  
 BLACKSBURG, VIRGINIA

DIAGRAM OF EXPERIMENTAL EQUIPMENT SHOWING  
 LAYOUT AND FLOW

SCALE:  $1\frac{1}{2}'' = 1'-0''$   
 DRAWN BY: JMB.  
 APPROVED BY: JWB.

CASE No. 46  
 FILE No. 539  
 DWG. No. 3



### LEGEND

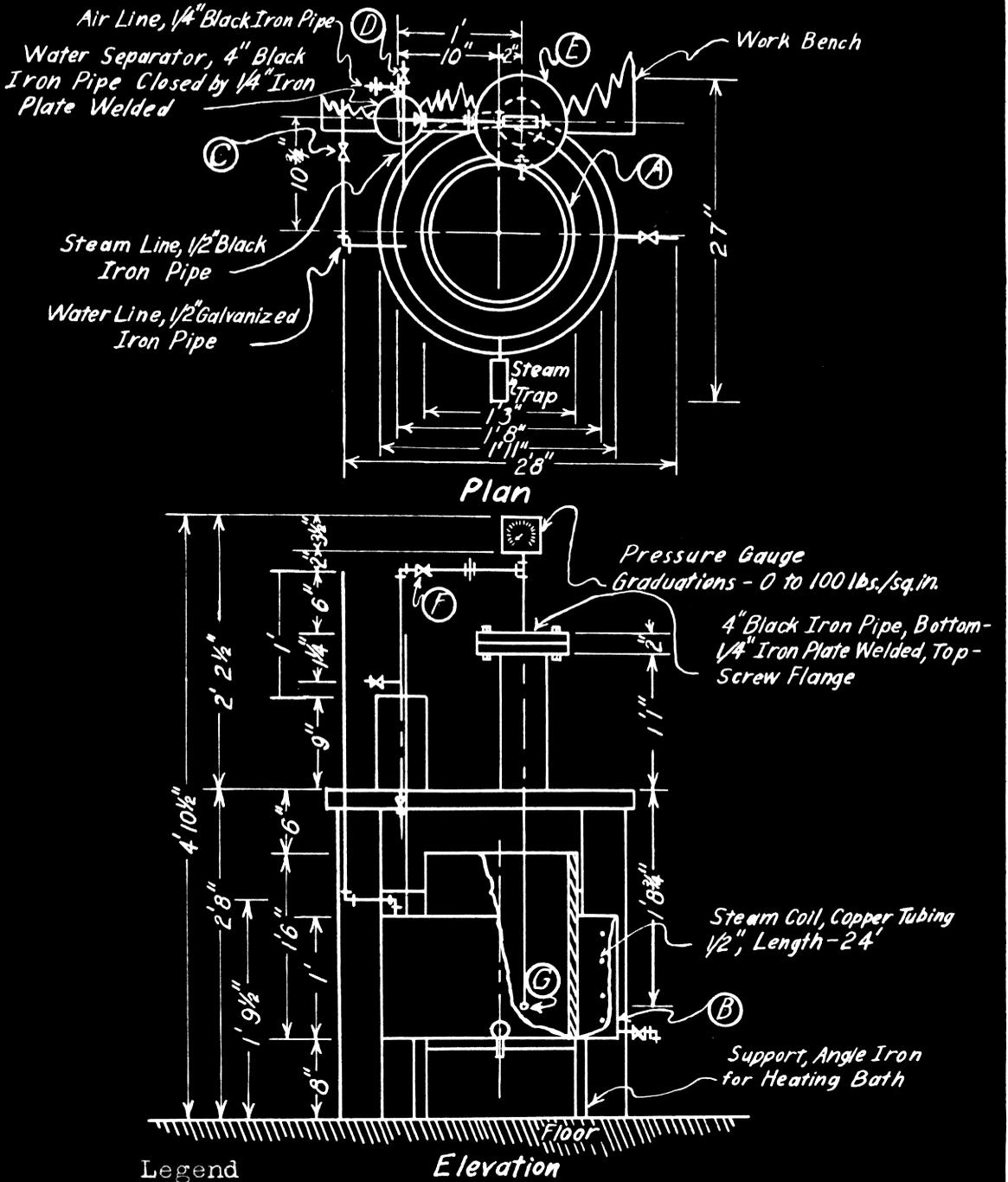
- A. 6-LITER FEED JAR
- B. CONSTANT HEAD WEIR
- C. LIEBIG CONDENSER JACKETS
- D. SCREW CLAMPS
- E. 500ML. GRADUATED CYLINDERS
- F. 2-LITER OVERFLOW JAR

