1855/2102

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Date of request 6/22/95 Expected receipt of document 7/30/95

Document number A-3761 Date of document

Title and author (if document is unnumbered)

(This section to be completed by Document Center)

Date request received 6/27/95

Date submitted to ADC 6/30/95

Date submitted to HSA Coordinator 6/27/95

(This section to be completed by HSA Coordinator)

Date submitted to CICO ~~6/30/95~~ 7/17/95

Date received from CICO 7/17/95

Date submitted to ChemRisk/Shonka and DOE 7/17/95

(This section to be completed by ChemRisk/Shonka Research Associates, Inc.)

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1855 | 2102

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A-3761

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PLANT RECORDS DEPT.
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X-REF.
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A-7361 — DECLASSIFIED —
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CG-PGD-4 Classification Specialist
(Authorized Declassifier's name and organization)
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Summary Laboratory Report

The Kellex Corporation

CARBON TRAPS

Classification changed to: UNCLASSIFIED
(level and category)
Thomas W. Kelley 7/12/95
AEC or ADD signature (first reviewer) Date
7/17/95
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Carbide and Carbon Chemicals Corporation Operating Contractor for the U.S. Atomic Energy Commission.

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Jersey City, N. J.
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TABLE OF CONTENTS

Subject Index

		Page No.
I.	Introduction	1
II.	Scope of Work	2
III.	Summary of Conclusions	3
	A. Solid C-616 Absorbents	3
	B. Selection of Carbon Trap Charge	3
	C. Protective Diluents	4
	D. Segregation of Carbon-Alumina Mixtures	4
	E. Analysis of Exit Gases from Carbon Traps During C-616 Absorption	4
	F. Mechanism and Products of C-616 Absorption by Carbon	5
IV.	Experimental Data and Conclusions	6
	A. Selection of C-616 Absorbent	6
	1. Activated Carbon	7
	a. High Activity Carbon	8
	b. Low Activity Carbon	8
	c. C-716 Impregnated Carbon	9
	d. Combination Carbon-Alumina Particles	10
	2. Florite Dessicant	11
	3. Silica Gel	11
	4. Sodium Fluoride	12
	5. Other Absorbents	13
	B. Choice of Diluent	13
	1. Reason for Use of Diluent	13
	2. Desired Diluent Characteristics	13
	3. Tests of Diluent Properties	15
	4. Effect of Dilution on Temperature Rise	16
	C. Selection of Carbon for Use in Traps	17
	1. Heat of Absorption	18
	2. Effect of Carbon Activity and C-616 Absorption Efficiency	19
	3. Effect of Dilution	20
	4. Effect of Other Factors on Caking	20
	5. Summary	21
	D. Selection of Charges for Plant Carbon Traps	21
	1. Basis for Selection	21
	2. Experimental	22
	3. Section 100	23
	4. Section 600	26
	a. Standard Charge	26
	b. 6/8 Mesh High Activity Carbon	27
	c. Very High and Low Activity Carbon	27
	5. Section 300	29

SECRET

UNCLASSIFIED

Subject Index (cont)

	Page No.
6. Mobile Carbon Trap	30
7. Summary of Conclusions	31
E. Protective Diluent	32
1. Possibilities	32
2. Boron Carbide	32
a. Reaction with C-616	32
b. Conclusion	34
3. Cadmium Oxide	34
a. Cadmium Oxide Coating of Aluminum	34
b. Reaction with C-616	34
c. Testing of Cadmium Content	35
F. Segregation of Carbon-Alumina Mixtures	36
G. Performance of Charges Under Pilot Plant Conditions	42
1. Apparatus	42
2. Technique	45
3. Experimental	46
a. Section 600	47
b. Section 300	48
4. Conclusions	51
H. Long Exposure of Carbon to C-616	52
I. Analysis of Exit Gases from Carbon Traps	53
J. Absorption of C-616 by Activated Carbon	56
1. Experimental	57
2. Materials Used	57
3. Results of Experiments	58
a. Activity of Carbon	58
b. Size of Carbon Particles	59
c. Type of Carbon Particles	60
d. Moisture Content of Carbon	60
e. Temperature of Carbon	61
f. Partial Pressure of C-616	61
g. Mass Velocity of C-616	62
h. Rate of C-616 Absorption	63
i. Reversibility of C-616 Absorption	66
j. Fluorocarbon Production	66
k. X Analysis on Carbon after Exposure to C-616	71
4. Interpretation of Results	73

