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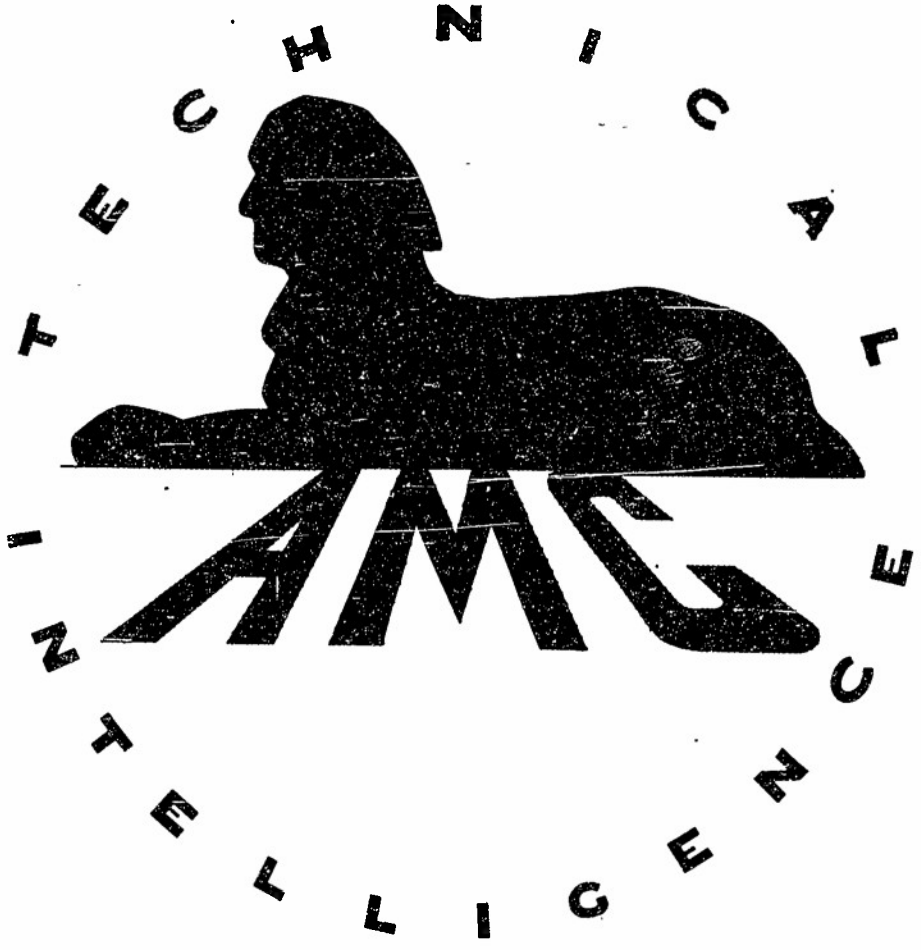

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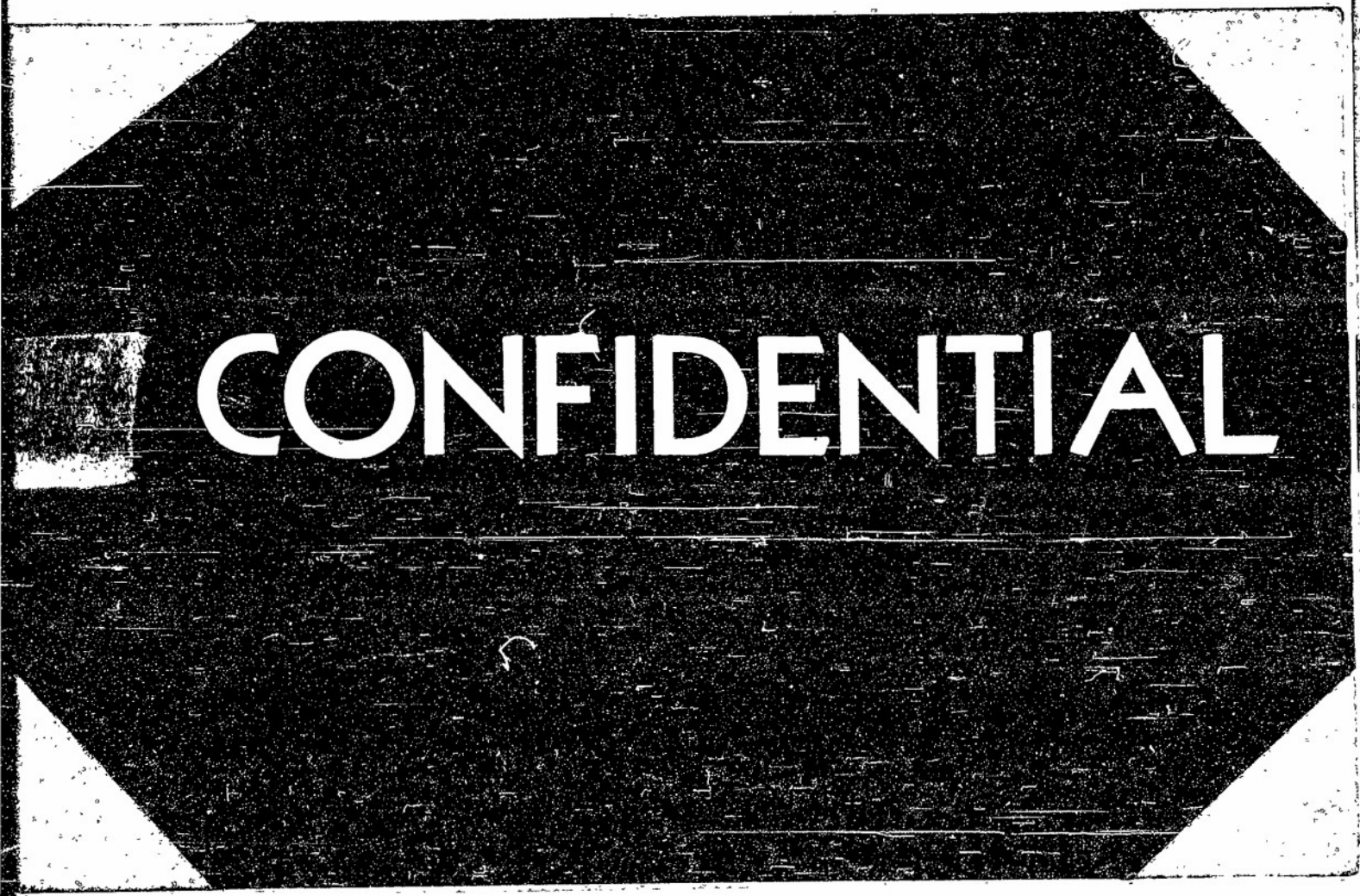
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Gas and Chemical Activation of Charcoal

32734

York, Robert, Jr.
Carnegie Institute of Technology
Office of Scientific Research and Development, NDRC, Div 10

(None)

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OSRD-5278

June '45 Confd'l U.S. Eng. 96 tables, graphs, drwg

The report summarizes useful information on charcoal activation and covers such subjects as the correlation of variables in the steam activation of char prepared from compressed wood and the mechanism of activation of wood by such chemicals as zinc chloride. An investigation as to the influence of different inorganic compounds showed that zinc chloride and phosphoric acid are the only effective activating agents among the common chemicals tried and that zinc chloride is by far the better of the two. Explanations of the effect of time of digestion on the reactivity of the final char are suggested.

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GAS AND CHEMICAL ACTIVATION OF CHARCOAL

to
July 31, 1942 - August 31, 1944
by
Robert York, Jr., et al

Report OSRD No. 5278
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GAS AND CHEMICAL ACTIVATION OF CHARCOAL

Service Directives: CWS-7, NL-B26, NL-B28, NL-B29, NL-B34
(The last four are now replaced by NS-338)

Endorsement (1) W. L. McCabe, Division Member to W. A. Noyes, Jr., Chief, Division 10.

Forwarding report and noting:

1. The report summarizes work on: I., the mechanism of activation; II. the correlation of variables in the steam activation of char prepared from compressed wood; and III. the mechanism of activation of wood by such chemicals as zinc chloride.

2. Part I correlates, by means of an hypothesis proposed by the present contract, data obtained by other OSRD projects or found in the published literature. The hypothesis is used as a basis for the discussion of the experimental work reported in Parts II and III.

3. Part II reports an experimental study of the affects of the operating variables important in steam activation on the relative internal and external weight losses, the development of base charcoal properties, and the rate of activation.

4. Part III summarizes experimental work on the activation of wood flour by chemicals. It is shown that zinc chloride and phosphoric acid are the only effective activating agents among the common chemicals tried, and that zinc chloride is by far the better of the two. Explanations of the effect of time of digestion on the reactivity of the final char are suggested. The results confirm and extend those reported in Report OSRD No. 432a (Contract 10-477, OEMsr-1200).

(2) from W. A. Noyes, Jr., Chief, Division 10 to Dr. Irvin Stewart, Executive Secretary of the National Defense Research Committee.

Forwarding report and noting:

"This report summarizes much useful information on charcoal activation. It is presented as a final summary of this phase of the work and should be of interest to those who continue to be interested in gas mask absorbents."

This is a final report under Contract 10-57, OEMsr-580 with Carnegie Institute of Technology.

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GAS AND CHEMICAL ACTIVATION OF CHARCOAL

by

Robert York, Jr., et al.

covering the period

July 31, 1942 - August 31, 1944

ABSTRACT

The presentation of results obtained in this program is divided into three sections. The theoretical discussion presented in the various sections is intended to apply not only to those results in particular but also to charcoal activation in general. The three parts are as follows:

Part I - The Activation Mechanism in Charcoal

The results of the current development of X-ray diffraction technique in the determination of physical structure of charcoal have been confirmed by nitrogen adsorption measurements. It is possible to account for all of the internal adsorptive area in charcoal in terms of graphite plane layers.

The activation process is definitely determined to be due to the opening of passageways to voids already existing in the primary char. It is not possible to produce more area in the activated particle than was available in the surfaces of the voids in the primary char particle. In the gas activation process there is a relationship between the external weight loss and the mesh size loss from which the product yield can be calculated from the apparent density and a correlation of size loss with volume loss.

Part II - Variables of the Charcoal Activation Process Studied in the Laboratory Jiggler

1. The laboratory jiggler described previously in OSRD Formal Report No. 956 and OSRD Formal Report No. 1521 has been used for an investigation of the variables in the jiggler process for compressed wood char made by the Carlisle process.

2. In this report the reproducibility and correlation of jiggler results are discussed.

Conclusions (for Part II):

1. In the jiggler activation of Carlisle wood charcoal between 80 and 30% yield the yield-time, apparent density (A.D.)-time, and A.D.-yield relationships are linear and may be represented by equations of the form $y = ax + b$.

2. The b values in these equations give some indication of the weight and volume relations during the inception of the activation reaction.

3. The calculated volume losses agree with the observed per cent size losses.

4. In the relations $dm = \rho\ dV + V\ d\rho$, where dm is the mass change, ρ the density, and V the volume, $\rho\ dV$ is the external and $V\ d\rho$ the internal burning. The data show that the ratio of external to internal burning is approximately unity in the region between 80 and 30% yield.

5. The Arrhenius constant for the increase of reaction velocity with temperature between 1450 and 1650°F. is 47,500 cal./mol and is in agreement with measurements reported in the literature on Acheson graphite.

6. The maximum PS life developed has been correlated with activating gas velocity, gasification rate, temperature of activation, and time of activation.

7. At the high gasification rates which are obtained in the jiggler process, the effects associated with the gaseous environment are important in determining the pore size distribution of the activated product from Carlisle process primary char.

8. In the gas activation of 12-30 US mesh particles the high gas velocities in the jiggler process reduce the diffusion resistance and promote the formation of adsorptive surface within the particle.

Part III - Laboratory Investigations on Chemical Activation

An investigation of all the recent data in the literature on the influence of different inorganic compounds as activating agents supplemented by runs in the laboratory has shown that zinc chloride is by far the most suitable activating agent.

1. In the dehydration mixing of wood flour-water-zinc chloride mixtures the time-temperature curve of the mix goes through a peak at times corresponding to large changes inthephysical appearance of the mix. The most probable possibility is that the temperature drop is connected with the colloidal properties of the sawdust.

2. In studying the chemical activation of wood flour a laboratory sized break mixer is being used for the wood flour impregnation operation preliminary to carbonization. A number of runs have been made to ascertain the degree of control necessary for reproducibility of briquetting characteristics with time of dehydration.

3. The physical appearance of the mixture during the impregnation operation provides sufficient significant information to enable the operator to predict mechanical characteristics of the briquet.

4. In the dehydration of zinc chloride-wood flour-water mixtures the ratio of the densities before, during, and after compression to 4000 lbs./in.^{2} are shown to be related to the mechanical characteristics of the briquet.

5. A study of the variation in density of the briquet under 4000 lbs./in.^{2} with time of mixing and percentage of zinc chloride would be necessary to determine the absolute amount of swelling of the wood.

6. A study of the reactivity of the mix to oxygen in various stages of dehydration has been made. This investigation shows that increasing the concentration of the zinc chloride in the mix and increasing the time of mixing decreases the amount of volatile matter in the mix and raises the ignition temperature.

RECOMMENDATIONS

None.

INTRODUCTION

Previous results on this contract have been reported in the following reports of Division 10, NDRC:

OSRD Formal Report No. 956 (July 31, 1942): "Activation of Gas Charcoal by a New Jiggler Process."

OSRD Formal Report No. 1521 (April 1, 1943): "Further Developments of a Laboratory Jiggler for Activating Gas Charcoal, and Tentative Results on Gasification Rate Studies."

OSRD Formal Report No. 4011 (August 12, 1944): "Activation of Charcoal in a Boiling Bed Furnace."

and Informal Progress Reports:

10.4-14 (April 1, 1943): "Weight and Size Losses During Laboratory Activation of PCI Char."

10.4-18 (May 1, 1943): "Activation of Charcoal in a Boiling-Bed Furnace."

10.4-20 (April 1, 1943): Same as OSRD No. 1521

10.4-30 (August 1, 1943): "Composition of Gases Evolved During Activation."

10.5-1 (September 1, 1943): "Activation of Carbonized Presto-Logs."

10.5-6 (November 12, 1943): "Activation of Charcoal in a Boiling-Bed Furnace II."

10.5-7 (December 1, 1943): "Effect of the Activation Process on the Nitrogen Adsorption and Whetlerite Properties of PCI and Carlisle Charcoals."

10.5-9 (December 15, 1943): "Activation of Carbonized Peach Pits and Black Walnut Shells in PCI Retorts."

10.5-14 (February 1, 1944): "An Hypothesis of the Activation Mechanism in Charcoal."

10.5-18 (March 1, 1944): "A Modified Boiling-Bed Furnace for Charcoal Activation by Steam III."

and the Monthly Summary Reports of Sections 10.1 and 10.5.

The program on this contract on the activation of charcoal has been on the application of the fundamental research to the large-scale activation of charcoal and to the improvement of charcoal quality through the activation process.

We are indebted to Dr. P. H. Emmett of the Johns Hopkins University and to Dr. T. F. Young of the University of Chicago, both Official Investigators on NDRC, for their able assistance and helpful advice during the progress of the work. Any contributions of our project to the knowledge of the activation mechanism are largely due to the advances which their projects have made.

Part I. Activation Mechanism in Charcoal

(Tables and Figures are at end of Part I.)

One of the requisites of a gas mask charcoal is a large internal surface area for gas adsorption. In the activation of charcoal, by oxidation of the primary char in a gaseous atmosphere, the internal surface area is produced by opening passages to small voids which are present in all forms of carbonized materials.

These voids are produced in the carbonization process. As carbonaceous materials are subjected to progressively higher temperatures, they tend to approach natural graphite as a limiting state of aggregation. In the process, elements such as oxygen, hydrogen, and nitrogen, which were present in the raw material, are volatilized. At a temperature of 1000°C. substantially all of the foreign elements present, except ashes, have been eliminated; and the carbon present is chemically in a graphitic condition. X-ray analysis has shown, however, that the physical structure is highly disordered. The individual graphite plane layers, instead of being arranged in the graphite lattice, are collected into small packets.[1] The voids in charcoal result from the crevices between the packets as they compose the gross particle.

Figure 1[3] shows the crystallattice of graphite according to Hofmann[2]. Figure 2 shows the arrangement of the packets of Diameter A and Height C.

The distance between plane layers in the packets is slightly larger than that for true crystalline graphite (3.35Å), due to the random orientation of one plane layer network with respect to the adjacent plane layer

1- See references in Table I.
2- Ergebnis. exakt. Naturwiss. 18, 229 (1939).
3- Figures and tables for each part are collected at the end of the part.

network. The interplanar distance in graphite represents the approximate distance of closest approach, due to physical attraction, of any atom center to the graphite plane. In charcoal the planes do not approach quite as closely because they are not oriented in positions for maximum attraction. X-ray measurements have furnished this picture of the packet as the elementary aggregate in charcoal structure. The packets are randomly arranged and interlocked together to form the gross charcoal particle. The space between the packets constitutes the pore space. It should be emphasized that this picture of the charcoal structure is based on information from X-ray and electron diffraction measurements on samples which did not have gases physically adsorbed on them. That physical adsorption of gases could take place between plane layers of less than 4 Å separation is an impossibility. If interplanar penetration did occur to any large extent during physical adsorption, the packets would have to expand accordionwise. A certain amount of this might occur without a noticeable increase of the gross volume of the particle. The highly interlocked structure of the charcoal would tend to retard such an expansion, however. No X-ray measurements have been made yet to show whether plane layers do increase their separation during the adsorption process.

Table I[1] shows values obtained for the lattice axes and packet dimensions by various investigators. Table II shows the areas of activated charcoals determined from nitrogen adsorption by Emmett compared with the areas calculated for the packets measured by Clark and Johnstone. These measurements by Emmett are the first area determinations to confirm the X-ray predicted area by an agreement within 100%.

This calculation is only an example of how X-ray data can be treated. The significant fact is that the X-ray calculated areas are proportional to

1 - Figures and Tables for each part are collected at the end of the part.

the nitrogen areas over a range of 1000 sq.m./gm. Considerable refinement is necessary before the absolute agreement can be considered more than fortuitous. However, it is important to note that the area of the packets is sufficient to account for the necessary adsorption area.

It has been stated in the literature[3] that one gram of black carbon is limited in its maximum area to that obtained for both sides of a single graphite plane layer of one gram weight. This figure is 2640 sq.m. This, however, is for a continuous plane. When the plane is broken up into plates of 20 Å diameter the additional area on the edges increases the total to 4410 sq.m.

The presence of voids may be confirmed by helium density measurements. The X-ray measurements have already determined that the density of the carbon phase is about 2.0 g./cc. Any large departure of the density by helium displacement below this figure must be due to closed voids that are unavailable to helium penetration. The fact that the helium density increases during activation has frequently been cited as evidence that the activation process opens up passageways to these voids.

Activation Process

A brief summary of the mechanism of gas activation is as follows: Starting with the primary char which has been heated to at least 1000°F. in the carbonization process, a char has already been formed which has a considerable capillary system interwoven between aggregations of carbon about 1000 Å in diameter or larger. These aggregations are composed of the graphite plane layer packets interlocked together. The oxidizing gases attack the aggregates in all directions from the original capillary system. Oxidation

3- Berl, Andress, Reinhardt, and Herbert, Z. f. Physik Chem., A158, 273 (1932).

proceeds most rapidly at the plane layer edges where a few atoms of hydrogen and oxygen remain, and at centers of ash concentration where the ash constituents catalyze the attack on the graphite plane layers. The other regions of the graphite plane layers are more stable and are unaffected by the oxidation reactions. The penetration of the aggregates follows the crevices between packets and rapidly extends the capillary system throughout all the void spaces. As the capillary system is being extended, the oxidation also begins to attack the walls of the capillaries which are alreadyformed, burning away carbon and increasing the diameter of the capillaries. This oxidation does not contribute any new area; in fact, it tends to decrease the area.

It should be emphasized that oxidation does not create any capillaries in the solid phase. All attempts recorded in the literature[4] to activate macrocrystalline graphite have only resulted in reducing the surface area. All attempts to make capillaries in macro-crystalline graphite by oxidation have been unsuccessful. This is an entirely different idea, however, from that of penetrating voids that are already present.

In the initial stages of the activation this loss of area due to capillary enlargement is not serious. The rate of loss of area increases, however, and eventually passes the rate of increase of area (per cc. of charcoal). Further activation beyond this point decreases the adsorptive power of the charcoal and the charcoal is characterized by an excessively low apparent density.

Simultaneously with the oxidation effect there is a temperature effect in the activation process. If the char was carbonized at 1000°F. and is activated at 1800°F., the increase in temperature causes considerable shrinkage. This increases the grams of carbon per cc. and also the adsorptive capacity per cc.

4- Ruff and Backe, Kolloid Z, 38, 59 (1926).
Bach and Levitin, ibid. 64, 22 (1933).

Figure 3 is an illustration of the way in which these different effects might be manifested in the properties of the activated char. (This figure is taken from Informal Progress Report 10.5-9, "Activation of Carbonized Peach Pits and Black Walnut Shells in PCI Retorts.") At low activation temperatures the pore-widening oxidation takes place predominantly giving large apparent density losses with small area increases. At 1700°F. the optimum combination of penetrating void spaces at a high rate and thermal shrinkage of the charcoal takes place, giving larger area and tending to counteract apparent density loss and resulting in the optimum PS adsorptive characteristics.

In addition to the oxidation of material out of the inside of the particles, a certain amount of charcoal is burned away from the circumference of the particle resulting in a total loss of material. From data taken on the activation of carbonized black walnut shells reported in NDRC Informal Progress Report 10.5-9 and a correlation of the external weight loss of particles with size loss a calculation of the yield has been made. The yield values in Figure 3 were obtained in this manner. The following discussion explains how this calculation was made using the black walnut data for purposes of example. Let the following nomenclature be used:

m = mass, grams

y = % yield, gram basis

ρ = apparent density, grams/cc

v_o = apparent specific volume of charcoal as charged, cc/gram

v = apparent specific volume of charcoal, cc/gram

ΔS = % size loss, wt. % of material originally held on given screen now passing that screen. All sizes U.S. mesh.

External Weight Loss

The equation for the fractional yield at time t for a one gram charge is:

$$y = 1 + \Delta\rho \Big]_o^t v_o + \Delta v \Big]_o^t \rho_t \qquad (1)$$

This relation assumes that the weight loss occurs by the successive steps of (a) the original bulk volume of charge undergoes an A.D. loss from ρ_o to ρ_t (b) the original bulk volume of charge now at ρ_t undergoes a volume loss from V_o to V_t. The path by which the reduction of particle size and weight is followed is immaterial.

In equation (1) the only unknown term is $\Delta v \Big]_o^t$ which is equal to

$$\left[\frac{(\Delta v)}{(v_o)\ (\Delta S)}\right]\left[(\Delta S)(v_o)\right] \qquad (2)$$

The first bracket in expression (2) is determined as a single factor from experimental data obtained on these shells in laboratory activation where the charge is 100% 12-16 mesh and whose volume is m/ρ. A correlation of the % loss of volume with the % loss of 12-16 mesh for this particular sample shows that the volume loss is 0.7 x the size loss. In other words, if all of the original charge of 12-16 mesh has been reduced to below 16 mesh the volume of this product is only 0.3 times the original volume. This is confirmed by a calculation of the loss of volume necessary to reduce a frustum of a cone having bases whose diameters correspond to 12 and 16 mesh, to a frustum of a cone whose largest base corresponds to 16 mesh, by reducing all dimensions the same amount. This gives a ratio of 0.8 in substantial agreement with the experimentally observed ratio of 0.7.

In order to employ the % size loss of Term (2) in equation (1) the per cent size loss can be expressed in terms of apparent density loss, by the

equation:

$$I - \rho_t = c\,\Delta S \qquad (3)$$

where I is the intercept at 100% 12-16 mesh and c is the slope of the curve of ρ versus mesh size. This slope has been determined from the calculated % loss of 12-16[5] and ρ of 12-16 particles obtained in the 4 qt. samples taken from the PCI retorts during activation. The use of this equation for the volume loss would be entirely misleading in the range where devolatilization is taking place, i.e. the early stages of the run, since during shrinkage ρ is increasing while the volume decreases. However, this equation is theoretically exact in the range where the volume loss is linear function of ρ and gives exactly the overall volume loss since charging, as will be shown below.

The initial volume per gram v_o in equation (2) is equal to 1/A where A is the arbitrary ρ_o since that represents a density at which the commencement of external burning has arbitrarily been set. It will be shown later that the final equation for the yield depends only on this arbitrary value for ρ_o which is assigned to the sample as charged, consistent with a balance in the total process of manufacture.

Term (2) thus becomes:

$$\frac{(0.7)(I - \rho_t)}{c \qquad A} \qquad (4)$$

3. The procedure followed for getting samples was to take a given volume of charcoal from the retort for each sample. A screen analysis by weight of this constant volume of sample then is equal to the screen analysis by volume providing ρ of the fractions classified is the same. A corollary of size theory is that packed spheres always have the same percentage of 5 - This calculation will be discussed in Part 3 below.

void spaces. Measurements of ρ of 12-16 and 16-20 fractions show that variations of ρ are irregular from one size to another. It will be assumed, therefore, that the 12-16 ρ holds for 16-20 and 20-30. The per cent fines is negligible as far as variation in ρ is concerned. By using the size analysis of the original retort charge the per cent 12-16 mesh of subsequent retort samples may be placed on a basis of 100% 12-16 mesh charged, corresponding to the laboratory data used for correlating size loss with volume loss. One further correction must be made to the per cent 12-16 mesh obtained for retort samples. This is necessitated bythe fact that in withdrawing a constant volume of sample,the volume loss from the volume decrease of 12-16 mesh particles is filled up by particles which were originally over 16 and not included in the original volume basis of 100% 12-16. In other words, some extraneous particles came in which were not made by activating down the particles taken on the original basis. This makes the observed per cent of 12-16 obtained in the analysis smaller than it should be on the original basis.

If x is the actual fraction of 12-16 and R is the observed fraction of 12-16 and the volume loss is 0.7 times size loss, then

$$x = R\left[1 + 0.7(1 - x)\right] \qquad (5)$$

A plot of $x = f(R)$ was made and the correct x values read from this plot and used as described in Part 2.

4. Using the term (4), the yield may now be evaluated by equation (1). Substituting: where A is arbitrary original density

$$y_{12\text{-}16} = 1 - \frac{(A - \rho t)}{A} - \frac{(0.7)(I - \rho t)(\rho t)}{c \quad A} \qquad (6)$$

$$y_{12\text{-}16} = \frac{A - A + \rho t - 0.7/c\, I\rho t + \rho t^2}{A} \qquad (7)$$

$$y_{12\text{-}16} = \frac{\rho t\left[1 - 0.7/c(I - \rho t)\right]}{A} \qquad (8)$$

The yield obtained by this equation will be the yield of 12-16 mesh particles in the charge. The loss of weight by the smaller sizes will probably parallel this. The accuracy of the equation is ±5% and is limited by the term $0.7/c\ (I - \rho_t)$. This equation may be used to calculate the yield of spot samples taken from large scale activating processes using the correlation of size loss with volume loss determined empirically in a laboratory model of such a process.

TABLE I

LATTICE AXES AND PACKET DIMENSIONS OF BLACK CARBONS

Sample	C-C Distance(A°)	Interplanar Distance(A°)	Packet Dimensions(A°) Diameter A	Height C	Calc. Area m^2/gr.	Investigators
Ceylon Graphite	1.42	3.35		200	-	Hofmann and Wilm(1)
AKT IV Charcoal	-	3.7	18	8	2300	" " "
Supranorite Charcoal	-	3.7	24	7	2200	" " "
Carboraffin Charcoal	-	3.7	19	7	2400	" " "
Degoa Charcoal	-	3.6	17	8	2250	" " "
Sugar Carbon	-	3.6	18	7	2270	" " "
Cellulose C(1000°C.)	-	-	20	10	-	Blayden, Riley and Taylor(2)
Lignin C(1000°C.)	-	-	20	10		Blayden, Riley and Taylor(2)
Cabot carbon black	1.42	3.4	20	13	-	Biscoe and Warren (3)
Methane carbon black	1.42	3.6	20	8	-	White and Gerzer(4)
USN-1 Coconut	-	3.82	36	10	-	Johnstone and Clark(5)
PCI 35	-	3.70	38	9	-	" " "
CWSN-55	-	3.74	24	9	-	" " "(6)
CWSN-111A	-	3.76	25	29	-	" " "

(1) - Z. Elektrochem. 42, 504 (1936).
(2) - J. Am. Chem. Soc. 62, 180 (1940).
(3) - J. Applied Physics 13, 364 (1942).
(4) - J. Chem. Phys. 9, 942(1941)
(5) - OSRD Formal Report No. 1143
(6) - Unpublished data.

TABLE II

COMPARISON OF SURFACE AREAS CALCULATED FROM X-RAY DATA WITH AREA MEASURED BY N_2 ADSORPTION AT -195°C.

Data from Johnstone and Clark, OSRD Formal Report No.1143

Sample	Interplanar Distance(A°)	Packet Dimensions(A°) Diameter A	Height C	Calc.Area $m^2/gm.$	Area by N_2 ads. $m^2/gm.$
CWSN 31	3.7	20	10	1970	2040
32	3.7	34	10	1560	2015
33	3.8	45	10	1460	1770
34	3.8	62	9	1370	1670
C1	3.8	38	11	1470	1580
19	3.7	32	11	1530	1710
CWSE 1	3.9	28	11	1640	1550
CWSC 11	3.8	54	11	1230	1610
CWSC 542	3.5	52	12	1150	1180
Lot B-255	3.8	30	10	1640	1679

CWSN = $EnCl_2$ - wood flour carbon.

CWSC = Compressed wood char.

CWSE = Coconut shells.

FIG. 1 Crystal Structure of Graphite

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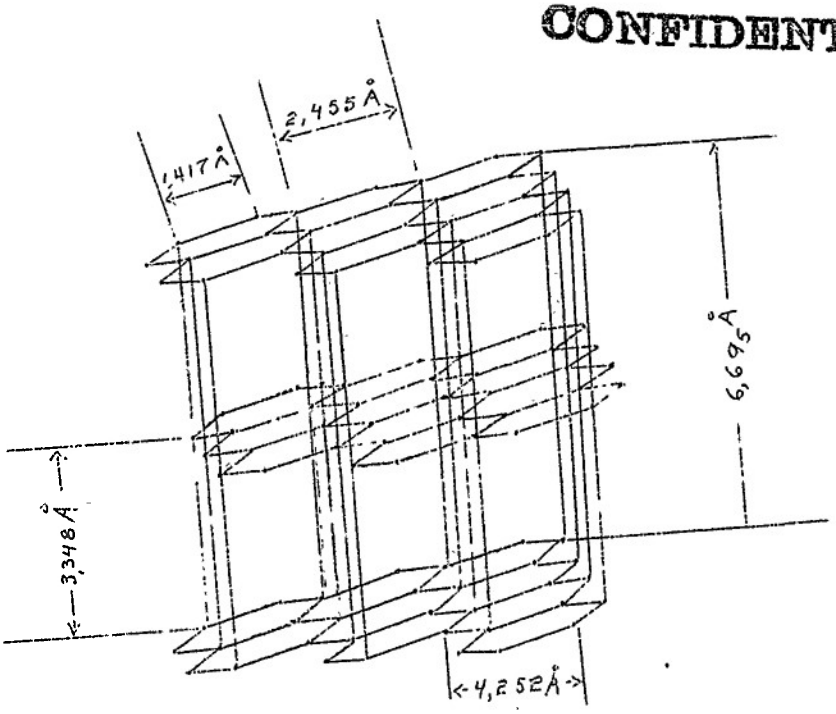

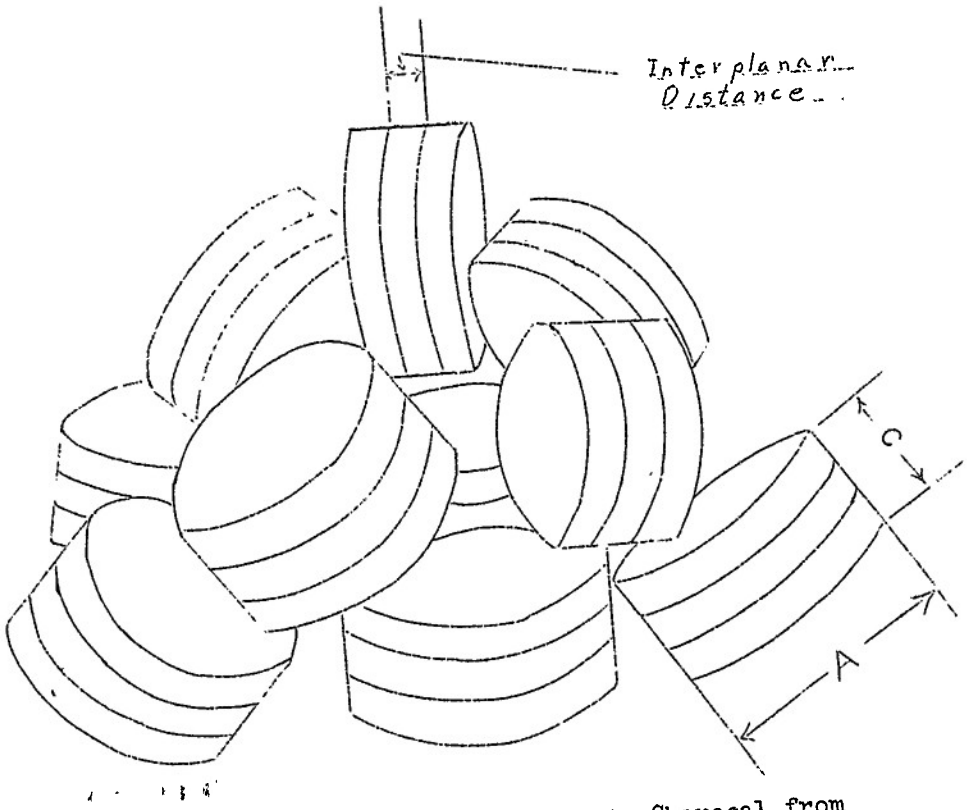

FIG. 2 Picturization of Packet Structure in Charcoal from X-ray Measurements.

Figure 3

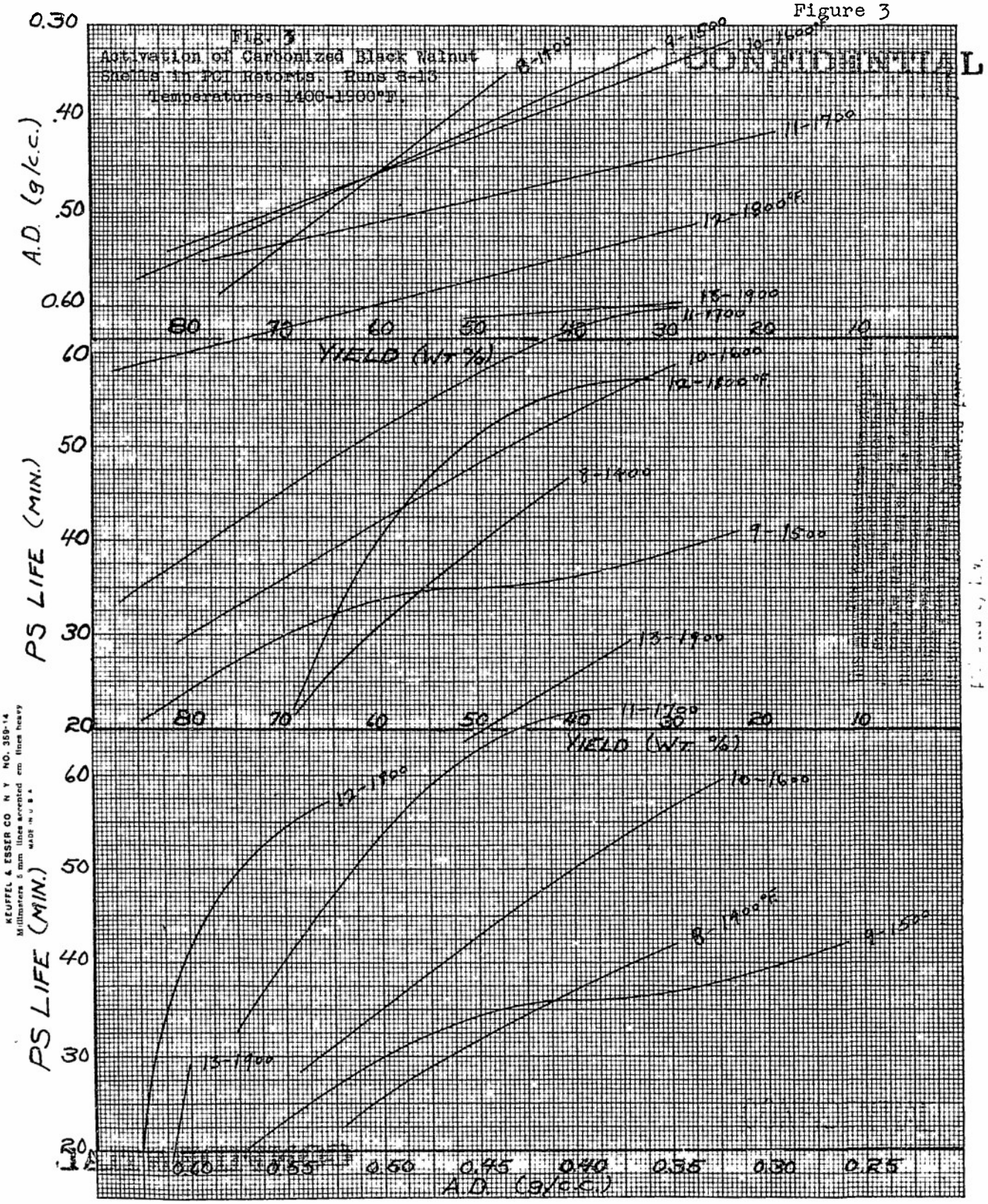
Fig. 3
Activation of Carbonized Black Walnut Shells in PCT Retorts. Runs 8-15
Temperatures 1400-1900°F.
A.D. (g/c.c.)
PS LIFE (MIN.)
YIELD (WT %)
0.30
.40
.50
0.60
80
70
60
50
40
30
20
10
8-1400°
9-1500°
10-1600°
11-1700°
12-1800°F.
13-1900
14-1900
15-1900
KEUFFEL & ESSER CO N Y NO. 359-14
Millimeters 5 mm lines accented cm lines heavy
MADE IN U S A

Part II. Variables of Charcoal Activation Process as studied in the Laboratory Jiggler

(Tables and Figures are at end of Part II)

INTRODUCTION

Activated charcoal may be produced by passing oxidizing gases such as oxygen, carbon dioxide and steam over the primary char at elevated temperatures. As the entering gases meet the charcoal, the resultant burning forms oxidation products such as CO, CO_2, and H_2 which are given off into the gas stream. As this gas stream proceeds from one layer of charcoal to the next the concentration of reaction products progressively increases. Each oxidizing gas has a specific activation effect on charcoal depending upon the temperature and ash constituent of the charcoal. Factors connected with diffusion in the gas phase must be considered as determining the gas composition at the solid gas interface. The activation temperature of the charcoal is important in determining not only the characteristics of the oxidation reaction but also the constitution of the finished charcoal, unless the primary char has been previously formed at a higher temperature.

Due to this effect of variation in the composition of the activating gases, commercial processes employ different methods of stirring to insure uniformity of oxidation history. The requisite oxidation conditions for the satisfactory activation of a primary char are: (1) that a certain amount of carbon be removed from the interior of the particle by (2) a combination of activating gases that will produce the desired pore structure. This second condition usually fixes the rate of activation and determines the time necessary to accomplish the first.

For charcoals which must be activated at a low rate, mechanical stirring is most desirable. Some charcoals, however, can be activated at a high rate of gasification and for these chars the jiggler stirring process is applicable. In the jiggler process the charcoal is stirred by holding the charcoal in a vertical activating retort and passing the activating gases from the bottom of the retort at a velocity high enough to lift and circulate the charcoal. A detailed photographic study of the fluid flow mechanics of the jiggler process has been reported in our Formal Report OSRD No. 1521. This study showed that there were two types of fluid action possible in the jiggler: one at velocities above 10 ft./sec. with light loadings, less than 6 lbs./cu.ft. of 8-30 mesh, the other at velocities below 10 ft./sec. and heavier loadings. The first condition of agitation looks like a snowstorm of charcoal particles and is called "jiggling." The second condition is entirely different in appearance. Here the charcoal lies in a solid bed and is agitated by slugs or bubbles of gas rising through the bed in a manner similar to boiling water, and has been termed "Boiling Bed." The development of this process is described in OSRD Formal Report No. 4011 (Aug. 12, 1944) "Activation of Charcoal in a Boiling Bed Furnace."

The laboratory jiggler for the activation of gas charcoal was first reported by Proposal 10.5-57 (B-57) in OSRD Report No. 956 of July 31, 1942. That report describes a retort for the activation of charcoal in which the charcoal is lifted and circulated in a stream of oxidizing gases flowing upward at a relatively high velocity. The results of that work showed that primary char from widely varied raw materials could be activated rapidly in pure steam to give a product whose PS life was a function of the amount of material gasified, reaching a characteristic maximum value for each char.

The photographic study OSRD No. 1521 of the fluid flow characteristics existing in the laboratory jiggler was made to provide the information necessary to improve the reproducibility of the temperature measurements. The report at hand gives the results of several hundred activations in a study of the variables of the activation process in the laboratory jiggler.

Operating Procedure.

The procedure by which the activation data in Table I were obtained for this report is as follows: The primary char to be charged to the jiggler was dried at 150°C. A weighed sample of this char (12-16 U.S. mesh) was loaded at the separator cone with commercial cylinder nitrogen flowing through the jiggler. The temperature was determined by a chromel-alumel thermocouple in the charcoal bed. When this temperature reached the desired activation temperature, the nitrogen flow was stopped and the steam flow started. At this time the activation period was considered to have started. The heating period usually took about four minutes. During the activation run the temperature of the thermocouple was maintained constant. Upon the elapse of the predetermined activation time the steam was shut off and the charge blown out into a receiver with a stream of nitrogen. The product was weighed, screened to 12-16 U.S. mesh, reweighed for yield of 12-16, and dried before testing by the accelerated PS tube test (10 cm. bed, 47 mg./l., 1 liter/$(\text{c.m.})^2(\text{min.})$.

In all of the runs a 50 gram charge was used with the exception of J 679-82, 696-97, and 711-14 in which the charge was 75 grams.

In all of the runs the charcoal was held in the jiggler tube by the lower screen at the bottom of the tube. In the runs marked SCREEN the upward travel of the charcoal was restricted by another screen placed in the upper part of the tube (above the charcoal) after the charcoal was charged.

By this arrangement the charcoal was allowed to circulate only between the two screens about six inches apart. This did not give a fixed bed (with reference to fluid characteristics). The rate of steam input is given in Table I, but owing to the fact that the gas velocity was so high most of the meshes in the upper screen were quite forcibly plugged by charcoal particles, and a considerable amount of steam probably channeled around the sides of the charge. As far as most of the charcoal particles were concerned, the relative gas velocity was effectively zero.

Reproducibility of Jiggler Measurements.

In measuring the time of activation, variations in handling procedure do not cause an error of more than 1% in the time. The inaccuracy of our weighing and PS life testing procedure for a single determination of yield, apparent density (A.D.) and PS life is less than 3%.