DEPARTMENT OF CHEMICAL ENGINEERING  
 VIRGINIA POLYTECHNIC INSTITUTE  
 BLACKSBURG, VIRGINIA

LAYOUT AND FLOW DIAGRAM FOR  
 FILTRATION OF VISCOSE SPIN BATH

DRAWN BY: JMB.  
 APPROVED BY: JMB.  
 SCALE:  $1\frac{1}{2}'' = 1'-0''$

CASE NO. 96  
 FILE NO. 539  
 DWG. NO. 7



- A Spinning Bath, Earthenware Crock, 10 Gallon
- B Heating Bath
- C Water Valve, 1/2" Globe
- D Steam Valve, 1/2" Globe
- E Pressure Chamber
- F Air Valve, 1/4" Needle
- G Spinneret, 1/4" Black Iron Cap, 1/16" Drilled

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APPARATUS FOR SPINNING VISCOSE

Drawn By: P.N.P.	Date: 1/12/47	Scale: 3/4" = 1'
Checked By: P.N.P.	Date: 1/12/47	Drawing No.
Approved By: J.W.B.	Date: 1/13/47	5

#### IV DISCUSSION

##### A. Discussion of Results

Surface Area of Coal Samples: It was thought that one of the contributing factors to adsorption of colloidal particles by coal was the surface area available. A search of the literature revealed several methods (7) (27) of determining the total surface area (internal and external) of coal, but it is believed that the internal surface area is probably not useable in the adsorption of colloidal particles, because of the small diameter of the capillaries which make up this internal surface area. King and Wilkins (24) calculated the average diameter of capillaries in coal to be  $6 \times 10^{-7}$  cm. The tannin extract and viscose spinning bath solutions used in this investigation were cloudy when freshly prepared; however, the colloidal particles, causing this cloudiness, would settle on standing (three or four days) leaving the solution clear. This indicated that the particles in the testing solutions were either in the upper range of colloidal particle size ( $1 \times 10^{-4}$  cm. diameter (18)) or larger. From this observation and the work of King and Wilkins, it would definitely appear that the internal capillaries of the coal particles were not available for adsorption of the colloidal particles thus necessitating the determination of external surface only.

A search of the literature revealed a method (9) of determining external surface area of various shaped particles. This method, with modifications, was used in determining the surface area of the

coal samples. It was necessary to use a manometer (E, Drawing No. 2, page 85) inclined at  $45^{\circ}$  rather than a vertical one as recommended (Drawing No. 1, page 20) to increase the accuracy of measurements of the resistance drop across the sample bed from  $\pm 0.074$  to  $\pm 0.045$  cm. of water. Even with this modification an error of one half of a scale unit (1 unit = 0.0358 in.  $H_2O$ ) affected the specific surface area of the sample to the extent of  $3 \text{ cm}^2/\text{cm}^3$ . It was also necessary to boil the coal samples in distilled water until all occluded air was eliminated before making surface area determinations. It was found that the presence of occluded air in the sample bed caused an error in these determinations of  $\pm 10 \text{ cm}^2/\text{cm}^3$ .

Keyes (23), in referring to a method of determining surface areas similar to the one used, pointed out that the surface area calculated might be substantially less than it actually is when the particles measured contain internal angles or fissures. This may be true with the coal samples tested, but it is believed that all measurements on the coals were comparable.

It was found that the maximum deviation between measurements on one coal sample was  $\pm 5 \text{ cm}^2/\text{cm}^3$ . Each coal sample was tested four times, and it is believed that the average of these four measurements reduced the maximum error to  $\pm 2 \text{ cm}^2/\text{cm}^3$ .