UNCLASSIFIED

~~SECRET~~

Index to Figures

Figure No.	Title	File #
1	Adiabatic Temperature Rise in Alumina-Activated Carbon Bed Mixtures	Y-3054
2	Reactor for Testing Carbon Trap Charges	Y-3083
3	Glass Jar for Particle Count	Y-1747
4	Apparatus for Determining the Efficiency of Carbon as Absorbent for C-616	Y-1878
5	C-616 Saturator	Y-1896
6	3/4" O.D. C-616 Absorption Tube	Y-1929
7	Detail of Exit Carbon Trap	Y-1897
8	Effect of Carbon Activity on Absorption of C-616	Y-3084
9	Effect of Carbon Particle Size on Absorption of C-616	Y-3052
10	Effect of Carbon Particle Size on Initial Absorption of C-616	Y-3085
11	Effect of Carbon Temperature on C-616 Absorption	Y-3048
12	Effect of C-616 Partial Pressure on Absorption Efficiency of Carbon	Y-1790
13	Effect of Time on C-616 Absorption by Activated Carbon	Y-3086
14	Apparatus for Fluorocarbon Production	Y-1975
15	Production of Heavy Fluorocarbons as a Function of Carbon Temperature	Y-3087
16	Transmission of Carbon Extract vs. Amount Extracted	Y-3088
17	X Analysis on Carbon after C-616 Exposure	Y-3053

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SECRET

Index to Tables

Number	Title
I	Properties of Solid C-616 Absorbents
II	Comparison of Temperature Rises in Charges of High Activity Carbon Diluted with Alumina with Charges of Low Activity Carbon
III	Tests on Diluents
IV	Tests on Different Types of Carbon
V	Preliminary Tests - Section 100 Conditions
VI	C-616 Absorption - Section 100 Conditions
VII	C-616 Absorption - Section 600 Conditions
VIII	C-616 Absorption in Charges Containing 6/8 Mesh High Activity Carbon - Section 600 Conditions
IX	C-616 Absorption by Low and Very High Activity Carbon
X	C-616 Absorption - Section 1 Recovery System
XI	C-616 Absorption - Section 300-2 Recovery System
XII	C-616 Absorption - Section 300-3 Recovery System
XIII	C-616 Absorption - Mobile Carbon Trap Conditions
XIV	C-616 - B_4C Reaction Rates
XV	Analyses of CdO Coated Alumina
XVI	Check on Method of Computation of Mixing Results
XVII	1:2 Mixture, 4 Mesh Al_2O_3: 2/4 Mesh High Activity Carbon Pellets
XVIII	1:1 Mixture, 4 Mesh Al_2O_3: 6/8 Mesh High Activity Carbon Pellets
XIX	1:1 Mixture, 6/8 Mesh Al_2O_3: 6/8 Mesh High Activity Carbon Pellets
XX	Segregation in a 1.5:1 Mixture of 4 Mesh Alumina and 6/8 Mesh High Activity Carbon Pellets

SECRET

UNCLASSIFIED

Index to Tables (cont)

Number	Title
XXI	Segregation in a 2:1 Mixture of 4 Mesh Alumina and 2/4 Mesh High Activity Carbon Pellets
XXII	Segregation in a 2:1 Mixture of 2 Mesh Alumina and 2/4 Mesh Hi h Activity Carbon Pellets
XXIII	C-616 Absorption - Pilot Plant Runs Under Section 600 Conditions
XXIV	C-616 Absorption - Pilot Plant Runs Under Section 300 Conditions
XXV	Absorption of C-616 Under Continuous Purge Unit Conditions
XXVI	C-616 Concentrations - Entrance and Exit Gas Under Continuous Purge Unit Conditions
XXVII	Exit Gas Analysis as Function of C-616 Absorbed - Section 300 ± 1
XXVIII	Exit Gas Analysis as Function of Absorbed - Section 600, Trap F-605
XXIX	Experimental Conditions - Section 300 ± 1 Runs for Exit Gas Analysis
XXX	Experimental Conditions - Section 600 Runs for Exit Gas Analysis
XXXI	Chemical Analysis of Activated Carbon
XXXII	Effect of Carbon Activity on Absorption of C-616
XXXIII	Effect of Carbon Particle Size on Absorption of C-616
XXXIV	Comparison of C-616 and CCl_4 Absorption on Activated Carbon, Showing Effect of Type of Carbon on the Absorption of C-6 6
XXXV	Effect of Drying Carbon on C-616 Absorption
XXXVI	Effect of Carbon Temperature on Absorption of C-616
XXXVII	Effect of C-616 Partial Pressure on Absorption Efficiency of Carbon
XXXVIII	Rate of C-616 Absorption by Activated Carbon
XXXIX	Effect of Time on C-616 Absorption by Activated Carbon
XL	Distillation of 100 cc of Fluorocarbons from C-616, Carbon Reaction