The observed spread of data shown in the odd numbered Figures 1-25, "Activation of Charcoal," shows the approximate magnitude of the standard deviations to be for the yield ± 2.5%, for the A.D. ± 12.5%, and for PS ± 5%. The temperature reproducibility is demonstrated in Figure 23 showing a series of 75 runs between J 913 and J 1023 in which 38 different production samples were activated at 1400°F. to give identical gasification rates.

The question of reproducibility of fluid flow characteristics was discussed in OSRD Formal Report No. 1521. Recalibration of boiler heater, replacement of burned out heater and new calibration at J 1358, frequent inspection of jiggler for leaks, and the replacement of the screen to maintain constant position of the bed relative to the thermocouple insured the continued reproducibility of the mechanical action of the jiggler.

Recycle.

One feature of the mechanical action, the full importance of which was not

realized when described in the last formal report (OSRD No. 1521), is the recycle (or shuttling) between different temperatures. In the gas-fired furnace system which heats the jiggler the jiggler tube is maintained at its highest temperature at the bottom and falls to about 200°F. at the separator cone. Charcoal is heated to its highest temperature at the screen and cools progressively as it is blown up the tube. The charcoal which is blown into the cone at high gas velocities may fall to as low a temperature as 500°F. The comparison of gasification rates at different gas velocities for the same temperature at the screen shows that the temperature history effect on the burning of carbon is slight; i.e., other kinetic resistances (gas films) in carbon gasification are so great that some cyclical cooling does not decrease burning. This indicates that perhaps the maximum temperature during the cyclical process is of the most significance. In the extreme case where carbon cools in the cone in an atmosphere of more than 80% water, water vapor is drawn into the pores. As the particles fall to the hot tube some of this water adsorbed may react before it can escape resulting in oxidation of as much carbon as if the particles had stayed hot all the time. However, the magnitude of the effect at a recycle frequency of 10 per minute and a temperature variation of 1000°F. would not account for more than 5% of steam reacting with the carbon. It has not been proven as yet whether or not recycle has any effect on the quality of the product.

Analyses of composition of the activating gas atmosphere in the jiggler were reported in Division 10 Informal Report 10.4-30 of August 1, 1943. The analyses were those of the effluent gas where the influent gas was steam, entering with a velocity of 3 feet per second through a charcoal charge of 50 grams initially. These data show that the charcoal particles are in an atmosphere of more than 80% steam as calculated by the equation:

$$\% \text{ steam decomposed} \propto \frac{(\% \text{ charcoal decomposed by steam/hr.})}{(\text{steam input rate})} \times \frac{(\text{mole } \% H_2 \text{ in leaving gas})}{(\text{mole } \% CO + \text{mole } \% CO_2)} \times \frac{(\text{mol.wt.} H_2O)}{(\text{at.wt. C})}$$

This steam concentration is high enough so that, considering the effect of the composition of the activating gases on the activation process as a first order effect, variations in composition during a series of runs are only of second order magnitude.

For the screen runs discussed above, the composition of the gaseous environment is probably quite different from that encountered in jiggling and this should be borne in mind in comparing results.

Correlation of Results

With these limits of reproducibility in mind, a discussion of the technique or correlating the results is an integral part of the activation method.

The following variables are to be considered for correlating activation data:

1. Type of char activated.
2. Time of activation period.
3. Velocity of activating gas.
4. Charcoal temperature (maximum).

The properties to be observed are:

1. Weight of 12-16 U.S. mesh charged.
2. Weight % yield.
3. Weight % of 12-16 U.S. mesh in yield
4. Apparent density of 12-16 mesh product.
5. PS life of 12-16 U.S. mesh product.

Since the time is most accurately measured, the observed properties may well be

plotted as a function of time and smoothed values read from the curves. This reduces the probable error. Then the average values of the properties can be correlated. The correlations of the different properites are discussed in detail below:

Yield-Time.

Figure 23 shows 75 runs between J 913 and 1023. The yield-time relationship is, as near as can be practically determined, linear. As a result of this evidence it has been assumed that this relationship held for all runs herein reported, and the reader may draw his own conclusions as to whether the deviations of the points in odd-numbered Figures 1-25 justify this assumption.

A.D.-Time.

The observed points for the A.D. versus time for the 75 runs mentioned above did not so conclusively give a linear relationship. In view of the large spread of the points as mentioned in the discussion of reproducibility, it is believed that a linear relationship is as good a fit as any other. Furthermore, there seems to be no theoretical basis for predicting a more complex function. It developed later that other data have confirmed the assumption of linearity to some extent. Hinshelwood in his report[7] has also assumed thatthe straight curve for A.D. versus time holds for coal chars activated by a different process than those of this report. Theoretical considerations which will be discussed later also show that no simple assumptions will give a function which will differ enough from a straight line in the region measured to make any significant difference.

7- "The process of charcoal activation in relation to the properties of the product." Oxford Physical Chemistry Laboratory, 1942.

PS Life-Time

The PS life versus time is of course hardly ever a straight line and the best that can be done is to follow the experimental points to the best ability of drawing a curve.

Graphing Procedure

In correlating the data the procedure used was as follows: A graph for three properties of yield, A.D., and PS life versus time was drawn for each continuous series of activation runs in which all conditions except time were constant. This resulted in over 100 separate graphs on which was only one set of points. For example, sample X72 is run for 1.0, 0.50, 0.75, and 1.25 hours, at a temperature of 1500°F., and a steam velocity of 3.5 ft./sec. resulting in four products J 909, 910, 911, and 912 in the order of times given above. (That is, an increasing order of time was not always followed.) This gives four values of yield, A.D., and PS each of which are then plotted against time. The best curves are drawn through the points and no further points are plotted on this sheet. The figures in this report were obtained by superimposing these separate graphs representing the variables to be correlated and transferring the best average line by inspection to the master graph for the report. The data points are also plotted as a check for the master curve. But in case the reader seems to detect unnecessarily large deviations, it should be emphasized that the curves in the figures bear only second-order relations to the points, these having come directly from a collection of individual curves. This procedure results in a truer average slope.

From the master curves of yield, A.D. and PS versus time values for the curves of A.D. versus yield, PS versus yield, and PS versus A.D. were taken and plotted giving the smooth curves of the even-numbered figures 2-26. The experimental data were again plotted to check the curves. The fit of these

points which are now three times removed from the curves is in general acceptable.

The value of this procedure will be appreciated when it is realized how erratic individual points might be on an A.D.-yield relationship making it a very difficult task to pass any kind of average curve through such a plot having only a few points in a series of runs. In the case of a large number of points such as the 75 runs previously referred to, the time parameter curve does not fit the A.D.-yield plotted points exactly. By looking at Figure 24 the reader may get some idea as to how accurate this method is likely to be in predicting the proper slopes on the A.D. versus yield, PS versus yield, and PS versus A.D. bases.

Size Loss Versus Yield.

The per cent of 12-16 mesh in the yield is plotted against per cent yield. The accuracy and the significance of the size losses are not sufficient to warrant such extended treatment as that described for the PS, A.D., yield time data.

Conclusions from Experimental Data.

The experimental data on which this report is based are presented in Table I. For a graphic picture of the relations found the data were plotted in Figures 1 through 35, according to the method described in the previous section. The discussion below relates to the conclusions drawn from these figures.

Weight and Volume Losses.

Between 80 and 30% yield the yield-time curve is a straight line and the A.D-time curve is a straight line and of necessity the yield-A.D. curve is a straight line, as shown by Figures 1-26 and 32. The slopes for the curves are listed in Table II.

The intercepts at zero time and 100% yield, as well as the average values for the curves on each graph, are listed in Table III. There are six possible

cases for the positions of the intercepts obtained in Table III. The interpretation of these cases with regard to the charcoal burning process is as follows:

(1) Yield-time intercept above 100%: steam oxidation reaction starts slowly and reaches maximum value after an induction period.

(2) Yield-time intercept below 100%: loss of volatile matter commencing as temperature of last heat treatment is passed in heating up period. Most of this volatile matter will be discharged before the steam is admitted (i.e., zero time) and there may be as in (1) an induction period before the steam oxidation reaction reaches its maximum value.

(3) A.D.-time intercept above initial A.D.: probably shrinkage. Of course there is no way of telling what the actual A.D. is in the jiggler. It can only be observed that the curve of A.D.-after-cooling extrapolates to a higher A.D. than it had before charging.

(4) A.D.-time intercept below the initial A.D.: the shrinkage is probably negligible. In both cases three and four other factors such as loss of volatile matter and carbonization treatment must be considered. It can only be observed with certainty that the curve is convex or concave ot the abscissa at the origin. Whether it crosses the abscissa or not is uncertain since no measurements were taken in that range.

(5) A.D.-yield intercept above initial A.D.: represents shrinkage, but does not necessarily delineate the actual path by which the shrinkage occurred.

(6) A.D.-yield intercept below initial A.D.: represents no shrinkage.

The average values for the intercepts and slopes of the A.D.-yield curves from Table III are plotted in Figure 32.

The curves of per cent volume yield versus per cent weight yield were calculated by the equation:

$$\frac{(W\%)_t}{(AD)_t} = \frac{(V\%)_t}{(AD)_o}$$

using 0.60 and 0.50 as AD_o; where m is the mass, V the volume, ρ the apparent density at any time, and ρ_o the apparent density at zero time.

A disagreement of the size loss data with the calculated volume loss, shown as solid lines in Figs. 27, 28, 29, 30 and 31, is caused by attrition of the particles. Where the size per cent is higher than the predicted volume per cent the softer particles have suffered disproportionate amounts of attrition and burning. Where the size per cent is lower the effect of attrition has been general. The per cent of 12-16 mesh in the yield versus the per cent yield is plotted in Figures 27-31. The curves shown in Figures 27-31 are calculated volume per cent yields taken from appropriate corresponding AD-yield curves in Fig. 16-26. The per cent size loss of 12-16 is theoretically equivalent to about 75% of the volume loss. The per cent volume curve calculated from the average AD-yield curve 1-13 in Fig. 32 is shown in Fig. 28 composite. The shape of the per cent volume curve is very sensitive to the slope of the AD-yield curve, so that too close correspondence to the size loss cannot be expected. The agreement which is obtained therefore in Figures 29-31 can be considered excellent. The above effect of gas velocity on the attrition produced is shown in Figure 28 composite. Such a velocity distribution effect is undoubtedly due to the fluid action in the laboratory jiggler.

THEORETICAL SHAPE OF AD-YIELD CURVES

The AD-yield and volume are mathematically related by the following equation:

Nomenclature:

Let m = mass, grams
v = volume, ccs.

ρ = apparent density (A.D.)

R = ratio factor, $\frac{dm}{dm_i}$

c = integration constant

a = constant, slope = $\frac{d\rho}{dm}$

e = constant, intercept on density axis of density vs. yield relation

γ = ratio factor

$$\frac{m}{V} = \rho \tag{1}$$

The path followed during activation may be described differentially by

$$dm = \rho\, dV + V\, d\rho \tag{2}$$

We may arbitrarily designate the quantities occurring in the equation as external m_e and internal m_i.

$$dm = dm_e + dm_i \tag{3}$$

by the following transformation

$$\frac{dm}{dm_i} = \frac{dm_e}{dm_i} + 1 = R = \frac{dm}{V\, d\rho} \tag{4}$$

substituting from (1)

$$\frac{\rho\, dm}{m\, d\rho} = \frac{d \ln m}{d \ln \rho} = R \tag{5}$$

Let R be constant and integrate

$$m = c\rho^R \tag{6}$$

Equation (6) is obtained where c is a constant of integration.

Let a second case be considered where the apparent density vs. yield curve is linear and m_o = 1 gram.

$$\rho = am/m_o + e \tag{7}$$

Let $d\rho/dm = a$. a is the slope of the apparent density vs. mass yield curve. Substituting in (4) we obtain

$$R = \frac{1}{aV} \tag{8}$$

Substituting (8) and (1) in (7) we obtain

$$amR = am + e \qquad (9)$$

$$am = (R - 1)e \qquad (10)$$

$$R - 1 = \frac{dm_e}{dm_i} = Y \qquad (11)$$

and

$$m = \frac{e}{aY} \qquad (12)$$

In brief, the log relationship requires that the ρ go down with large curvature below $m = 0.2\ m_o$ (20% yield) so that ρ will go to 0, and the first order relations require that the external burning increase as the volume goes to 0. Here yield and volume may go to zero having a perfectly finite apparent density. Both relations fit the data equally well, and the data do not furnish a basis for extrapolation.

Rate of Gasification.

From the burning characteristics of these samples several conclusions can be drawn concerning the oxidation of the charcoal.

1. A constant burning rate with time must mean that the effective area taking part in the oxidation is approximately constant.

2. The Arrhenius constant for the reaction is 47500 cal./mol. (See Table IV.) This indicates the diffusion is not controlling and this value is in good agreement with Mayers[8].

3. The composition of the gases evolved during activation of the pressed wood char as reported in Informal Progress Report 10.4-30 agrees with those reported by Mayers and also approaches the value for the water gas equilibrium (See Table IV). This indicates that the charcoal is behaving like graphite in this reaction, as contrasted to PCI char which shows the effect of its ash

8 - J. Am. Chem. Soc. 56, 1879 (1934).

constituent by the large percentage of CO in the reacted gases.

PS Life.

The observed PS lives have been correlated with thetime of activation, yield of product, and apparent density of product, as shown by Figures 1-26.

The general shape of the PS curve in these correlations is one which rises to a maximum and then levels off or descends slightly.

PS-Yield

The rate of rise of PS life with decreasing yield on the ascending portion of the PS-yield curve is regular and reproducible for the No. 109 samples, as shown by the even-numbered Figures 2-14. From the CR, CM, CL and 108 samples there is a much larger spread of values in this region (see Figures 16-26). In Figures 24 and 26 the 24A and 26A and B curves reach the maximum very rapidly while the 24B and 26D curves have a much smaller slope to the maximum. For the 109 char the PS-yield curves are flat between 50% and 30% yield forming a symmetrical family. For the laboratory carbonized Presto-logs the above relationship is approximately true, but in some cases the maximum life is higher and is reached at lower yields. Refer to Figures 20, 24 and 26.

PS-AD

The PS-AD curves are generally of the same type as the PS-yield. The runs numbered J 1291-96, 1334-37 with screen in Figures 6 and 10 show that under some conditions the PS life increases within a very narrow range of AD. A correlation between initial AD and PS life developed is presented in Figure 24 of OSRD Report No. 1856 on "The Preparation of Wood Charcoal Suitable for Activation." The figures in our report compare only graphs for activation of materials with the same initial AD.

The relation of the adsorption isotherm to the adsorptive capacity of charcoal has been under investigation in NDRC Section 10.5. The PS life has

been correlated with the volume of nitrogen adsorbed at -195°C. under 0.4 relative pressure per cc. of charcoal by the equation.

$$\text{PS life (min.)} = 0.53\ (V_{0.4} - 48) \quad ^{9}$$

Nitrogen isotherms for Carlisle char reported in Informal Progress Report 10.5-7 show that for jiggler activation of this char the V_s values are approximately proportional to the apparent density of the sample. V_s is the volume of N_2 adsorbed at saturation and is a measure of the total pore volume. Figure 36 illustrates three variations of the shape of the PS curves with the corresponding nitrogen adsorption isotherms, the shape of which is deduced from the above assumption that V_s is proportional to the AD.

If the sample has the PS-AD curve I then in activating from point 2 to point 4 the shape of the isotherm does not change and the proportion of large sized pores is constant. If the sample has the II PS-AD curve the isotherm changes from 2 to 3 during activation and the number of larger sized pores has been increased during activation. The activation process for curve III has been such that large sized pores were present even in the early stages of activation.

PS-Time

In Table II are given the maxima for the PS curves for 109 and the times at which these maxima are reached. Figure 35 is a plot of these maximum lives versus the log of time. The solid and dotted lines show the approximate zones of constant temperature, constant gasification rate and constant activating gas velocity. The curves represented in Figure 35 having low maxima are ones having greater development of the large sized pores. In effect Figure 35 shows roughly the influence of temperature of activation, gasification rate, and activating gas velocity on the pore size distribution developed

9 - OSRD Formal Report No. 1777.

in the activation of this char.

Figure 34 is a plot of the PS maxima in Table II against the yield and AD at which these maxima were reached. This is another method of representing the manner in which different amounts of internal and external burning affect the PS life.

CONCLUSIONS

Previous investigations[10] of the effect of gas velocity on the activity of the charcoal produced have resulted in the conclusion on the part of several authors that increasing the gas velocity improves the activity of the charcoal for a given amount of gasification.

Bach and Zolotarevskaya [11] using pulverized sugar carbon sifted through silk report the contrary which seems to be merely further confirmation of the effect of the gross structure of the primary char on the potential adsorptive capacity of the finished char.

We conclude that in gas activation of 12-30 US mesh particles the high gas velocities in the jiggler process reduce the diffusion resistance and promote the formation of adsorptive surface within the particle.

10- Ruff and Backe, Kolloid Z. 38, 59, (1926).
Kozakovitch and Ismailoff, ibid. 48, 241 (1929), 57, 294, (1931).

11- J. Phys. Chem. (U.S.S.R.) 7, 54 (1936).

Table I
Results on Activation of Charcoal in Laboratory Jiggler

Sample 108 Compressed wood char from Carlisle Company carbonized to 1200°F. received March 1943. AD 12-16 U.S. mesh 0.49 g./cc.

Sample 109 Compressed wood char from Crown Zellerbach Company (Carlisle process) electrically calcined at 2000°F. Received June 1943 AD 12-16 U.S. mesh 0.60 g./cc.

Samples CM, CR, and CL Compressed wood char from proposal 10-171 Carnegie Institute of Technology (Carlisle process; see OSRD Report No. 1002.).
CR carbonized to 1200°F. AD 12-16 U.S. mesh 0.52 g./cc.
CM muffle calcined 1800°F. AD 12-16 U.S. mesh 0.58 g./cc.
CL electric calcined 1800°F. AD 12-16 U.S. mesh 0.60 g./cc.

Symbols and Units for Table I

Column	Symbol
(1)	Sample = Sample number, described above.
(2)	Run = Run number.
(3)	t = temperature of charcoal bed, °F.
(4)	s = steam rate, lb./(hr.)(lb.charge)
(5)	u = gas (steam) velocity, ft./sec.
(6)	~~θ~~ = time, hours.
(7)	w = gasification rate, lb. carbon gasified/(hr.)(lb.charge)
(8)	Y = yield, weight per cent.
(9)	y = weight per cent of 12-16 U.S. mesh fraction in yield Y.
(10)	ρ = apparent density, gm./cc.
(11)	PS = chloropicrin life (accelerated), minutes. (10 cm. bed; 47 mg./l.; 1 liter/($cm.^2$)(min.).)

Table I
Results on Activation of Charcoal in Laboratory Jiggler

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
109	1551	1450	8.3	1.5	2.0	7.7	84.6	98.0	0.52	25
	1552				3.0	9.33	72.0	95.8	0.46	30
109	1553	1450	20.0	3.6	1.5	9.86	85.2	98.5	0.54	22
	1554				2.75	11.5	68.4	98.5	0.47	35
	1555				3.50	9.3	67.6	97.5	0.43	35
109	1556	1450	37.7	6.8	2.5	9.4	76.6	97.8	0.50	27
	1557				4.0	10.3	59.0	94.3	0.42	42
	1559				4.5	11.4	48.6	88.1	0.40	42
	1558				5.0	12.1	39.4	84.7	0.36	47
109	1560	1450	30.5	5.5	4.0	10.1	59.6	92.6	0.40	39
	1561				5.5	9.4	48.4	86.8	0.39	40
109	1526	1500	0.34	0.062	1.00	7.0	93.0	98.2	0.54	4
	1527		2.32	0.42	2.00	12.4	75.2	98.5	0.43	29
	1528		4.55	0.82	2.00	15.9	68.2	97.4	0.39	32
	1529		7.20	1.3	2.00	17.3	65.4	97.2	0.41	39
	1533		8.30	1.5	2.5	15.5	61.4	93.8	0.37	34
	1530		8.30	1.5	3.0	18.1	45.8	92.1	0.33	36
	1532		8.30	1.5	3.25	16.8	45.6	91.7	0.34	30
	1531		8.30	1.5	3.5	18.1	36.8	87.4	0.30	--
109	1470	1500	8.30	1.5	1.75	20.6	63.9	98.4		
	1468				2.00	22.2	55.6	96.8	0.39	32
	1469				2.25	21.4	51.8	95.0	0.39	32
	1466				2.50	23.3	41.8	90.8	0.31	35
	1467				3.0	23.3	30.0	86.0	0.29	36
109*	1268	1500	19.4	3.5	1.0	16.8	83.2	98.8	0.50	22
	1269				2.0	19.4	61.2	95.4	0.40	37
	1271				2.50	19.4	51.4	89.6	0.34	38
	1273				2.75	23.0	36.8	77.7	0.33	40
	1272				3.00	19.4	42.0	78.5	0.31	42
	1270				3.25	20.1	34.8	80.5	0.31	43
109	1370	1500	20.0	3.6	1.0	35.0	65.0	94.8	0.46	35
	1366				1.25	33.8	57.8	92.0	0.40	41
	1367				1.50	33.6	49.6	89.5	0.34	40
	1369				1.625	37.9	38.4	82.8	0.34	45
	1368				1.75	38.3	33.0	75.3	0.31	34
109	1475	1500	30.5	5.5	1.25	29.3	63.4	93.2	0.40	39
	1472				1.50	26.5	60.2	93.5	0.39	39
	1473				1.75	28.2	50.6	89.0	0.36	46
	1474				1.875	31.7	40.6	78.9	0.33	45
	1476				1.925	31.7	39.0	84.1	0.34	43
	1471				2.0	36.4	27.4	62.8	0.30	37

*Not plotted on Figure 3

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Table I (Continued) pg. 2

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) ϵ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
109	1371	1500	30.5	5.5	1.25	33.0	58.8	89.8	0.42	35
	1372				1.50	31.1	53.2	91.5	0.41	43
	1373				1.75	32.5	43.2	85.2	0.34	45
	1374				2.0	33.0	34.0	76.0	0.33	46
109	1375	1500	37.7	6.8	1.0	27.2	72.8	96.3	0.44	31
	1376				1.5	31.6	52.6	90.1	0.39	48
	1378				1.75	31.0	45.6	85.9	0.34	37
	1377				2.0	31.6	36.8	80.5	0.33	42
109	1482	1500	37.7	6.8	1.25	25.0	68.8	89.1	0.45	29
	1477				1.50	28.6	57.0	93.0	0.42	42
	1479				1.75	28.5	50.2	90.0	0.36	39
	1480				1.875	29.0	45.4	89.8	0.38	--
	1478				2.00	32.6	34.6	78.2	0.31	41
	1481				2.00	28.5	43.0	85.6	0.36	--
	1483				2.25	26.9	39.4	84.8	0.34	36
109	1382	1500	47.1	8.5	1.0	35.8	64.2	87.8	0.39	34
	1381				1.25	37.6	53.0	89.0	0.39	40
	1379				1.50	34.5	48.2	85.5	0.37	41
	1380				1.75	36.8	35.6	74.6	0.30	38
109	1453	1500	56.0	10.1	0.75	25.6	81.8	93.5	0.48	24
	1454				1.00	24.4	75.6	91.5	0.46	28
	1455				1.25	25.0	68.8	90.9	0.46	36
	1456				1.50	27.5	58.8	88.1	0.47	38
	1459				1.75	28.8	49.6	82.0	0.37	39
	1457				2.00	28.7	42.6	80.6	0.35	40
	1458				2.25	27.9	37.2	71.5	0.30	40
109	1460	1500	77.5	14.0	1.50	20.7	69.0	88.7	0.43	34
	1461				2.00	19.2	61.6	83.4	0.38	38
	1462				2.50	20.8	47.8	83.0	0.34	37
	1463				3.00	18.4	44.8	76.6	0.35	40
	1464				3.50	16.9	40.8	67.1	0.34	40
	1465				4.00	17.2	31.2	50.7	0.28	35
109	1292	1550	30.5	screen*	1.0	35.2	64.8	96.6	0.43	28
	1291			(5.5)	1.25	39.2	51.0	91.4	0.39	34
	1293				1.375	39.5	45.6	90.7	0.40	35
	1294				1.50	35.6	46.6	90.1	0.40	33
	1295				1.625	36.1	41.4	89.8	0.38	37
	1296				1.75	34.3	40.0	93.5	0.37	33

*In these runs the circulation of the charcoal was restricted to that possible between two screens 6 inches apart so that the jiggling gas velocity is effectively zero. The figures in parentheses are the entering gas velocities.

Table I (Continued) pg. 3

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) ~~e~~	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
109	1302	1550	36.0	Screen*	1.0	34.0	66.0	95.8	0.40	30
	1307			(6.5)	1.25	38.0	52.4	93.3	0.39	37
	1303				1.50	33.1	50.3	93.2	0.39	30
	1308				1.50	37.0	44.6	94.0	0.40	33
	1306				1.625	38.0	38.2	91.2	0.37	35
	1304				1.75	36.4	36.4	94.0	0.40	31
	1305				1.865	34.1	36.4	89.0	0.36	35
109	1281	1550	7.76	1.4	1.0	28.0	72.0	99.3	0.45	29
	1286				1.0	31.6	68.4	96.5	0.43	30
	1285				1.25	33.1	58.6	94.1	0.39	39
	1282				1.5	34.4	48.4	93.0	0.32	34
	1284				1.75	33.0	42.2	95.7	0.30	40
	1283				2.0	32.3	35.4	91.5	0.28	38
109	1276	1550	19.4	3.5	0.75	40.8	69.4	96.8	0.45	30
	1275				1.0	38.2	61.8	94.8	0.42	40
	1278				1.16	42.5	50.4	92.4	0.37	34
	1279				1.33	35.2	47.2	92.7	0.36	38
	1277				1.50	47.8	28.2	71.6	0.29	34
	1280				1.50	43.7	34.4	86.0	0.31	45
	1274				2.00	46.3	7.4	32.4	--	--
109	1287	1550	30.5	5.5	1.0	38.6	61.4	90.3	0.35	34
	1299				1.0	33.2	66.8	98.5	0.45	34
	1290				1.125	43.0	51.6	93.0	0.35	37
	1289				1.25	42.0	47.5	86.5	0.35	40
	1298				1.25	36.0	55.0	96.0	0.37	38
	1297				1.375	35.5	51.2	95.3	0.39	36
	1288				1.50	42.2	36.8	74.0	0.33	37
	1301				1.50	37.9	43.2	82.4	0.42	43
	1300				1.75	39.4	31.0	82.5	0.31	40
109	1312	1550	36.0	6.5	0.75	44.0	67.0	94.4	0.42	35
	1309				1.00	52.0	48.0	90.0	0.39	40
	1310				1.25	42.3	47.2	91.1	0.36	38
	1313				1.30	45.9	40.4	81.6	0.34	40
	1311				1.50	44.5	33.2	74.2	0.31	37
109	1351	1550	36.0+	10.0	1.00	42.6	57.4	87.0	0.41	31
	1349				1.25	42.3	47.2	82.7	0.37	32
	1352				1.375	42.2	42.0	79.0	0.35	38
	1350				1.50	40.8	38.8	76.3	0.33	33

* In these runs the circulation of the charcoal was restricted to that possible between two screens 6 inches apart so that the jiggling gas velocity is effectively zero. The figures in parentheses are the entering gas velocities.

+ Gas velocity raised to 10.0 ft./sec. by addition of nitrogen.

Table I (Continued) pg. 4

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) FS
109	1545	1550	2.22	0.4	1.5	21.4	67.8	96.8	0.46	26
	1542				2.0	23.4	52.8	96.5	0.39	31
	1544				2.25	24.3	45.4	95.6	0.40	31
	1543				2.50	27.3	31.8	93.1	0.32	31
109	1537	1550	5.54	1.0	1.25	29.4	63.2	96.2	0.39	29
	1535				1.5	31.6	52.6	95.0	0.42	30
	1536				1.75	31.1	45.6	93.5	0.37	32
	1534				2.0	31.5	37.0	92.5	0.33	29
109	1387	1550	8.31	1.5	0.75	45.4	66.0	82.1	0.42	32
	1383				1.0	51.8	48.2	87.6	0.33	36
	1386				1.125	50.3	43.4	86.6	0.26	37
	1384				1.25	48.2	39.8	86.3	0.27	30
	1388				1.375	51.0	29.8	86.5	0.27	37
	1385				1.5	53.2	20.2	68.3	0.28	31
109	1393	1550	20.0	3.6	0.625	60.7	62.0	94.8	0.34	36
	1390				0.75	64.0	52.0	93.3	0.36	36
	1391				0.875	65.0	43.2	88.4	0.31	36
	1389				1.0	62.8	37.2	84.5	0.30	36
	1392				1.125	66.7	24.8	67.7	0.27	36
109	1397	1550	30.5	5.5	0.625	55,3	65.4	93.6	0.44	36
	1396				0.75	60.3	54.8	89.0	0.39	36
	1395				0.875	64.0	44.0	85.4	0.35	36
	1394				1.0	63.0	37.0	77.3	0.32	43
109	1400	1550	37.7	6.8	0.75	63.3	52.6	89.4	0.37	40
	1401				0.625	88.0	45.0	91.5	0.37	24
	1399				0.875	64.0	44.0	86.0	0.32	11
	1398				1.0	62.4	37.6	76.1	0.31	29
109	1404	1550	47.1	8.5	0.60	62.0	62.8	84.5	0.43	38
	1405				0.675	63.7	57.0	88.4	0.35	28
	1403				0.75	79.8	40.2	83.6	0.36	24
	1402				1.0	76.0	24.0	62.5	0.29	35
109	1447	1550	56.0	10.1	0.50	46.4	76.8	92.5	0.44	30
	1448				0.75	56.6	57.6	90.4	0.33	39
	1449				0.875	56.3	50.8	89.0	0.35	31
	1450				1.0	49.8	50.2	86.0	0.34	31
	1451				1.25	48.8	39.0	74.4	0.30	32
	1452				1.50	47.9	28.2	58.8	0.26	30

PART II

Table I (Continued), pg. 5

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
109	1442	1550	77.5	14.0	0.875	40.7	64.4	88.2	0.40	28
	1440				1.0	36.2	63.8	89.6	0.42	34
	1441				1.25	38.8	51.6	82.9	0.36	38
	1443				1.375	41.6	42.8	77.5	0.32	35
	1444				1.50	40.0	40.0	70.0	0.31	28
	1445				1.75	34.2	40.0	73.6	0.32	38
	1446				2.00	29.8	40.6	65.0	0.31	38
109	1334	1600	30.5	Screen*	0.50	69.7	65.2	92.7	0.44	29
	1335			(5.5)	0.625	70.3	56.2	93.5	0.43	33
	1336				0.75	74.7	44.0	93.7	0.39	33
	1337				0.875	69.0	39.6	93.9	0.40	34
109	1347	1600	36.0	Screen*	0.50	67.8	66.6	94.3	0.41	28
	1346			(6.5)	0.625	80.0	50.0	95.3	0.47	31
	1345				0.75	71.2	46.6	92.2	0.42	31
	1344				0.875	69.1	39.6	91.9	0.41	31
	1348				1.0	73.2	26.8	93.2	0.39	31
109	1316	1600	7.76	1.4	0.75	52.6	60.6	96.7	0.41	32
	1317				0.875	53.5	53.2	96.6	0.42	33
	1314				1.0	55.4	44.6	92.5	0.33	36
	1315				1.25	53.7	32.8	85.9	0.29	35
109	1326	1600	19.4	3.5	0.50	73.5	63.2	90.0	0.39	32
	1327				0.625	73.4	54.2	93.4	0.37	31
	1319				0.75	68.2	48.2	88.5	0.35	37
	1322				0.75	69.5	47.8	89.3	0.40	39
	1325				0.767	79.8	38.8	84.5	0.31	32
	1324				0.794	75.7	40.0	83.0	0.32	31
	1323				0.816	76.8	37.2	81.2	0.29	33
	1320				0.875	72.3	36.6	81.3	0.30	37
	1318				1.0	73.2	26.8	67.1	0.27	34
109	1329	1600	30.5	5.5	0.50	81.0	59.5	93.0	0.38	33
	1333				0.567	82.5	53.3	90.2	0.40	33
	1330				0.625	86.2	46.2	86.7	0.37	37
	1332				0.667	80.8	46.2	85.3	0.34	34
	1331				0.70	87.2	39.0	82.0	0.34	36
	1328				0.75	85.3	36.0	80.0	0.33	35
	1338				1.00	73.2	26.8	63.4	0.30	33

* In these runs the circulation of the charcoal was restricted to that possible between two screens 6 inches apart so that the jiggling gas velocity is effectively zero. The figures in parentheses are the entering gas velocity.

Table I (Continued), pg. 6

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
109	1341	1600	36.0	6.5	0.50	77.2	61.4	95.7	0.43	36
	1340				0.625	79.6	50.2	89.2	0.39	36
	1343				0.70	83.4	41.6	83.7	0.34	40
	1339				0.75	79.0	40.8	83.8	0.34	38
	1342				0.875	82.1	28.2	68.8	0.30	34
109	1357	1600	36.0*	10.0	0.625	63.3	60.4	92.0	0.38	30
	1355				0.75	67.5	49.4	87.5	0.36	33
	1356				0.875	69.0	39.6	83.8	0.32	35
	1354				1.00	67.8	32.2	68.8	0.29	37
	1353				1.25	60.8	24.0	50.0	0.24	31
109	1546	1600	2.22	0.4	1.00	31.6	68.4	96.8	0.43	25
	1549				1.5	38.3	42.6	95.4	0.37	30
	1548				1.75	37.4	34.6	97.7	0.34	27
	1547				2.0	34.0	32.0	86.9	0.28	30
	1550				1.25	37.8	54.0	99.3	0.42	30
109	1541	1600	5.54	1.0	0.75	40.0	70.0	97.3	0.46	29
	1540				1.0	44.0	56.0	95.6	0.43	30
	1539				1.25	43.9	45.2	94.2	0.35	32
	1538				1.5	45.6	31.6	92.4	0.30	31
109	1409	1600	8.31	1.5	0.50	69.2	65.4	82.3	0.46	29
	1407				0.75	73.4	45.0	86.7	0.32	28
	1408				0.80	78.8	37.0	86.5	0.30	32
	1406				1.0	75.6	24.4	77.1	0.22	26
109	1412	1600	20.0	3.6	0.50	80.0	60.0	94.6	0.42	26
	1414				0.683	61.7	57.8	95.6	0.39	33
	1413				0.567	89.0	49.6	90.3	0.39	31
	1411				0.625	92.0	42.6	86.3	0.34	25
	1410				0.75	101.5	23.8	68.0	0.26	28
109	1415	1600	30.5	5.5	0.50	58.5	70.8	97.0	0.46	28
	1416				0.75	60.0	55.0	93.3	0.41	35
	1419				0.80	64.5	48.4	87.6	0.35	38
	1418				0.875	61.0	46.6	85.5	0.35	38
	1417				1.0	64.0	36.0	82.7	0.32	39
109	1523	1600	30.5	5.5	0.60	49.4	70.4	96.0	0.47	34
	1524				0.70	52.8	63.0	95.5	0.41	38
	1522				0.80	65.0	48.0	90.0	0.38	42
	1525				1.125	58.7	34.0	81.7	0.31	41
	1521				1.0	60.2	39.8	84.5	0.34	39

* Gas velocity raised to 10.0 ft./sec. by addition of nitrogen.

Table I (Continued), pg. 7

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
109	1424	1600	37.7	6.8	0.50	58.0	71.0	95.5	0.45	35
	1423				0.625	56.7	64.6	93.9	0.42	36
	1422				0.75	73.9	44.6	85.2	0.35	38
	1421				0.875	72.2	36.8	77.1	0.34	38
	1420				1.0	69.6	30.4	72.4	0.31	39
109	1429	1600	47.1	8.5	0.50	98.4	50.8	80.7	0.38	38
	1430				0.70	94.4	34.0	68.8	0.33	32
	1431				0.60	91.7	45.0	76.9	0.35	40
109	1435	1600	56.0	10.1	0.333	74.5	75.2	86.3	0.41	25
	1432				0.50	90.8	54.6	81.7	0.38	37
	1434				0.70	79.7	44.2	75.6	0.32	37
	1433				0.75	84.6	36.6	66.1	0.28	31
109	1436	1600	77.5	14.0	0.75	59.3	55.6	88.2	0.37	31
	1439				0.933	58.4	45.6	81.1	0.33	34
	1438				0.875	62.1	45.4	83.8	0.34	33
	1437				1.0	68.4	31.6	50.6	0.30	26
109	1569	1650	2.22	0.4	0.625	62.4	61.0	96.0	0.39	21
	1565				0.75	62.5	53.2	97.0	0.40	23
	1566				0.865	60.3	48.0	97.0	0.37	28
	1568				0.93	70.4	34.6	92.5	0.35	29
	1562				1.0	47.2	52.8	96.2	0.42	29
	1567				1.0	66.0	34.0	93.0	0.36	29
	1564				1.25	62.0	22.4	91.0	0.36	34
	1563				1.50	55.0	17.4	87.4	0.32	35
109	1570	1650	4.43	0.8	0.50	72.8	63.6	96.6	0.41	25
	1574				0.55	83.7	54.0	98.4	0.43	28
	1572				0.625	86.7	45.8	92.5	0.35	28
	1573				0.70	83.4	41.6	96.2	0.36	31
	1571				0.75	83.0	37.8	92.5	0.33	29
109	1579	1650	8.86	1.6	0.40	87.5	65.0	100.0	0.43	26
	1575				0.50	88.4	55.8	96.8	0.38	28
	1578				0.55	86.6	52.4	97.7	0.41	30
	1577				0.625	89.6	44.0	97.0	0.37	31
	1576				0.70	91.0	36.4	93.4	0.29	30
109	1585	1650	20.0	3.6	0.33	66.2	78.0	84.5	0.41	30
	1583				0.50	100.0	50.0	93.2	0.35	34
	1584				0.55	100.3	44.8	98.3	0.33	33
	1582				0.625	111.3	30.4	83.5	0.30	34
	1581				0.75	108.4	18.6	67.7	0.25	33
109	1590	1650	30.5	5.5	0.25	110.0	72.6	97.6	0.41	27
	1589				0.33	117.8	60.8	96.9	0.41	32
	1588				0.40	118.1	52.8	93.1	0.42	35
	1586				0.50	118.5	40.8	85.6	0.32	35
	1587				0.625	118.9	25.8	70.5	0.28	33

Table I (Continued) p. 8

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
109	1594	1650	37.7	6.8	0.25	139.2	65.2	95.8	0.44	32
	1593				0.33	134.0	55.4	92.5	0.37	34
	1592				0.40	136.6	45.4	88.0	0.33	35
	1591				0.50	128.8	35.6	78.0	0.30	36
109	1609	1650	56.0	10.1	0.33	105.0	65.0	96.5	0.36	33
	1608				0.40	116.7	53.4	93.2	0.38	33
	1610				0.50	115.6	42.2	87.6	0.33	35
	1611				0.625	113.9	29.0	75.1	0.25	33
CR 116 -173	823	1500	55.5	10	0.125	214.0	73.2	70.8	0.48	20
	819				0.25	165.5	58.6	66.5	0.42	31
	821				0.33	144.0	52.0	60.0	0.39	36
	822				0.42	137.0	42.8	54.0	0.35	36
	818				0.50	122.0	38.0	49.2	0.34	39
	820				0.63	111.0	30.6	33.3	0.30	37
CR 116 -173	827	1500	19.4	3.5	0.33	133.0	59.0	59.6	0.43	31
	826				0.42	124.5	48.2	60.5	0.38	34
	824				0.50	113.5	43.2	61.5	0.35	33
	825				0.625	104.0	35.0	45.2	0.31	39
CR 116 -173	836	1400	55.5	10.0	0.75	53.8	59.6	61.1	0.45	37
	837				1.25	43.0	46.2	52.8	0.40	43
	838				1.50	37.9	43.2	48.5	0.37	44
	839				2.00	33.0	34.0	29.4	0.35	42
	840				2.00	32.0	36.0	38.3		
CM 37 -57	652	1500	20	3-	0.5	89.6	55.2	77.5	0.42	30
	655				0.50	110.0	45	70.7	0.38	37
	654				0.583	108.4	36.8	62.0	0.35	39
	653				0.75	106.5	20.2	36.6	--	--
CM 37 -57	657	1500	50	10-	0.33	135.7	54.8	69.7	0.43	36
	658				0.417	129.4	46.0	63.4	0.41	38
	656				0.5	116.4	41.8	53.6	0.37	38
CM 37 -57	714	1500	12	3-	0.75	53.4	60.0	77.5	0.45	34
	712				1.0	51.2	48.8	73.2	0.39	34
	713				1.25	47.8	40.1	56.7	0.39	40
	711				1.5	47.2	29.1	34.0	0.34	39
CM 37 -57	815	1500	55.5	10-	0.33	102.2	66.0	81.8	0.45	32
	814				0.50	88.8	55.6	76.2	0.41	37
	817				0.63	92.3	42.4	57.5	0.38	41
	813				0.75	79.6	40.4	57.4	0.38	41
	816				0.88	80.7	29.4	36.7	0.31	41
	811				1.0	73.8	26.2	29.8	0.32	43
	812				1.0	73.8	26.2	29.8		

Table I (Continued) pg. 9

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
CM 37	1014	1450	55.5	10	1.00	49.0	51.0	65.0	0.41	44
-57	1016				1.25	44.0	45.0	59.0	0.39	43
	1017				1.375	41.3	43.2	54.0	0.38	44
	1015				1.50	41.7	37.6	48.4	0.38	44
CM 37	862	1400	55.5	10	1.0	30.2	69.8	82.3	0.46	29
-57	865				1.02	35.6	63.8	77.5	0.44	29
	863				2.0	25.0	50.0	66.0	0.41	41
	864				2.5	22.7	43.2	59.7	0.38	37
	866				3.0	21.6	35.2	40.9	0.38	48
CM 37	913	1400	55.5	10	1.50	29.2	56.2	68.7	0.43	36
-57	912				1.75	29.3	48.8	65.5	0.40	39
	911				2.0	27.6	44.8	60.7	0.40	42
	909				2.5	25.4	36.6	42.6	0.38	45
	910				3.0	22.7	32.0	36.3	0.38	46
CL 18	697	1700	13	3	0.33	130.0	56.8	76.5	0.42	34
-29	696				0.417	138.0	42.4	67.9	0.39	40
CL 18	680	1600	13	3	1.0	44.0	56.0	71.0	0.46	33
-29	681				1.25	46.2	42.4	64.0	0.43	38
	682				1.35	43.0	42.0	64.8	0.43	38
	679				1.5	47.3	29.0	53.0	0.38	38
CL 18	835	1500	55.5	10	0.33	66.0	78.0	82.2	0.51	26
-29	828				0.5	92.0	64.0	83.1	0.49	35
	832				0.75	55.5	58.4	73.9	0.44	41
	833				0.75	55.7	58.2	72.5	0.42	39
	829				1.0	53.2	46.8	60.0	0.41	43
	831				1.25	46.1	42.4	59.4	0.37	46
	834				1.375	48.6	33.2	51.8	0.36	46
	830				1.5	47.9	28.2	41.2	0.33	50
108	1214	1500	7.75	1.4	0.25	92.8	76.8	85.1	0.43	11
	1267				0.25	85.6	78.6	93.6	0.46	10
	1213				0.375	81.6	69.4	82.5	0.41	17
	1266				0.375	73.1	72.6	91.8	0.43	15
	1212				0.50	75.2	62.4	82.3	0.39	23
	1265				0.50	71.2	64.4	82.7	0.39	21
	1211				0.625	72.6	54.6	77.6	0.42	26
	1264				0.625	65.3	59.2	81.7	0.37	21
	1210				0.75	66.9	49.8	77.5	0.33	27
	1263				0.75	63.0	52.8	81.1	0.36	29
	1209				0.875	63.8	44.2	65.6	0.32	29
	1262				0.875	60.3	47.2	76.7	0.32	30