Adsorption Tests Using Tannin Extract Solution: Couper (14), in previous investigations of the adsorption of colloidal particles from tannin extract solution by coal, stated that iron in the coal might react with the tannin in solution to precipitate iron tannates.

To eliminate this possibility, all coal samples tested, except one (coal sample No. 2) were leached, prior to testing, with 10% HCl. The leaching was continued until there was no change in the appearance of a sample of tannin extract solution left in contact with a sample of coal for twenty-four hours. The one sample (No. 2, low volatile bituminous) which was not leached with 10% HCl, but washed with tap water to eliminate dust, did not discolor the tannin extract solution when tested with it. Referring to TABLE XVI, page 62, it is seen that the maximum deviation of total solids in treated samples (coal sample no. 2) from total solids in the feed sample was  $-0.03\%$  which is considered to be within the limit of experimental error (48). This test on the one coal does not preclude the possibility that the other coal samples would affect the testing liquid.

It was thought that the leaching and subsequent washing process would eliminate the possibility of dust being retained by the coal sample. However, in one particular instance, notably sample no. 6, a maximum deviation in solid content of  $\pm 0.10\%$  was noted. This was encountered in the first sample passed through the coal bed. Since this was an anthracite sample, relatively hard and non friable, the only explanation for such an increase in solid content of treated sample (over and above experimental error,  $\pm 0.03\%$ ), is that the sample was not washed sufficiently in its preparation, with the consequent retention of dust particles by the coal.

It may also be seen in TABLE XVI, page 62, that there was

no appreciable adsorption of colloidal particles by the coals. With the exception of coal sample no. 6, there was no appreciable difference in the total solids content of the treated samples and that of the feed samples used for comparison. All of the deviations were within or very near the allowable limits of experimental error  $\pm 0.03\%$ . Even an increase of surface area did not affect adsorption as evidenced by coal sample no. 4<sup>+</sup> (semi-anthracite, -16 +40 mesh, U. S. Standard Screen). Further, a decrease in velocity from 2600 to 500 cm.<sup>2</sup>/min. of the tannin extract solution through the bed did not affect adsorption of colloidal particles. These flow rates correspond to  $\frac{1}{4}$  -  $1\frac{1}{4}$  gals./sq. ft. of filter bed/min; while commercial filtering rates are recommended as 1 to 2 gals/sq.ft./min.

Some of the adsorption tests conducted required a period of three to four hours for completion. Over this period of time at such flow velocities (11 to 16 ml./min.) of tannin extract solution, it was noticed that some solids had settled to the bottom of each adsorption chamber. It is believed that if the flow of testing solution had been in a downward direction through the coal beds, these settling solids would have provided a filtering medium to aid further in separation of the solid content.

Even though the results shown in TABLE XVI may seem somewhat erratic and in some instances difficult to explain, they may nevertheless be considered accurate in a comparable sense. This consideration is based on the results obtained when one set of tannin extract samples,

from coal bed no. 1, was analyzed by the investigator and a duplicate set was analyzed by the Mead Corporation, Tannin Extract Division, Lynchburg, Virginia. It will be noted that the results obtained by the investigator were higher by a consistent per cent within the allowable range of experimental error.

Specific Gravities of Coal Samples: It was thought that the leaching of the coal samples with 10% HCl would change their specific gravities. Since the coal samples tested with tannin extract solution were leached with 10% HCl, and those tested with viscose spinning bath solution were not, two sets of specific gravity determinations were made. Determinations on six of the coals were within allowable limits of experimental error,  $\pm 0.01$  (2). While three sets of determinations differed by 0.03, 0.03, and 0.02, these results showed that there was no appreciable change in specific gravity when the coals were leached with 10% HCl.

Viscose Spinning Bath as Testing Solution: After no apparent separation of solids from tannin extract solution was obtained by the different coals, it was decided to use viscose spinning bath as the adsorbate solution. It was known that the American Viscose Corporation, Roanoke, Virginia uses coal to clarify their spinning baths. Therefore, this solution should be a good liquid for testing the adsorbent properties of coal.