Index to Tables(cont)

Number	Title
XLI	Production of Heavy Fluorocarbons in the C-616, Carbon Reaction
XLII	X Analyses on Carbon after C-616 Exposure
XLIII	X Analyses on Carbon-Special Test

CARBON TRAPS

I. Introduction

On the basis of preliminary experimental work, the Process Engineering Group designed carbon traps for use at various points in the plant for C-616 removal. It was planned at the time to charge these traps with average activity carbon. Later, calculations from the measured heat of absorption of C-616 on activated carbon showed that temperatures of 600°F or higher might be attained in these traps in service. Since no experimental work had been performed on the reaction of C-616 with carbon at that temperature, it was decided to perform experiments to determine in advance the effect of exposing carbon to C-616 both at 400° and at 600°F. These experiments showed that under these conditions a badly caked bed would result. If the reaction were allowed to proceed long enough, a complete plugging of the bed occurred.

No other satisfactory solid absorbents for C-616 had been found at the time of starting these experiments. It was, therefore, decided to try to adapt a carbon absorbent in some way to make it suitable for use in the already designed traps at the site. As a possible means of avoiding the large temperature rises occurring as a result of C-616 absorption on carbon, it was suggested that an inert diluent be added to the charge. This would serve to absorb the heat of the reaction without permitting an excessive temperature rise.

II. Scope of the Work

Experiments were performed to discover a suitable diluent for carbon. As a result of these experiments it was decided that crystalline alumina would be the best of the materials tested for this purpose. After selecting the diluent, laboratory scale runs were made using a series of carbon-alumina mixtures under conditions comparable to those which would occur in carbon traps at site during typical services. From the results of these experiments charges for the different traps at the site were tentatively selected. Later these charges were tested on a pilot plant scale in order to observe under conditions more nearly analogous to those to be met in practice, the operation of these traps.

While conducting experiments on the selection of charges, work was also undertaken to obtain answers to related problems such as choosing a protective diluent for use in traps handling enriched C-616, developing means of charging mixtures of carbon and alumina without segregation, and developing means of removing condensed C-616 from the traps. In addition, other solid C-616 absorbents were tested and a relatively thorough investigation was made on the phenomenon of C-616 absorption by activated carbon.

Completion of the work on a solid C-616 absorbent occupied from June 1, 1944 to June 15, 1945.

III. Summary of Conclusions

A. Solid C-616 Absorbents

The following solid materials have been tested as C-616 absorbents:

1. Activated Carbon: In this category are included many shapes, sizes and grades of activity of carbon. Also included are activated carbon pretreated with an inert liquid (C-716) and a combination activated carbon-alumina mixture.

2. Silica Gel: Both Santocel, an expanded silica gel, and the ordinary Davco product were tested.

3. Activated Alumina

4. Fluorite Dessicant

5. NaF and $NaHF_2$ Pellets

Of these materials high activity carbon was the most efficient and the quickest acting C-616 absorbent under normal conditions. Fluorite Dessicant showed some promise but absorbed C-616 relatively slowly. The other materials did not appear to have desirable properties from the point of view of possible plant use, although by special drying or use in vacuum activated alumina or NaF might prove useful for this job in the laboratory.

B. Selection of Carbon Trap Charge

A mechanical mixture of particles of high activity carbon and crystalline alumina has been shown to be useable in the traps at the K-25 plant. A combination pellet containing carbon, alumina and cadmium would be less difficult to charge; this material is in the process of development by the National Carbon Company.

The optimum proportion of carbon to alumina was found to vary in the different services for which carbon traps are used.

C. Protective Diluents

Cadmium oxide, although perhaps reacting with C-616, does not form a volatile cadmium compound as a result of the reaction. It was therefore selected as a suitable protective diluent for use in carbon traps. It was added as a coating on the alumina used as diluent.

Boron carbide reacts to some extent with C-616 forming a volatile boron compound.

D. Segregation of Carbon-Alumina Mixtures

When a uniform mixture of activated carbon and crystalline alumina is disturbed, as in pouring, serious segregation will occur. Methods of charging traps have been developed which minimize this effect.