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Table I (Continued) pg. 10

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
108	1208	1500	7.75	1.4	1.0	62.4	37.6	57.3	0.29	29
	1261				1.0	59.6	40.4	70.7	0.30	31
	1215				1.125	64.0	28.0	60.0	0.24	37
108	800	1500	19.4	3.5	0.25	100.8	74.8	82.7	0.49	15
	791				0.25	91.2	77.2	81.4	0.44	17
	788				0.375	99.2	62.8	73.3	0.42	25
	789				0.375	98.1	63.2	74.4	0.43	26
	773				0.50	85.6	57.2	79.0	0.39	26
	780				0.50	82.0	59.0	71.3	0.40	27
	781				0.50	83.0	58.6	73.1	0.39	28
	778				0.625	83.8	47.6	66.4	0.34	31
	779				0.625	76.5	52.2	68.1	0.38	30
	784				0.625	78.1	51.2	64.8	0.38	31
	782				0.75	70.7	47.0	62.1	0.38	33
	783				0.75	76.3	42.8	62.6	0.37	31
	786				1.00	75.0	25.0	39.2	0.27	30
	787				1.00	73.0	27.0	45.2	0.28	32
	785				1.25	69.4	13.2	12.1	--	--
108	1207	1500	19.4	3.5	0.125	141.0	82.4	85.2	0.45	4
	1252				0.125	131.2	84.6	88.9	0.50	4
	1206				0.25	105.7	73.6	83.4	0.41	17
	1253				0.25	92.8	76.8	81.8	0.48	15
	1205				0.375	103.0	61.4	73.9	0.40	27
	1254				0.375	87.1	67.3	79.8	0.44	21
	1204				0.50	81.6	59.2	73.4	0.39	30
	1255				0.50	91.6	54.2	87.1	0.42	25
	1203				0.625	86.7	45.8	66.3	0.36	36
	1256				0.625	75.0	53.2	74.5	0.43	27
	1202				0.75	80.8	39.4	62.4	0.31	33
	1257				0.75	73.0	45.2	68.2	0.34	35
	1201				0.875	81.9	28.4	50.6	0.27	31
	1258				0.875	65.0	43.2	64.3	0.32	35
	1200				1.00	79.4	20.6	34.0	--	--
	1259				1.00	66.8	33.2	51.2	0.34	30
	1260				1.125	66.1	25.6	43.8	0.28	43
108	1225	1500	30.5	5.5	0.125	127.2	84.1	87.5	0.50	29
	1243				0.125	129.5	83.8	86.5	0.47	4
	1223				0.25	96.0	76.0	82.4	0.44	28
	1244				0.25	96.0	76.0	86.5	0.43	11
	1222				0.375	84.8	68.2	77.4	0.45	18
	1245				0.375	81.1	69.6	87.5	0.42	17
	1221				0.50	74.3	62.8	75.7	0.41	26
	1246				0.50	67.2	66.4	81.8	0.39	23
	1220				0.625	71.7	55.2	75.0	0.38	28

Table I (Continued) pg. 11

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
108	1247	1500	30.5	5.5	0.625	81.2	49.2	98.3	0.39	28
	1218				0.75	65.5	50.8	75.0	0.39	31
	1249				0.75	61.8	53.6	75.8	0.39	31
	1217				0.875	62.6	45.2	70.8	0.34	30
	1250				0.875	54.8	52.0	72.5	0.37	30
	1216				1.00	59.8	40.2	69.1	0.33	26
	1248				1.00	65.2	34.8	86.1	0.36	29
	1219				1.125	60.6	31.8	65.5	0.36	31
	1251				1.125	55.0	38.2	58.6	0.38	38
108	1227	1500	36.0	6.5	0.125	147.2	81.6	87.0	0.48	5
	1242				0.125	121.5	84.8	88.0	0.40	5
	1228				0.25	107.8	73.2	84.8	0.47	16
	1241				0.25	93.0	76.8	86.0	0.47	13
	1229				0.375	93.9	65.2	81.0	0.44	23
	1240				0.375	82.7	69.0	86.6	0.44	22
	1230				0.50	81.4	59.3	76.7	0.41	25
	1239				0.50	74.4	62.8	82.4	0.40	33
	1231				0.625	76.0	52.5	76.5	0.39	27
	1238				0.625	64.3	59.8	80.0	0.40	23
	1232				0.75	70.4	47.2	73.7	0.34	27
	1237				0.75	68.5	48.6	74.5	0.36	34
	1236				0.875	59.8	47.6	69.3	0.39	33
	1234				0.875	65.8	42.4	72.6	0.34	30
	1224				1.00	63.2	36.8	70.6	0.33	24
	1226				1.125	65.4	26.4	72.0	0.34	31
	1235				1.125	55.7	37.4	71.6	0.34	28
108	798	1500	55.5	10	0.25	120.0	70.0	78.8	0.48	23
	799				0.25	119.0	70.2	78.0	0.47	22
	797				0.375	107.2	59.8	73.6	0.43	32
	796				0.375	92.7	65.2	79.8	0.41	26
	775				0.50	96.8	51.6	74.0	0.38	32
	795				0.50	99.2	50.4	73.7	0.39	34
	777				0.625	92.2	42.4	52.3	0.35	33
	776				0.75	83.8	37.2	58.5	0.33	34
	792				0.75	83.7	37.2	54.2	0.35	37
	793				1.00	73.8	26.2	34.4	--	--
	794				1.00	68.8	31.2	36.5	0.31	37
CR 206	919	1400	55.5	10	0.75	52.5	60.6	70.0	0.43	34
-208	914				1.0	45.6	54.4	62.4	0.42	35
	915				1.25	42.3	47.0	54.7	0.40	40
	916				1.50	39.5	40.8	49.5	0.37	40
	917				1.75	36.6	36.0	41.7	0.35	45
	918				2.0	34.2	31.6	31.0	0.34	48
	949				2.0	35.2	29.5	28.0		

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Table I (Continued) pg. 12

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
CR 240	925	1400	55.5	10	0.625	59.9	62.6	66.2	0.45	32
-242	924				0.833	53.8	55.2	61.2	0.42	39
	920				1.0	49.6	50.4	61.8	0.39	43
	921				1.25	45.5	43.2	51.9	0.38	39
	922				1.50	39.3	41.0	48.8	0.37	42
	923				1.75	37.7	34.0	3818	0.34	42
CR 196	926	1400	55.5	10	0.75	46.4	65.2	64.8	0.45	27
-198	927				1.0	40.8	59.2	60.5	0.45	34
	931				1.25	40.0	50.0	55.6	0.39	39
	928				1.50	35.5	46.8	50.9	0.40	47
	930				1.75	33.9	40.6	46.3	0.36	43
	929				2.0	32.9	35.4	35.6	0.37	45
CR 193	938	1400	55.5	10	0.667	56.1	62.6	67.4	0.43	32
-195	932				1.0	46.4	53.6	56.3	0.41	38
	933				1.25	42.4	47.0	51.9	0.38	42
	934				1.50	38.7	42.0	51.3	0.37	42
	936				1.75	31.8	34.4	43.0	0.32	38
CR 237	939	1400	55.5	10	0.667	56.1	62.6	68.1	0.45	30
-239	937				1.0	50.2	49.8	55.4	0.39	39
	940				1.0	48.2	51.8	58.3	0.41	39
	943				1.316	38.5	49.8	55.4	0.40	40
	941				1.50	39.2	41.2	48.0	0.37	42
	942				1.75	36.2	36.6	41.5	0.34	42
CR 212	948	1400	55.5	10	0.667	56.9	62.0	62.6	0.41	30
-214	944				1.0	45.2	54.8	59.5	0.41	34
	945				1.25	41.3	48.4	52.8	0.39	38
	946				1.55	38.9	39.8	46.3	0.35	37
	947				1.75	35.4	38.0	41.1	0.34	42
CR 246	965	1400	55.5	10	0.667	57.0	62.0	62.3	0.45	28
-248	961				1.0	47.6	52.4	54.9	0.21	38
	964				1.25	40.8	49.0	49.8	0.39	39
	962				1.50	39.5	40.8	43.2	0.38	46
	963				1.75	36.8	35.6	39.3	0.36	46
CR 199	970	1400	55.5	10	0.667	55.1	63.2	67.7	0.43	26
-201	966				1.0	45.8	54.2	56.8	0.41	34
	969				1.25	42.8	46.4	55.5	0.40	38
	967				1.50	40.0	40.0	44.5	0.39	41
	968				1.80	35.8	35.6	44.4	0.36	39
CR 190	976	1400	55.5	10	0.667	55.1	63.2	65.2	0.43	26
-192	971				1.0	45.6	54.4	58.1	0.40	36
	972				1.25	41.0	48.8	54.9	0.39	40

Table I (Continued) pg. 13

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
CR 190	973	1400	55.5	10	1.50	37.5	43.8	48.4	0.37	43
192	974				1.75	35.6	37.8	39.1	0.36	44
	1151				2.00	29.4	41.3	44.7	--	--
CR 243	981	1400	55.5	10	0.667	56.6	62.2	65.9	0.46	33
-245	977				1.0	45.8	54.2	57.5	0.42	36
	978				1.5	38.9	41.6	44.7	0.37	38
	979				1.85	36.8	32.0	32.5	0.35	45
	980				1.85	36.7	32.2	31.1		
CR 247	985	1400	55.5	10	0.5	65.6	67.2	65.5	0.48	27
-249	982				1.0	50.2	49.8	53.0	0.40	36
	983				1.50	40.8	38.8	42.3	0.36	44
	984				1.75	35.1	38.6	41.5	0.37	39
CR 209										
-211	992	1400	55.5	10	0.667	60.8	59.4	61.2	0.45	36
	986				1.00	46.4	53.6	53.2	0.40	38
	988				1.25	45.8	42.6	47.8	0.36	38
	989				1.50	31.6	42.6	47.8	0.35	43
	990				1.75	38.8	32.2	31.7	0.34	47
	991				1.75	38.4	32.8	39.3		
CR 218	1004	1400	55.5	10	0.667	58.8	60.8	66.1	0.42	31
-220	997				1.00	51.6	48.4	55.3	0.38	38
	998				1.50	37.2	44.2	46.1	0.37	42
	1000		44.7	10	1.75	38.8	32.0	27.3	--	--
	1003		55.5	10	1.75	34.8	39.2	42.8	0.35	40
	999				2.00	32.9	34.2	37.0	0.34	38
	1152		45.8	10	1.50	39.5	40.8	48.0	--	--
CR 187	1023	1400	55.5	10	0.667	50.0	66.6	69.0	0.45	27
-189	1020				1.00	45.0	55.0	63.6	0.42	39
	1021				1.50	36.8	45.8	55.0	0.38	41
	1022				2.00	30.0	40.0	46.0	0.37	42
CM 76	1081	1450	55.5	10	0.667	55.3	63.0	73.3	0.47	37
	1078				1.0	49.2	50.8	66.5	0.42	43
	1080				1.25	45.9	42.6	54.0	0.41	49
	1079				1.50	43.5	34.8	43.6	0.38	47
CM 64	1085	1450	55.5	10	0.50	50.4	74.8	78.7	0.54	20
	1082				1.00	46.4	53.4	69.2	0.44	40
	1084				1.25	41.1	48.6	63.7	0.44	42
	1083				1.50	40.8	38.8	49.5	0.40	48
CM 77	1088	1450	55.5	10	0.667	55.4	63.0	74.4	0.50	37
	1086				1.0	47.4	52.6	63.1	0.43	42
	1087				1.30	44.3	42.4	53.7	0.41	48
	1089		65.0	10	1.60	35.4	43.4	51.8	0.42	41

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Table I (Continued) pg. 14

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
CM 71	1068	1450	55.5	10	0.667	47.4	68.4	76.0	0.49	34
	1065				1.0	47.6	52.4	70.0	0.42	40
	1066				1.25	42.7	44.6	64.0	0.41	44
	1067				1.50	38.4	42.4	58.4	0.40	43
	1069				1.75	39.3	31.2	41.6	0.38	53
CM 62	1073	1450	55.5	10	0.50	54.8	72.6	81.3	0.52	26
	1072				1.0	44.6	55.4	70.0	0.43	36
	1071				1.25	42.1	47.4	61.6	0.41	39
	1070				1.50	40.5	39.2	51.0	0.39	41
CM 72	1045	1450	55.5	10	0.667	51.9	65.4	74.2	0.47	35
	1042				1.00	45.4	54.6	67.0	0.44	42
	1044				1.25	45.9	42.6	56.8	0.41	42
	1043				1.50	40.8	38.8	53.5	0.39	45
CM 78	1049	1450	55.5	10	0.667	55.0	63.2	74.8	0.48	40
	1046				1.00	47.4	52.6	67.2	0.43	40
	1048				1.25	48.3	39.6	53.5	0.40	46
	1047				1.50	42.4	36.4	48.3	0.39	48
CM 75	1053	1450	55.5	10	0.50	60.8	69.6	74.7	0.49	30
	1050				1.00	49.0	51.0	59.6	0.42	43
	1052				1.25	47.1	41.2	48.5	0.39	41
	1051				1.50	48.7	37.0	42.2	0.38	48
CM 65	1059	1450	55.5	10	0.50	45.9	77.1	80.0	0.52	24
	1054				1.0	46.4	53.6	67.8	0.47	40
	1055				1.35	38.8	47.6	57.9	0.43	41
	1056				1.75	32.6	43.0	52.1	0.42	54
	1057				2.25	31.8	28.4	35.2		
	1058				2.25	34.2	23.2	29.3	0.35	43
	1149				2.25	32.5	27.0	31.9		
CM 73	1033	1450	55.5	10	0.667	52.8	64.8	71.3	0.46	36
	1029				1.00	49.0	51.0	58.0	0.42	41
	1031				1.25	45.0	43.8	51.1	0.38	40
	1030				1.50	40.9	38.6	43.5	0.36	41
	1032				1.625	40.8	33.6	36.3	0.36	46
CM 63	1105	1450	55.5	10	0.667	50.0	66.6	75.1	0.48	33
	1102				1.0	46.2	53.8	65.4	0.43	42
	1103				1.33	41.1	45.2	56.6	0.41	42
	1104				1.60	37.3	40.4	51.4	0.41	43

Part II

Table I (Continued) pg. 15

(1) Sample	(2) Run	(3) t	(4) s	(5) u	(6) θ	(7) w	(8) Y	(9) y	(10) ρ	(11) PS
CM 80	1122	1450	55.5	10	0.60	49.4	20.4	77.8	0.50	30
	1119				1.00	47.8	52.2	69.7	0.41	38
	1120				1.33	40.3	46.2	58.9	0.42	44
	1121				1.667	37.6	37.4	50.1	0.38	45
CM 61	1093	1450	55.5	10	0.75	58.9	55.8	66.6	0.45	41
	1090				1.00	40.6	59.4	62.8	0.44	42
	1092				1.25	43.9	45.2	56.7	0.43	42
	1091				1.50	41.4	38.0	51.0	0.40	40
CM 79	1101	1450	55.5	10	0.50	55.6	72.2	77.5	0.52	31
	1098				1.00	50.8	49.2	66.7	0.42	41
	1099				1.25	44.7	44.0	57.2	0.42	43
	1100				1.50	51.8	37.2	49.4	0.39	47

PART II

Table II. Summary of Graphical Correlations

Sample 109 Carlisle calcined 2000°F. A.D. = 0.60 gm./cc. described in Table I.
Sample CR 116-173 Pres-to-logs carbonized 1200°F. A.D. = 0.52 gm./cc.
Sample CM 37-57 from CR sample 116-173 muffle calcined 1800°F. A.D. = 0 58 gm./cc.
Sample CL 18-29 from CR sample 116-173 electrically calcined 1800°F. A.D. = 0.60 gm./cc.
Sample 108 Carlisle carbonized 1200°F. A.D. = 0.49 gm./cc.
Sample CR 187, etc. Pres-to-logs carbonized 1200°F. A.D. = 0.52 gm./cc.
Sample CM 61, etc. Muffle calcined at 1800°F. A.D. = 0.58 gm./cc.

Symbols and Units for Table II

Column	Symbol		
(1)	Fig.	=	figure number in this report.
(2)	Run	=	Jiggler run number, J- , as J-1551
(3)	t	=	temperature of charcoal bed, °F.
(4)	u	=	gas (steam) velocity, ft./sec.
(5)	w	=	gasification rate, lb. carbon gasified/(hr.lb.charge)
(6)	Y_o	=	yield extrapolated to zero time, weight per cent
(7)	$\dot{\rho}$	=	time rate of density loss, gm./(cc.)(hour).10
(8)	ρ_o	=	apparent density extrapolated to zero time, gm./cc.
(9)	$d\rho/dY$	=	slope of apparent density (ρ) vs. yield (Y) curve.
(10)	ρ_{100}	=	apparent density extrapolated to 100% yield, gm./cc.
(11)	θ_{PS}	=	activation time to give maximum PS life, hours.
(12)	PS_{max}	=	maximum PS life developed for runs in question (constant temperature and constant steam velocity)
(13)	Y_{PS}	=	yield at maximum PS life, weight fraction
(14)	ρ_{PS}	=	apparent density at maximum PS life, gm./cc.

Table II
Summary of Graphical Correlations

(1) Fig.	(2) Run	(3) t	(4) u	(5) w	(6) Y_o	(7) $\overline{\rho}$	(8) ρ_o	(9) $d\sigma/dY$	(10) ρ_{100}	(11) ϑ_{PS}	(12) PS_{max}	(13) Y_{PS}	(14) ρ_{PS}
				Sample 109									
1 & 2	1551-1561	1450	6.8 (1.5, 3.6 5.5)	15	114	0.6	0.66	0.40	0.59	4.5	45	0.45	0.35
3 & 4	1526-1533	1500	1.5-1[x]	21	113	0.8	0.57	0.33	0.51	2.75	35	0.45	0.32
	1466-1470		1.5 -2[x]	27	110	1.0	0.59	0.33	0.51	2.75	35	0.45	0.32
	1268-1273		3.5	(20)	--	--	--	0.41	0.58	3.25	43	0.35	0.30
	1366-1370		3.6	38	103	1.6	0.59	0.41	0.58	1.75	44	0.40	0.34
	1471-1476		5.5	35	108	1.5	0.62	0.41	0.58	1.75	44	0.40	0.34
	1371-1374		5.5	35	108	1.5	0.62	0.41	0.58	1.75	44	0.40	0.34
	1375-1378		6.8	35	108	1.5	0.62	0.41	0.58	2.0	40	0.45	0.35
	1477-1483		6.8	35	108	1.5	0.62	0.41	0.58	2.0	40	0.45	0.35
	1379-1382		8.5	38	103	1.6	0.59	0.41	0.58	1.5	40	0.45	0.35
	1453-1459		10.1	35	108	1.5	0.62	0.41	0.58	2.0	40	0.45	0.35
	1460-1465		14.0	15	92	0.5	0.49	0.33	0.51	3.0	40	0.45	0.35
5 & 6	1291-96, 1302-08	1550	screen 5.5,6.5)	42	107	0.5	0.46	0.15	0.46	1.4	35	0.45	0.39
	1281-1286		1.4	32	100	1.7	0.60	0.51	0.60	1.5	40	0.44	0.35
	1274-1280		3.5	43	102	1.7	0.57	0.34	0.54	1.5	40	0.40	0.35
	1287-90 1297-1301		5.5	43	102	1.7	0.57	0.34	0.54	1.5	40	0.40	0.35
	1309-1313		6.5	48	104	1.5	0.53	0.34	0.54	1.0	40	0.40	0.35
	1349-1352		10.0	43	102	1.7	0.57	0.34	0.54	1.0	32	0.57	0.41
7 & 8	1542-1545	1550	0.4	29	112	0.9	0.59	0.45	0.62	2.0	31	0.55	0.43
	1534-1537		1.0	34	104	1.8	0.70	0.45	0.62	1.6	31	0.60	0.43
	1383-1388		1.5	54	105	2.3	0.57	0.45	0.57	1.25	38	0.55	0.35
	1389-1393		3.6	74	110	2.3	0.53	0.32	0.52	0.6	36	0.55	0.35
	1394-1397		5.5	74	110	2.3	0.53	0.32	0.52	0.6	36	0.55	0.35
	1398-1401		6.8	74	110	2.3	0.53	0.32	0.52	0.6	36	0.55	0,35
	1402-1405		8.5	98	121	2.3	0.53	0.32	0.52	0.6	36	0.55	0.35
	1447-1452		10.1	39	87	1.6	0.50	0.41	0.56	0.5	31	0.70	0.44
	1440-1446		14.0	40	101	1.9	0.59	0.45	0.57	1.25	38	0.55	0.35
9 & 10	1334-37, 1344-48	1600	screen 5.5,6.5	79	106	1.0	0.49	0.12	0.49	0.6	33	0.50	0.41
	1314-1317		1.4	55	100	2.3	0.57	0.33	0.53	0.9	35	0.50	0.35
	1318-20, 1322-27		3,5	80	101	2.6	0.53	0.33	0.53	0.7	35	0.50	0.35
	1328-33,38		5.5	80	101	2.6	0.53	0.33	0.53	0.7	35	0.50	0.35
	1339-1343		6.5	80	101	2.6	0.53	0.33	0.53	0.7	35	0.50	0.35
	1353-1357		10.0	74	106	2.4	0.54	0.33	0.53	0.9	35	0.40	0.30

x = curve designation on figures.

Table II (Continued) pg 2

(1) Fig.	(2) Run	(3) t	(4) u	(5) w	(6) Y_o	(7) σ	(8) ρ_o	(9) $d\rho/dY$	(10) ρ_{100}	(11) θ_{PS}	(12) PS_{max}	(13) Y_{PS}	(14) ρ_{TS}
11 & 12	1546-1550	1600	0.4	44	111	1.2	0.54	0.25	0.51	1.5	30	0.50	0.37
	1538-1541		1.0	50	108	2.2	0.63	0.47	0.61	1.2	30	0.50	0.37
	1406-1409		1.5	79	104	4.7	0.69	0.48	0.61	0.5	30	0.50	0.37
	1410-1414		3.6	141	131	6.4	0.74	0.47	0.61	0.5	30	0.50	0.37
	1415-1419		5.5	76	111	2.8	0.60	0.47	0.61	1.0	40	0.40	0.32
	1521-1525		5.5	76	111	2.8	0.60	0.47	0.61	1.0	40	0.40	0.32
	1420-1424		6.8	76	111	2.8	0.60	0.47	0.61	1.0	40	0.40	0.32
	1429-1431		8.5	85	94	2.6	0.51	0.47	0.61	0.6	40	0.40	0.32
	1432-1435		10.1	87	103	3.1	0.52	0.36	0.52	0.6	40	0.55	0.35
	1436-1439		14.0	76	111	2.8	0.60	0.47	0.61	0.9	33	0.50	0.37
13 & 14	1562-1569	1650	0.4	64	101	0.8	0.45	0.11	0.45	1.5	35	0.20	0.32
	1570-1574		0.8	96	105	3.4	0.57	0.33	0.54	0.7	30	0.50	0.35
	1575-1579		1.6	96	105	3.4	0.57	0.33	0.54	0.6	30	0.50	0.35
	1581-1585		3.6	112	104	3.8	0.54	0.36	0.54	0.5	35	0.50	0.35
	1586-1590		5.5	124	104	4.3	0.54	0.36	0.54	0.5	35	0.50	0.35
	1591-1594		6.8	127	96	5.0	0.54	0.36	0.54	0.5	35	0.50	0.35
	1608-1611		10.1	124	104	4.3	0.54	0.36	0.54	0.5	35	0.50	0.35
	Sample CR 116-173												
15 & 16	818-823	1500	10.0	83	80	3.4	0.51	0.41	0.59	0.5	40	0.40	0.34
	824-827	1500	3.5	85	86	4.2	0.56	0.41	0.59	0.6	40	0.35	0.31
	836-840	1400	10.0	18	71	0.8	0.50	0.43	0.63	1.5	45	0.45	0.39
	Sample CM 37-57												
17 & 18	652-655	1500	3-1*	94	91	3.6	0.56	0.34	0.57	0.5	40	0.35	0.35
	656-658	1500	10-1	71	77	3.6	0.55	0.34	0.56	0.5	40	0.40	0.38
	711-714	1500	3-2	41	91	1.2	0.54	0.27	0.54	1.5	40	0.35	0.35
	811-817	1500	10-2	65	88	2.2	0.52	0.27	0.54	1.0	43	0.40	0.36
	1014-1017	1450	10	28	80	0.8	0.49	0.27	0.54	1.0	43	0.50	0.41
	862-866	1400	10-1	15	76	0.4	0.49	0.27	0.54	3.0	47	0.35	0.39
	909-913	1400	10-2	16	82	0.4	0.49	0.26	0.55	3.0	47	0.35	0.39
	Sample CL 18-29												
19 & 20	696-697	1700	3	159	108	3.2	0.52	0.21	0.51	0.4	40	0.40	0.39
	679-682	1600	3	54	110	1.6	0.62	0.30	0.59	1.3	38	0.40	0.41
	828-835	1500	10	40	89	1.6	0.57	0.39	0.61	1.5	50	0.30	0.34
	Sample 108												
21 & 22	1208-1215	1500	1.4	55	91	2.1	0.50	0.37	0.53	0.9	30	0.40	0.30
	1261-1267		1.4	55	91	2.1	0.50	0.37	0.53	0.9	30	0.40	0.30
	778-791		3.5	55	86	2.1	0.50	0.36	0.55	1.0	35	0.35	0.32
	1200-1207		3.5	55	86	2.1	0.50	0.36	0.55	1.0	35	0.35	0.32
	1252-1260		3.5	55	86	2.1	0.50	0.36	0.55	1.0	35	0.35	0.32
	1216-1225		5.5	50	85	1.1	0.46	0.21	0.49	0.9	30	0.40	0.35
	1243-1251		5.5	50	85	1.1	0.46	0.21	0.49	0.9	30	0.40	0.35

* A curve designation for figures.

(1) Fig.	(2) Run	(3) t	(4) u	(5) w	(6) Y_o	(7) ϕ	(8) ϕ_o	(9) $d\phi/dY$	p_{100}	θ_{PS}	$Y_{max\ PS}$	ϕ_{PS}
21 and 22 (cont)	1228-1242	1500	6.6	47	88	2.3	0.50	0.36	0.55	0.75	32	0.35 0.36
	775-777		10	69	86	2.1	0.50	0.29	0.53	0.75	36	0.55 0.32
	792-800		10	69	86	2.1	0.50	0.29	0.53	0.75	36	0.35 0.32
			Sample CR 187-201, 206-214, 218-220, 237-251.									
			Curve A col. 5 all samples except 187, 196.									
			Curve A col. 11 samples 187, 237, 240.									
			Curve C col. 11 samples 193, 199, 218.									
			Curve B col. 11 remainder.									
23 and 24	913-48	1400	10.0	A25	80	A 0.7	0.49	A 0.30	0.55	A 1.0	40	0.55 0.42
	961-92,			B25	84					B 1.75	45	0.32 0.34
	998-1004,									C 1.5	42	0.45 0.38
	1020-25											
			Sample CR 61-65, 71-73, 75-80.									
			Curve B col. 5 samples 61, 73, 75, 79.									
			Curve C col. 5 sample 65.									
			Curve A col. 5 remainder.									
			Curve B col. 7 samples 64, 65, 80.									
			Curve A col. 7 remainder (not 61, 65, 71, 72).									
			Curve C col. 7 samples 61, 63, 71, 72.									
			Curve B, col. 9 samples 64, 65, 80.									
			Curve C col. 9 samples 61, 63, 71, 72.									
			Curve A col. 9 remainder.									
			Curve B col. 11 samples 75-78.									
			Curve C col. 11 sample 62.									
			Curve D col. 11 samples 64, 71, 79, 80.									
			Curve A col. 11 remainder.									
25 and 26	1029-33	1450	10.0	A33	90	A 1.3	0.57	A 0.42	0.64	A 1.25	43	0.55 0.43
	1042-59			B33	85	B 1.0	0.56	B 0.30	0.60	B 1.50	47	0.35 0.38
	1065-73			C33	94	C 0.7	0.51	C 0.20	0.53	C 1.50	40	0.40 0.38
	1078-94									D 1.75	52	0.35 0.36
	1098-1105											
	1119-22.											

Table III
Average Values of Activation Variables determined from Figures 1-25 (odd numbers)

Fig No.	Ave. Yield Extrapolated to 0 time		Average AD Extrapolated to 0 time		Average AD Extrapolated to 100 %		Average AD-Yield Slope	
1	114		0.65		0.59		0.40	
3	106		0.59		0.56		0.39	
5	103	Ave	0.57	Ave	0.55	Ave	0.37	Ave
7	107	106	0.56	0.58	0.56	0.56	0.39	0.38
9	102		0.54		0.53		0.33	
11	110		0.60		0.59		0.44	
13	103		0.55		0.54		0.35	
15	79		0.52		0.60		0.42	
17	84	Ave	0.52	Ave	0.55	Ave	0.29	Ave
21	85	83	0.49	0.51	0.53	0.56	0.32	0.32
23	82		0.49		0.55		0.30	
25	87		0.55		0.59		0.31	
19	102		0.57		0.57		0.30	

Table IV
Change of Gasification Rate with Temperature

t_F	t_C	T	1/T	G	log G
1450	788	1061	0.000943	15	1.176
1500	816	1089	0.000917	35	1.544
1550	843	1116	0.000897	43-74	1.633-1.869
1600	871	1144	0.000873	80	1.903
1650	899	1172	0.000854	104	2.017

Slope taken from plot on Figure 33.

$$\frac{2.0 - 1.2}{(8.70 - 9.47)10^{-4}} = -\frac{A}{4.575}$$

$$A = 47500 \text{ cal./mol.}$$

Table V

Composition of Gases Evolved Compared with Water Gas Equilibrium

Sample	Temp.	% Steam Decomposed	CO/CO_2 (observed)	CO/CO_2 (Mayers) on graphite	CO/CO_2 (equilibrium)
CM	1600	13	0.52	0.5	0.3
CL	1700	22	0.90	1.0	0.4
PCI	1600	8	2.0		0.1
PCI	1600	9	2.7		0.1
PCI	1700	13	2.2		0.2

Figure 1

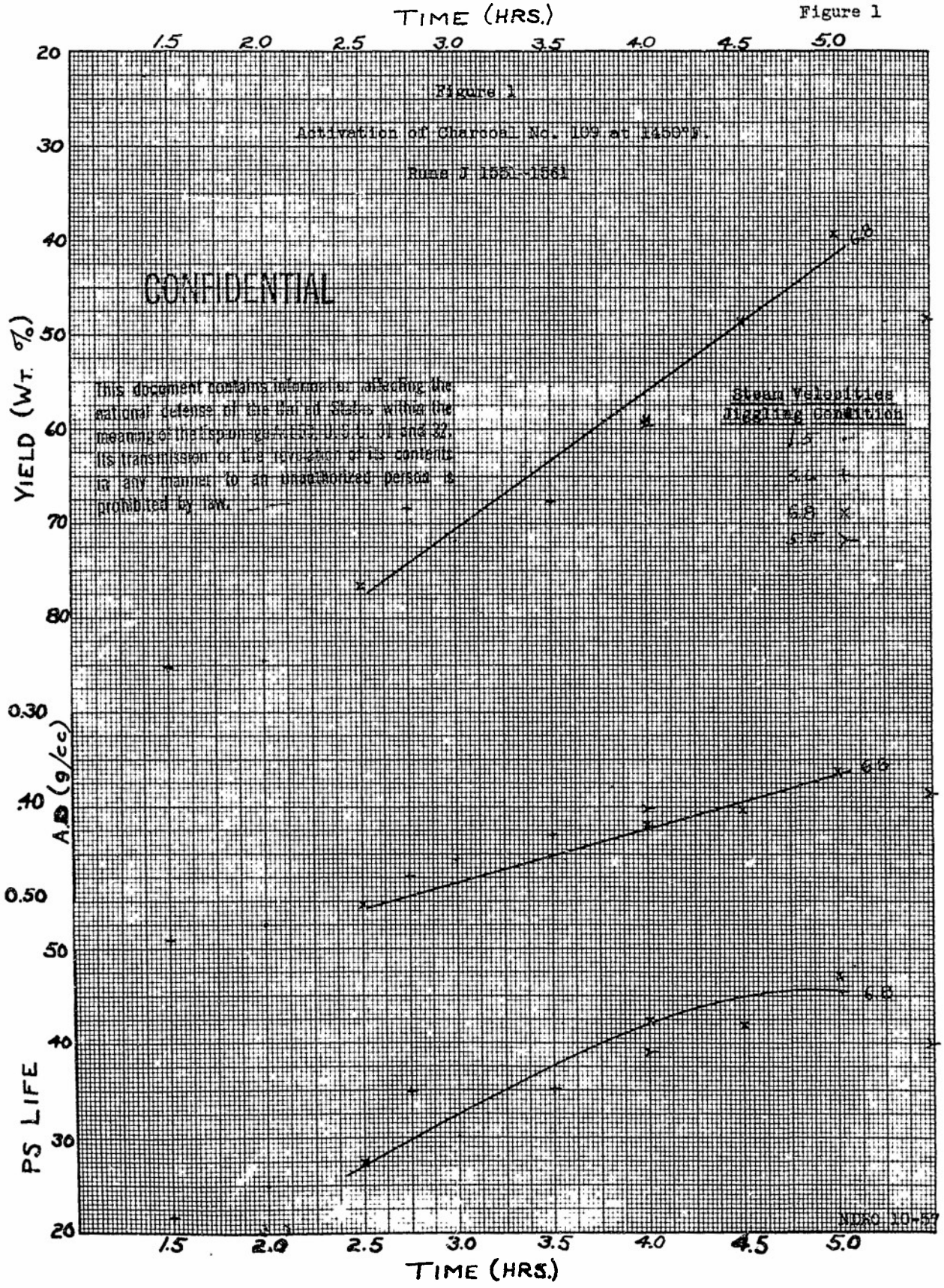
Figure 1
Activation of Charcoal No. 109 at 1450°F.
Runs J 1561-1661
CONFIDENTIAL
This document contains information affecting the national defense of the United States within the meaning of the Espionage Act, U.S.C. 50 and 32. Its transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.
Steam Velocities Heating Condition
TIME (HRS.)
YIELD (Wt. %)
PS LIFE
A.D. (g/cc)
NUKO 20-57

Figure 2

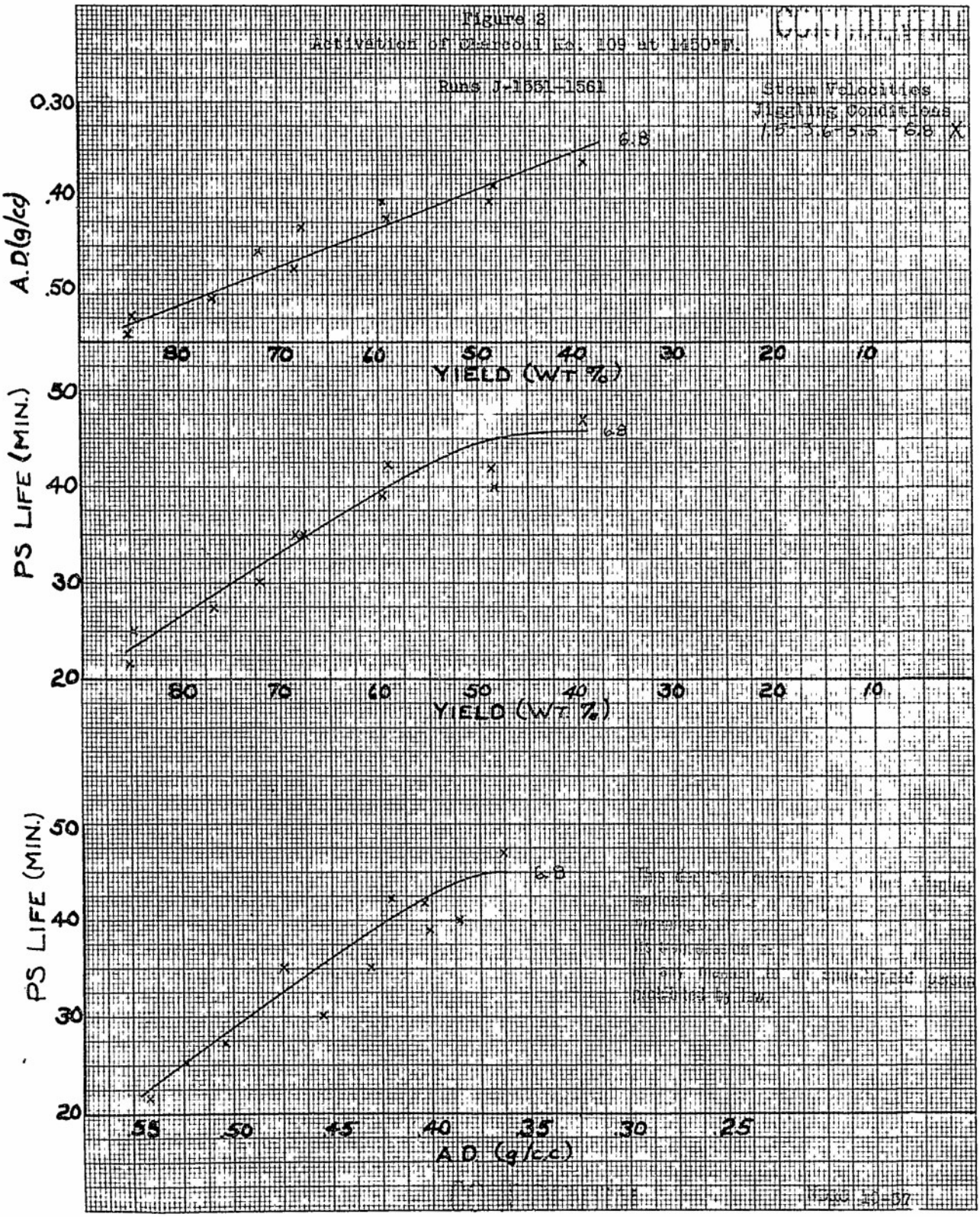
Figure 2
Activation of Wilcoal Lo. 109 at 1450°F.
Runs J-1551-1561
Steam Velocities
Blasting Conditions
1.5 - 3.6 - 5.5 - 6.8 X
A.D(g/cc)
0.30
.40
.50
6.8
80 70 60 50 40 30 20 10
YIELD (WT %)
PS LIFE (MIN.)
50
40
30
20
6.8
80 70 60 50 40 30 20 10
YIELD (WT %)
PS LIFE (MIN.)
50
40
30
20
6.8
55 50 45 40 35 30 25
A.D. (g/c.c)

Figure 3

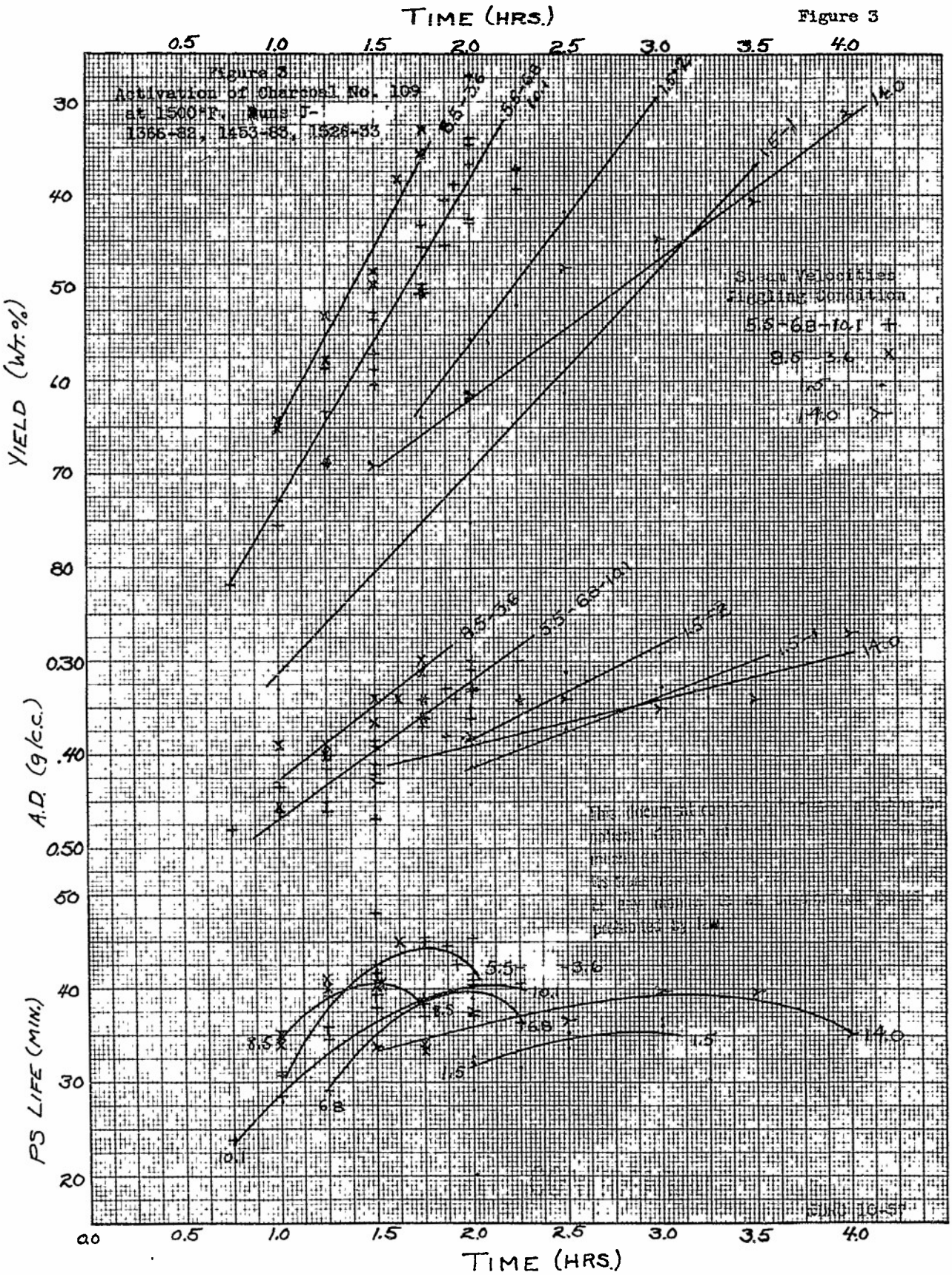
Figure 3
Activation of Charcoal No. 109
at 1500°F. Runs T-
1366-82, 1453-85, 1526-33
TIME (HRS.)
YIELD (WT.%)
A.D. (g/c.c.)
PS LIFE (MIN.)
Steam Velocities
Fluidizing Condition
5.5-6.8-10.1
3.5-3.6
14.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
20
30
40
50
60
70
80
0.30
.40
0.50
60