Viscose Spinning Bath Composition: Since it was impossible to obtain any spinning bath solution from the American Viscose Corporation, Roanoke, Virginia for testing purposes, it was necessary to

prepare small (10 liter) batches of spinning bath. Without the necessary temperature controls (none were available) in the preparation of viscose itself, each batch varied, consequently affecting the nature and properties of the exhausted spinning bath solution. It was noted that in some cases the solids in the final testing liquid settled slowly (3 to 4 days) while others required 12 to 16 hours.

The variations in the properties of the viscose spinning bath solution made it necessary to run as many adsorption tests as possible using the same testing solution. The data reported on coal samples no. 2 through 9 (TABLE XIV, page 65) were obtained using one viscose spinning bath solution. The data reported on all sizes of coal sample no. 1 were obtained using another viscose spinning bath solution.

Preliminary Adsorption Tests: Several preliminary tests were made to determine optimum conditions of adsorption tests using viscose spinning bath solution as the testing medium. The first test was made on coal sample no. 1 (anthracite) with rates of coverage by the testing liquid of 1000 cm.<sup>2</sup>/min., 1750 cm.<sup>2</sup>/min. and 3000 cm.<sup>2</sup>/min. corresponding to  $\frac{1}{2}$ ,  $\frac{3}{4}$ , and  $1\frac{1}{2}$  gals./sq.ft./min. - the range of rates used by the American Viscose Corporation. As expected, it was found that the solids adsorbed increased (45.8%, 64.3%, and 68.0%) with decreasing velocity of testing liquid. It was decided to use a rate of coverage of 1750 cm.<sup>2</sup>/min. for the majority of the tests on the different coals.

Analysis of Solids in Viscose Spinning Bath Solution Samples: Two feed samples of each of the two viscose spinning bath so-

lutions used in the final adsorption tests were analyzed. In each case one sample was analyzed by the investigator and the other by F. W. Bell. The investigator's analyses showed more solids present in the solutions than did the analyses of Bell in each case, 39.1 p.p.m. to 27.8 p.p.m. and 36.9 p.p.m. to 32.0 p.p.m. solids. Bell used two crucibles in analyzing each sample, while the investigator used one for each sample. It was thought that the division of the samples by using two Gooch crucibles for filtering and washing each sample might introduce an error in analysis. To check the reproducibility of analyses, three samples of viscose spinning bath solution, from one spinning bath, were analyzed for solids by the investigator. (See TABLE XV, page 66). The calculated solids were 90.4, 88.0, and 90.3 p.p.m. These results indicate that the accuracy of analyses of solids in viscose spinning bath solution samples was  $\pm 2$  p.p.m. This variation in analyses may be due to:

1. Inaccuracy of weighing crucibles and samples.
2. Loss of solids while filtering and washing in Gooch crucibles.
3. Insufficient washing of solids in the Gooch crucibles.
4. Oxidation of solids while drying to a constant weight.

Since all samples of solids were washed until free of sulfates, as indicated by 10%  $\text{BaCl}_2$  in a boiling sample of wash water, sufficient washing was obtained. This leaves the other three possibilities to create inaccuracies.

Adsorption and Porosity of Coal Bed: To eliminate the effect of surface area on tests of the different coals using viscose spinning bath solution, the solids removed were calculated to percent per 1000 cm.<sup>2</sup> of surface area of coal in the adsorption chambers. The effect of velocity of the testing solution passing a unit surface area was eliminated by calculating velocities from 1750 cm.<sup>2</sup>/min:

$$\text{cm.}^2/\text{min.} \div \text{cm.}^2/\text{cm.}^3 = \text{cm.}^3/\text{min.}$$