E. Analysis of Exit Gases from Carbon Traps During C-616 Absorption

The exit gases from carbon traps have been analyzed for both HF and C-616 as functions of time during C-616 absorption. Results obtained on absorbing C-616 from 10% C-616 in G-74 mixture flowing at 0.16 ft./sec. are fairly typical. After a preliminary period during which C-616 concentration may run 1.0 mg./liter or higher, the concentration of C-616 in the exit gases drops to less than 0.02 mg./liter. The concentration remains at or below this level until the trap is nearly exhausted, then it begins to increase rapidly. HF concentration varies in much the same manner with time, but the concentration of HF in the exit gases starts to rise rapidly before that of C-616.

F. Mechanism and Products of C-616 Absorption by Carbon

The data obtained in small scale runs indicate that between 75°F and 350°F the major part of the C-616 is held to the carbon by activated adsorption. Above 350°F, chemical reaction begins to become important and at 500°F, and higher, reduction of C-616 and production of fluorocarbons is the dominating factor for exposures longer than a very few minutes.

In the range of temperature where activated adsorption dominates, there is little fluorocarbon production and most of the X present on the carbon is in the +6 valence state. When chemical reaction occurs, XF_4 is formed and a mixture of fluorocarbons, ranging from CF_4 to a dark resinous substance which is brittle at room temperature, is produced. Around 400°F there is evidence that one of the reaction products is $(CF)_X$.

IV. Experimental Data and Conclusions

A. Selection of C-616 Absorbent

The primary criterion for selection of a given material as an absorbent for C-616 is the weight of C-616 absorbed by a given quantity of absorbent before significant amounts of C-616 issue in the exit gases of the absorption trap. This criterion is termed the efficiency. Thus, selection of a C-616 absorbent for the plant primarily involved measurement of efficiency under various conditions of temperature, pressure, dilution and flow rate expected for plant service. Required plant efficiencies were, in general, very high. These stringent requirements almost immediately eliminated a large group of possible absorbents.

Another consideration in the selection of an absorbent is the ease of handling, both before and after C-616 absorption. In connection with ease of handling after C-616 absorption, the reversibility of the C-616 absorption is of interest, and so also is HF generation during and after C-616 absorption. Knowledge of the temperature rise as a result of C-616 absorption, together with the effect of high temperatures on the absorbent in the presence of C-616, and the rate of the absorption reaction is also necessary in the selection of an absorbent.

In Table I is given a summary of the information available on these factors for all the absorbents tested in these laboratories. It will be seen from the tabulated data that,

of the absorbents tested, the various forms of activated carbon, Florite Dessicant and Santocel have efficiencies which are satisfactory for large scale use of an absorbent for recovering plant waste C-616. The following considerations detail individual factors which apply to the results of Table I.

1. Activated Carbon

Because of its extremely satisfactory efficiency, activated carbon represented an obvious best choice for a plant waste gas absorbent. However early experiments showed that if a carbon trap approached temperatures of 300-400°F, the absorbent bed became plugged with a fused cake. Further study indicated that these temperatures could easily be approached and possibly exceeded in plant service. Absorption of C-616 by activated carbon was found to be a process which, at temperatures between room temperature and 300°F involved no change in the valence of the C-616. Above 400°F, chemical reduction of C-616 to XF_4 or XF_5 occurred. Evidence was obtained that the latter chemical reaction caused caking and plugging through the formation of an XF_4-fluorocarbon resin cement which bound the carbon particles together in a hard solid mass. Acceptance of this hypothesis leads to the conclusion that beds cake because the heat of absorption raises the temperature of the carbon to a point where reaction can occur rapidly.

Thus if a large temperature rise could be avoided in

plant traps, activated carbon might be satisfactory for plant use. For this reason, further work on this absorbent centered on means to reduce this temperature rise.

a. High Activity Carbon

One of the most obvious methods for reducing the temperature rise was to reduce the amount of C-616 absorbed by any given mass of carbon or mixed carbon and diluent. This was possible since most of the traps at the site were designed with a relatively large factor of safety in capacity. The use of alumina as a diluent for high activity carbon was tried and found to be a practical method for eliminating caking without an excessive reduction in absorptive capacity, at least on a laboratory scale. It was found that a mixture of one part of high activity carbon with one and a half to two parts by volume of crystalline alumina gave a bed with the desired C-616 absorptive properties. Mixtures of this type were recommended for use in the traps at the site. This type of C-616 absorbent charge will be discussed in more detail in the section on selection of charges.