Figure 4

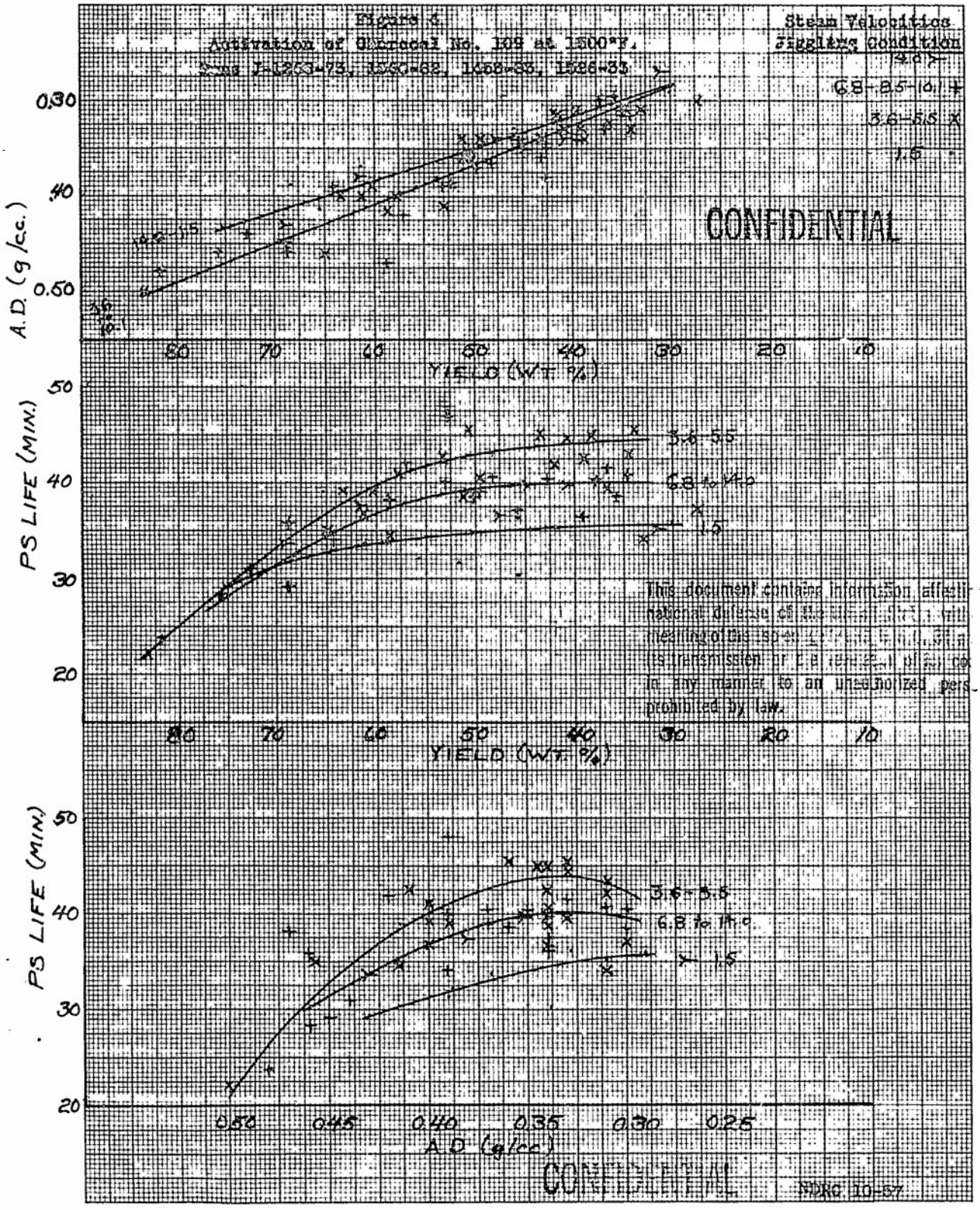

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Figure 5

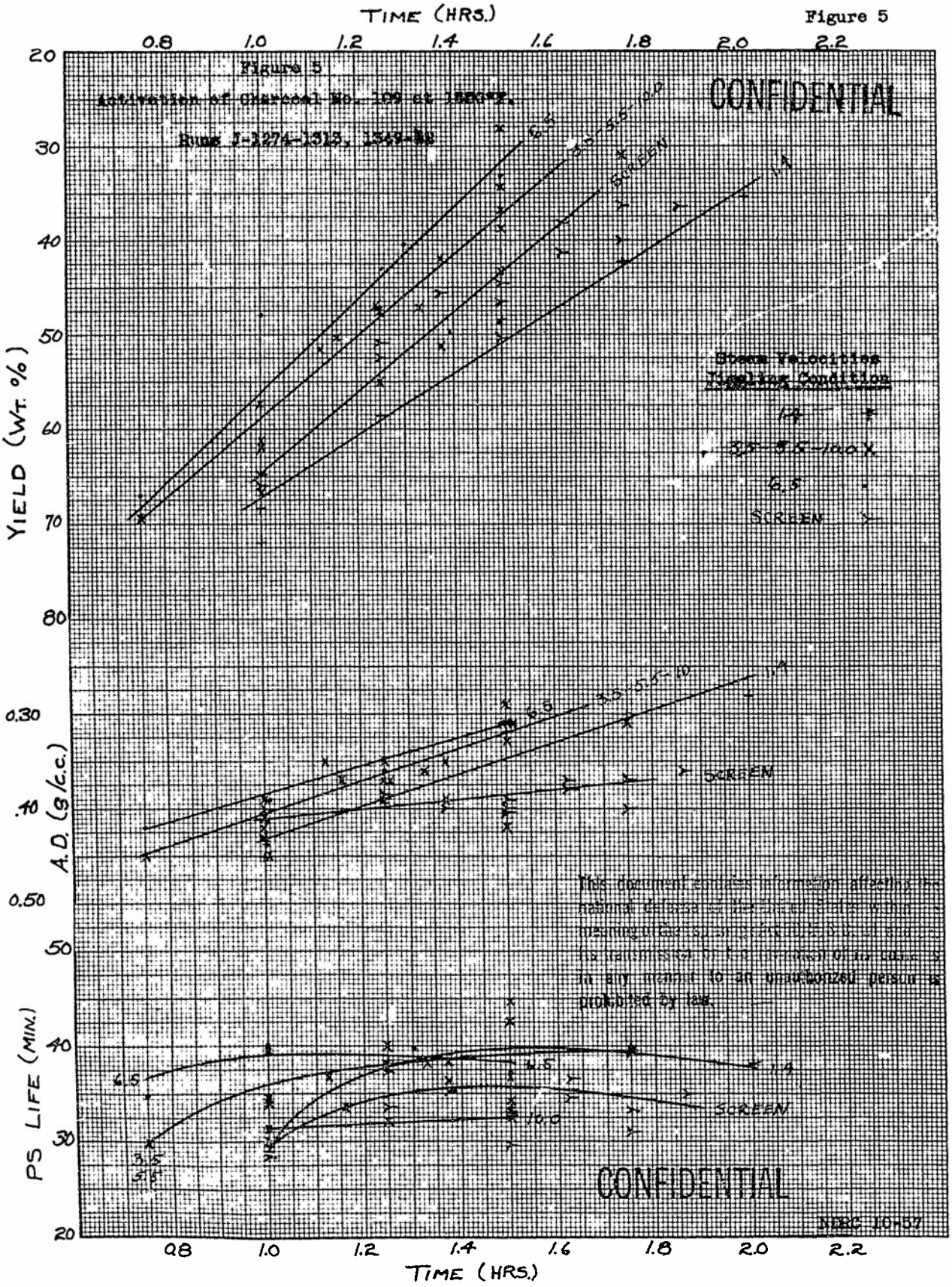
Figure 5
Activation of Charcoal No. 109 at 1580°F.
Runs J-1274-1312, 1349-58
CONFIDENTIAL
Steam Velocities
Fluidizing Condition
1.4
3.5-5.5-10.0
6.5
SCREEN
YIELD (WT. %)
A.D. (g/c.c.)
PS LIFE (MIN.)
TIME (HRS.)
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
20
30
40
50
60
70
80
0.30
.10
0.50
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Figure 6

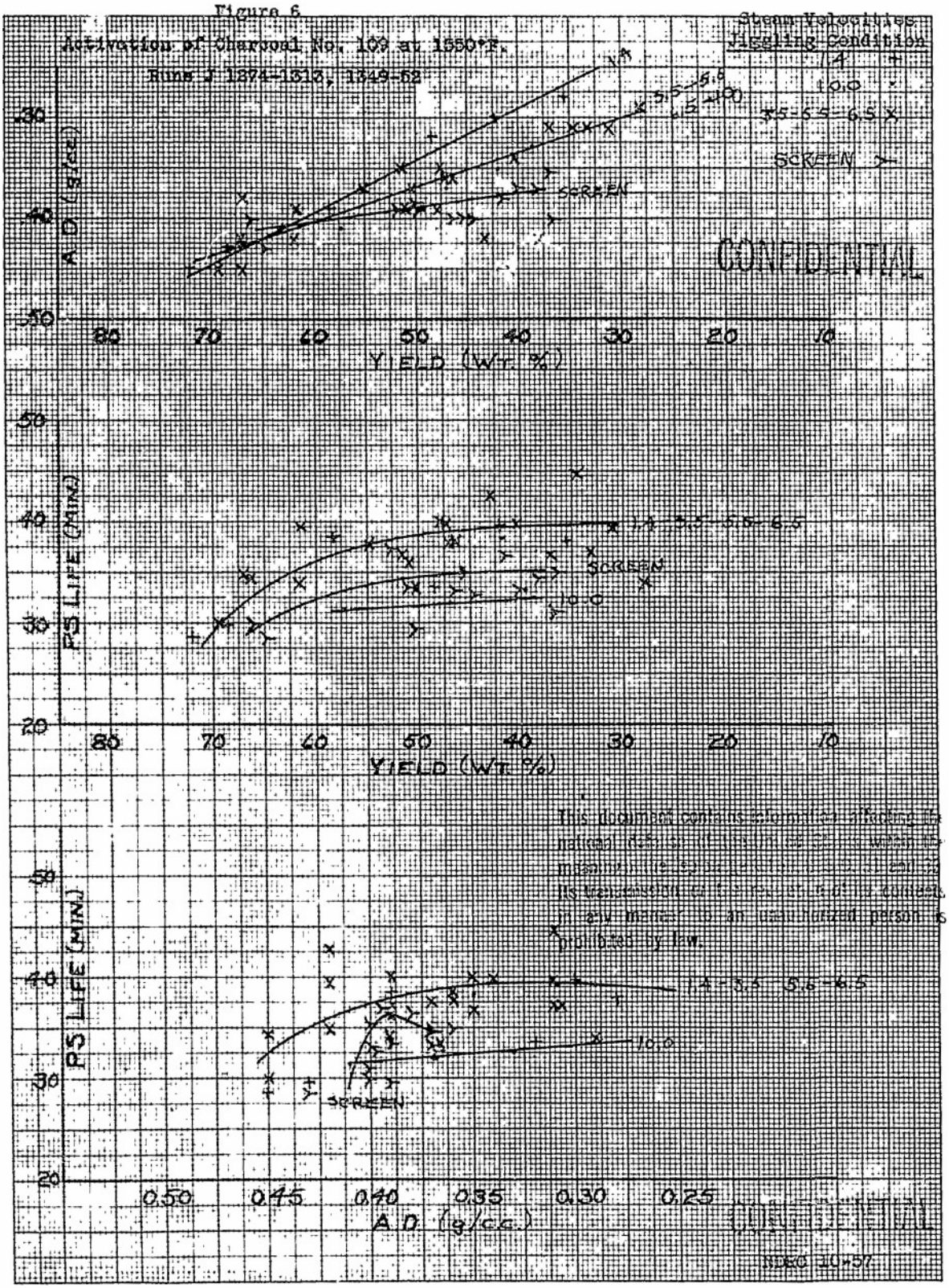

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Figure 7

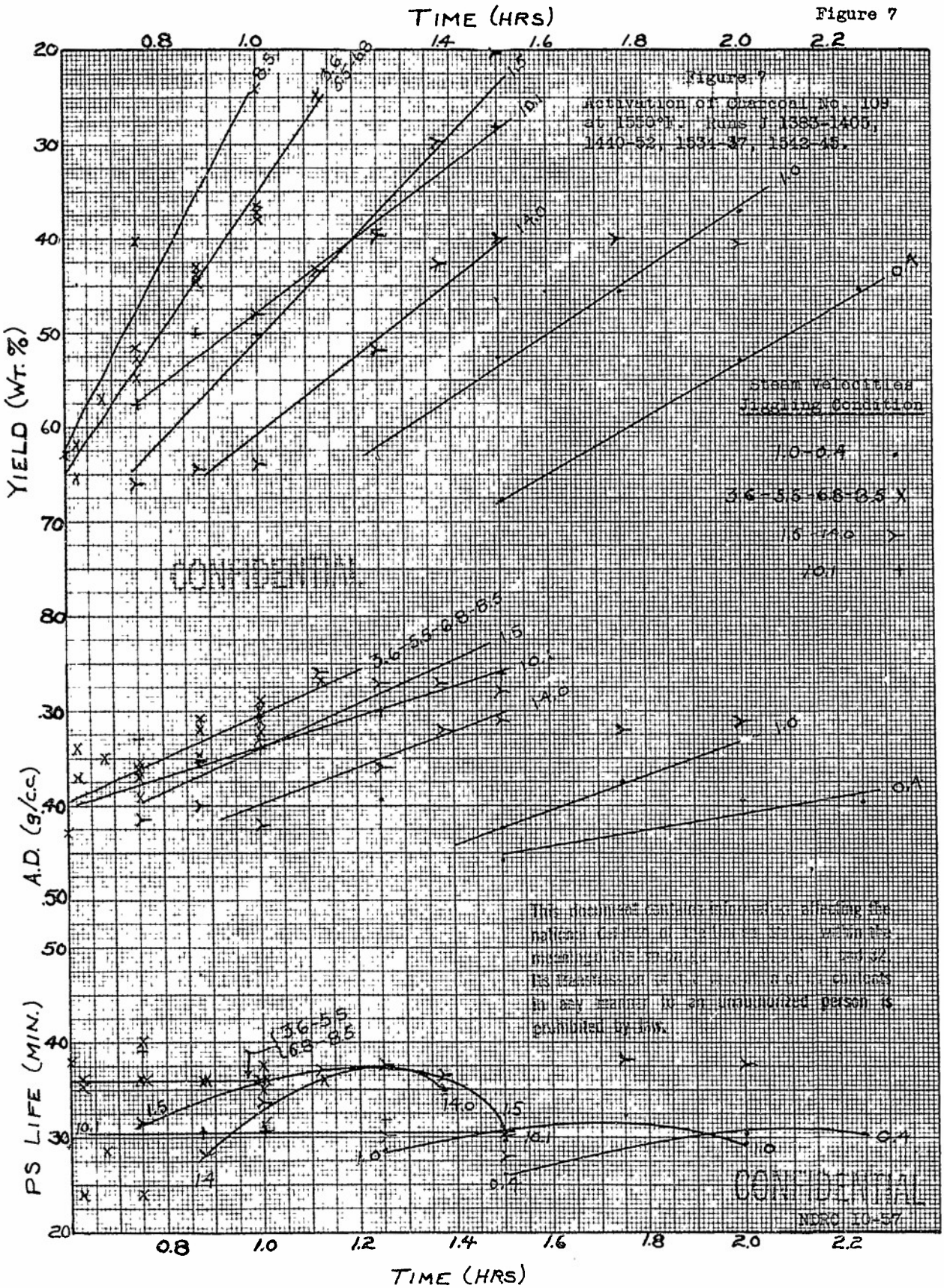
Figure 7
Activation of Charcoal No. 109 at 1550°F., runs 1383-1405, 1440-52, 1531-37, 1542-46.
Steam Velocities at Flooding Condition
1.0-0.4
3.6-5.5-6.6-8.5
1.5 1.0
10.1
0.4
YIELD (WT. %)
A.D. (g/c.c.)
PS LIFE (MIN.)
TIME (HRS)
TIME (HRS)
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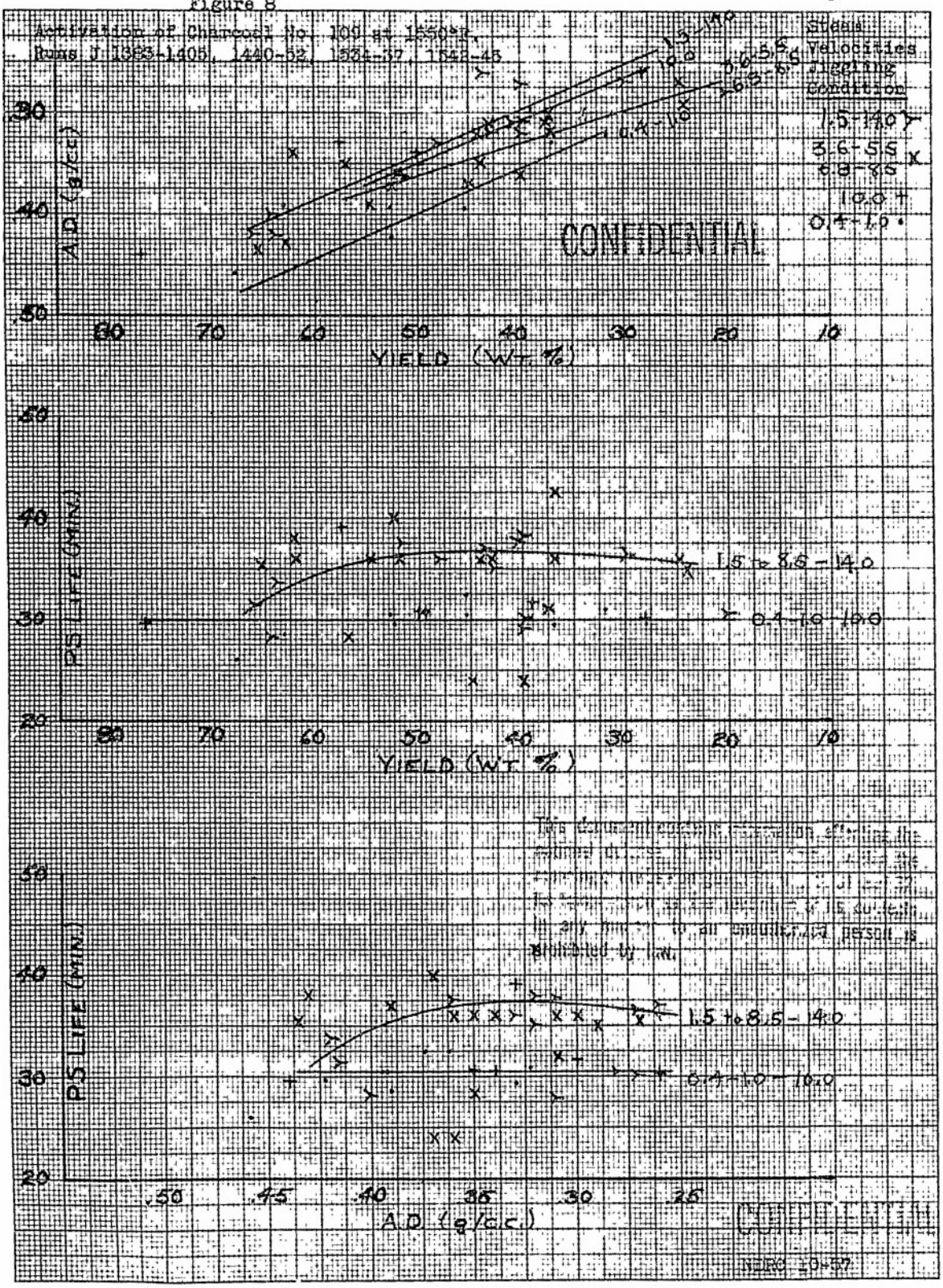
Figure 8


Activation of Charcoal No. 100 at 1550°F.
Runs J 1363-1405, 1440-52, 1534-37, 1542-45
Steam Velocities Plugging condition
1.5-14.0 Y
3.6-5.5 S
6.8-8.5 X
10.0 +
0.4-1.0 •
CONFIDENTIAL
AD (g/cc)
YIELD (WT. %)
DISTIL. (MIN.)
1.5 - 8.5 - 14.0
0.4 - 1.0 - 10.0
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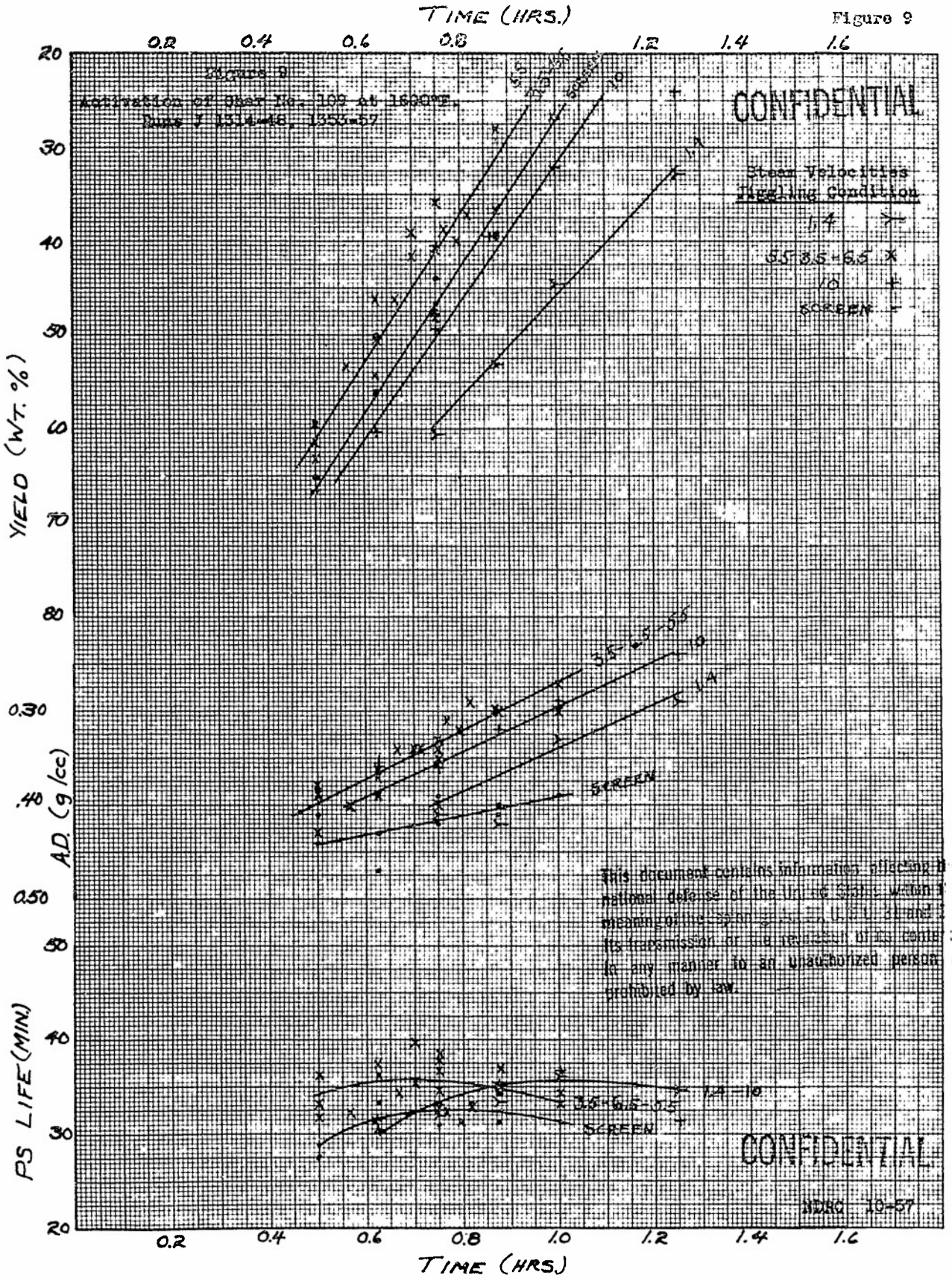
NAVEXOS 2052

Figure 9


TIME (HRS.)
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
Figure 9
Agitation of Heat No. 109 at 1600°F.
Runs I 1314-48, 1350-57
CONFIDENTIAL
Steam Velocities
Toggling Condition
1.4
5.5 3.5-6.5
1.0
SCREEN
YIELD (WT. %)
20
30
40
50
60
70
80
0.30
.40
0.50
A.D. (g/cc)
PS LIFE (MIN)
20
30
40
50
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1.4-1.0
3.5-6.5-5.5
SCREEN
CONFIDENTIAL
NDRC 10-57
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
TIME (HRS.)

Figure 10

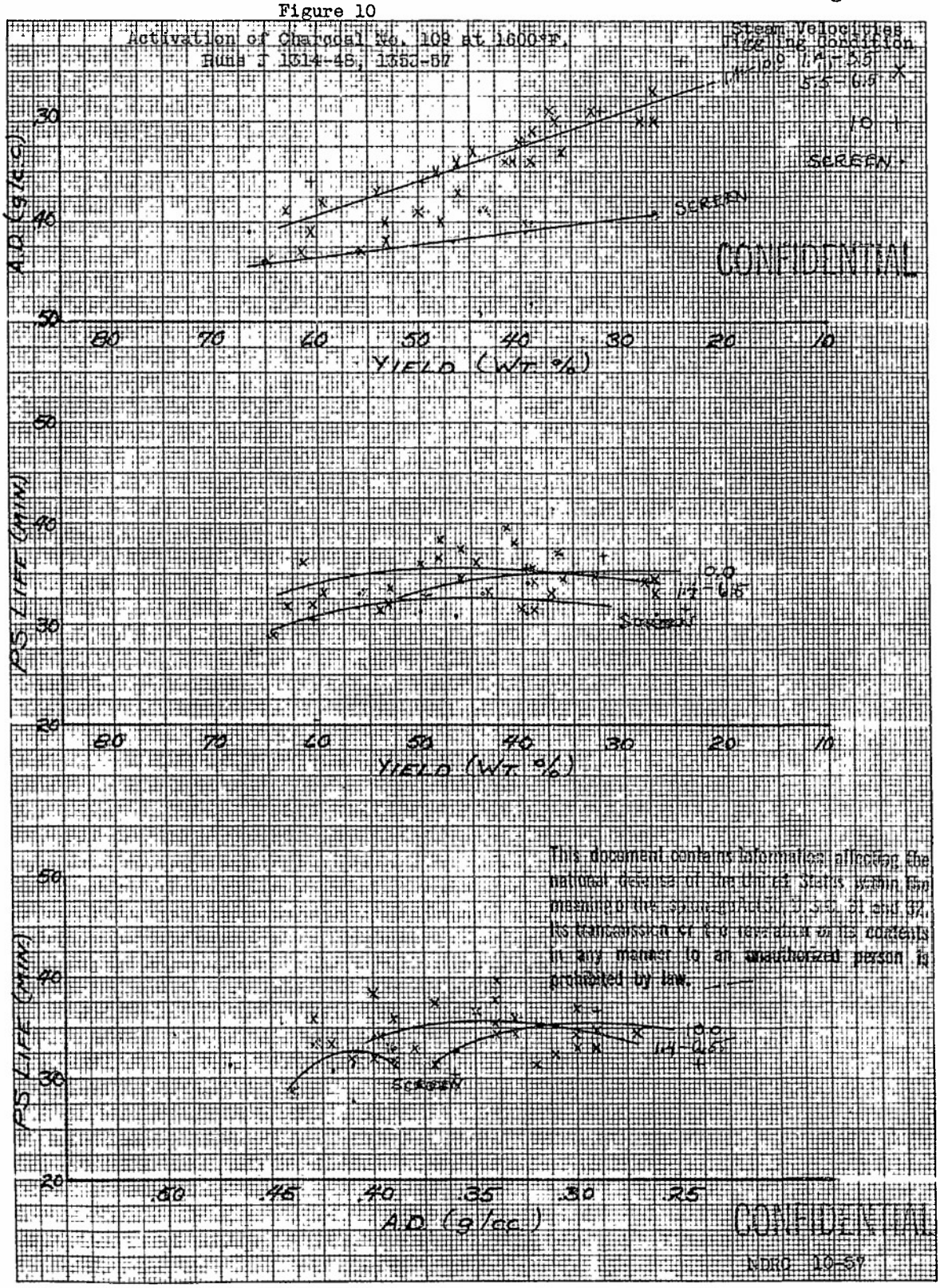
Activation of Charcoal No. 109 at 1600°F.
Runs J 1514-48, 1552-57
Steam Velocities
Wetting Condition
5.5 - 6.5
SCREEN
AD (g/cc)
YIELD (WT %)
PS LIFE (MIN)
0.0
1.4 - 6.5
Screen
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Figure 11

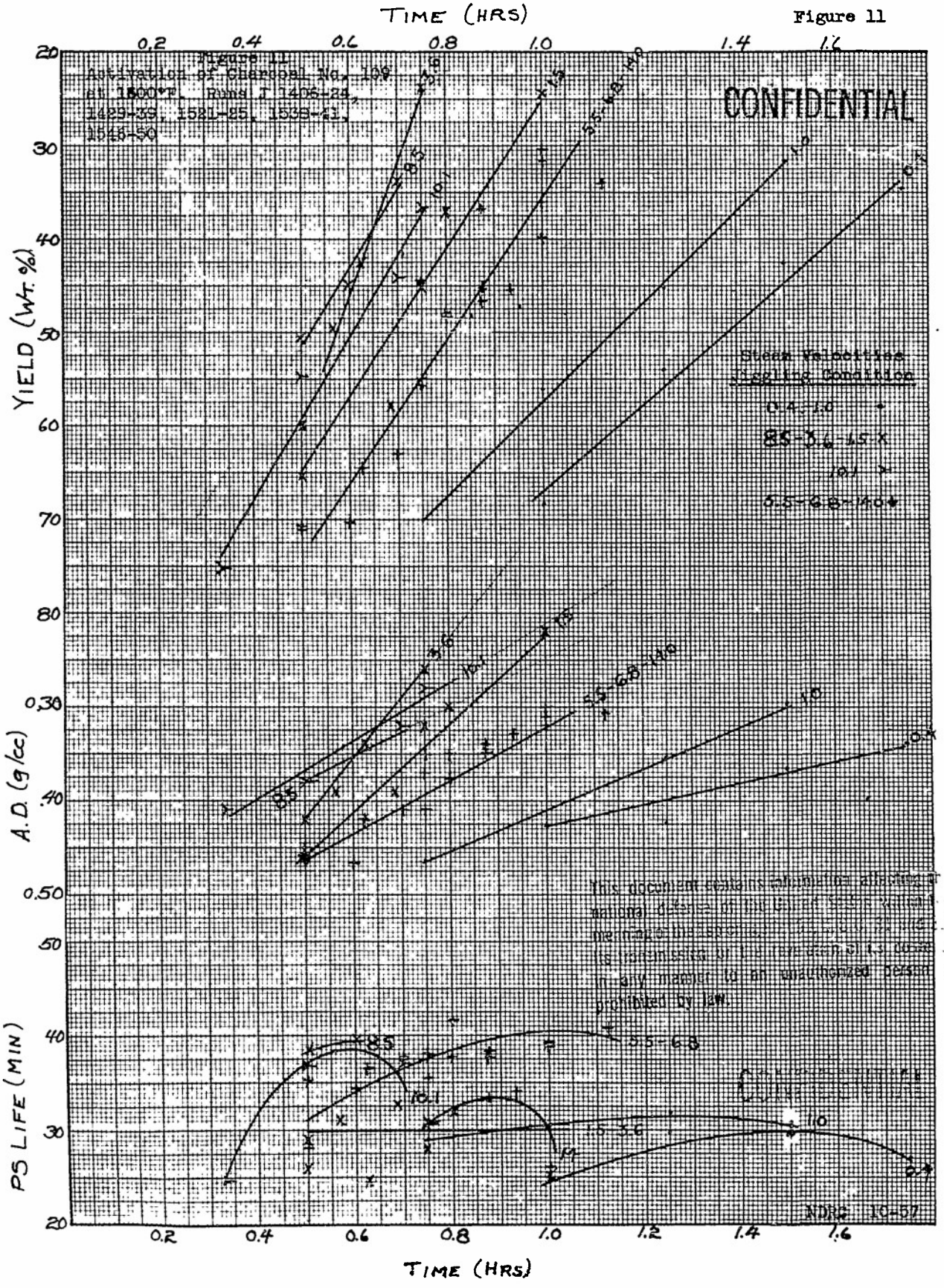
Figure 11
Activation of Charcoal No. 109
at 1800°F. Runs J 1406-24,
1429-39, 1521-25, 1538-41,
1546-50
CONFIDENTIAL
Steam Velocities
Yielding Conditions
0.4-1.0
8.5-3.6-4.5
10.1
5.5-6.8-14.0
TIME (HRS)
YIELD (WT. %)
A.D. (g/cc)
PS LIFE (MIN)
TIME (HRS)
NDRC 10-57
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Figure 12

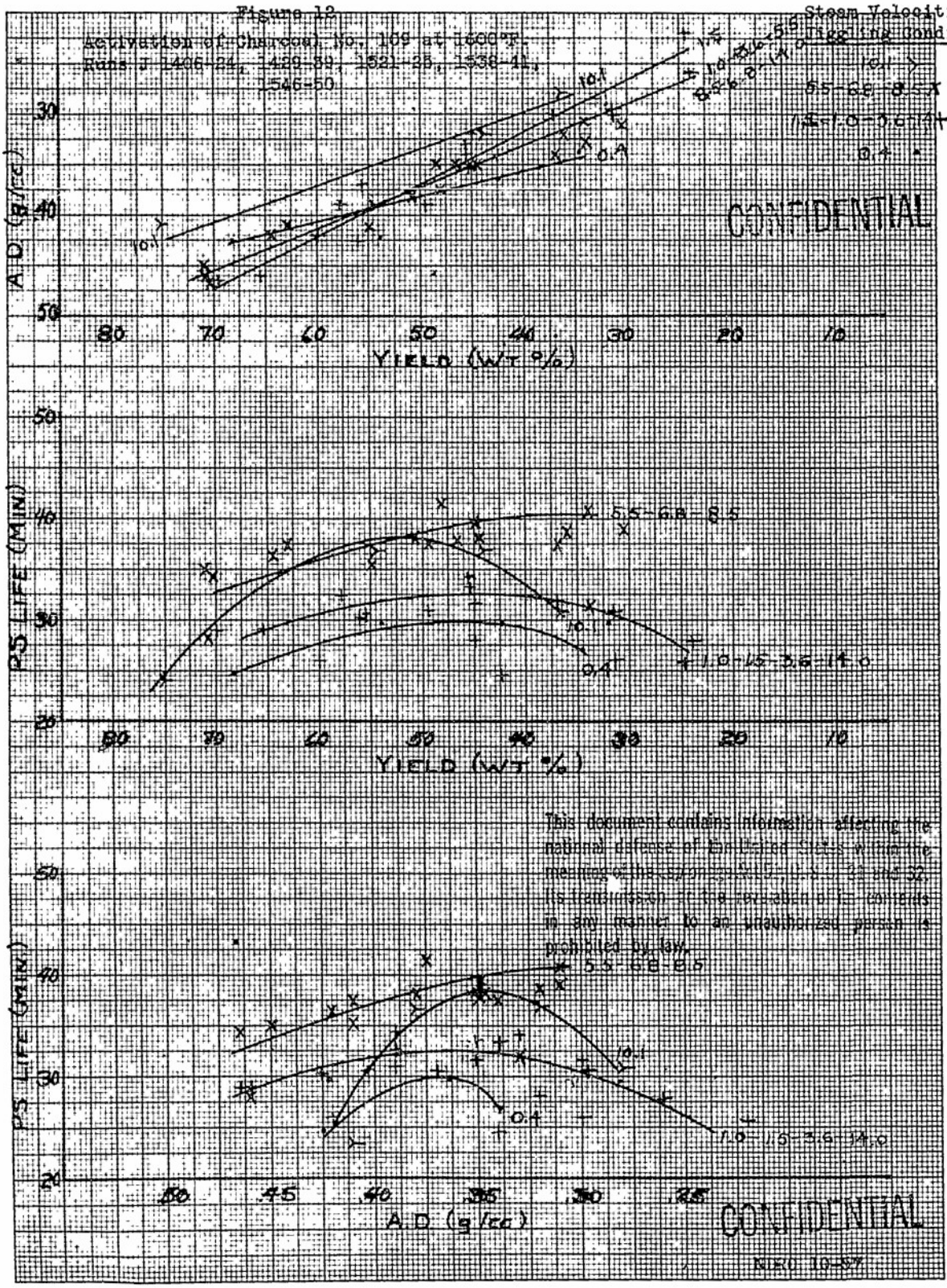
Figure 12
Activation of Charcoal No. 109 at 1600°F.
Runs J-1406-24, 1429-39, 1521-25, 1538-41, 1546-50
Steam Velocities
Rigging Condition
5.5-6.8-8.5
1.0-1.5-3.6-14.0
0.4
AD (g/cc)
PS LIFE (MIN)
YIELD (WT %)
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Figure 13

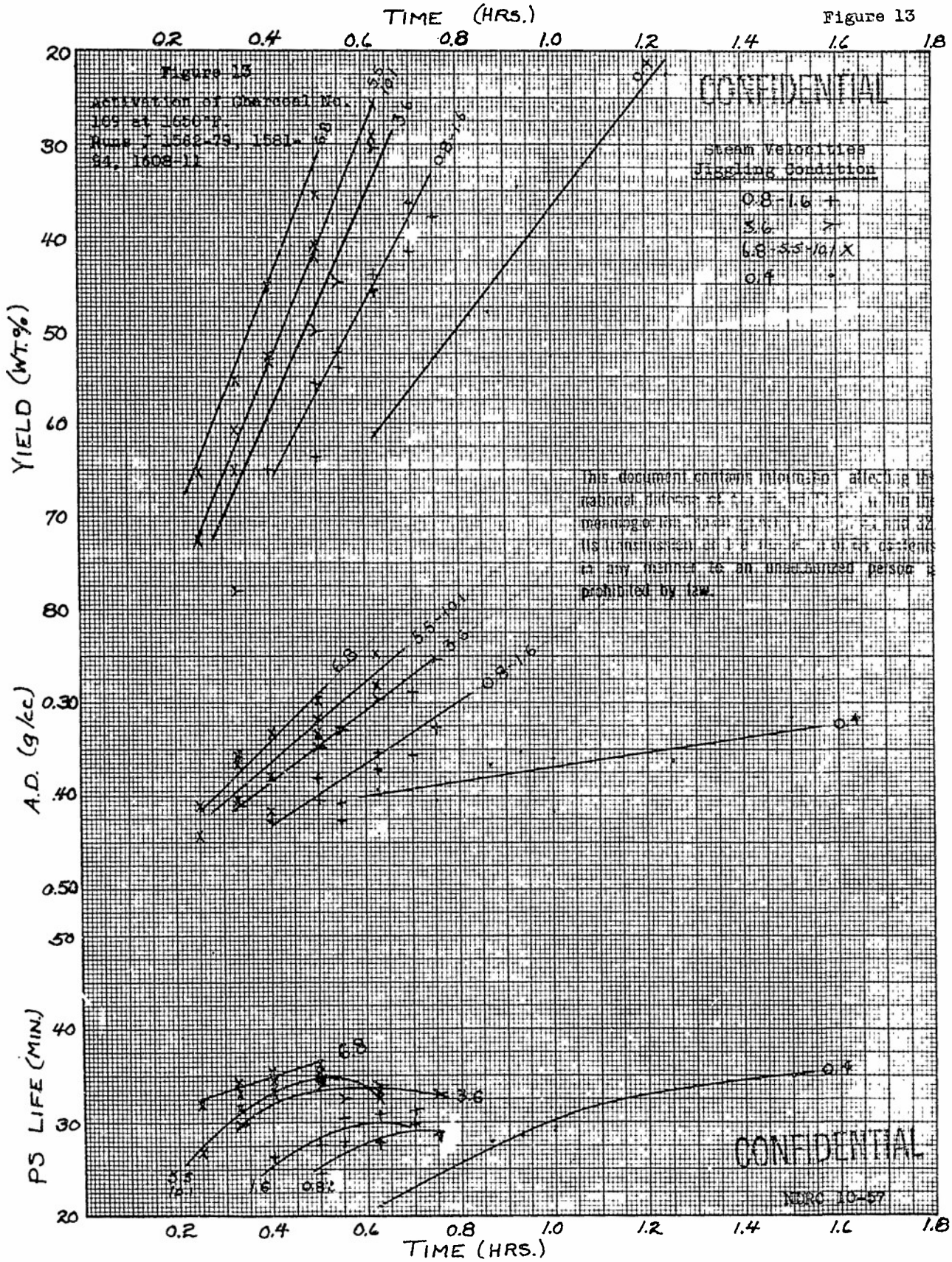
Figure 13
Activation of Charcoal No. 109 at 1650°F.
Runs 7, 1582-78, 1581-6, 94, 1608-11
CONFIDENTIAL
Steam Velocities
Jigging Condition
0.8-1.6 +
3.6 ▽
6.8-5.5-10.1 X
0.4 •
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TIME (HRS.)
YIELD (WT.%)
A.D. (g/cc)
PS LIFE (MIN.)
CONFIDENTIAL
NDRC 10-57

Figure 14

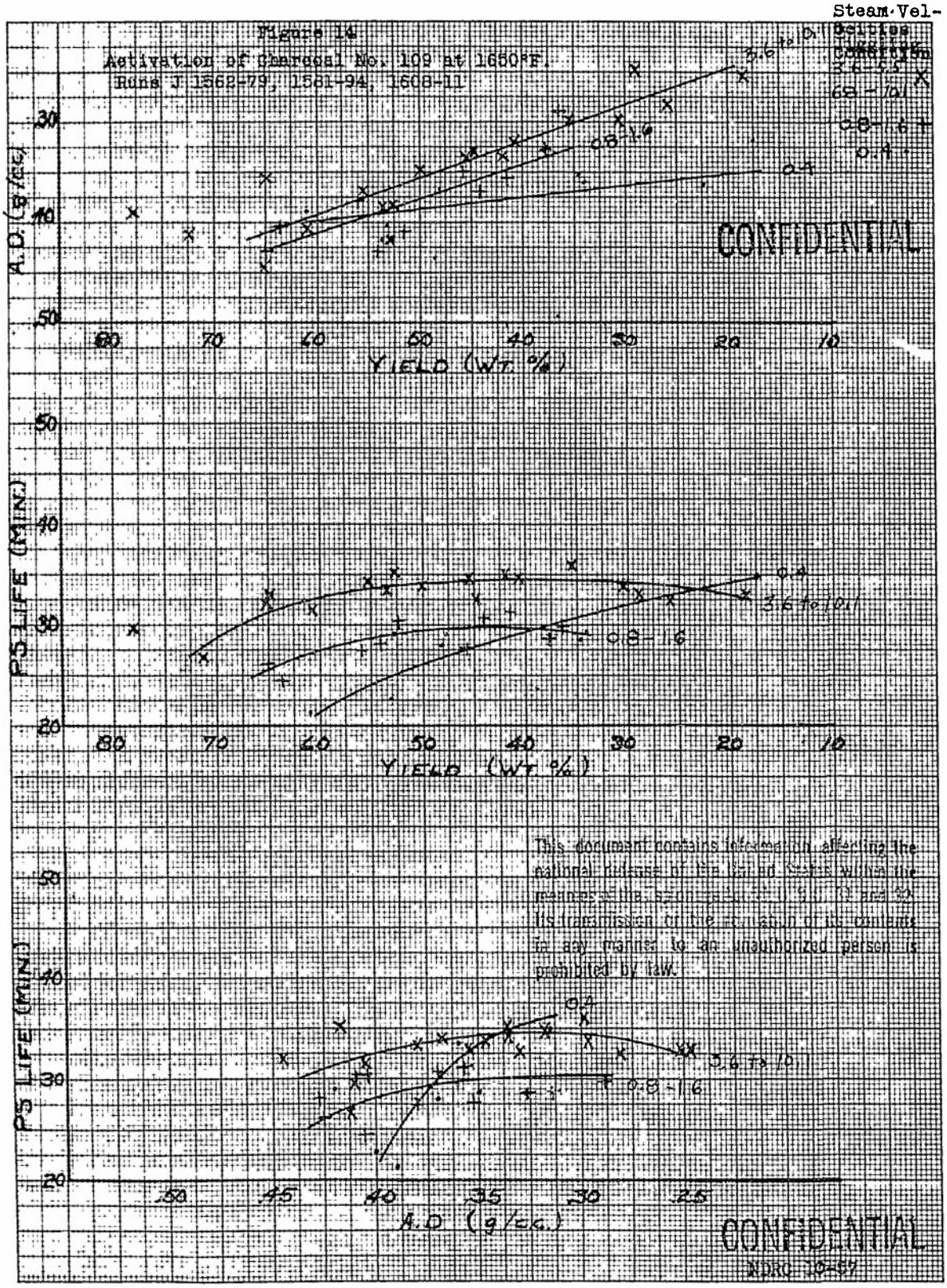
Figure 14
Activation of Charcoal No. 109 at 1650°F.
Runs J 1562-79, 1581-94, 1608-11
Steam Velocities
Superficial
Contacting
3.6 - 5.5
6.8 - 10.1
0.8 - 1.6
0.4
3.6 to 10.1
0.8 - 1.6
0.4
3.6 to 10.1
0.8 - 1.6
PS LIFE (MIN.)
A.D. (g/cc.)
YIELD (WT. %)
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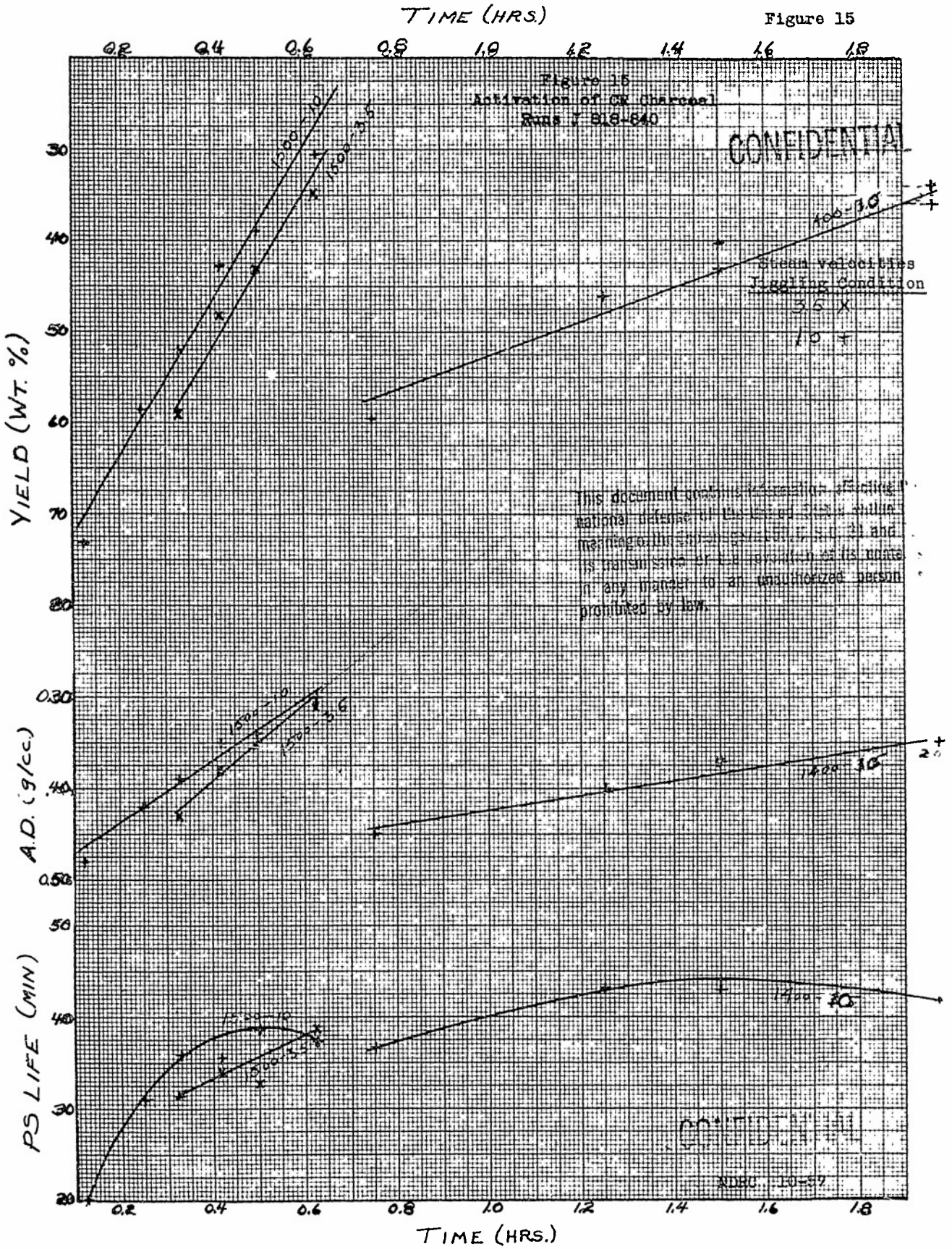
Figure 15


TIME (HRS.)
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
Figure 15
Activation of CK Charcoal
Runs J 818-840
CONFIDENTIAL
Steam velocities
Toggling condition
3.5 X
1.0 +
YIELD (WT. %)
A.D. (g/cc.)
PS LIFE (MIN)
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TIME (HRS.)