For example, the specific surface area of coal sample no. 1 was 73 cm.<sup>2</sup>/cm.<sup>3</sup> (see TABLE XVIII, page 76). Therefore, the velocity of testing solution through this coal was maintained at:

$$\frac{1750}{73} = 24 \text{ cm.}^3/\text{min.}$$

The two variables in the experiments were the kind of coal tested and porosity of coal bed. The various coals exhibited variations in roughness of surface and chemical composition. All of the coals tested were -8 +16 mesh (U. S. Standard Screen), except coal sample no. 6 (anthracite) which was approximately -8 +20 mesh, and an attempt was made to pack the coal beds to the same extent. Since the size of the particles was nearly the same in each case, the diameter of the pore spaces between the particles of coal was more a function of shape than size of the particles of coal. If porosity of the coal bed is a function of pore space diameter, then, as the porosity decreases, the pore space diameter decreases. Even though the decreased diameter of the pore space would undoubtedly increase the velocity of the liquid

through the pores, it would also cause more of the testing liquid to come into intimate contact with the coal surface. If filtering is a function of surface contact, then more solids should be removed from the testing solution by the least porous bed. This is evidenced by FIGURE 1, page 77, which shows a plot of solids removed from spinning bath solution by the different coals per 1000 cm.<sup>2</sup> of surface area (per cent) versus porosity of coal sample bed (per cent volume). The curve obtained shows an increase in the solids removed from 9.7% to 14.7% by the coal as the porosity of the coal bed decreased from 52.3% to 47.2%.

Adsorption and Roughness of Surface of Coal: Referring to TABLE XVIII, page 76, it is seen that coal sample no. 1 (anthracite) and coal sample no. 9 (high volatile bituminous B) had the same surface area (6,690 cm.<sup>2</sup>) and practically the same porosity of bed (52.3% and 52.1%, respectively). Coal sample no. 1 removed 23.9 p.p.m. solids while coal sample no. 9 removed 30.7 p.p.m. solids from the testing liquid. A comparison of photographs 1 (coal sample no. 1) and 9 (coal sample no. 9) page 80, shows that the surface of the particles of coal sample no. 1 appears relatively smooth and shiny, and was arbitrarily given a relative roughness rating of 1; while the surface of the particles of coal sample no. 9 appears dull and rough and was given a relative roughness rating of 2. These results are not conclusive, however, because a different spinning bath was used in testing each coal sample.

A comparison of coal sample no. 3 (high volatile bituminous C) and coal sample no. 8 (high volatile bituminous C) with surface areas of 6,100 and 6,130  $\text{cm}^2$ , porosities of 50.5% and 50.0%, p.p.m. solids removed from the testing liquid of 29.3 and 26.7 and relative roughness of 1.5 and 2 (photographs 3 and 8, page 81), respectively, shows that the rougher coal (coal sample no. 8) removed 3 p.p.m. less solids than coal sample no. 3, the smoother of the two. These apparent erratic results may be attributed to an error in analysis or an error in classifying the coals according to roughness. This also applies to coal samples no. 5 and 7 with surface areas of 5,700 and 5,880  $\text{cm}^2$ , porosities of 48.2% and 49.2%, p.p.m. solids removed from the testing liquid of 27.7 and 29.4, and relative roughnesses of 3 and 1.5 (photographs 5 and 7, page 82) respectively.

A comparison of the adsorptive abilities of coal samples no. 6 (anthracite) and 2 (low volatile bituminous) after eliminating the effects of surface area, shows that the rougher coal (sample no. 2) relative roughness of 3, removed 14.7% solids per 1000  $\text{cm}^2$  of surface area. The smooth coal, coal sample no. 6, relative roughness of 1, removed 9.7% solids per 1000  $\text{cm}^2$  of surface area. However, the porosities of the beds of coal samples no. 2 and 6 was 47.2% and 51.7% respectively. Since the rougher coal had the least porosity, it would be impossible to say whether pore space or roughness was the predominating factor in the adsorption of colloidal particles.

Coal sample no. 4 with the greatest relative roughness (3.5)

removed the least amount of solids, 9.6% per 1000 cm.<sup>2</sup> of surface area. While analyzing the testing solution treated with this coal, it was noticed that some of the precipitate was black and crept up the sides of the crucible, used in testing, like coal dust. It is believed that some coal dust was carried into this testing solution. This may account for the low per cent removal of solids mentioned above.