Figure 16

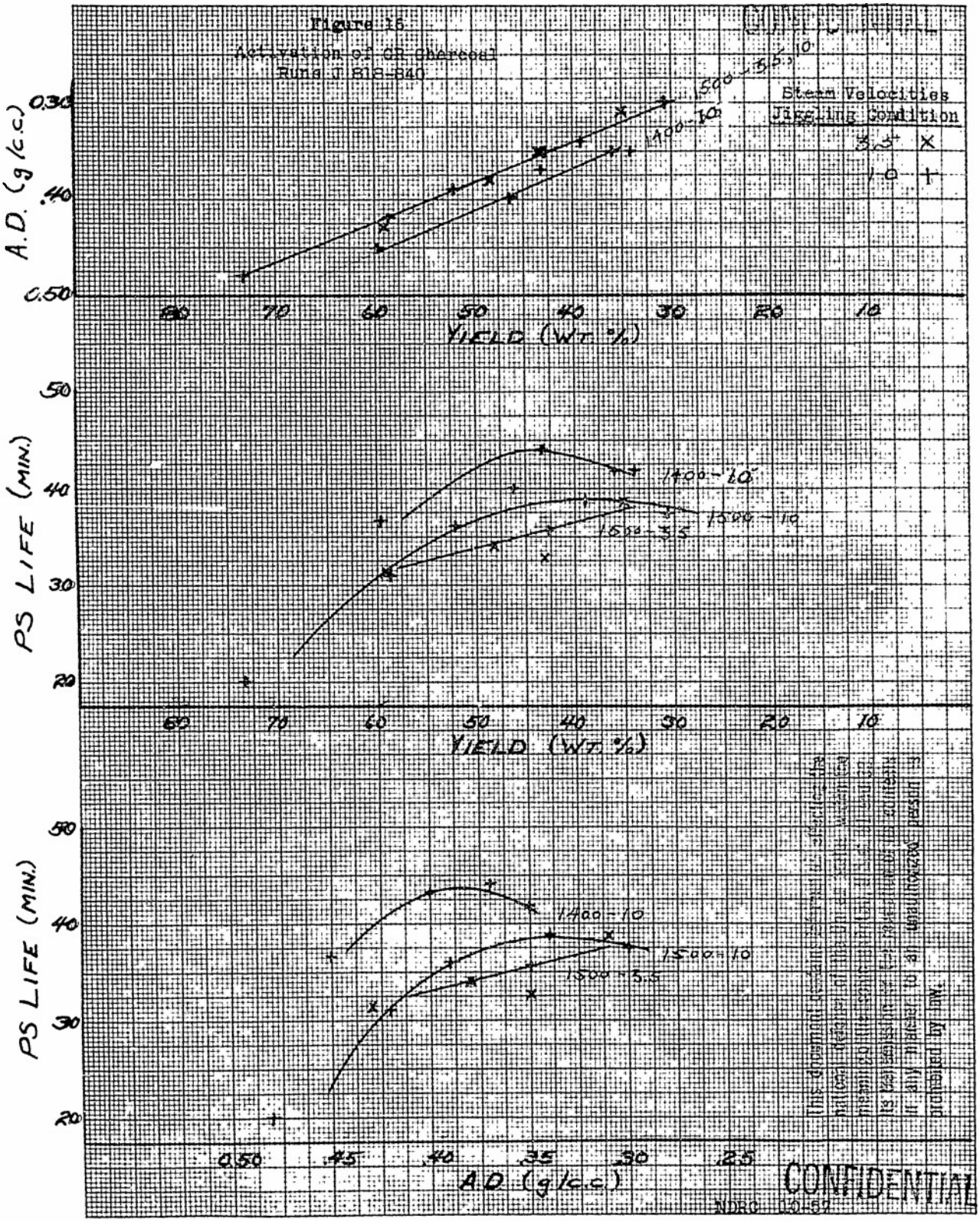
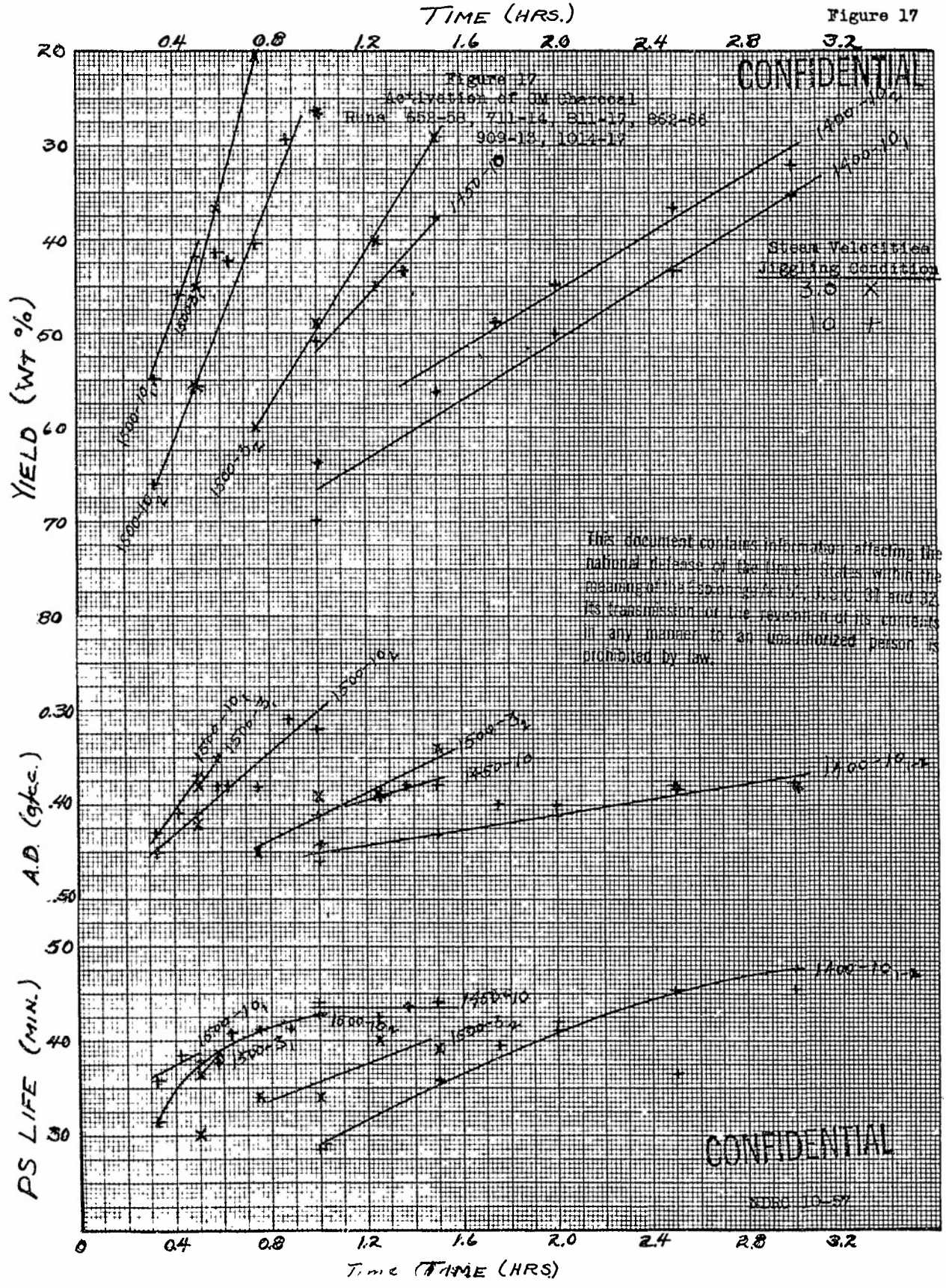


Figure 16
Activation of GR Charcoal
Runs J 818-840
Steam Velocities
Jacketing Condition
3.5 X
1.0 +
1400-10
1500-10
1500-3.5
A.D. (g/c.c.)
PS LIFE (MIN.)
YIELD (WT %)
0.30
0.40
0.50
60
70
50
40
30
20
10
45
35
25
CONFIDENTIAL
NDRC 05-57

Figure 17


TIME (HRS.)
0.4
0.8
1.2
1.6
2.0
2.4
2.8
3.2
CONFIDENTIAL
Figure 17
Activation of GM Charcoal
Runs 558-58, 711-14, 811-17, 862-66
909-18, 1014-17
Steam Velocities
Jiggling condition
3.0 X
1.0 +
YIELD (WT %)
A.D. (gkc.)
PS LIFE (MIN.)
20
30
40
50
60
70
80
0.30
.40
.50
30
40
0
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NEDRC 102-57
Time (TIME (HRS)

Figure 18

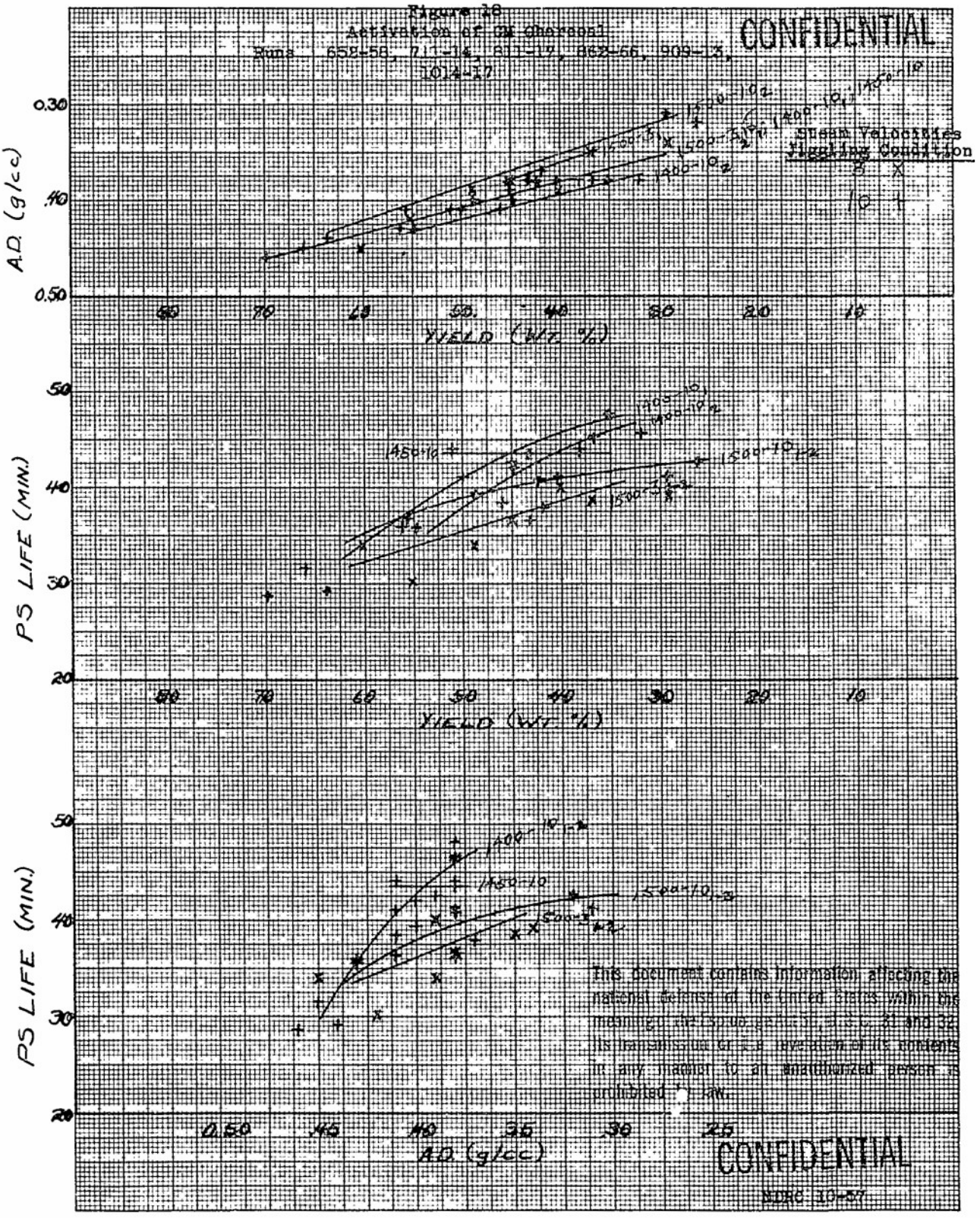
Figure 18
Activation of GX Charcoal
Runs 652-58, 711-14, 811-17, 862-66, 909-13, 1012-17
CONFIDENTIAL
Steam Velocities
Toggling Condition
A.D (g/cc)
YIELD (WT. %)
PS LIFE (MIN.)
YIELD (WT. %)
PS LIFE (MIN.)
A.D (g/cc)
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NDRC 10-57

Figure 19

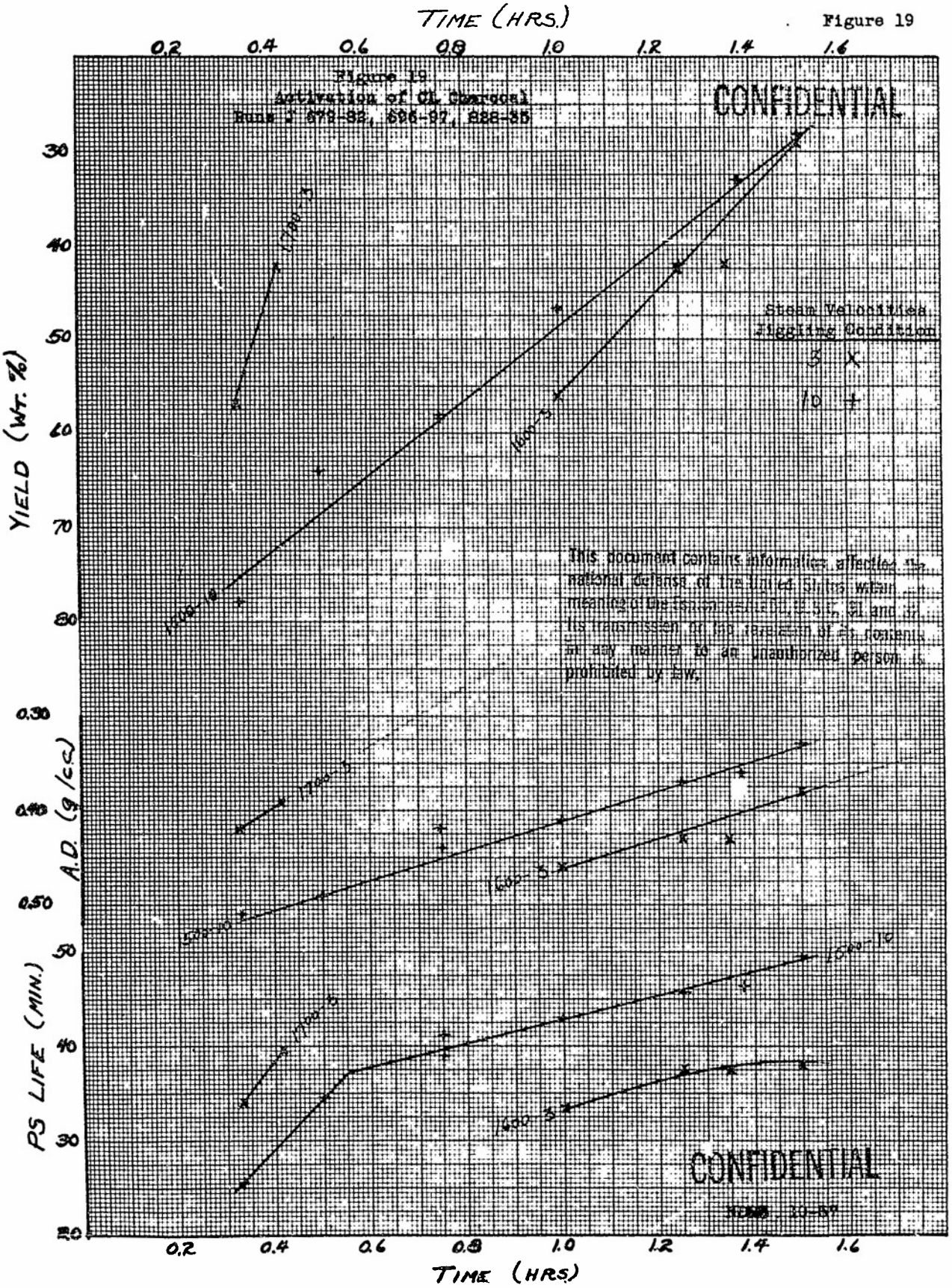
Figure 19
Activation of CL Charcoal
Runs : 879-82, 896-97, 828-85
CONFIDENTIAL
Steam Velocities
Fluidizing Conditions
3 X
10 +
1700-10
1700-3
1700-5
1600-5
1600-10
1600-3
1700-5
YIELD (WT. %)
A.D. (g/cc)
PS LIFE (MIN.)
TIME (HRS.)
TIME (HRS)
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
30
40
50
60
70
80
0.30
0.40
0.50
50
40
30
20
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Figure 20

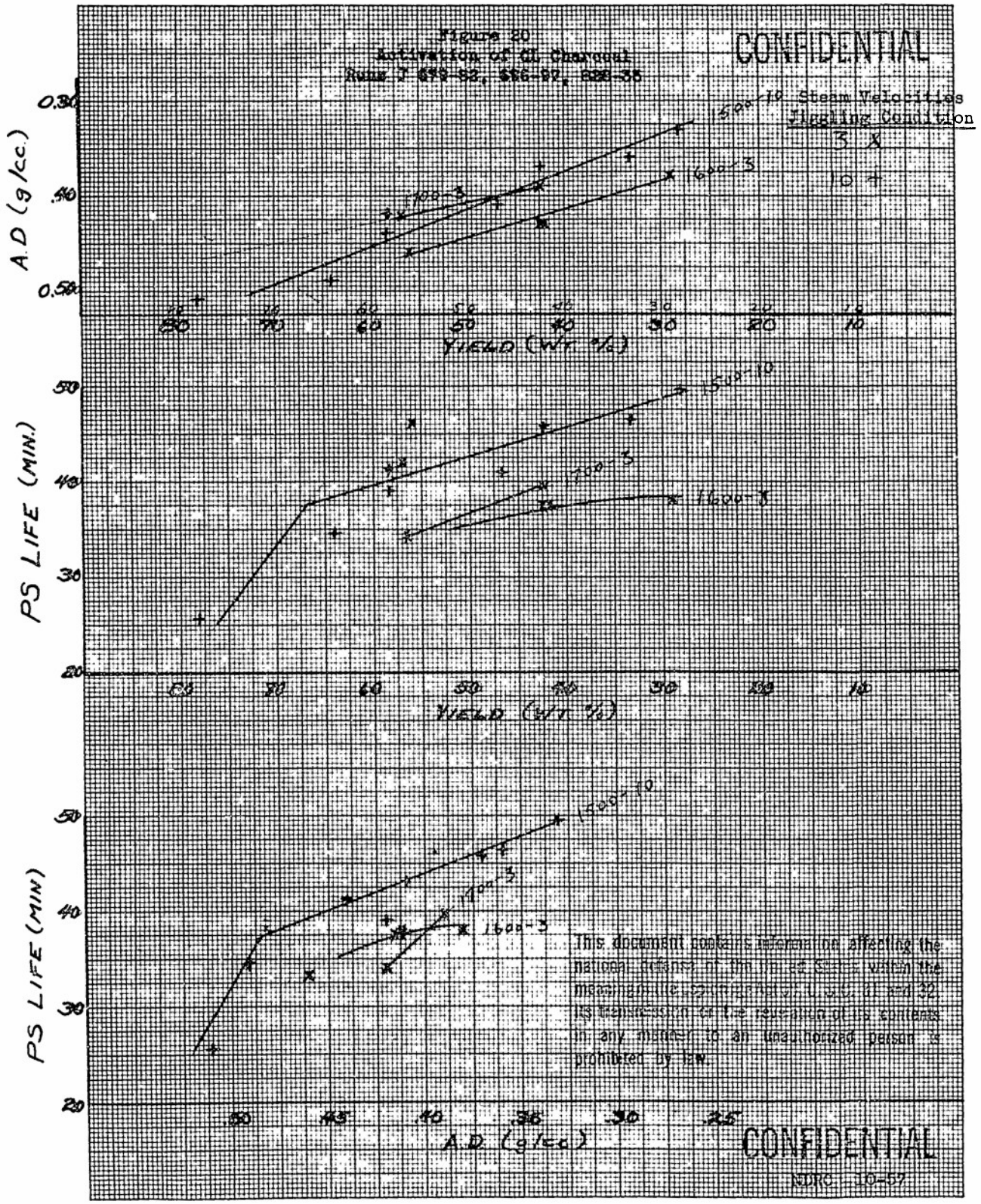
Figure 20
Activation of GL Charcoal
Runs T 618-82, 886-92, 828-38
CONFIDENTIAL
1500-10 Steam Velocities
Flooding Condition
3 X
10 +
1600-3
1400-3
A.D (g/cc.)
0.30
.40
0.50
YIELD (WT %)
PS LIFE (MIN.)
50
40
30
20
1500-10
1400-3
1600-X
YIELD (WT %)
PS LIFE (MIN)
50
40
30
20
1500-10
1400-3
1600-3
A.D (g/cc)
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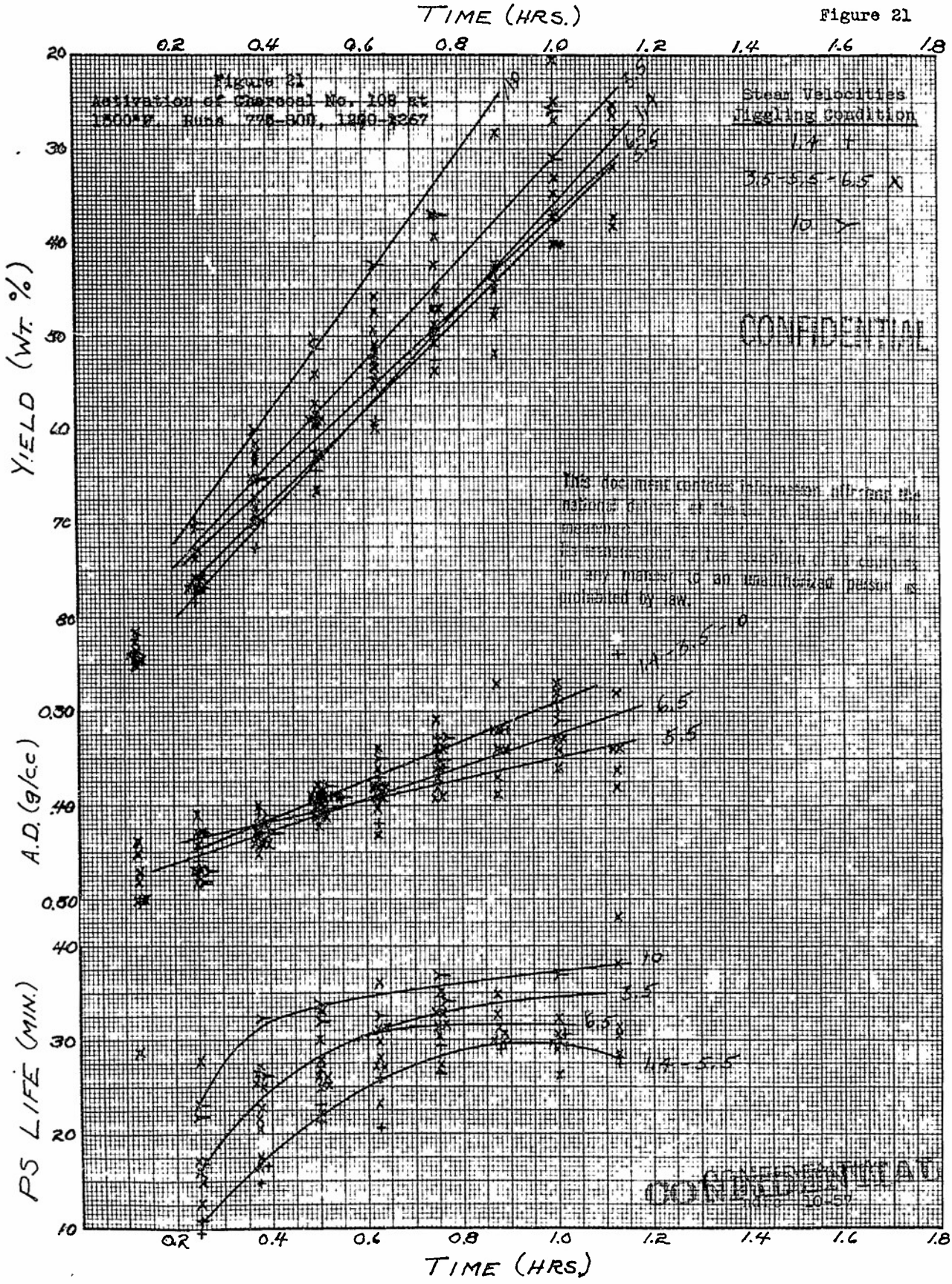
Figure 21


TIME (HRS.)
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
Figure 21
Activation of Charcoal No. 108 at 1500°F. Runs 778-800, 1260-1267
Steam Velocities
Jiggling Condition
1.4 +
3.5-5.5-6.5 X
10. Y
CONFIDENTIAL
YIELD (WT. %)
20
30
40
50
60
70
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A.D. (g/cc)
0.30
0.40
0.50
PS LIFE (MIN.)
40
30
20
10
10
6.5
3.5
1.4-5.5
CONFIDENTIAL
Declassified 10-50
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
TIME (HRS)

Figure 22

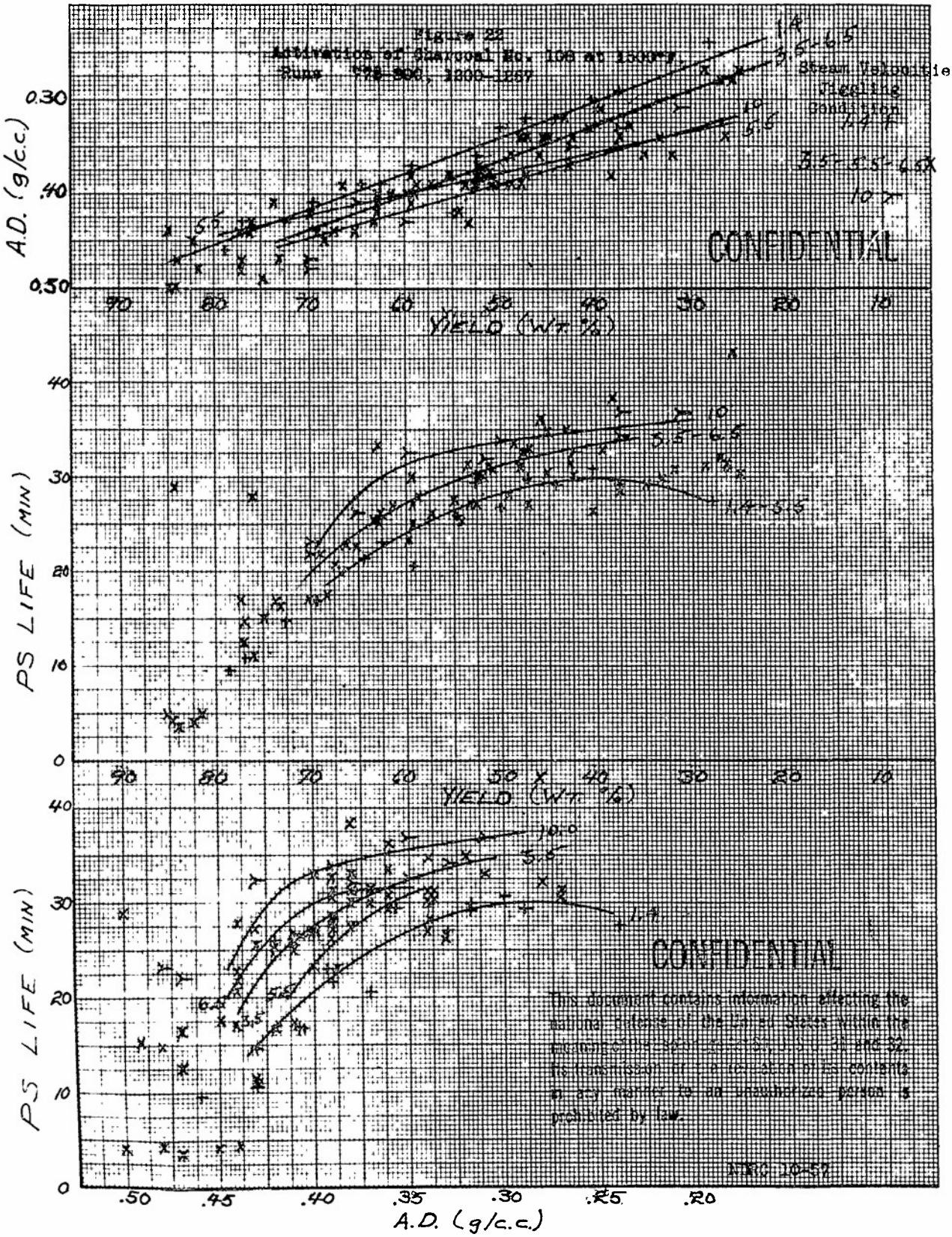
Figure 22
Activation of Charcoal No. 108 at 1500°F
Runs 778-800, 1800-1867
Steam Velocities
Variable
Conditions
A.D. (g/c.c.)
YIELD (Wt. %)
PS LIFE (MIN)
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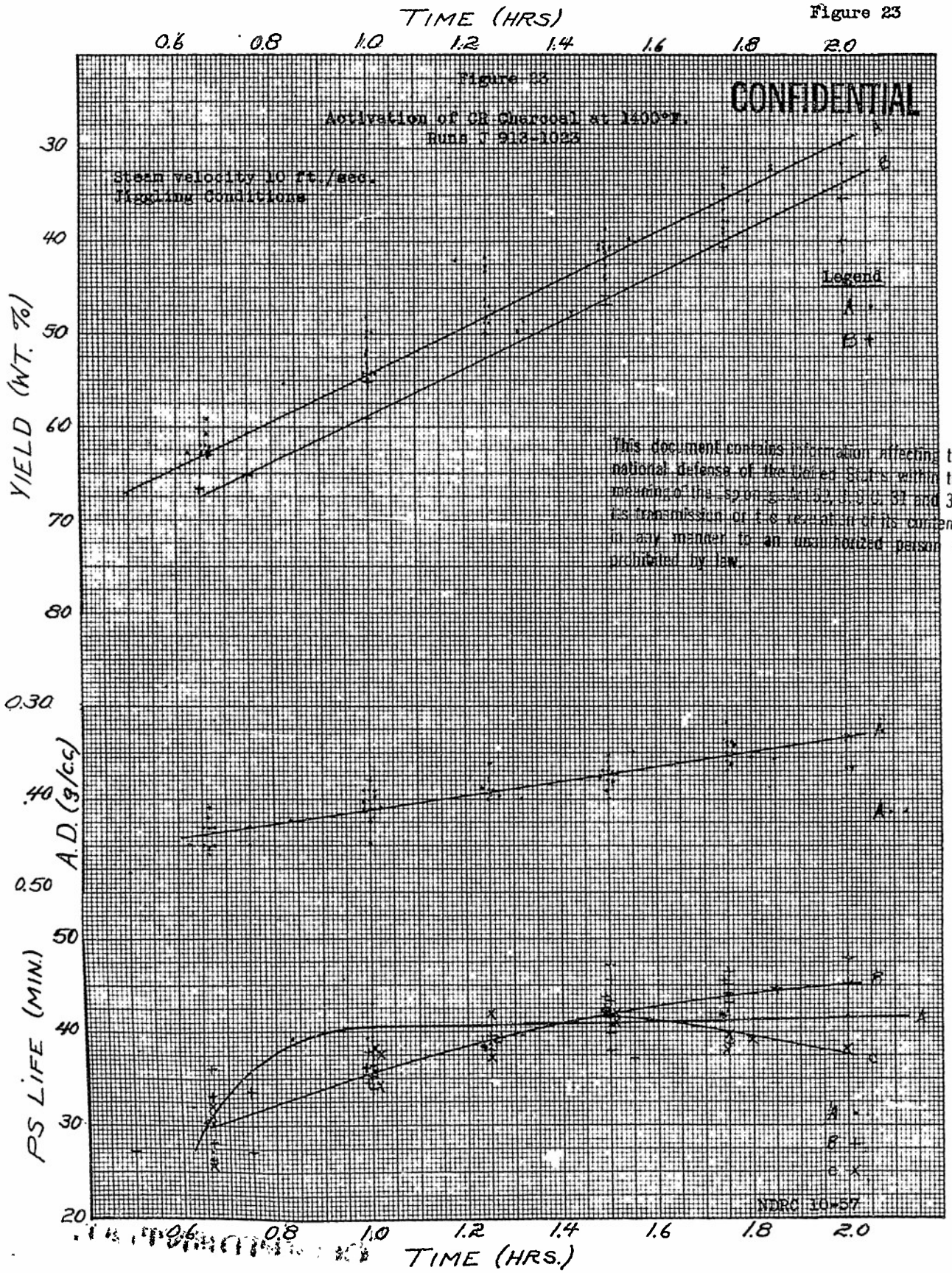
Figure 23


TIME (HRS)
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
Figure 23
CONFIDENTIAL
Activation of CR Charcoal at 1400°F.
Runs T 918-1023
Steam velocity 10 ft./sec.
Fluidizing Conditions
Legend
A
B
YIELD (WT. %)
30
40
50
60
70
80
A.D. (g/c.c.)
0.30
.40
0.50
PS LIFE (MIN.)
20
30
40
50
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NDRC 10-57
TIME (HRS.)