Adsorption and Surface Area of Coal: Referring to TABLE XIX, page 78, and FIGURE 2, page 79, it may be observed that as the surface area of coal sample no. 1 (anthracite) was increased, by decreasing the size of the particles of coal, the solids removed from the viscose spinning bath solution increased. The -4 +8 mesh (U. S. Standard Screen) sample, having a total surface area of 2740 cm.<sup>2</sup> and a porosity of bed of 51.4%, removed 10.3 p.p.m. or 27.9% of the solids from the testing solution. As the surface area increased from 2,740 cm.<sup>2</sup> to 11,340 cm.<sup>2</sup> the removal of solids from the testing solution increased nearly three fold from 10.3 p.p.m. or 27.9% to 26.0 p.p.m. or 70.4%. The -30 +40 mesh sample, having a total surface area of 22,940 cm.<sup>2</sup> and a porosity of bed of 56.2%, removed 34.1 p.p.m. or 92.4% of the solids from the testing solution. The rate of increase of removal of solids decreased by 50% between surface areas of 11,340 and 22,940 cm.<sup>2</sup>. These results were not corrected for changes in porosity of the coal beds, nor was the decrease in pore space diameter, due to decrease in the size of the coal particles, accounted for. Therefore the increased adsorptive properties of the coal with increased surface

area was actually due to two factors, i. e. increased surface area and decreased pore space diameter. These two factors increased the possibility of more solids contacting the coal surface and thus making a more effective removal of solids possible. These results bear out the work of Hassler (20) and McMinn (21) who found that surface area influences adsorption to a considerable extent.

It might appear that, as the number of particles in a coal bed increased with decreasing size, the greater number of angular projections produced would contribute to increased adsorptive properties. This may or may not be true since in one instance during this investigation a -4 +8 mesh "Rounded" (see item 4, page 39) sample of coal no. 1 exhibited greater adsorptive properties than did the "unrounded" duplicate sample. In viewing photograph no. 10 and 11, page 84, it will be noted that the surface conditions of the two specimens are somewhat different, i.e. the treated specimen appears as rounded, dull particles indicating a roughened surface, while the untreated specimen shows angular bright surfaced particles indicating a smoother surface condition. To what extent the surface condition affected the adsorptive properties was not determined.

The testing beds produced from these two samples - "unrounded and rounded" - exhibited almost identical surface areas of 2,740 and 2,670 cm.<sup>2</sup>, respectively, but different porosities, 51.4% and 41.7% respectively. The increase in adsorptive power of the "rounded" specimen as compared to the "unrounded" specimen probably cannot be attributed

entirely to a decrease in porosity because of the condition of the surface produced by the grinding action in rounding the particles. However, as mentioned before in this discussion, if the size of the particles remain the same but the shape is varied as in the case now under discussion, the pore space diameter is a function of particle shape. This would explain the decrease in porosity as the particles approach a spherical shape. This decrease in porosity due to the elimination of the angular projections probably accounts for the greater adsorptive properties of the rounded specimen.

From the above discussion it may be postulated that variations in the adsorption of colloidal particles from spinning bath solution by different coals are due to physical factors only and not the chemical compositions of the coals. As the shape of the coal particles changes approaching the shape of a sphere, or the size of the coal particles become smaller, the voids between the particles become smaller. This in effect causes the action of the coal bed to be analagous to that of different weaves of a filter cloth in a filter press. The filtering surface in a filter press is a function of the size of the press. Likewise, in a coal bed the surface is a function of particle size and/or depth of bed. The capacity of a filter press is a function of the pressure, filtering area, volume of solids that can be held, and the ease with which the press may be cleaned (or regenerated). Likewise, the capacity of a coal bed is comparable. Even though in this work angularity of particles proved detrimental to solid removal from

adsorbate, it may be advantageous in increasing the ease of regeneration of the bed and may explain why so many industries have made a switch over from the long established spherical particled sand bed to the relatively new angular particled coal bed.