Figure 24

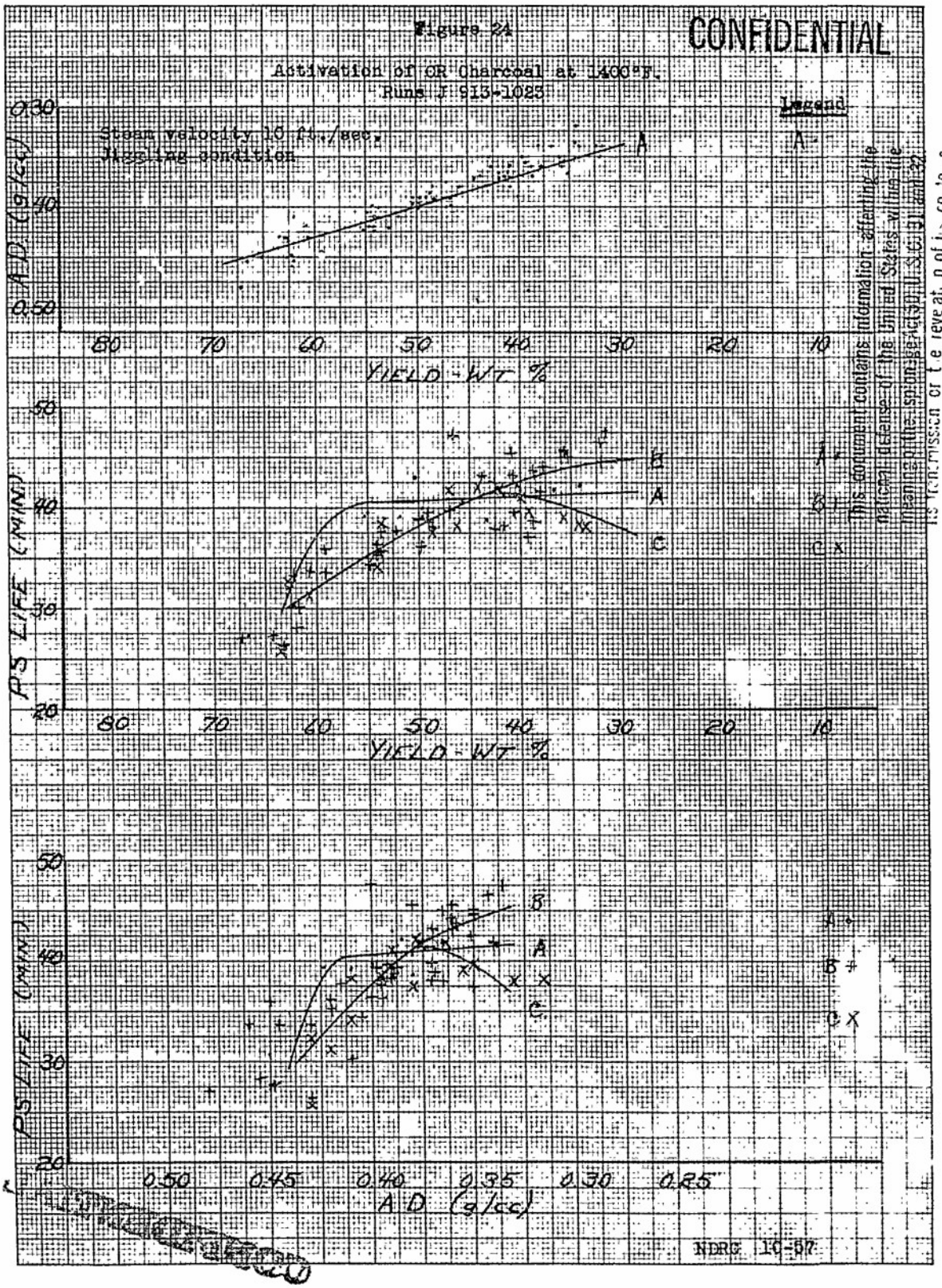

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Figure 25

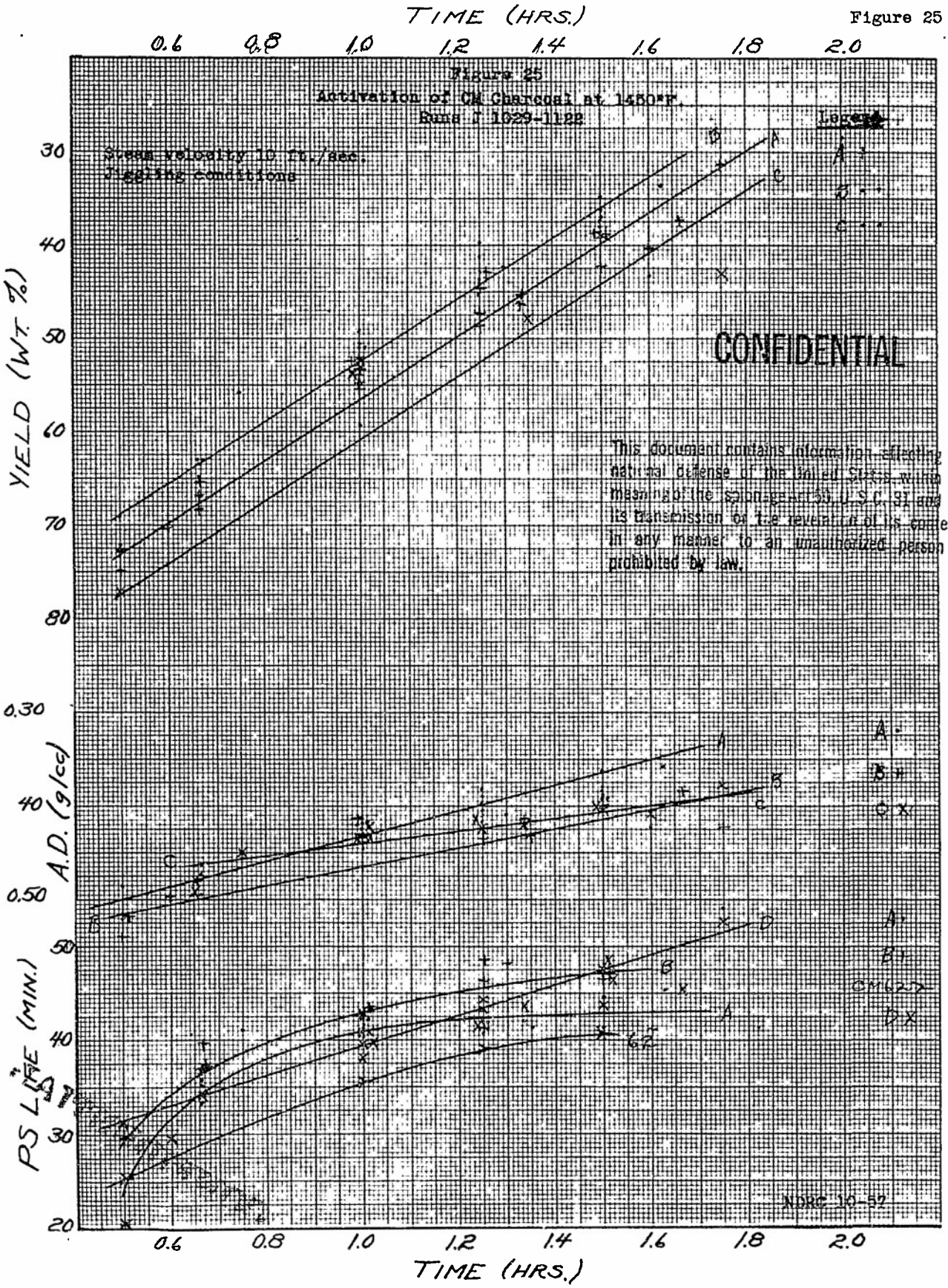
Figure 25
Activation of CM Charcoal at 1450°F.
Runs 1029-1122
Legend
A
B
C
Steam velocity 10 ft./sec.
Fluidized conditions
YIELD (WT %)
A.D. (g/cc)
PORE VOLUME (CC/G)
PORE LIFE (MIN.)
TIME (HRS.)
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
20
30
40
50
60
70
80
0.30
0.40
0.50
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Figure 26

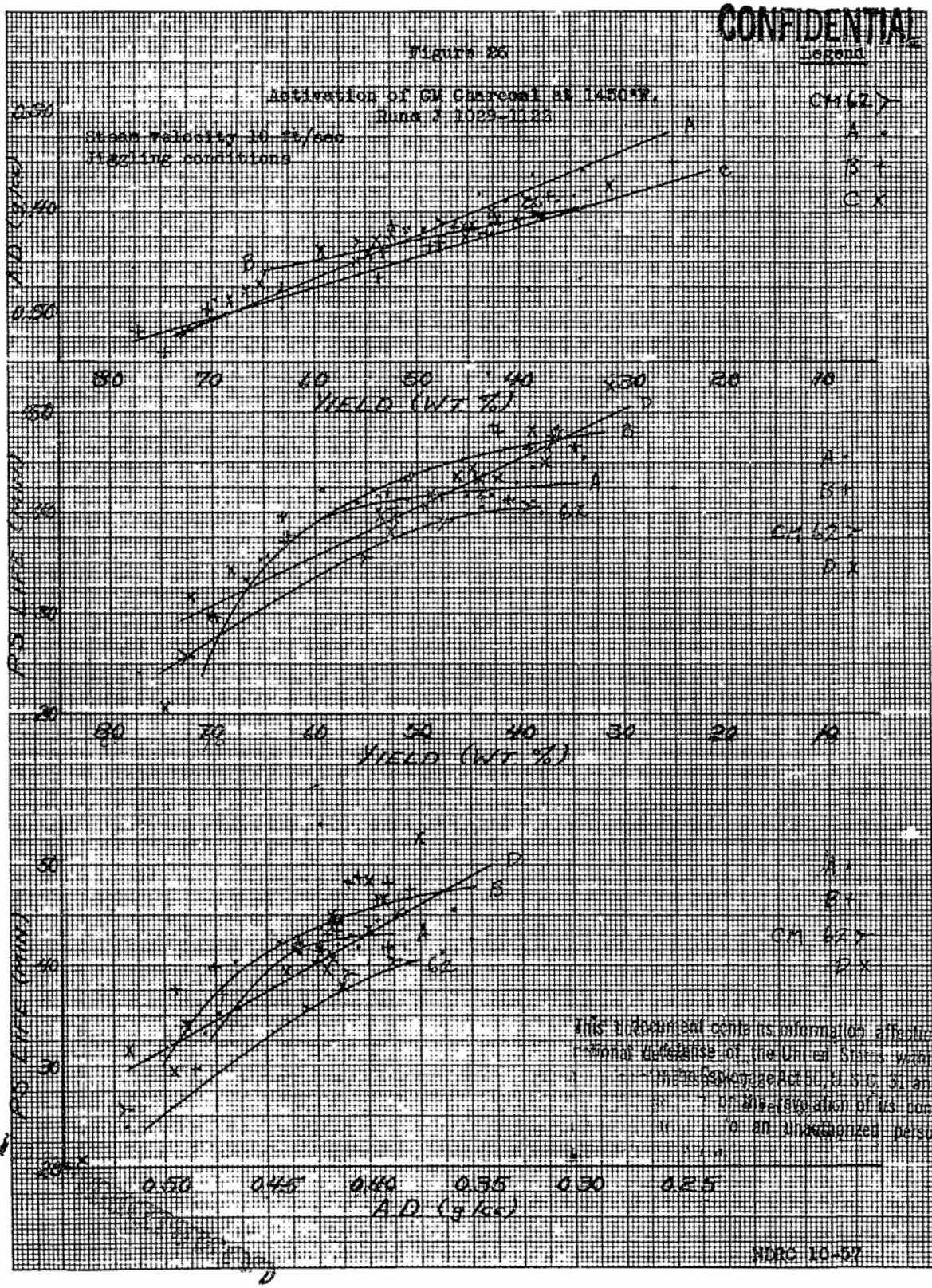

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Figure 27

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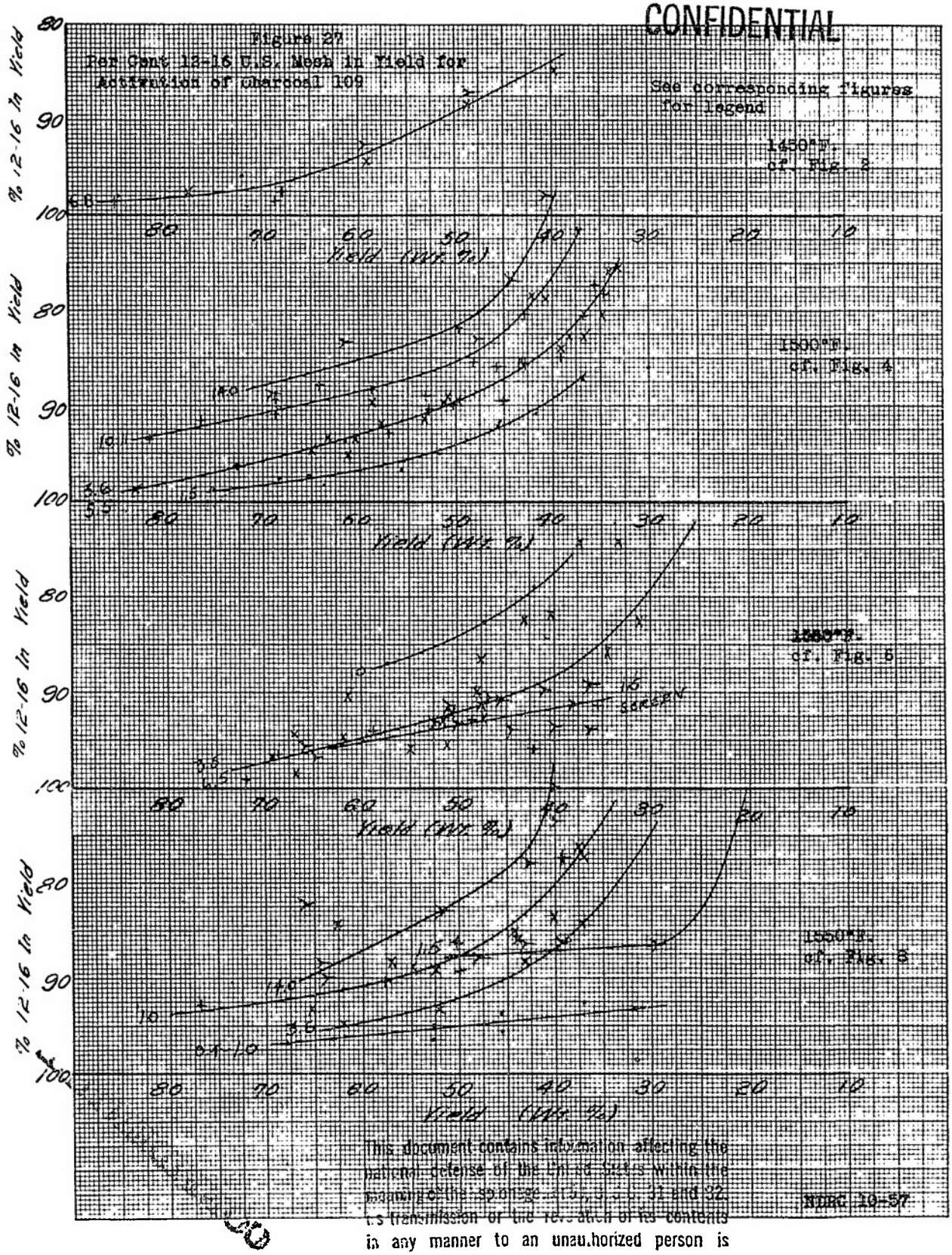

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Figure 28

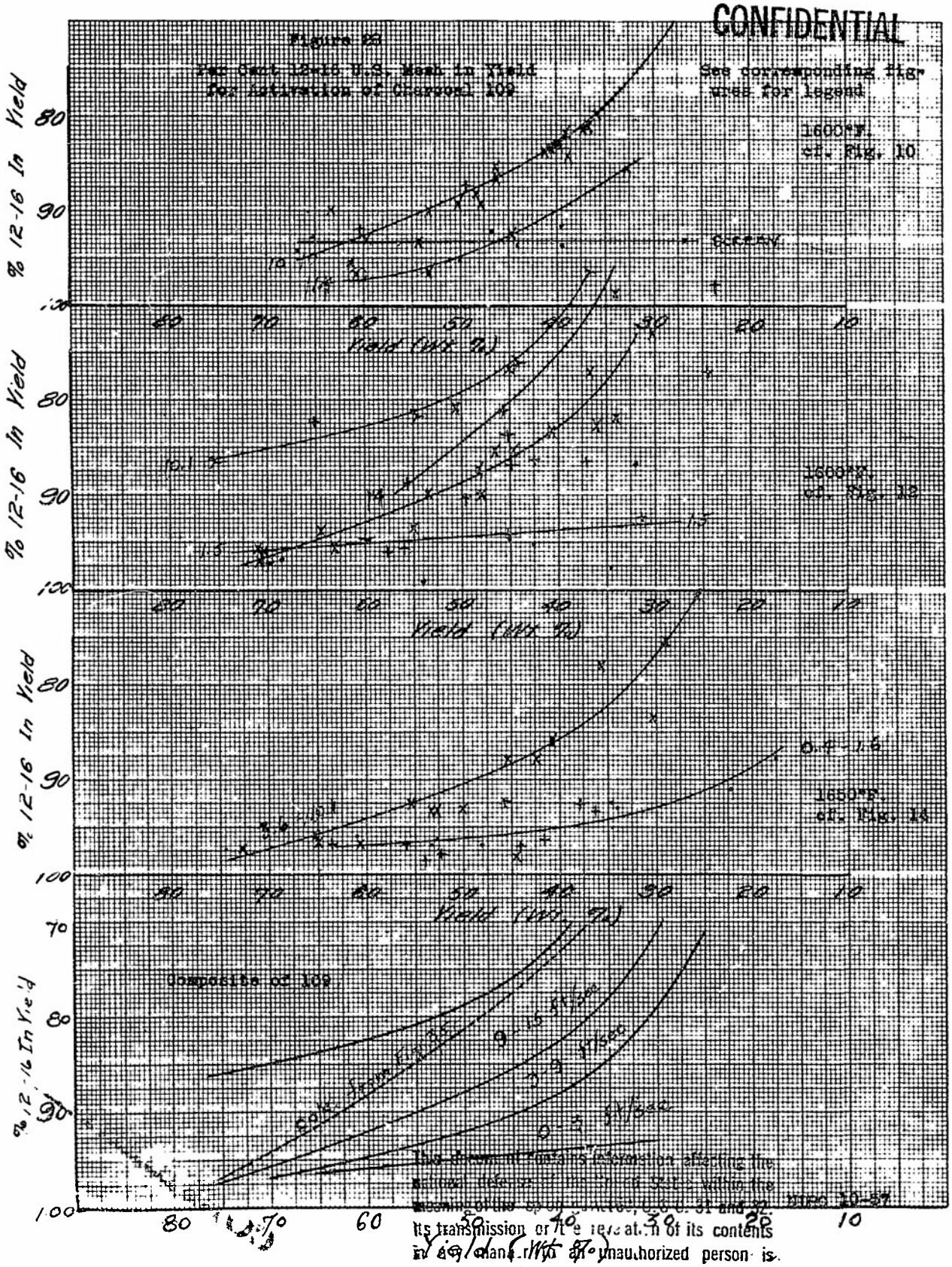
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Figure 28
Yield Curve 12-16 U.S. Mesh in Yield
for Extraction of Charcoal 102
See corresponding figures for legend
1600°F.
cf. Fig. 10
1600°F.
cf. Fig. 12
1650°F.
cf. Fig. 14
% 12-16 In Yield
Yield (wt% Pd)
Composition of 102
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Figure 29

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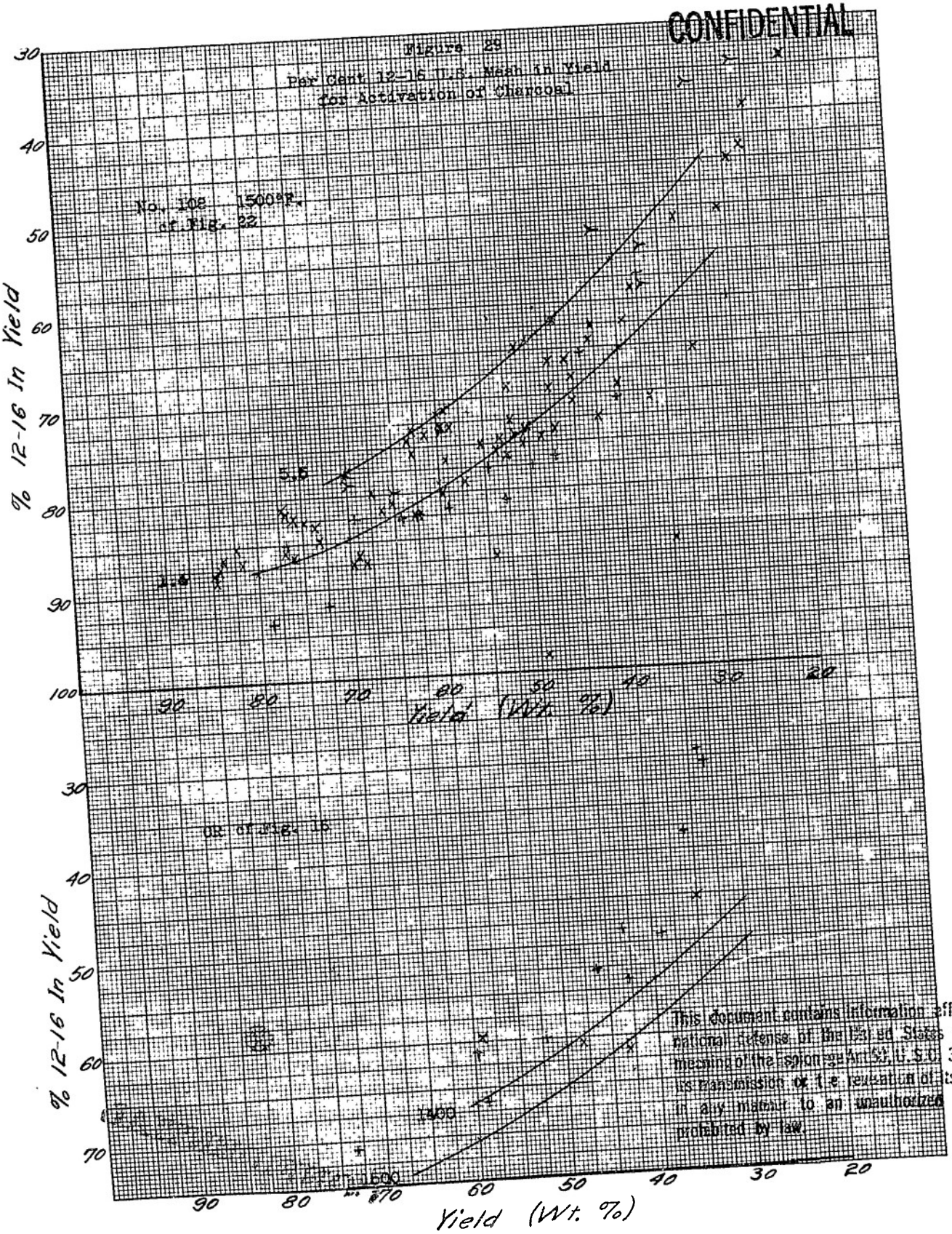

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meaning of the Espionage Act, 50 U.S.C. 31 ar
its transmission or the revelation of its co
in any manner to an unauthorized pers
prohibited by law.

Figure 30

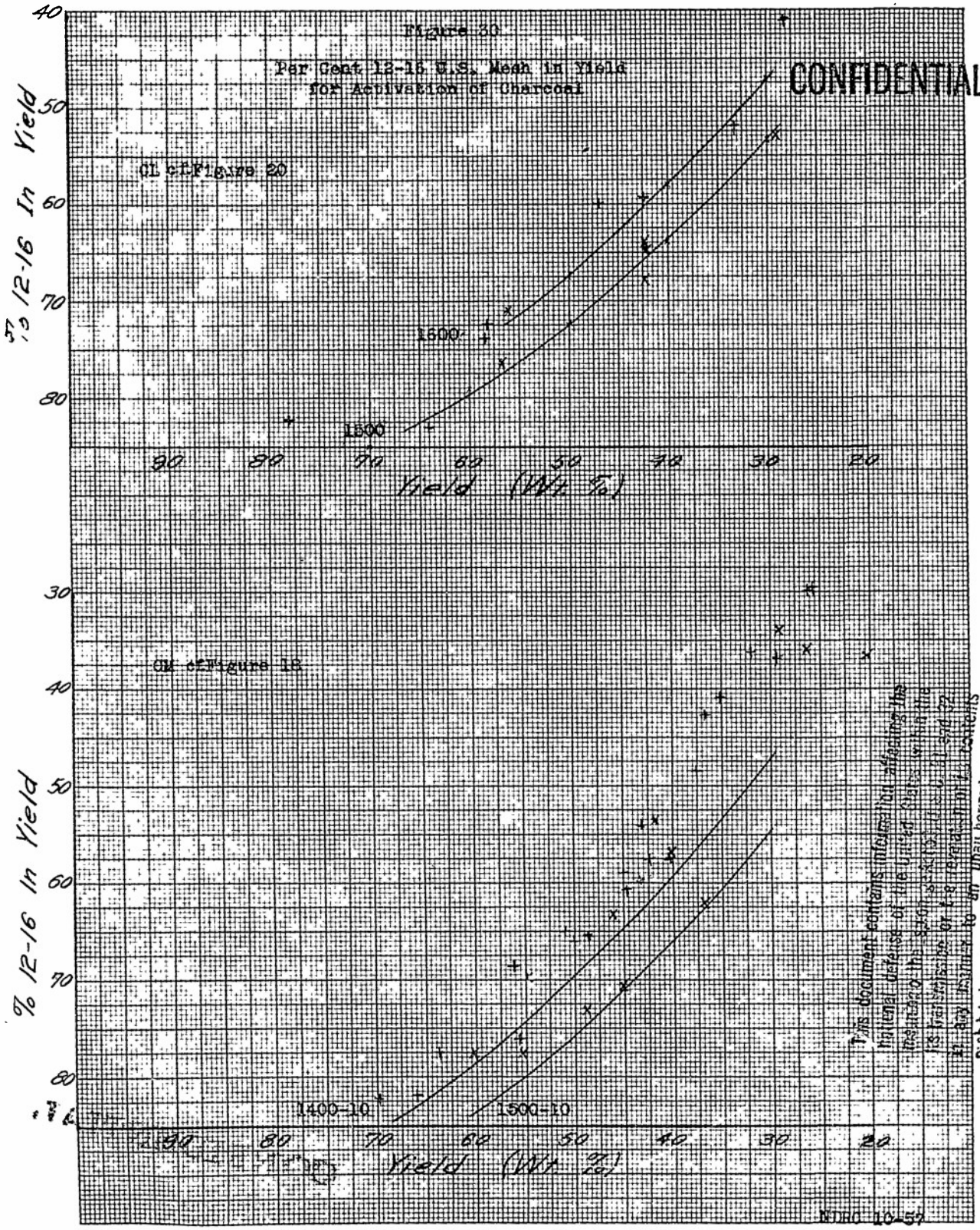

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Figure 31

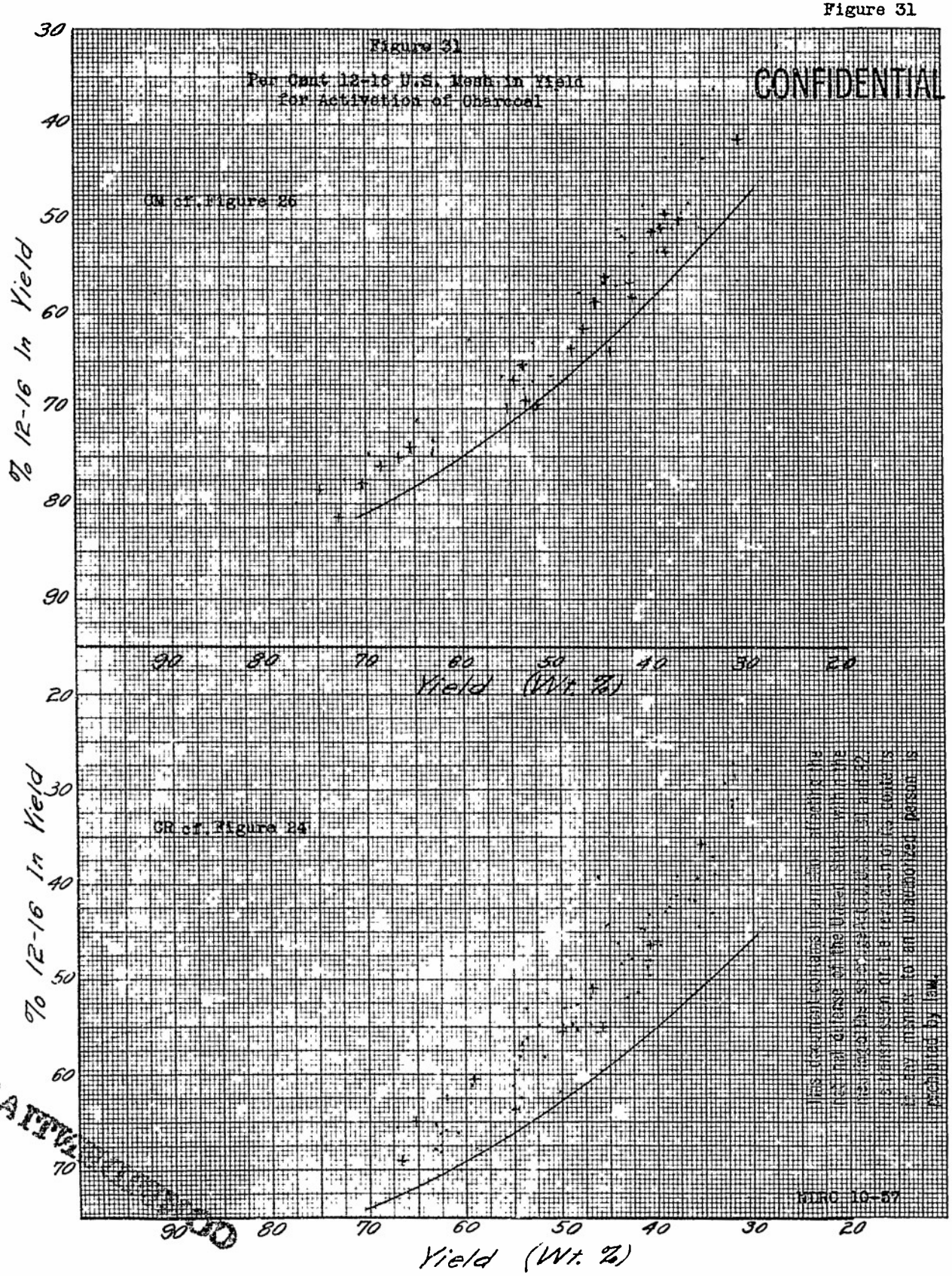
Figure 31
Per Cent 12-16 U.S. Mesh in Yield
for Activation of Charcoal
CONFIDENTIAL
GM cf. Figure 26
% 12-16 In Yield
Yield (Wt. %)
GR cf. Figure 24
% 12-16 In Yield
Yield (Wt. %)
WIRO 10-57

Figure 32

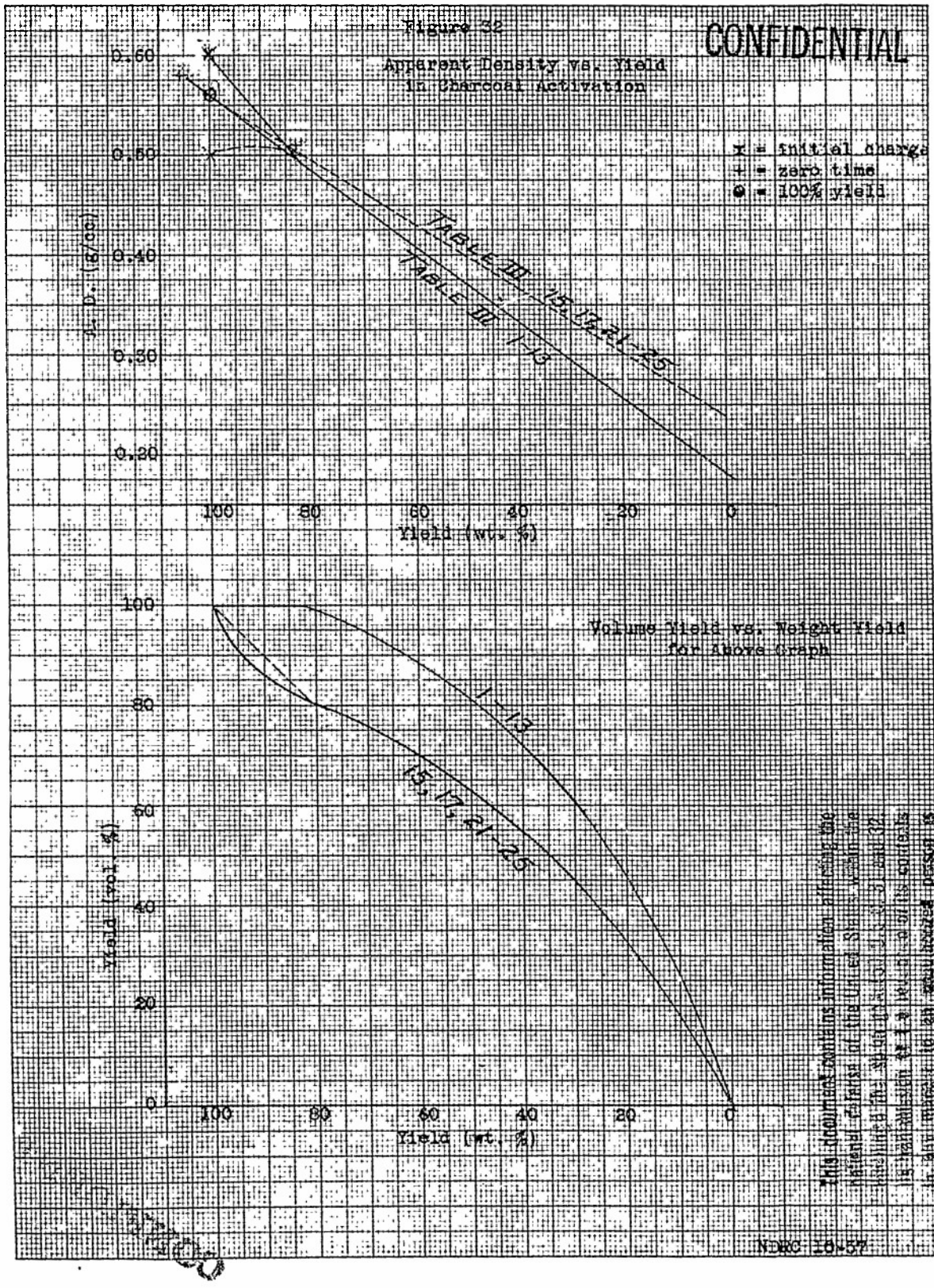
Figure 32
CONFIDENTIAL
Apparent Density vs. Yield
in Charcoal Activation
x = initial charge
+ = zero time
● = 100% yield
Table XV
Table XIII
(15, 17, 21-25)
Yield (wt. %)
Volume Yield vs. Weight Yield
for Above Graph
Table XIII
(15, 17, 21-25)
Yield (wt. %)
NDRC 10-527

CONFIDENTIAL

Figure 33
Log Gasification Rate vs. 1/T for Steam Oxidation of Charcoal 109 1450°-1650°F.

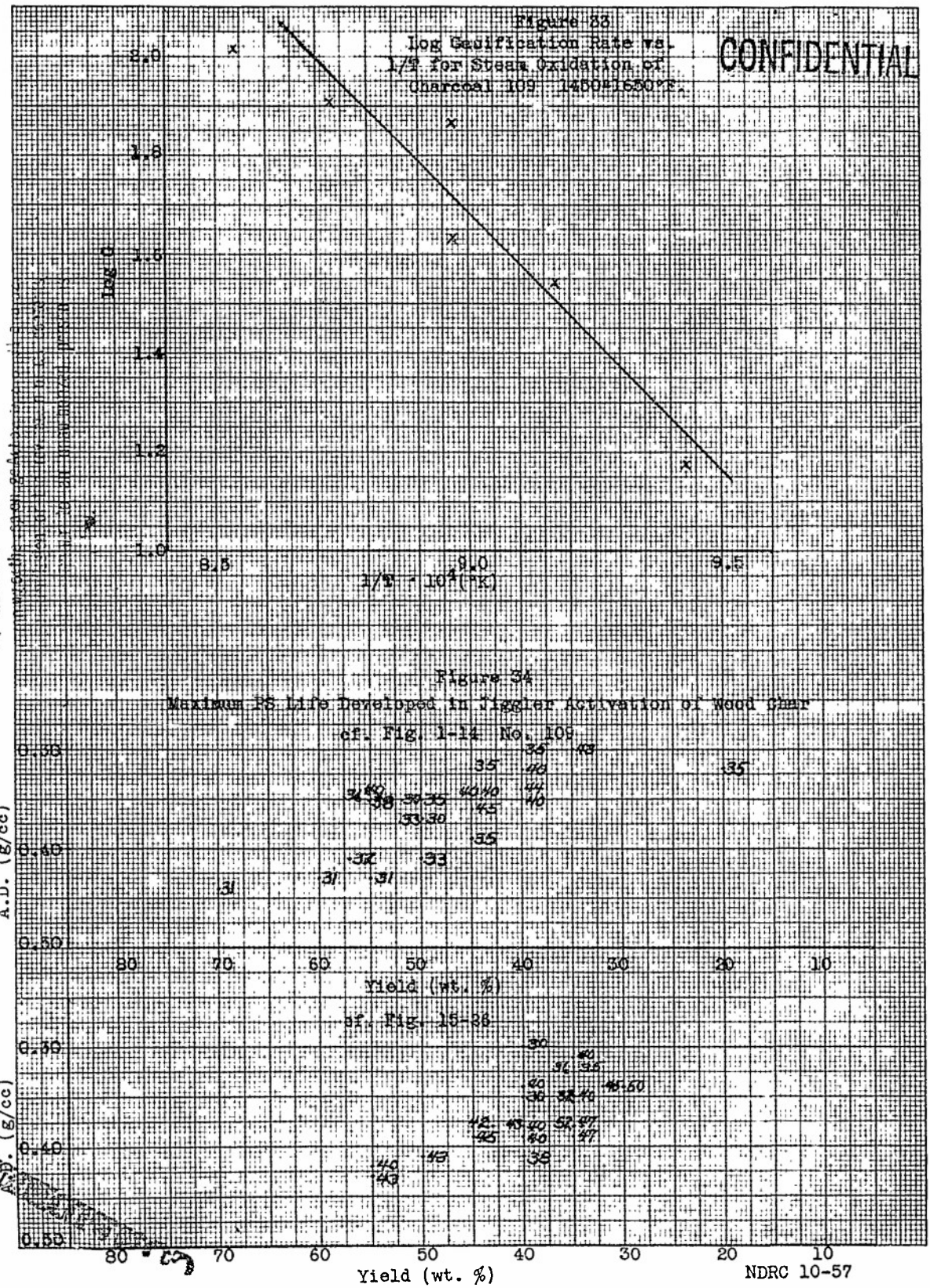

Figure 34
Maximum RS Life Developed in Ziegler Activation of Wood Char
cf. Fig. 1-14 No. 109

cf. Fig. 15-38

NDRC 10-57

Figure 35

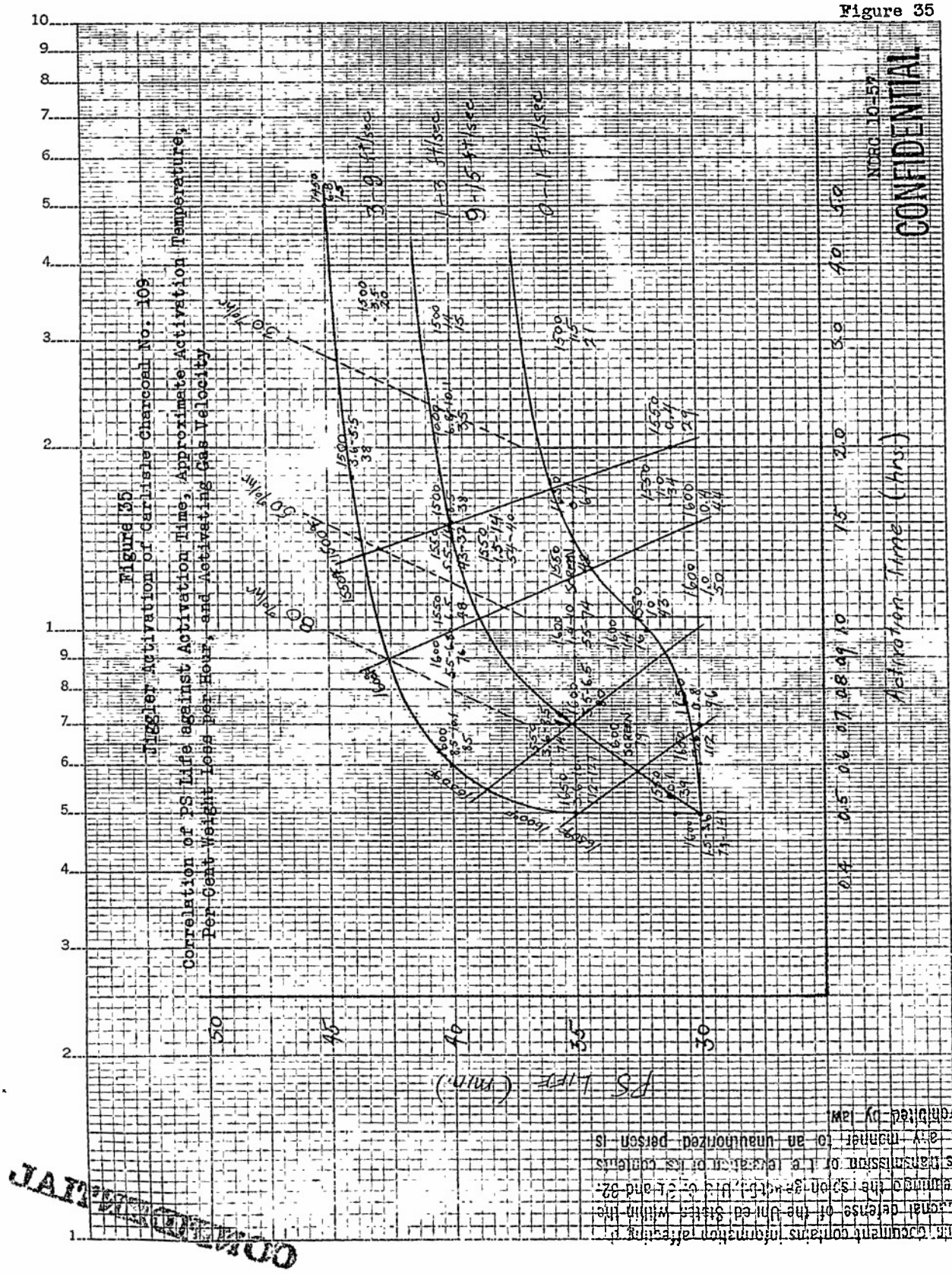

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Figure 36

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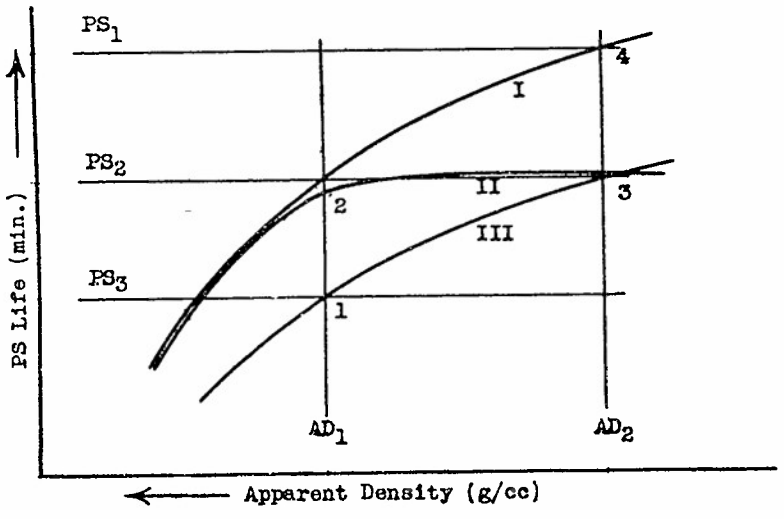

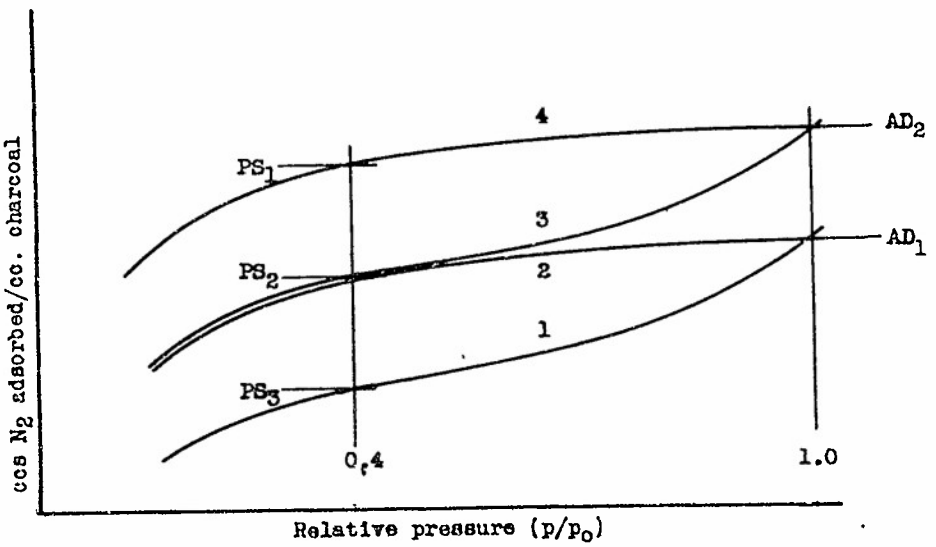

Figure 36
Nitrogen Isotherms Corresponding to PS-AD Correlation

NIRC 10-57

Part III Laboratory Investigation on Chemical Activation

(Tables and Figures are at end of Part III)

CHEMICAL ACTIVATION RESEARCH

Some preliminary research has been done on the activation of wood flour by chemical agents. The principal problem to be solved in connection with the production of gas mask charcoal by this process was the production of charcoal having a satisfactory ASC whetlerite performance. Although improvement in manufacturing procedure has been able to produce such a material, the fundamental problem has not, in our opinion, been solved. There are two aspects to the question: One is measuring the properties of the finished material upon which the whetlerite performance is dependent. The other is tracing the development of these properties during the course of the manufacturing process.

The essential difference between chemical activation and gas activation as discussed above is that the chemical agent prevents coking during calcination and the decomposition of the chemical agent oxidizes the char and produces a capillary system exposing the adsorptive area.

The mechanism of the chemical activation process is, according to our present views, as follows: As the wood flour, water, zinc chloride, and hydrochloric acid are mixed, there is only a slight reaction with the wood flour, no zinc chloride is adsorbed by the wood flour.[1] The acidic zinc chloride solution dissolves a few low molecular weight constituents producing a colored solution with an odor characteristic of tree sap. As the temperature is raised in the mixer, the hydrophobic protection of the cellulose is dissolved and the water swells the cellulose. Zinc chloride may be adsorbed at this point on the cellulose micelles. Under the catalytic influence of hydrochloric

1- Shown by experimental work in this laboratory.

acid some of the wood cellulose is hydrolyzed to six and twelve carbon units and may be further oxidized. The lignin is not dissolved and thereby prevents some of the cellulose from being acted upon, since it binds it in place. A solvent for lignin such as pyridine or morpholine should allow complete swelling of the cellulose. The cellulose is dissolved only in the sense that it is peptized. Upon drying, peptized cellulose will be precipitated. In the mixer, however, as before mentioned, hydrolysis has broken up some of the cellulose chains. In addition to these effects, the thermal effect of elevated temperatures becomes more pronounced now that the cellulose and lignin constituents are separated. Smith and Howard[2] have shown that prolonged heating below 200°C. will result in the decomposition of cellulose into carbonic radicals. In the mixing process, as the temperature of the mix rises, the mix becomes quite plastic and homogeneous at one point; further evaporation of water causes the mix to break up into a fine powder similar to the original wood flour size. Simultaneously with this powdering the hydrophilic nature of the wood flour is lost and upon the further addition of water the plastic condition cannot be attained again. Also the remaining cellulose constituent has now been precipitated out. At this point the mix is briquetted and dried at 200°C. and more of the cellulose constituent is lost by decomposition. As the product is heated above 200°C. the cellulose changes into a aromatic type of compound which is the nucleus of the graphite plane layer in the final product [2,3]. The lignin is already largely composed of humic acids[4]. As the

2- J. Am. Chem. Soc. 59, 234-6 (1937).
3- Hofmann and Sinkel, Z. anorg. allgem. chem. 245, 85 (1940).
4- Sedletzky and Brunowsky, Kolloid Z, 73, 90 (1935).

temperature is raised various organic groups are volatilized[5]. The zinc chloride reacts with these groups that are given off decomposing them and preventing them from solvenizing the char which, if it became plastic, would become bubbly and unsuitable for charcoal. Eventually the zinc chloride is decomposed to zinc oxide and the chlorine goes off as hydrogen chloride.

At 700°C. the char undergoes a profound chemical and physical transition, common to all kinds of carbonaceous material. At this point the charcoal becomes chemically the same as a charcoal from any raw material except for the ash composition.

Physically the pore structure consists of a macro and micro pore system. The micro pore system is independent of the process of carbonization but dependent upon the final temperature. The macro pore system is the result of the path of the process. The surface condition of the charcoal is independent of the path and dependent only upon conditions of the final step.

The important question to be investigated relating to the formation of the capillary system is the conditions affecting the formation of the aggregates of packets of about 1000 Å in diameter or larger mentioned in Part I.

CHEMICAL ACTIVATING AGENTS

The effects of different agents have been treated to some extent in the literature, as described below.

Interplanar Compounds with Graphite. In a process in which active charcoal is produced in the presence of inorganic compounds the effect of the formation of graphite interplanar compounds should be considered. The graphite compounds which have been investigated are: C_8K, C_8Rb, C_8Cs, $C_{16}K$, $C_{16}Cs$, CF, $C_{16}Rb$, C_2O; $C_{24}^{+} \cdot HSO_4^{-} \cdot 2H_2SO_4$, corresponding to graphite bisulfate there is biselenate, perchlorate, nitrate, phosphate, pyrophosphate, arsenate; graphite $FeCl_3$ and

5 - Sedletzky and Shmakova, Compt. rend. acad. sci. (U.S.S.R.) 35, 265 (1942).

graphite $FeCl_3(NH_3)_6$, graphite Br_2. The following do not form graphite compounds: AsI_3, SbI_3, BiI_3, $AsBr_3$, $SbBr_3$, $BiBr_3$, $AsCl_3$, $BiCl_3$, $AlCl_3$, $CrCl_3$, and $CoCl_2$.

The penetration of these constituents between the planes causes a considerable increase in the separation of the graphite plane layers. Separations of 4.5 Å for O up to 16.9 Å for $FeCl_3(NH_3)_6$ have been determined. This increase in layer separation is accompanied by a corresponding increase in gross volume of the carbon particles. These graphite compounds were all prepared under special conditions of pressure and control of reaction rate using high temperature carbons; that is, carbons from which most of the organic impurities have been removed by high temperature treatment. The stability of these graphite compounds is such that they all separate to graphite and inorganic constituents attemperatures above 500°C.

In the carbonization of organic materials in the presence of inorganic constituents, no graphite compounds would be liable to be formed because the temperature at which the graphite constituent begins to appear is too high for the formation of the graphite compound.

Properties of Chars at Temperatures above 700°C. Only above 700°C. do the graphitic properties of charcoal such as low electrical resistance, high helium density, ability to react as a laminar system to oxidizing agents, begin to appear.

X-ray measurements[6] on filter paper soaked in H_3BO_3, $ZnCl_2$, $AlCl_3$, HCl (gas), $CaCO_3$, SiO_2, Fe_2O_3, Na_2CO_3, K_2CO_3, and carbonized at 600 and 1000°C. in N_2, NH_3 and HCl atmospheres show no differences in packet sizes or layer distances for any of these treatments.

6 - Blayden, Gibson, Riley and Taylor, Fuel 19, 24 (1940).

It has been suggested in the literature that inorganic constituents increase the activity per gram by spreading the layer planes apart. The preceding considerations show this to be improbable. What is more essential than spreading the planes apart is to increase the extent of the pore system so that all of the surfaces of the graphite packets are accessible to the atmosphere.

Soare and Georgescu [7] have shown that among NaCl, $CaCl_2$, $FeCl_3$, $ZnCl_2$, NaOH, H_2SO_4, and H_3PO_4 only $ZnCl_2$ and H_3PO_4 produce sufficient activity to be applicable to the production of gas mask charcoal.

Our work on H_3BO_3 shows that it is not applicable to the present process because it does not give a briquettable material from the mixing step. Alekseevski and Plachenoff [8] have shown that varying the concentration of $ZnCl_2$ affects the pore structure of the finished material and that the pore structure is dependent on the composition of the wood with respect to lignin and cellulose.

LABORATORY PROCEDURE FOR DEHYDRATION OF MIXTURES OF ZINC CHLORIDE AND WOOD FLOUR

The first operation in the production of charcoal chemically activated with $ZnCl_2$, starting with wood flour, is the dehydration of a mixture of wood flour, $ZnCl_2$, water and HCl. The mixes were made in a stainless-steel lined, steam-jacketed Read mixer of six-liter capacity. A steam pressure of 80 psi. in the jacket was maintained by a mercury pressure switch on a 4 kilowatt electric steam boiler. The $ZnCl_2$ and wood flour were intimately mixed, then dumped into the hot mixer, and the water and HCl were poured in afterwards. The mix slowly turns brown then black and in a short time becomes plastic. At approximately 40-50 minutes after the mix has been started the plastic mass again breaks down to a soft black powder, the particles of which become finer

7 - Bul. chim. Soc. romane chim. 37, 79, 199, 227, 253 (1934).
8 - J. Applied Chem. (U.S.S.R.) 10, 589 (1937).

and harder if allowed to stay in the mixer. This was determined by the abrasive feel when rubbing the particles between the fingers.

In order to insure reproducibility in the various runs, the steam boiler and jacket system were bled before each run to sweep out any non-condensable gases which might possibly interfere with the heat transfer. The inside of the mixer was also washed by filling it with water and allowing it to boil for a short time. This removed any material which might have encrusted the walls and interfered with the heat transfer.

A further description of the laboratory procedure for completing the activation of wood flour is given in Appendix I.

TIME TEMPERATURE RELATIONSHIP DURING DEHYDRATION OF ZINC CHLORIDE WOOD FLOUR MIXES

A series of runs were made to determine the time temperature relationships of wood flour zinc chloride mixes. Several runs were also made using coal dust with zinc chloride to serve as a control.

The experiments were carried out in the Read mixer with the steam pressure at 80 psi. gauge. A three-hole lid was placed on the mixer. One hole served for placing a thermocouple well down into the mix. This well just cleared the rotating blades. The other holes were kept open to allow vapor and reaction products, if any, to escape. In all cases a batch of raw materials large enough to cover the thermocouple well at all times was used. A chromel-alumel thermocouple in conjunction with a Leeds and Northrup portable potentiometer was used to measure the temperature.

Several mixing runs were made using coal dust instead of wood flour. The zinc chloride, it was expected, would not react to any appreciable extent with the coal, and give a time-temperature relationship indigenous to the mixer. Figure 1 shows this relationship. An attempt to reproduce the physical phenomena observed in a woodflour mix was also made. In this,

coal dust and $ZnCl_2$ were used together with some starch. The starch formed a paste which kept the mix a plastic mass until most of the moisture had evaporated, at which time the mix broke down into powdery form. This served to duplicate the physical characteristics although the time of breakup was considerably different. This time-temperature curve is also on Figure 1. The coal-$ZnCl_2$ mix showed a sharp rise to a maximum temperature at which it remained with very little variation. The starch-coal-zinc chloride mix heated up more slowly and broke down into a powder at 100-120 minutes. An examination of the state of the mix was made periodically by inserting a stick in one of the vent holes and examining the state of the material which adhered to the end. In this manner the approximate time of the breakup to powder of the mix was determined.

The wood flour-zinc chloride mixes all showed a rapid temperature rise to a maximum of about 275°F., then a slow drop to a fairly constant temperature of about 260°F., as shown by Figure 2. This drop in temperature is accompanied by the breakup of the mix from a plastic mass into powder.

Two runs with a nitrogen and oxygen atmosphere produced time-temperature curves shown by Figure 3, very similar to those in which no special atmosphere was introduced. Gas analyses in the falling temperature zone showed the presence of considerable amounts of CO_2 with N_2 and O_2 atmospheres, and very little CO with the N_2 atmosphere, but an appreciable amount when O_2 was used.

In our opinion the rise of the time-temperature curve is connected with the boiling point elevation of the $ZnCl_2$ solution as it becomes more concentrated and the subsequent fall in the case of wood flour mixes at low water contents may be due to adsorption of zinc chloride on colloids in the

sawdust. This would affect the vapor pressure of the water remaining in the mix. Humic acid is one of the principal colloids in lignin. The discussion of the Thermodynamics of humic acid reactions by Fuchs [9] seems to provide a promising lead in regard to this question. Since water is still being vaporized from the mix, as shown by visual observation, for a considerable period after the peak of the time-temperature curve the presence or absence of small exothermic and endothermic reactions in the mix could have no effect on the temperature. They would affect the rate of vaporization, only if they were of a magnitude comparable to the heat of dilution of $ZnCl_2$ and the heat of vaporization H_2O. The heat of solution of $ZnCl_2$ in H_2O is 15.7 k cal/gram mole and the heat of vaporization of water about 9.0 k cal/gram mole. The coal dust dehydration runs show that the temperature drop is not due to a change of heat transfer conditions and the runs in N_2 and O_2 atmospheres show that changing the chemical reaction with the atmosphere does not affect the time-temperature curve. These fragmentary results are insufficient to answer the many contradictions which still arise, but the indications are that the colloidal nature of the wood is involved. When this problem is fully explored a much greater knowledge of the way in which the final structure of the char may be altered by mechanical treatment will be obtained.

BRIQUETTING OF MIXTURES OF ZINC CHLORIDE AND WOOD FLOUR

The mixes used were as follows:

A. 110% $ZnCl_2$ mix consisting of:

1000 gms. wood flour
1100 gms. $ZnCl_2$
80 ccs. HCl (to prevent $ZnCl_2$ from hydrolyzing)
1000 ccs. H_2O

9 - Fuel 22, 112 (1943).

B. 80% $ZnCl_2$ mix consisting of:

1000 gms. wood flour
800 gms. $ZnCl_2$
60 ccs. HCl (to prevent $ZnCl_2$ from hydrolyzing)
1000 ccs. H_2O

Previous preliminary experiments had been made to determine the best state of mix for briquetting. These runs were made to find a briquet which would be easy to handle; i.e., would be hard, firm, would not clog the die, and would comminute properly to give the best particle sizes. The best briquets, it was found, were made immediately after the mix broke into granules.

The only means of determining the similarity of reaction for various runs was by visual inspection. Accordingly, the times at which the plastic mass of the mix broke down to a powdery mass were observed and the reproducibility of the different runs determined on this basis. Thus for the 110% $ZnCl_2$ mix all runs broke down to powder at exactly 41 minutes. The efficacy of bleeding steam for removal of non-condensables in the jacket and also of washing the mixer was proven by these observations.

Compressibility Measurements

A property of these briquets which was measured was the compressibility. The equipment used was (1) a 2-1/4" diameter die set made by the Fred S. Carver Company, (2) a "T" expansion gage to measure the distance between the top platen and the lip of the die, in effect measuring the length of the die piston out of the die body, (3) a 1-1/2" to 2" micrometer for reading gage length, and (4) a ten-ton Carver laboratory hydraulic press.

The experimental procedure was as follows: A small amount of the mix was removed from the mixer, enough to almost fill the die, the die piston was then inserted and the assembly placed in the press. A pressure of

about 400 lbs./in.^{2} was then applied, and a measurement taken (L_o). The pressure was then raised to 16,000 pounds gage, 4000 lbs./sq.in., and another measurement taken (L). The pressure was then released to the original reading, and a third measurement (L') taken. The briquet was then removed, inspected, allowed to stand for several hours, and again inspected. This was repeated at various stages in the mixing operations.

Visual inspection of the briquet entailed examining it for surface hardness by touch, attempting to break the briquet by hand, attempting to deform it by hand, and attempting to crumble the edges. The briquet was allowed to stand for several hours, during which time many of the briquets were observed to crumble into powder spontaneously. Good briquets showed no change on standing other than a moistening of the surface due to the hygroscopic nature of the zinc chloride.

Discussion of Result's

A series of three duplicate runs (M47, M48 and M49) using a 110% zinc chloride mix were investigated first. The results are plotted on Figures 4 and 5. In Figure 4, $\frac{\text{length of compression stroke}}{\text{length of final briquet at 4000 lbs.}}$ was plotted against the time of removal of sample. The data show that the best briquets were made at times close to the point of inflection. The briquets made earlier were plastic and deformed on removal from the die while those made afterwards crumble to powder on removal from the die or shortly thereafter. Those made in the 42.5 to 52.5 minute interval were hard, held their shape and remained so indefinitely. The briquets attempted after the 52.5 minute critical point showed a large compression stroke but also gave a large decompression stroke, indicating no tendency to adhere or agglomerate.

Figure 5, the post compression graph of $\frac{\text{length of springback}}{\text{length of final briquet}}$ shows no point of inflection and for 110% $ZnCl_2$ mix the compression characteristic

would seem to be a better means for examining the briquettability of the mix. Data were not taken in the zero minutes to thirty minutes interval because the mix was so fluid that it flowed out of the die upon application of pressure.

The compression characteristics of 80% $ZnCl_2$ mixes (Figure 6, M50 and M51) were almost the same as that of the 110% $ZnCl_2$ mix, the good briquet range being slightly displaced to 45 and 57.5 minutes. Here, too, there is a point of inflection, slightly more marked than with the 110% mix. The post compression graph (Figure 7), however, shows a point of inflection at almost the same position as the compression graph. But since the points are rather scattered, it would still seem to be much more advisable to use Figure 6 or the compression data for an examination of briquetting characteristics.

Briquetting Temperature

A further investigation of the temperature during briquetting was undertaken. In M35 a 110% $ZnCl_2$ mix was removed from the hot mixer at a time when visual inspection indicated the operation had proceeded to the point where a good briquet would be made. Briquets were then made at various times while the mass was cooling down. A temperature variation from 211°F. to 98°F. was investigated. An examination of the $\frac{\text{length of compression stroke}}{\text{final length of briquet}}$ shows very little or no variation for the various temperatures. An examination of the $\frac{\text{length springback}}{\text{final length of briquet}}$ shows an erratic variation of small magnitude. This leads to the tentative conclusion that the temperature of the mix is not critical in the formation of good briquets.

It should be mentioned that these measurements show the compression of the mixture only in relation to the density of the briquet at 4000 lbs./in.^{2} for a given time of mixing. In order to determine the absolute amount of swelling of the wood, the density at 4000 lb./in.^{2} will have to be determined by a material balance.

REACTIVITY OF MIXES TOWARDS OXYGEN

In order that a comparison of a number of samples varying widely in their physical characteristics could be made, some means had to be devised for determining the rate of oxidation of both granular and plastic substances. At first it was thought that all samples could be obtained in the same form, if the more plastic members were solidified by cooling with solid CO_2 and ground to a standard size while still in a solid state. All such attempts failed; the plastic samples could not be sufficiently solidified to enable them to be crushed. Attempts were made to form briquets of the plastic samples, but this too failed when the semi-liquid material flowed out around the dies. From the above, it can be seen that no absolute rate of oxidation can be determined as the oxidation rate will depend upon the particle size, no matter what the material characteristics and the method are. The method finally adopted, although far from perfect, did permit a comparison of all types of samples. In principle, the method consisted of packing each sample into a small glass receptacle containing a thermocouple.

APPARATUS AND OPERATION

All samples used in this experiment were produced in a Read steam-jacketed mixer. The pressure in the steam jacket was maintained at eighty pounds per square inch, which corresponds to a temperature of 155°C. The charge consisted of 1000 cc. of tap water, 25 cc. of concentrated HCl (to prevent hydrolysis of the $ZnCl_2$), 1000 grams of wood flour, and the required amount of technical grade $ZnCl_2$ mixed together by hand. The mixture was poured into the mechanical mixer at operating temperature, and the stopwatch started. At twenty-minute intervals (and at other times when conditions warranted it) samples were removed and placed in small stoppered vials. After each run at a given $ZnCl_2$

percentage, the mixer was scraped clean.

Series of samples were run for mixtures using 60, 80, 90, 110, 130 and 150 per cent of zinc chloride based on the quantity of wood flour. The physical condition of each sample was noted as the sample was removed from the mixer and is reported in Table I along with the behavior of sample in the reactivity furnace.

The reaction furnace was merely an adaptation of the familiar combustion furnace commonly used for CO_2 determinations, as shown by Figure 8. Two furnaces were constructed and were run alternately. Each furnace contained one thermocouple in the gas stream and another that was built into the glass receptacle holding the sample. Both thermocouples were constructed from number 22 chromel and chromel-alumel alloys; the internal thermocouple rested on the furnace wall just in front of the position occupied by the glass receptacle. Oxygen was passed through the furnace. The oxygen flow was measured by means of a flow meter, dried by passing through a bottle containing flake sodium hydroxide, and supplied to the furnace tube, as shown in Figure 8. A variable resistance for controlling the furnace temperatures was provided; either furnace could be operated from this resistance. The emf. developed by the thermocouples was read by a Leeds and Northrup portable precision potentiometer No. 8662. A switch was provided to enable either the potential of the furnace thermocouple or the difference between the furnace thermocouple and the thermocouple in the glass receptacle to be read.

In a typical rate of oxidation run, enough of the sample to completely fill the receptacle was packed into the receptacle, completely covering the thermocouple. The plastic samples received no treatment before being placed in the receptacle. The harder and more solid samples were ground

into fine particles in a mortar and pestle and then packed into the receptacle. In all cases the thermocouple was surrounded by the sample and a flat surface was exposed to the furnace atmosphere. The receptacle was then placed in the proper end of the furnace, and O_2 flow set at one liter per minute, and the furnace switch turned on. For all samples the rate of heating of the furnace was arbitrarily set at an increase in the furnace thermocouple reading of 0.20 millivolts per minute or 4.9°C. per minute (based on an average value of 0.041 mv/°C. in the range 0°C to 400°C for a chromel-alumel thermocouple).

Readings of the internal thermocouple and the difference between the internal thermocouple and the thermocouple in the receptacle were read once every minute after the furnace temperature had reached a temperature of approximately 75°C., and were continued until the charge had either ignited or reached a temperature of approximately 325°C. Variations in the 0.20 millivolt difference per minute in the internal thermocouple readings were corrected by means of the variable resistance. The average maximum variation in rage of heating was 0.02 mv./min. All thermocouple readings were made with the reference junction set to zero. The room temperature was read for each furnace run. To convert a furnace thermocouple reading to a temperature reading, the room temperature was converted to millivolts by means of the tables for the thermocouple, and this reading was added to the thermocouple reading and converted to degrees centigrade. To determine a given charge temperature the difference between the two thermocouples was subtfacted (the charge thermocouple was wired so as to give a positive difference so long as the charge thermocouple was at a lower temperature than the furnace thermocouple) from the sum of the furnace thermocouple reading and the room temperature reading in millivolts, and this value converted to degrees centigrade.

All samples were tested in this manner with the exception of samples

Nos. 29-2, 30-2, 31-2, 33-2; these samples were so unworkable that they could not be broken up small enough to be packed into the glass receptacle. A run was also made on a sample of $ZnCl_2$ as it came from the container.

For each run a graph was plotted of values of differences, Δt, between the sample thermocouple, t_c, and the furnace thermocouple, t_f, versus the heating time, θ, in the furnace.

Results. The data obtained from the zinc chloride run showed that the temperature of an unreactive material follows the furnace temperature almost linearly; the value determined for the melting point of solid $ZnCl_2$ compares favorably with the accepted value, 262°C. This shows that any variation in the graphed data from a nearly horizontal line indicates some change in either the physical or chemical properties of the charge material. A rise in the graph indicates an endothermic process; a decline indicates an exothermic process.

Figure 9 shows the relation of the significant portions of the temperature curves to each other. The coordinates of points A, B, and C and other pertinent data are given in Table II. Point C is called the comparison point.

The comparison point of the rates of oxidation was arbitrarily set at the charge temperature which produced an increase in the temperature of the charge at the rate of 6.85°C./min. This signifies a lowering of 0.08 mv./min. in the difference in the charge and furnace thermocouples. Values of the furnace and charge temperatures at the comparison point are collected in the table, along with the charge temperature at the peak of the curve. A visual analysis of these values shows that the charge temperature at the comparison point and the charge temperature at the curve maximum both increase with the time of mixing. This indicates that the more easily oxidizable materials

are driven out of the mix in the earlier stages of the mixing process.

Comparison of the curves of a given mixer run with increasing time shows: (a) a dropping off of the height of the curve maximum (a lowering of the charge-furnace temperature difference), (b) a sharp rise followed by a marked decline, and (c) the occurrence of a dip in a few curves just before the rise (see sample 29-3). Phenomena (a) indicates a decrease in the heat capacity of the material. The sharp rise and fall (b) occurred in nearly all samples to some degree, but much more pronounced in the sample of short mixing time and decreasing as the mixer time increased. The sharp rise is caused by the vaporization of volatile materials; this assumption is borne out by the decrease in the rise in the case of samples of longer mixing time whose volatile matter was largely driven off in the mixing operation. The drop in the curve is caused by oxidation of the sample. The point at which oxidation begins is dependent on the mixing time, but the course of the oxidation from there on appears to depend on the physical form of the sample--the later, more finely divided samples increasing in temperature very rapidly in comparison to earlier samples. The occurrence of a dip (c) in a curve at approximately 150°C. results from the oxidation of one of the more easily oxidized substances before volatilization takes place. Any such reaction occurring during vaporization of volatile substances would be concealed by the endothermic character of the vaporization process. Both the charge temperature at the comparison rate of 6.85°C./min. rise in charge temperature and the charge temperature at the curve maximum are independent of the percentage of zinc chloride in the original mix.

PART III

Table I

Remarks on Physical Condition of Samples.

Sample no.	Remarks
29-1	Brown, granular, pre-plastic, ignited.
29-3	Granular, post-plastic, ignited.
29-4	Fine powder, ignited.
30-1	Plastic, did not ignite.
30-3	Slightly past plastic condition, ignited.
30-4	Fine powder, ignited.
30-5	Fine powder, ignited.
31-1	Plastic, did not ignite.
31-3	Slightly past plastic condition, ignited.
31-4	Fine powder, ignited.
31-5	Fine powder, ignited.
32-1	Plastic, ignited.
32-2	Plastic, ignited.
32-3	Slightly past plastic condition, ignited.
32-4	Fine powder, ignited.
32-6	Fine powder, ignited.
33-1	Plastic, did not ignite.
33-2	Slightly past plastic condition, ignited.
33-3	Granular, did not ignite.
33-4	Fine powder, ignited.
33-5	Fine powder, ignited.
34-1	Brown, granular, preplastic, did not ignite.
34-2	Plastic, did not ignite.
34-4	Plastic, did not ignite.
34-5	Plastic, did not ignite.
34-6	Less plastic, ignited.
34-7	Granular, ignited.
34-8	Fine powder, ignited.
34-9	Fine powder, ignited.

PART III — Table II — CONFIDENTIAL

Reactivity of $ZnCl_2$-Activated Charcoal with Respect to Oxygen.

Sample	% $ZnCl_2$	Mix Time Min.	θ Min.	A t_c °C	A t_F °C	A Δt °C	A θ Min.	B t_c °C	B t_F °C	B Δt °C	B θ Min.	C t_c °C	C t_F °C	C Δt °C
29-1	60	20	15	109	133	24	31	149	190	41	35	171	209	38
29-3	60	60	25	166	182	16	33	176	200	24	37	195	217	22
29-4	60	80	15	106	120	24	24	140	158	18	37	02	218	16
30-1	80	20	18	114	130	16	37	168	216	48	37	168	216	48
30-3	80	56	24	156	156	20	34	176	202	26	36	190	214	24
30-4	80	62	26	154	168	14	33	176	197	21	36	194	212	18
30-5	80	80	30	167	186	19	36	193	213	20	37	198	218	20
31-1	90	20	18	124	131	7	32	162	196	34	34	168	208	40
31-3	90	60	21	134	144	10	27	158	170	12	34	196	208	12
31-4	90	80	23	150	168	18	32	177	192	15	37	206	218	12
31-5	90	100	12	93	104	11	24	142	160	18	37	193	214	21
32-1	110	20	16	115	124	9	35	160	196	36	36	174	210	36
32-2	110	40	17	118	128	10	34	170	189	19	36	186	213	27
32-3	110	52	22	136	148	9	35	184	206	22	40	214	230	16
32-4	110	61	24	137	148	21	34	176	203	27	37	191	218	26
32-5	110	80	23	154	152	18	34	181	204	23	38	202	233	31
32-6	110	90	23	138	156	18	37	196	216	20	39	204	227	23
33-1	130	21	13	96	110	11	36	162	210	48	39	178	224	46
33-2	130	41	24	144	160	16	35	188	208	20	37	192	217	25
33-3	130	47	25	142	164	22	33	171	145	24	37	193	216	23
33-4	130	70	26	145	166	21	35	160	206	46	38	199	223	24
33-5	130	90	28	156	178	12	38	181	195	14	37	206	216	10
34-1	150	20	-	-	-	-	30	152	187	35	35	181	207	26
34-2	150	40	22	130	149	19	36	160	213	50	40	181	228	47
34-4	150	80	21	127	146	19	35	164	208	44	38	181	222	41
34-5	150	100	22	130	148	18	38	183	223	40	41	196	233	37
34-6	150	120	24	144	161	17	37	175	216	41	44	207	246	39
34-7	150	140	27	158	172	14	37	184	217	33	41	206	236	30
34-8	150	160	29	160	180	20	38	190	219	29	42	210	237	24
34-9	150	180	9	80	94	14	37	190	216	26	41	214	234	20

Figure 1

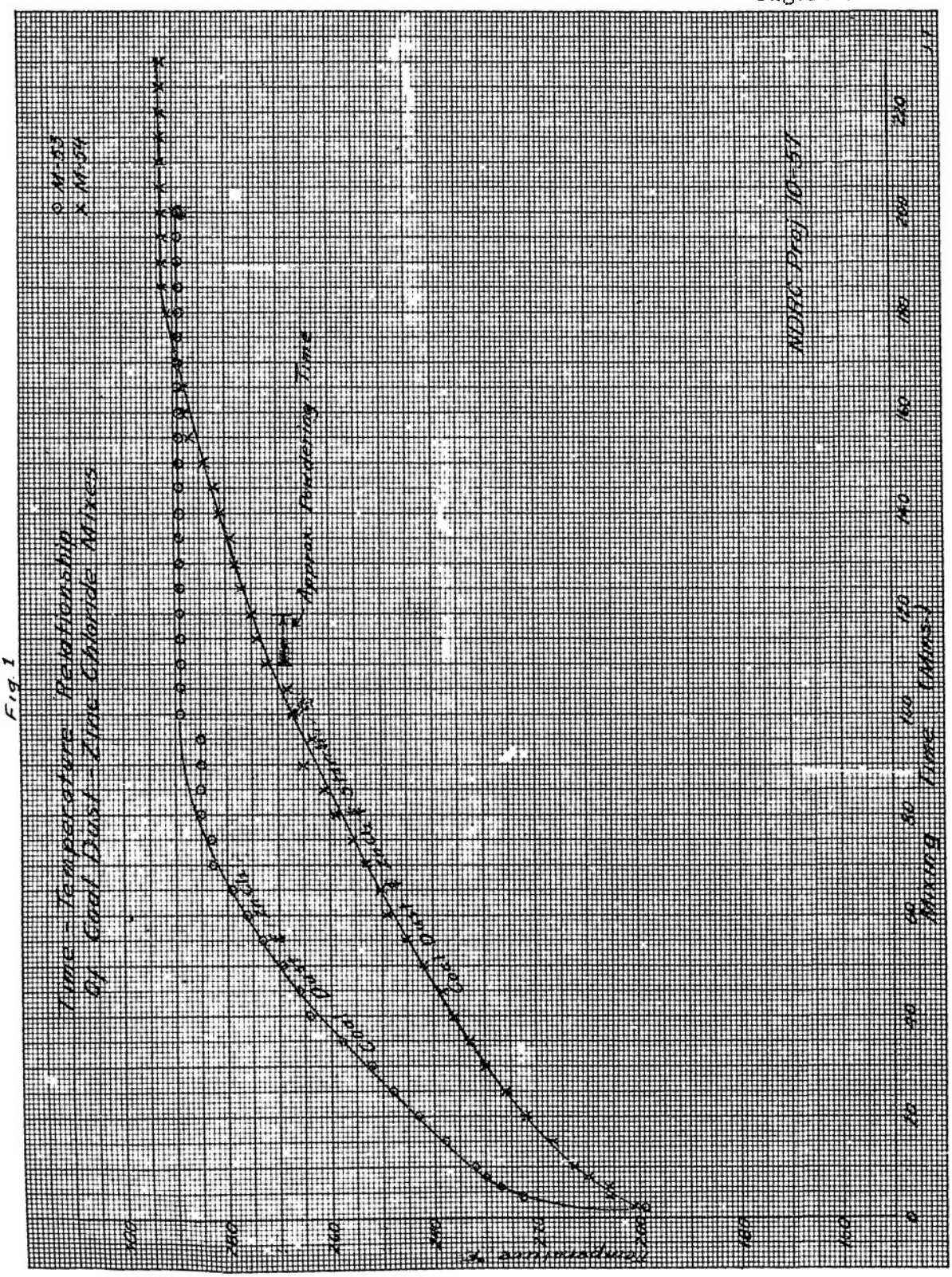
Fig 1
Time-Temperature Relationship
of Coal Dust-Lime Chloride Mixes
Approx. Preheating Time
Approx. Explosion Time
M-53
M-54
Minutes
Time (Minutes)
Temperature (°C)
KEUFFEL & ESSER CO. N Y NO 359-14
Millimeters, 5 mm. lines accented, cm lines heavy
MADE IN U S A

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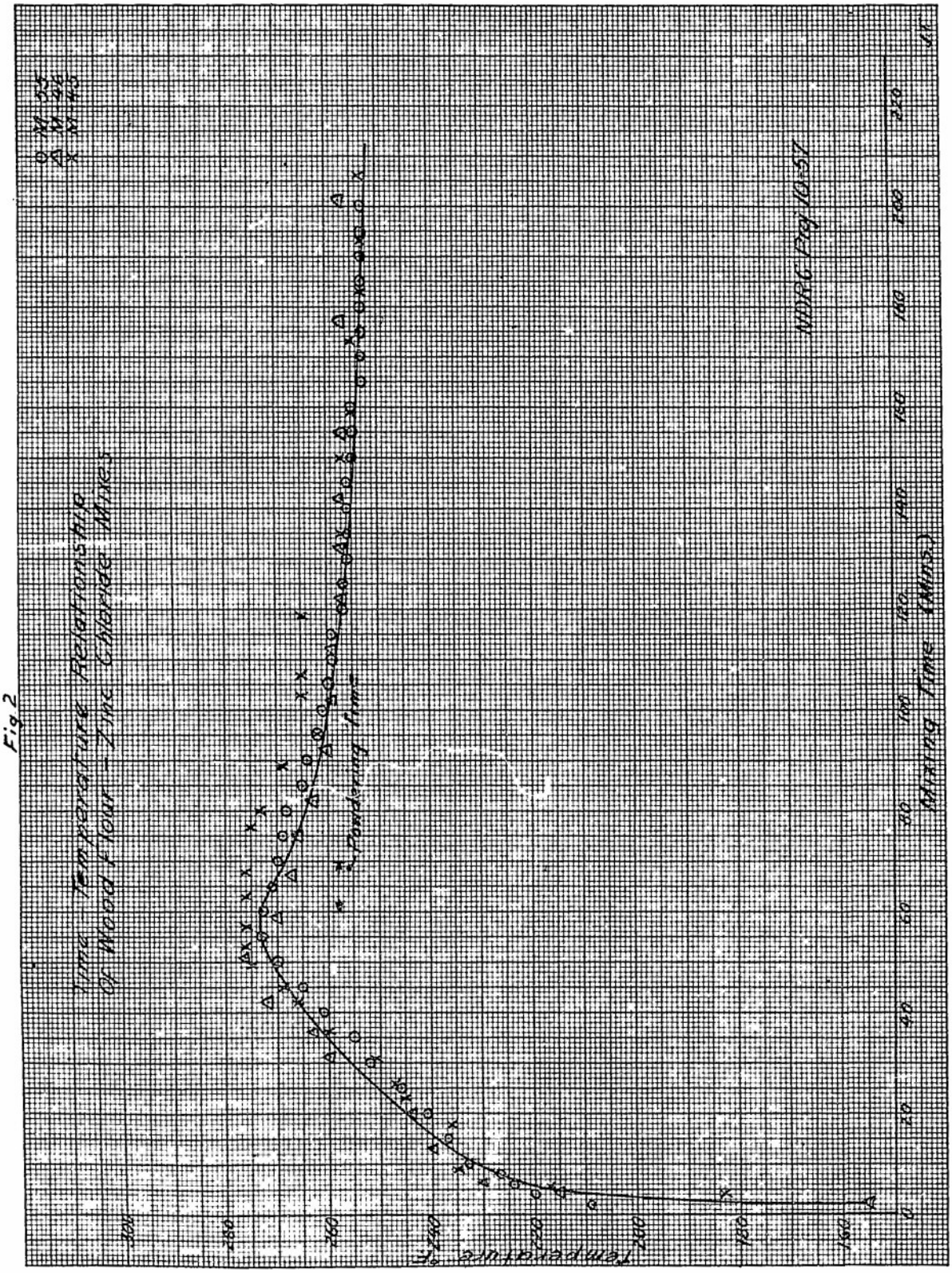
Figure 2



KEUFFEL & ESSER CO., N. Y. NO 359-14
Millimeters, 5 mm. lines accented, cm lines heavy.
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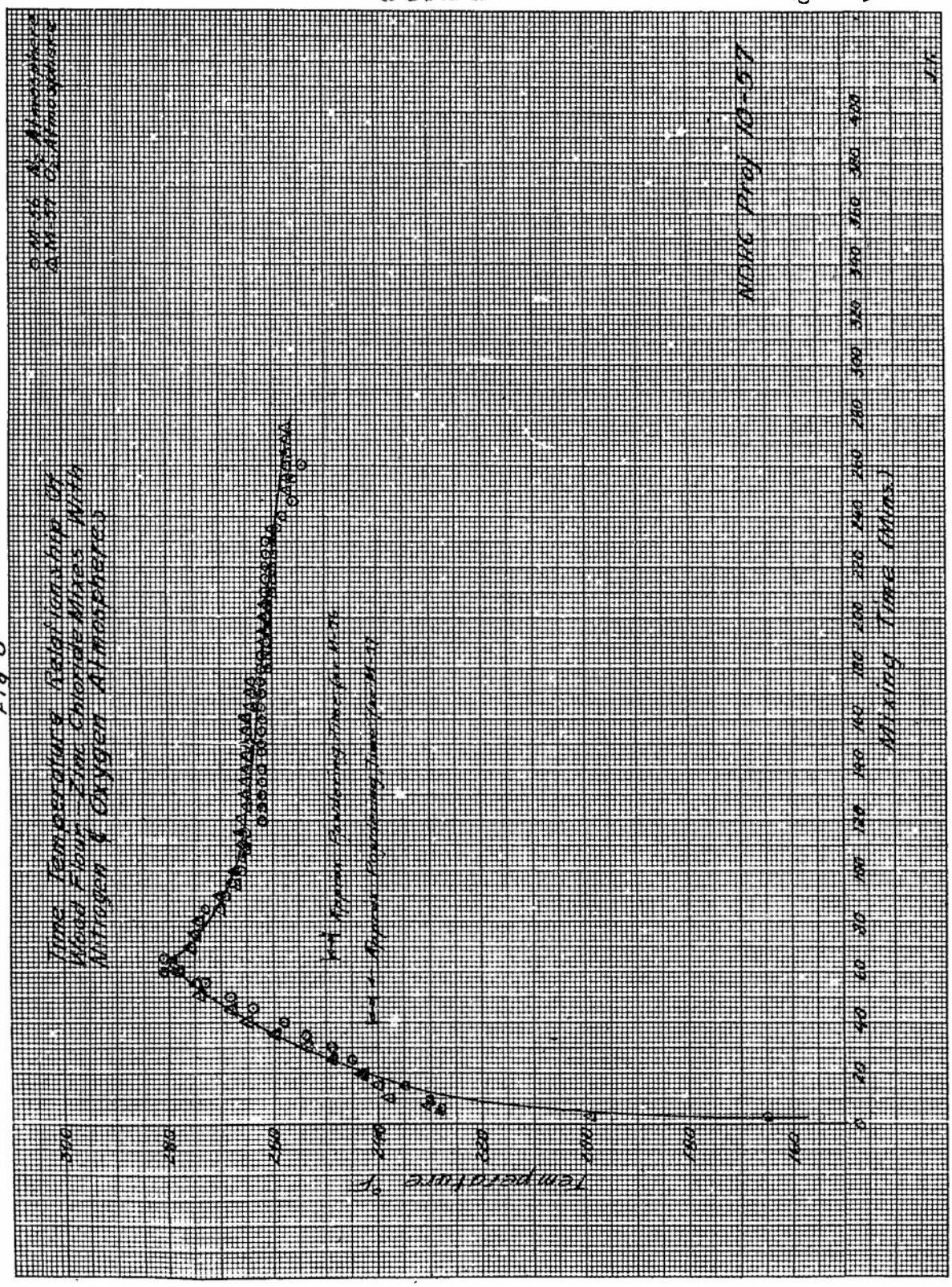
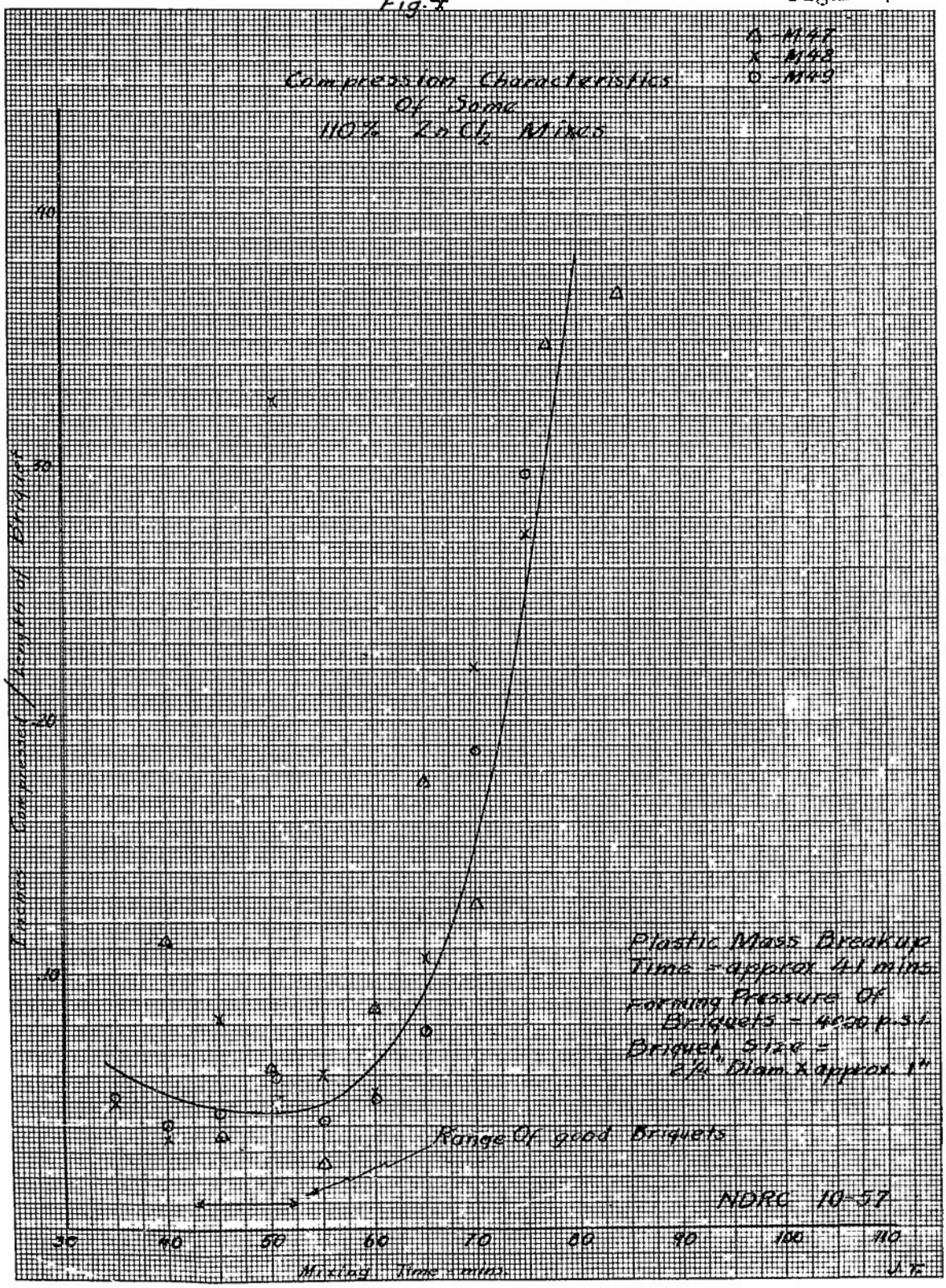
Figure 3



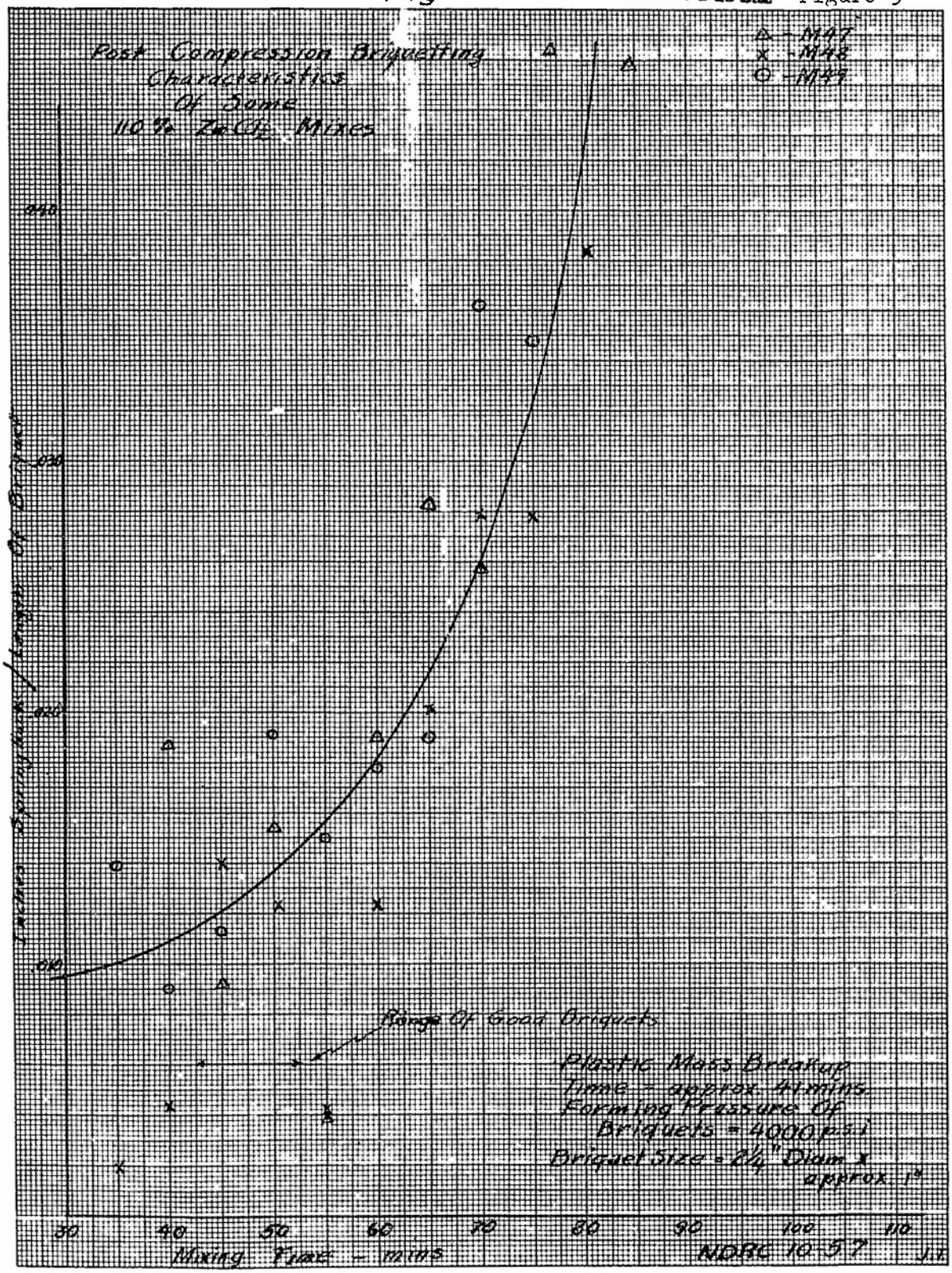
KEUFFEL & ESSER CO., N. Y. NO. 355-14
Millimeters, 5 mm. lines accented, cm lines heavy.
MADE IN U.S.A.