## B. Recommendations

General: It is recommended that all occluded air be eliminated from coal samples by boiling in water before tests are made in future investigations. It is believed that the presence of air in the coal bed will:

1. Increase the resistance to flow of the bed.
2. Cause channeling of the testing liquid.
3. Block some of the coal surface from the testing liquid.

Adsorption Tests on Coal Using Tannin Extract Solution: It is recommended that further investigations be conducted using tannin extract solution as the testing liquid. The coal tested should be in a continuous bed of three to four feet instead of two beds of ten inches each. It is thought that the increased depth of the bed would increase the separation of solids from solution. The flow of the testing solution should be down rather than up through the bed as it was in this investigation. It was noticed during the tests conducted that some solids had settled to the bottom of each adsorption chamber. If the flow had been in a downward direction, these settling solids would have provided a filtering medium to aid further in separation of the solid content.

Viscose Spinning Bath Solution: When viscose spinning bath solution is used as the testing liquid, attempts should be made to rigidly control all conditions in its preparation. Any one set of experiments should be run using one batch of testing solution. These

precautions would eliminate erroneous results due to variations in the testing liquid.

Adsorption and Surface Area: In this investigation correlation of surface area only with adsorptive properties of coal was not obtained, because pore space diameter was also a variable. It is recommended that tests be made varying surface area but eliminating pore space diameter as a variable. This may be accomplished by changing the quantity of coal used in each bed and holding the size of the coal and porosity of the beds constant. The velocity of the testing solution should be held constant also.

Adsorption and Pore Space Diameter: It is recommended that tests be conducted on one coal sample varying the porosity of the coal beds but keeping the quantity and size of coal tested constant. This will eliminate surface area as a variable and correlation of adsorptive properties with pore space diameter can be made. To maintain a constant velocity of testing solution through the pores the velocity should be varied directly with the porosity of the coal bed.

Adsorption and Conditions of Surface of Coal: Experiments should be conducted on different coal samples of the same size, calculating the quantity of coal to be used to give the same surface area in each test. The coal bed should not be tapped or packed in any way, thus giving the natural porosity of a bed of each particular fracture of coal. The quantity of solids adsorbed should be calculated to the same porosity of bed. The velocity of the testing liquid should

be varied directly as the porosity of the coal bed. The one variable would then be condition of surface of the coal. One factor in the condition of the surface of coal is roughness. An attempt to measure the roughness should be made by means of light reflection from the surface of the coal to a photoelectric cell or reflection of a beam of supersonic sound from the surface of the coal or both.

Capacity of Beds as a Function of Controlling Physical

Properties: The results of the tests recommended above will indicate the physical properties that control adsorption of colloidal particles by coal. It is recommended that experiments be carried out to correlate capacity of coal beds with these controlling physical properties. The capacity referred to should include the extent of solid removal from adsorbate, quantity of solids retained before regeneration is necessary, and the ease of regeneration of bed.

V. CONCLUSIONSA. Insoluble Solids in Tannin Extract Solution as Adsorbate

On the basis of results of the adsorption tests using the insoluble solids present in tannin extract solution produced by the Mead Corporation, Tannin Extract Division, Lynchburg, Virginia as the adsorbate and coals ranging in rank from anthracite to high volatile bituminous C and in mesh sizes of -8 #16 and -16 #40 as the adsorbents, using a tannin extract solution rate of coverage of 500 to 2600 cm.<sup>2</sup>/min. at  $24 \pm 4^{\circ}\text{C}$ , and a flow of testing solution in an upward direction through two  $10 \pm 1$  in. coal beds in series, the following conclusion was drawn:

1. The coals tested were non-effective to tannin extract solution clarification as evidenced by the fact that no insoluble solids were adsorbed by the coals.