Figure 4



KEUFFEL & ESSER CO., N Y NO 359-14
Millimeters, 5 mm. lines accented, cm lines heavy.
MADE IN U.S.A.

Fig. a CONFIDENTIAL Figure 5



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KEUFFEL & ESSER CO., N. Y. NO 359-14
Millimeters, 5 mm. lines accented, cm lines heavy.
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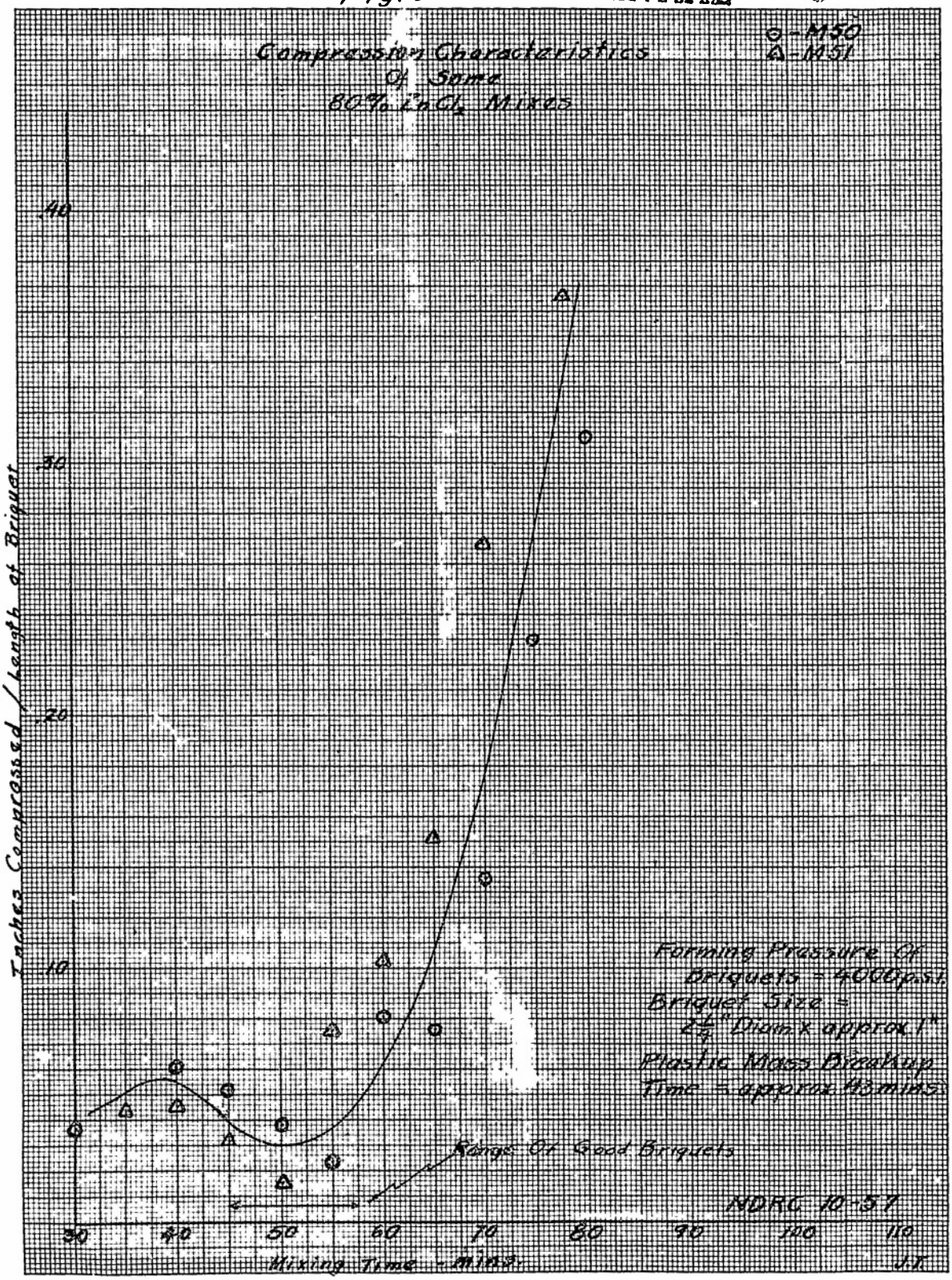
Fig. 6 CONFIDENTIAL Figure 6



KEUFFEL & ESSER CO., N. Y. NO 359-14
Millimeters, 5 mm, Heat scented, cm lines heavy.
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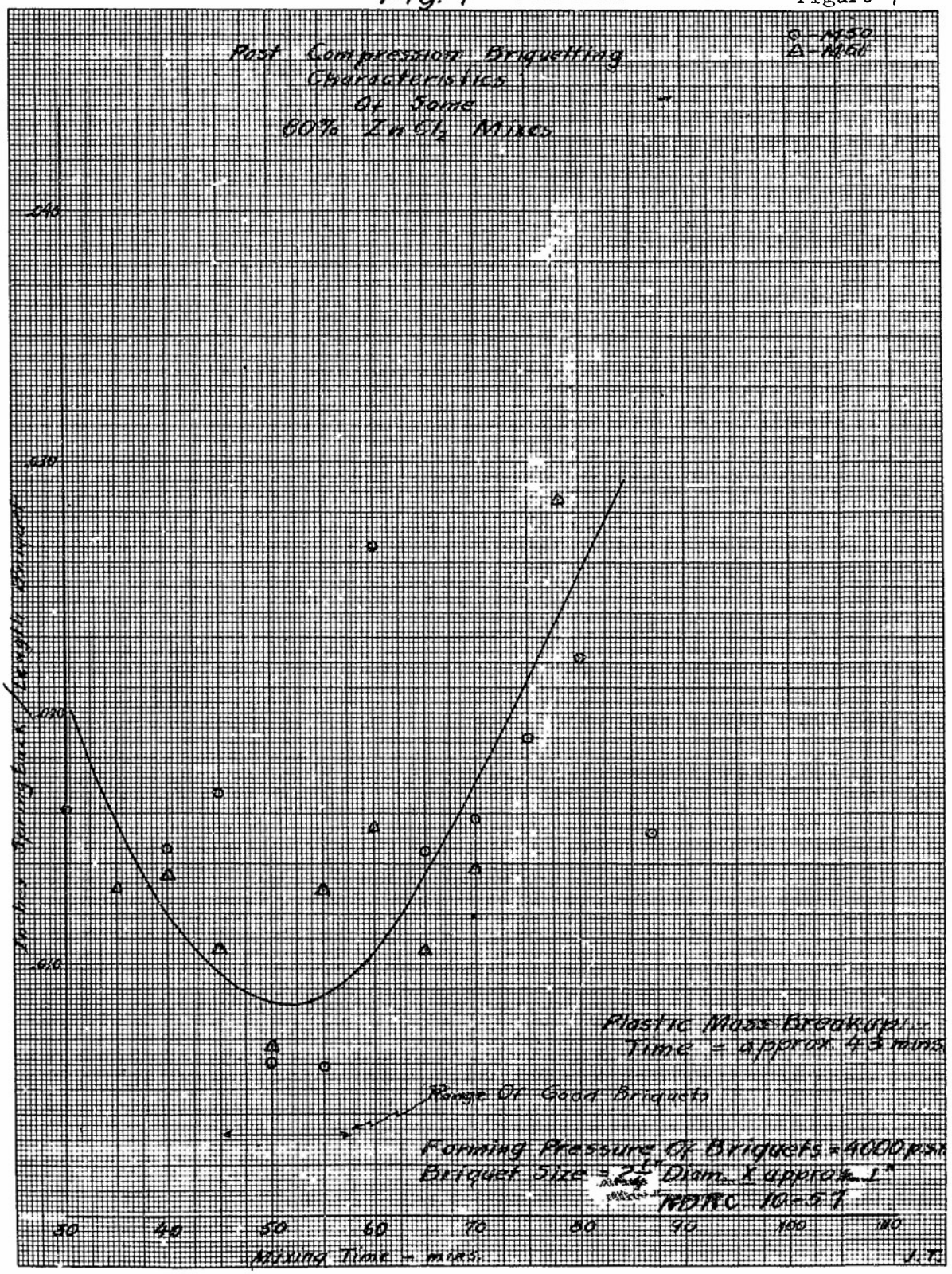
Fig. 7 CONFIDENTIAL Figure 7



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KEUFFEL & ESSER CO., N. Y. NO 359-14
Millimeters, 5 mm. lines accented, cm lines heavy
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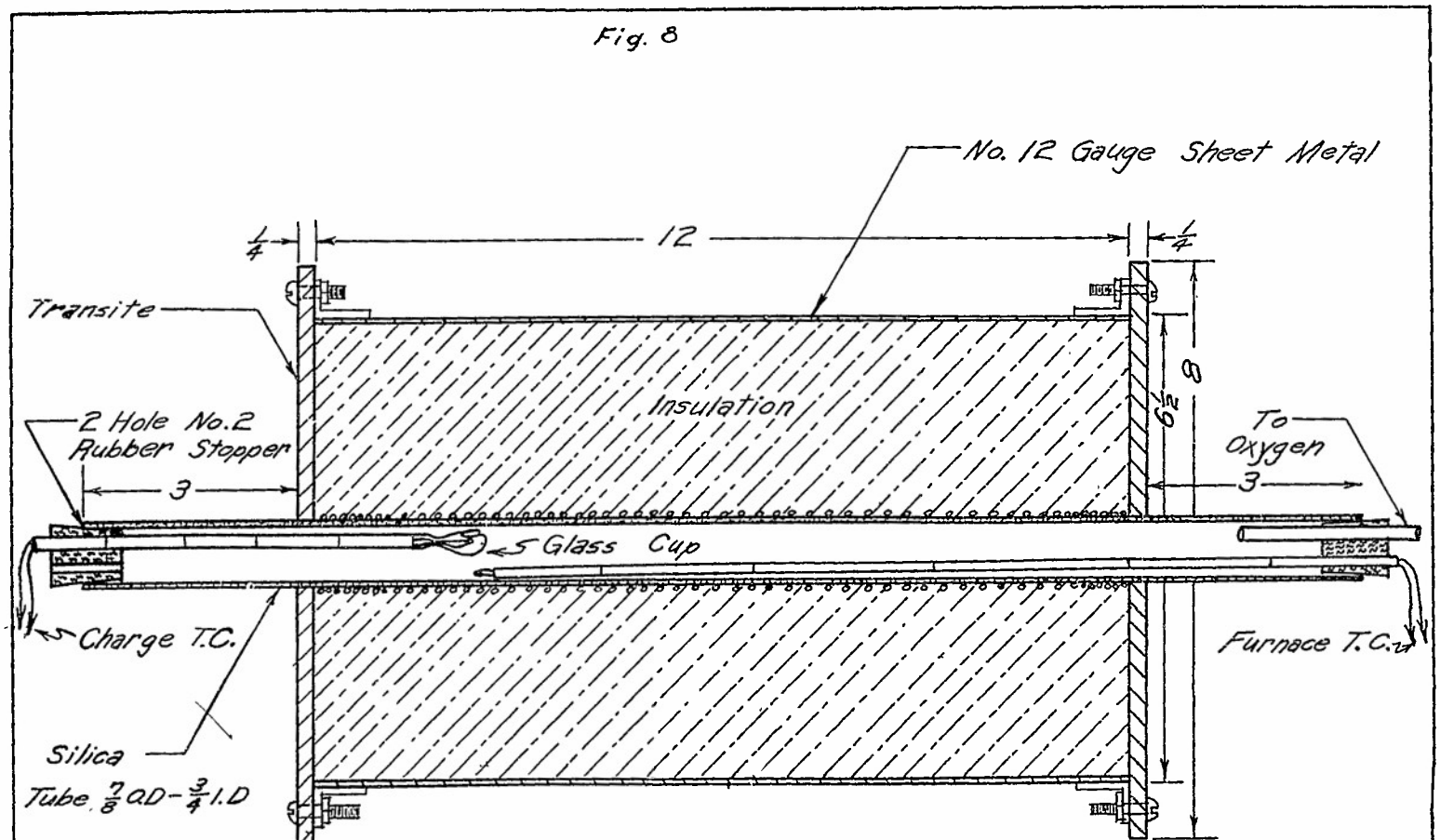
Fig. 8



Silica Core Wound With
No. 18 Chromel Wire

Scale : Half Size

OXIDATION FURNACE
N.D.R.C. 10-57
G.C.

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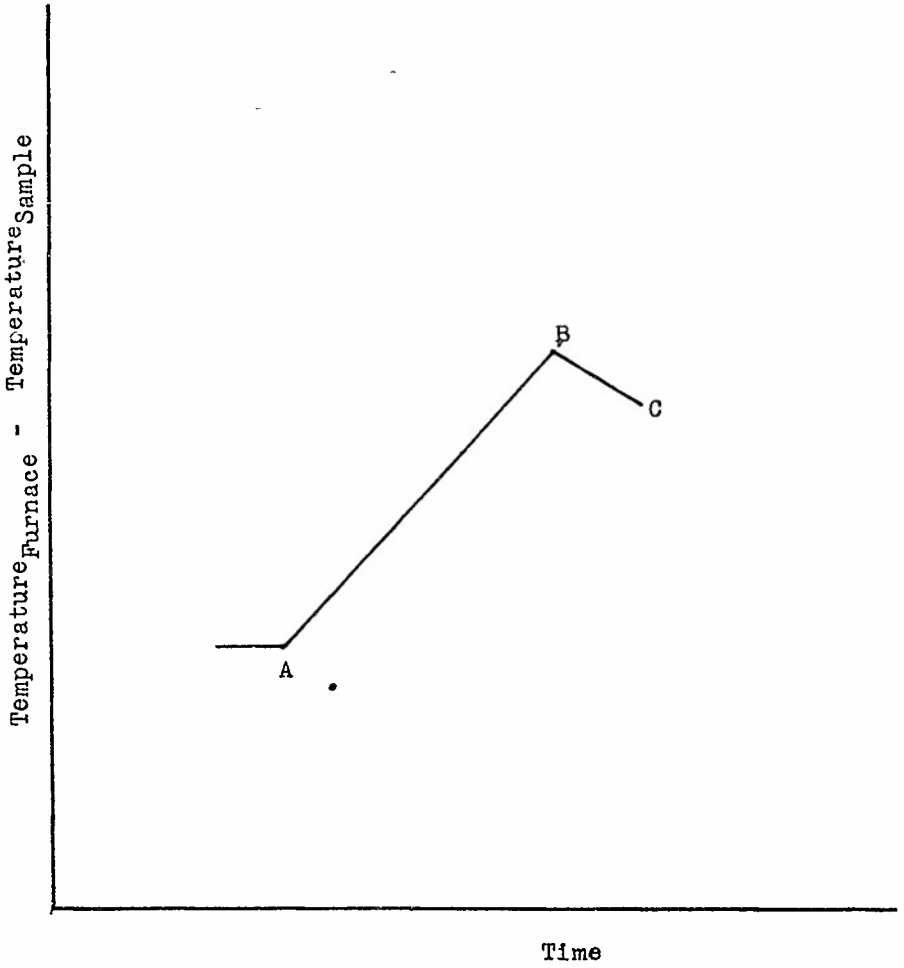
Figure 8

Figure 9

Shape of Curve of Temperature Difference of Sample below Furnace Temperature for Heating Run in Oxygen



APPENDIX I

Summary of Work on Portion of Chemical Activation

This appendix is supplementary to Part III of this report. In this there is presented the effects of changing the washing, grinding, and calcining procedure upon the adsorptive and whetlerite properties of wood charcoal. There are also given the results of using various chemicals of which only zinc chloride and phosphoric acid are satisfactory.

Conclusions. See pages 128 through 129, and pages 133 and 134.

VARIATIONS IN WASHING, GRINDING AND CALCINING PROCEDURE

Description of Apparatus and Operating Procedure. The mixing operation is performed in a Read steam jacketed mixer of six-liter capacity. The mixer is lined with stainless steel, and the mixing blades are of the same material. The steam is supplied by a small 7 kilowatt steam generator with an upper pressure limit of 100 lb./sq.in. The steam pressure is automatically controlled by a pressure-activated mercury switch.

Briquetting is done on a laboratory size Carver press of ten-ton capacity using a 2-1/4 inch die. The briquets are formed under a pressure of 4,000 lbs./sq.in.

The drying was at first done in a convection drying oven, but the acid fumes ruined the thermostat contacts, and it was decided to dry the briquets in specially constructed metal cans in a muffle furnace. The can covers have a tube welded to them which protrudes through the furnace door so that the fumes can exhaust to the atmosphere. This setup has worked very satisfactorily. Comminuting is done on a small jaw crusher, and then on a disk mill.

At first both calcinations were carried out in a chromax rotating retort. Later the first calcination was done in the old Presto-log carbonizing retort. This was modified by replacing the compression rod by a nitrogen inlet. Wire screen baskets made of stainless steel are used to hold the charge, and baffles are placed at the inlet and outlet to direct the flow of nitrogen so that it passes throughthe baskets and flushes off the vapors given off by the charge.

The second calcination, carried out at 1600°F., was done in the chromax retort. The retort itself is suspended in a Globar furnace by a hollow shaft. Inside the shaft there is a concentric pipe, and a packing gland arrangement allows nitrogen to be blown into the retort through the inner shaft, and the calcination products are drawn out through the annular space. There are no flights in the retort.

Washing of the first calcination product is done in a large battery jar over which is mounted a geared-head motor which drives a slowly rotating propeller shaft. The propeller on the end of this shaft disturbs the charcoal sufficiently so that intimate mixing is obtained with the acid and basic washes. An overflow arrangement is used to maintain a steady flow of washing fluids.

The summarized tabulation of operations is given by Tables IX and X.

Definition of Terms

Washing. The samples were washed in an acid solution (1 cc. HCl (conc.) : 100 cc. H_2O), then a basic solution (1 gm. NaOH : 500 gms. H_2O), and finally in a water wash. All mixes were given the same washing treatment.

Briquetting. See above.

Calcining. Calcined (1st)—the mix was calcined for one hour at 1100°F. (593°C.) in a nitrogen atmosphere.

Calcined (2nd)—the mix was calcined for one hour at 1616°F. (880°C.) in a nitrogen atmosphere.

Mixer Runs.

Six mixer runs, ABCDEF, were made with the following composition:

Woodflour	= 1000 gms.	
$ZnCl_2$	= 1100 gms.	jacket Steam/Pressure
Water (tap)	= 1000 ccs.	80 pounds
HCl (conc.)	= 80 ccs.	

The completion of the runs was judged by the appearance of the mix, although the time was fairly constant. The products briquetted with equal ease and formed good briquets. The time of mixing and the mass changes of these six mixer runs are shown in the following tabulation:

Run Number	Time in Mixer	Weight After Briquetting	Weight After Drying
M-37	41 min.	2,031 gm.	1,619 gm.
M-38	41	2,031	1,554
M-39	41.5	1,978	1,608
M-42	41	1,995	1,615
M-43	41	2,087	1,744
M-44	45	2,115	1,703
		12,237	9,843
Average	41.7	2,040	1,639

Samples M-ABCDEF were washed and then tested for PS life and apparent density (AD) with the following results:

	Zn	PS	AD
Unwashed	31.0	0	0.72
Washed	Trace	0	0.49

Treatment of Separate Samples from Mixer Runs M-A through M-F

M-A

Several briquets were taken from each mixer run after the products had been briquetted and dried. The briquets were calcined (1st) before being ground to (6-20) mesh. After grinding, a sample of fines (A_1) was taken to test for the zinc content. The charcoal was then washed and ground to 12-20 mesh, and a sample was taken (A_2) to test the AD, PS lives, and the Zn content. The charcoal was then calcined (2nd). The product (M-A) then was tested for the Zn content, PS life and AD. The results are reported in Table I.

TABLE I

Sample	% Zn	12-16 mesh PS	12-16 mesh AD	16-20 mesh PS	16-20 mesh AD
M-ABCDEF	31.0	0	0.72	-	-
$M-A_1$	32.5	-	-	-	-
$M-A_2$	6.2	43	0.30	-	-
M-A	0	45	0.33	48	0.31

M-B

Part M-BCDEF (the combined mixes (M-ABCDEF) minus M-A) was ground to 6-20 mesh size and dried. One fifth (1000 gms.) of M-BCDEF was separated and used as M-B. The M-B mix was washed and dried and a sample ($M-B_1$) was taken out for testing. Sample $M-B_2$ was taken after the first calcination. The second calcination was performed to give sample M-B. Tests on these samples are tabulated in Table II.

TABLE II

Sample	% Zn	12-16 mesh PS	12-16 mesh AD	16-20 mesh PS	16-20 mesh AD
M-ABCDEF	31.0	0	0.72	-	-
M-B_1	6.0	2	0.45	-	-
M-B_2	3.1	0	0.44	-	-
M-B	Trace	2	0.44	3	0.44

M-C and M-C_p

After M-B was taken out of M-BCDEF, M-CDEF (3968 gms.) was calcined (1st) and a sample (M-CDEF) was taken to be tested. Approximately one-fourth (640 gms.) of M-CDEF was taken and used as M-C. This part was re-briquetted and ground to 6-20 mesh where the sample M-C_1 was taken. M-C was then split and one-half continued as M-C and the other called M-C_p. M-C was washed and dried and ground to 12-20 mesh where sample M-C_2 was taken. M-C then was calcined (2nd) and tested as M-C. To M-C_p 47 gms. of pitch were added to aid briquetting and it was then calcined (2nd). The product was powder and therefore discarded without testing. Results on the other products are shown in Table III.

TABLE III

Sample	% Zn	12-16 mesh PS	12-16 mesh AD	16-20 mesh PS	16-20 mesh AD
M-ABCDEF	31.0	0	0.72	-	-
M-CDEF	30.2	0	0.81	-	-
M-C_1	21.7	-	-	-	-
M-C_2	14.6	40	0.44	-	Most iron present
M-C	0	51	0.35	51	0.35

M-D

After M-B was taken out of M-BCDEF, M-CDEF (3968 gms.) was calcined (1st) and a sample (M-CDEF) was taken to be tested. Approximately one-fourth (650 gms.) of M-CDEF was taken and used as M-D. This part was ground to 12-20 mesh and calcined (2nd) and tested (Sample $M-D_1$). M-D was then washed and tested ($M-D_2$). Test results are shown in Table IV.

TABLE IV

		12-16 mesh		16-20 mesh	
Sample	% Zn	PS	AD	PS	AD
M-ABCDEF	31.0	0	0.72	-	-
M-CDEF	30.2	0	0.81	-	-
$M-D_1$	4.7	45	0.37	-	-
$M-D_2$	Slight trace No iron	46	0.33	53	0.35

M-E

After M-C and M-D were taken out of M-CDEF, the remainder M-EF (1250 gms.) was washed and divided into equal parts, M-E and M-F, each 230 gms. Sample M-EF was taken from M-F after ground. M-E was then briquetted and ground to 12-20 mesh and the fines (192 gms.) taken off as $M-E_p$. After a sample was taken ($M-E_1$), M-E was calcined (2nd) and then tested, the results being shown as Table V.

TABLE V

Sample	% Zn	12-16 mesh PS	12-16 mesh AD	16-20 mesh PS	16-20 mesh AD
M-ABCDEF	31.0	0	0.72	-	-
M-CDEF	30.2	0	0.81	-	-
M-EF	6.0	47	0.32	-	-
$M-E_1$	4.8	49	0.34	-	-
M-E	0	47	0.34	48	0.36

$M-E_p$ and $M-E_{ph}$

The fines after the grinding of M-E were taken, and 66 gms. of pitch were added and the mixture called $M-E_p$. $M-E_p$ was briquetted cold and then ground (12-20) where a 16-20 sample ($M-E_{p1}$) was tested. The fines were again rebriquetted with 100 gms. of pitch added, and heat applied ($M-E_{ph}$). $M-E_{ph}$ was ground and also had a 16-20 mesh sample tested ($M-E_{ph1}$). Both $M-E_p$ and $M-E_{ph}$ were calcined (2nd) separately and tested. $M-E_{ph}$ made much better briquets than $M-E_p$. The results of these tests are shown in Table VI.

TABLE VI

Sample	% Zn	12-16 mesh PS	12-16 mesh AD	16-20 mesh PS	16-20 mesh AD
M-EF	6.0	47	0.32	-	-
$M-E_1$	4.8	49	0.34	-	-
$M-E_{p1}$			-	50	0.32
$M-E_p$				53	0.34
$M-E_{ph1}$				5	0.49
$M-E_{ph}$				0	0.51

content, PS life, and AD is shown in Table VIII.

TABLE VIII

	% Zn	PS	AD
CIR-21 (1st calcination)	5.9	46	0.29
CIM^2-29 (Finished product)	0	48	0.31

CONCLUSIONS

Mixing - The mixer step does very little, if anything, to develop a PS life whether the zinc chloride is washed out or not. The finished mix is black but will become brown if the $ZnCl_2$ is washed out at this point.

M-B shows that if the $ZnCl_2$ is washed out after the mixing step and the regular procedure of calcining followed, the PS life developed is very small, if any. The final products of M-B and M-F have the same outward appearances, however.

Calcining (1st) - From M-B it appears that the calcining (1st or 2nd) does little or no good toward developing a PS life if there is no zinc chloride in the sample. Results show that PS life is developed for the most part in the first calcination when $ZnCl_2$ is present during the calcining, but should be washed out before testing.

Calcining (2nd) - Has little effect on the PS life. It does, however, seem to get rid of any $ZnCl_2$ that is left in the carbon, if only a small amount is present.

There seems to be little difference whether or not the briquets are ground before the first calcination. The PS life and density are approximat ly the same: Example $\frac{M-A}{M-F}$. More $ZnCl_2$ is removed in the first calcination if the particles are finer.

Rebriquetting after the first calcination might improve the PS life slightly (M-C and M-E). When it is rebriquetted using a small amount of pitch the PS life seems to be good (a little higher than normal), but if a lot of pitch is added, the PS life drops considerably.

One mix was rebriquetted using a larger amount of pitch, with heat applied while briquetting. Hard briquets were formed, but after calcining the PS life was negligible.

A good PS life was developed in the charcoal that was not washed until after the second calcination. This could possibly mean that the washing step could be put off until the final step.

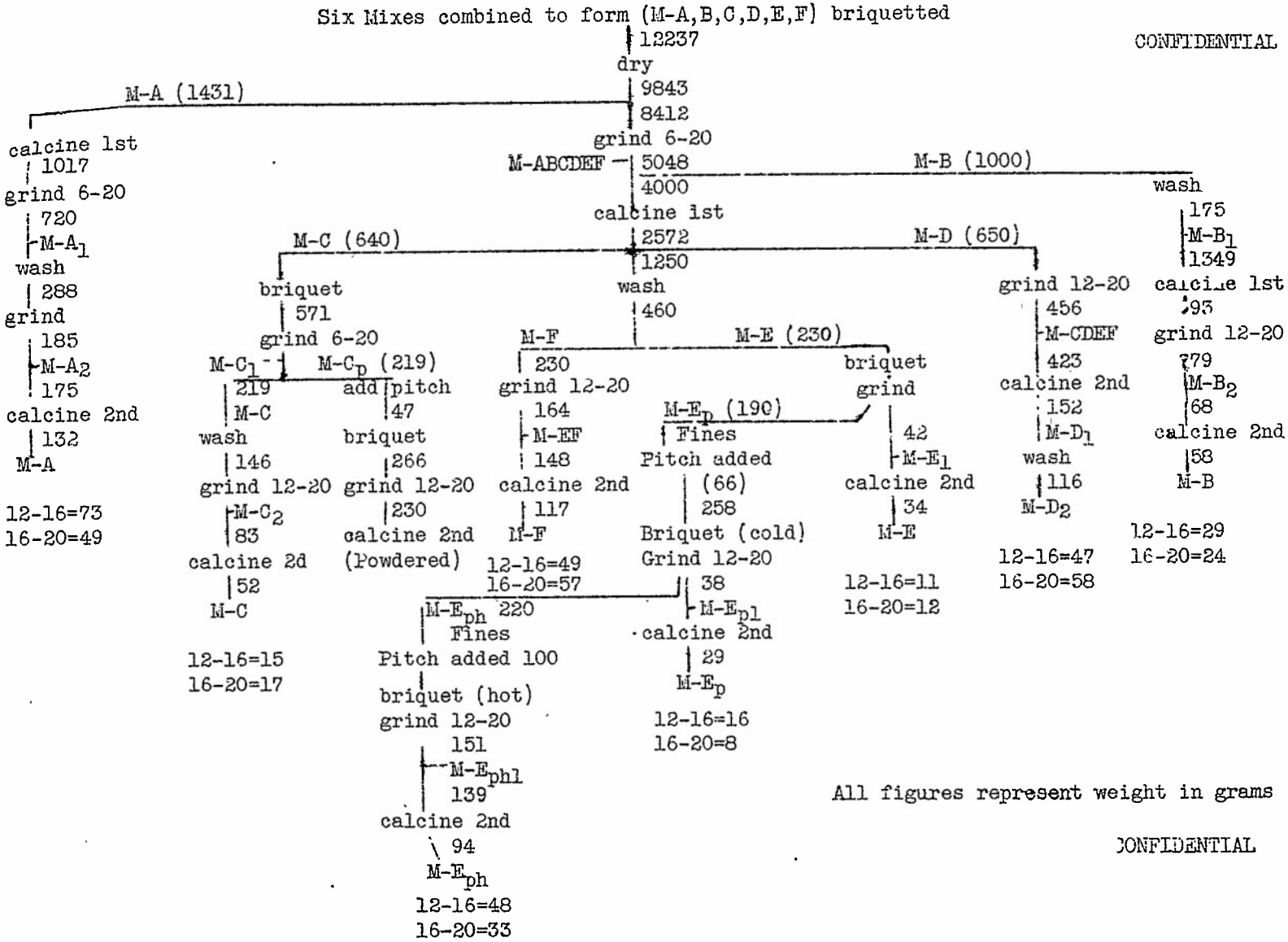
The results from M-40 and M-F checked fairly well. The M-40 mix was slightly easier to briquet. In both batches the second calcination increased the PS life and apparent density slightly.

When the woodflour, $ZnCl_2$, H_2O, and HCl are mixed by hand in an unheated container and then briquetted, the resulting mixture remains light brown and soft. This mix was then calcined (1st). (CIR-22--), the product was similar in appearance to Carlisle Presto-logs using little pressure (light, porous, and a large shrinkage). This was washed and calcined (2nd) and then tested.

Results: (12-20) PS = 26 min. A_1D = 0.21

The total yield of 12-20 of regular mixer runs is about 13%.

TABLE IX Variations in Washing, Grinding, and Calcining Procedure for Woodflour Activation



All figures represent weight in grams

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TABLE X

Data on Samples Taken from (M-ABCDEF)

Name of Sample Taken	Mesh size	12-16 PS min	12-16 AD g/cc	Zn %	16-20 PS min	16-20 AD g/cc
M-ABCDEF*	Fines & 12-16	0	0.72	31.0	-	-
$M-A_1$	Fines	-	-	32.5	-	-
$M-A_2$	12-16	43	0.30	6.2	-	-
$M-B_1$	12-16	2	0.45	6.0	-	-
$M-B_2$	12-16	0	0.44	3.1	-	-
M-CDEF	12-16	0	0.81	30.2	-	-
$M-C_1$	Fines	-	-	21.7	-	-
$M-C_2$	12-16	40	0.44	14.6 (most iron)	-	-
M-E,F	12-16	47	0.32	6.0	-	-
$M-E_1$	12-16	49	0.34	4.8	-	-
M-A	12-20	45	0.33	0	48	0.31
M-B	12-20	2	0.44	trace	3	0.44
M-C	12-20	51	0.35	0	51	0.35
$M-D_1$	12-16	45	0.37	4.7	-	-
$M-D_2$	12-20	46	0.33	slight trace no iron	53	0.34
M-E	12-20	47	0.34	0	48	0.36
M-F	12-20	48	0.34	slight trace	49	0.33
$M-E_{phl}$	16-20	-	-	-	5	0.49
$M-E_{ph}$	12-20	0	0.50	-	0	0.51
$M-E_{pl}$	16-20	-	-	-	50	0.32
$M-E_p$	12-20	48	0.34	-	53	0.34
*M-ABCDEF (washed)	12-16	0	0.49	trace	-	-

Experimental Work on $ZnCl_2$ Concentration

In the past several months some of the experimental work has consisted of varying the concentration of $ZnCl_2$ in the original mixer runs. The test results of these runs are not conclusive, but have been tabulated in Table XI.

TABLE XI

Variations in Zinc Chloride Concentrations

gms. $ZnCl_2$ /1000 gms. Woodflour	Time in Mixer min.	PS Life (min.) 12-16	PS Life (min.) 16-20	AD (g/cc) 12-16	AD (g/cc) 16-20	Mixer Run No.	Yield gms.
0*	46	3	3	0.66	0.67	M-79	211
500	45	42	47	0.49	0.49	67	179
700	42.5	40	48	0.39	0.40	66	148
900	41	46	48	0.38	0.38	65	143
1100	41.7	47	51	0.34	0.34	(M-37-43) M-D,M-F	
1500	47	58	63	0.34	0.35	68	150

*295 gms. (8.07 mols) of HCl were used.

Each mixer run used 1000 gms. of woodflour and 80 ccs. of HCl.

Experimental Work on Other Chemicals

In the past several months the experimental work has consisted of using chemicals other than $ZnCl_2$ in the mixer runs. The purpose of this work was to indicate whether other chemicals could be used to produce a satisfactory charcoal. In these runs a molar concentration of each chemical corresponding to the 110% $ZnCl_2$ (8.07 mols) mixes was used, with some exceptions. The chemicals used were: H_3PO_4, H_2SO_4, H_3BO_3, HCl, $CrCl_3$, $CuNH_4Cl$, $MgCl_2$, $CaCl_2$, $Ca(OH)_2$, NaOH, NaCl, $FeCl_2$, $FeCl_3$, and $Zn_3(PO_4)_2$. Another run was made in which a mixture of Na_2SiO_3 was added to the acidified water-woodflour mix.

A procedure similar to that of the $ZnCl_2$ mixes was followed, although there was some variation in the washing schedule. As shown previously, changing the washing step from after the first calcination to after the second calcination had no effect on the PS life. Therefore, the mixes M-61, M-62, M-64, and M-65 were washed after the second calcination, but the continuous clogging of the exit tube of the rotary retort (second calcination) necessitated the restoring of the washing to between the two calcinations. All mixes after M-65 were washed after the first calcination.

Conclusions. The only chemicals which developed a satisfactory PS life were $ZnCl_2$ and H_3PO_4. The charcoal made from the H_3PO_4 run had an excellent PS life (67 mins., for 12-16 mesh and 78 mins. for 16-20). Some additional experimental work on mixtures of these two compounds might be of value, for the HCl used to acidify the $ZnCl_2$-water-woodflour mixture could probably be replaced by the H_3PO_4, with a possible increase in PS life.

Some other chemicals ($CrCl_3$, $MgCl_2$, $CaCl_2$, $FeCl_2$, $FeCl_3$, NaCl, H_2SO_4, and HCl) reactedwith the woodflour and gave a briquettable product, although the PS tests showed no measurable activation. However, no analyses were made to determine the amount of chemical left in the char, and it is quite possible that the washing technique was unsatisfactory (though satisfactory for the $ZnCl_2$ runs). Also the amount of the various chemicals used was arbitrarily set as equal to the molar concentration of the 110% $ZnCl_2$ runs (8.07 mols.) but this need not be the point of best performance.

The following chemicals seemed to have little or no reaction with the woodflour: H_3BO_3, $Ca(OH)_2$, NaOH, and $CuNH_4Cl$ (with NH_4OH added).

Only 2.2 mols of $Zn_3(PO_4)_2$ were available for this mixer run, which showed no reaction with the woodflour. An increase in the amount of $Zn_3(PO_4)_2$ used may give more favorable results.

TABLE XII Experimental Results of Other Chemicals

Mix No.	Chemical Kind	gms.	mols	H_2O ccs	HCl ccs	Time in mix min.	PS min. 12-16	PS min. 16-20	AD g/cc 12-16	AD g/cc 16-20	Yield 12-20 gms	Remarks
79	HCl	295	8.1	1050	←	46	3	3	0.66	0.67	211	0% $ZnCl_2$ 100% HCl
75	HCl	103	2.8	1190	←	44	Passed normal end point. Powdered during drying					35% HCl
59	500 gms. woodflour			500	--	160	No change--discarded					0%$ZnCl_2$, HCl
58	$ZnCl_2$ See Remarks	1100	8.1	1000	80	53	--	53	--	0.40	131	50gms Na_2SiO_3 added
60	H_3PO_4	790	8.1	1140	--	48	Passed normal end point					Wouldn't briquet
61	H_3PO_4	790	8.1	1140	--	42	67	78	0.47	0.48	151	Watch for phosphorus burning.
62	H_2SO_4	790	8.1	1040	--	19	2	2	0.53	0.54	124	Mix turned black almost immediately
63	H_3BO_3	500	8.1	1000	--	530	No noticeable reaction					Slightly darker
72	$Ca(OH)_2$	600	8.1	2000	--	--	No noticeable reaction					
73	NaOH	323	8.1	1000	--	--	No noticeable reaction					Darker
64	$CrCl_3$	1280	8.1	3000	--	68	13	12	0.91	0.91	382	Cr very hard to wash out. Still lot Cr present.
69	$CuNH_4Cl$ See Remarks	1485	13.9	1000	--	--	No noticeable reaction					1000cc NH_4OH added
70	$MgCl_2 \cdot 6 H_2O$	1640	8.1	1000	10	112	2	2	0.37	0.37	151	HCl added after 20 min
71	$CaCl_2 \cdot 2 H_2O$	1200	8.2	1000 initially	See Remarks	203	Discarded because mix became so stiff that mixer was stopped.					Plastic at 45 min.(medium brown) Plastic & dry at 60 min.(dark brown)**
74	NaCl	482	8.2	1500 initially	0 initially	95	0	0	0.50	0.50	162*	dry & powdery at 60 min. 80 cc HCl and****
76	$FeCl_2$	1023	8.1	1000	80	40	0	0	0.36	0.37	147	After 1st calcination both mixes glistened as powdered iron***
77	$FeCl_3 \cdot 6 H_2O$	2180	8.1	1500	80	55	Powdered while washing					
78	$Zn_3(PO_4)_2$	855	2.2	2000	25	70	No noticeable reaction					Only 25% desired amt.$Zn_3(PO_4)_2$ available.

General Note: All mixes used 1000 gms. of woodflour (except M-59)

*Only 4/5 of mix was briquetted.
**Mixer stopped at 75 min., 500 cc H_2O added; Mixer stopped again, 1000 cc added. At 200 min. the mix was tarry.
***White flaky substance in exit tube.
****500 cc H_2O added. At 80 mix darker but powdery--80 cc HCl and 500 cc H_2O added mix turned dark brown immediately. No plastic state.

Whetlerite Results

Mixes M-58, M-61, M-65, M-66, M-67, and M-68 were whetlerized (ASC-1) individually using only the 12-16 mesh size samples. The results are given in Table XIII.

TABLE XIII

ASC-1 Whetlerization

Tests given		16-20 M-58	12-16 M-61	12-16 M-65	12-16 M-66	12-16 M-67	12-16 M-68
SA 80-80		146	1	162	177	55	6.0
CC 80-80	2.5 cm.	5	0	5	3	2	0
	3.5 cm.	16	0	14	9	3	2
	5.0 cm.	42	0	42	28	13	5
AC 80-80		110	10	104	94	67	72
% H_2O pickup		34.6	48.0	41.6	35.5	30.1	54.0