B. Insoluble Solids in Viscose Spinning Bath Solution as Adsorbate

On the basis of the results of the adsorption tests using the insoluble solids present in freshly prepared viscose spinning bath solution as the adsorbate and coals ranging in rank from anthracite to high volatile bituminous C as the adsorbents, and using a flow of testing solution in a downward direction through  $10 \pm 1$  in. coal beds, at  $35 \pm 6^{\circ}\text{C}$ , the following conclusions were drawn:

1. With a flow rate of adsorbate through the coal bed of  $42 \pm 2$  ml./min. and a porosity of coal bed of  $54 \pm 3\%$ , as the size of anthracite was decreased from  $-4 +8$  to  $-30 +40$  mesh (U. S. Standard Screen), thus decreasing the pore space diameter and increasing the surface area in the coal bed from  $2,740 \text{ cm.}^2$  to  $22,940 \text{ cm.}^2$ , the amount of insoluble solids adsorbed was increased from 10.3 p.p.m. or 27.9% to 34.1 p.p.m. or 92.4% of insoluble solids in the testing solution.
2. Using an adsorbate rate of coverage of  $1750 \pm 100 \text{ cm.}^2/\text{min.}$  for all coals tested in the size range of  $-8 +16$  mesh (U. S. Standard Screen), the solids adsorbed per  $1000 \text{ cm.}^2$  of coal surface increased from 9.7% to 14.7% as the porosity of the coal beds decreased from 52.3% to 42.7%.
3. Using an adsorbate rate of coverage of  $1750 \pm 100 \text{ cm.}^2/\text{min.}$  for all coals tested in the size range of  $-8 +16$  mesh (U. S. Standard Screen), no correlation of proximate chemical analysis or relative roughness with adsorptive power of coals could be obtained.
4. The method of determining surface area used in this investigation is a satisfactory means of obtaining relative external surface area of coal particles of the sizes tested.

VI. SUMMARY

The use of anthracite as a filter medium is gaining popularity throughout industry as evidenced by the fact that in recent years 1100 filter plants have been installed for clarifying water and other chemicals (51). Since little information is contained in the literature as to why anthracite has become so popular replacing the much used sand filters, this investigation was undertaken in an effort to determine the reason, if any, for this popularity of use. A number of factors such as rank, chemical composition, size, shape, and surface condition of coal could possibly affect its adsorptive properties.

Tests were made in an effort to determine if any relationship existed between these factors and adsorptive properties. In the experiments tannin extract solution and viscose spinning bath solution were used as the adsorbate and coals varying in rank from anthracite to high volatile bituminous C, in sizes of -4 +8, -8 +10, -8 +16, -10 +16, -16 +20, -20 +30, and -30 +40, as the adsorbent. The tests were made with the coal in the form of the conventional filter bed (10 ± 1 in. depth) in 1½" diameter by 12" length Liebig condenser jackets with the direction of flow of the adsorbate, in the case of the tannin extract solution in an upward direction, at a rate of coverage of 500 to 2600 cm.<sup>2</sup>/min., at 24 ± 4°C, and the viscose spinning bath solution in a downward direction, at a velocity of 19 to 44 ml./min., at 35 ± 6°C.

The results of the experiments indicated that in the case of the tannin extract solution the coals were not effective to tannin extract solution clarification; while in the case of the viscose spinning bath solution the following conclusions were drawn:

1. As the size of anthracite was decreased from -4 +8 to -30 +40 mesh (U. S. Standard Screen), thus decreasing the pore space diameter and increasing the surface area in the coal bed from 2740 cm.<sup>2</sup>, to 22,940 cm.<sup>2</sup>, the amount of insoluble solids adsorbed was increased from 10.3 p.p.m. or 27.9% to 34.1 p.p.m. or 92.4% of insoluble solids in the testing solution.
2. In testing all coals in the size range of -8 +16 mesh (U. S. Standard Screen), the solids adsorbed per 1000 cm<sup>2</sup> of coal surface increased from 9.7% to 14.7% as the porosity of the coal beds decreased from 52.3% to 47.2%.
3. In testing all coals in the size range of -8 +16 mesh (U. S. Standard Screen), no correlation of proximate chemical analysis or relative roughness with adsorptive power could be obtained.
4. The method of determining surface area used in this investigation is a satisfactory means of obtaining relative external surface area of coal particles of the sizes tested.

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Addendum

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