b. Low Activity Carbon

Instead of using a diluent it should be possible to use a lower activity carbon to reduce the amount of C-616 absorbed per unit volume. Although no data is available on the heat of absorption of C-616 on low

activity carbon, it might be expected that the heat evolved per mole of C-616 absorbed would be lower for low than for high activity carbon. This would to some degree offset the advantage in heat capacity gained by adding alumina to high activity carbon. However, laboratory experiments on which the data are presented in Table II indicate that for comparable values of the absorption per unit volume, a higher temperature is to be expected for low activity carbon. Although the caking, as a result, would probably be slightly worse, it might prove desirable to accept a further reduction in absorption efficiency to avoid caking in exchange for the much greater ease in charging a trap uniformly resulting from using a homogeneous charge.

c. C-716 Impregnated Carbon

One possible means of reducing the heat of absorption of C-616 on carbon is to absorb on the carbon another compound before exposing it to C-616. If this second compound is desorbed on exposure to C-616, that is, if C-616 is preferentially absorbed, the heat of absorption of C-616 will be reduced by an amount equal to the heat of absorption of the other compound.

Since C-716 is inert to C-616, it was felt that C-716 might be used in this manner. A test of absorption of C-716 on high activity carbon from a 10% C-716

in G-74 mixture showed that at room temperature it was absorbed to the extent of 36% of the original weight of the carbon. Exposure of the saturated carbon to 10% C-616 in G-74 at room temperature and collection of the products showed that at least half of the absorbed C-716 was desorbed. This indicated at least partial preferential absorption of C-616.

Calculations, however, make the process appear doubtful in value. It is assumed from analogy with other similar cases, that the heat of adsorption of C-716 on carbon is twice the heat of vaporization of the liquid, that is, approximately 1/3 of the heat of absorption of an equal weight of C-616, and that under the conditions of operation the carbon will have an absorption efficiency for C-616 of 2.5. On this basis, the heat of absorption of C-616 on carbon is reduced by only 10-15% by pretreating with C-716. This reduction is not enough to significantly affect the results.

d. Combination Carbon-Alumina Particles

Since the use of high activity carbon with an inert diluent of high density appears to offer advantages over the use of low activity carbon, with respect to absorptive capacity obtainable before caking occurs, efforts have been made to obtain both high heat capacity and high C-616 absorptive capacity by combining alumina

and carbon in single particles. Attempts were made in those laboratories to coat crystalline alumina with sugar carbon and then activate the carbon with air at 400° and 500°C. On one occasion a small amount of a nearly satisfactory product was obtained, but the technique of activation failed to give consistent results because of lack of adequate control.

The problem of making the combination particles was then given to the National Carbon Company, who are continuing the work at the present time.

2. Florite Dessicant

Florite Dessicant, an activated natural aluminum silicate, is one of a number of standard absorbent materials tested, and is a fairly satisfactory material for C-616 absorption. It has the disadvantage of a slow rate of C-616 absorption following the initial rapid uptake. Over short times it has only about half the absorptive capacity by volume of the carbon mixture scheduled for plant use. It is, however, much cheaper than activated charcoal. It does not cake at temperatures where carbon would cake badly. It also is safer to handle after C-616 absorption. Although it is a dessicant, and undoubtedly contains some moisture, there is practically no HF liberated as a result of C-616 absorption.

3. Silica Gel

Santocel has been eliminated from practical considera-

tion because of its form, (a very fine powder), and because it cakes badly. While experimental quantities of Santocel in a larger particle size were obtained and tested for C-616 absorption, the particles were very weakly bound, had a much lower capacity and still caked on exposure to C-616.

4. Sodium Fluoride

It was found on passing mixtures of HF and C-616 over anhydrous NaF pellets, that large quantities of C-616 were absorbed. This observation led to the trial of NaF and $NaHF_2$ as C-616 absorbents.

Tests on NaF without initial evacuation showed only slight absorption in the absence of HF. Since the pellets used were not dried, the observed absorption could be accounted for on the basis of the HF and TO_2F_2 formed by hydrolysis of the C-616.

Experiments were next performed at atmospheric pressure with pellets containing 75% $NaHF_2$. These, too, showed only slight absorption of C-616 (25% by weight of the pellets). By crushing the pellets, the absorption was increased but caking resulted. Caking also resulted when some of the NaF pellets were treated with HF and then with C-616. From the results of these last experiments, it has been concluded that very low melting mixtures of NaF and HF and of NaF, HF and C-616 exist, and that these mixtures are encountered when C-616 is added to $NaHF_2$.

5. Other Absorbents

The data given in Table I on the other absorbents tested is sufficient to condemn them.

B. Choice of Diluent

1. Reason for Use of Diluent

The reason for adding an inert diluent to activated carbon for plant C-616 traps has been discussed. In the absence of the diluent, it has been shown, both theoretically and experimentally, that a very high temperature will result from the absorption of C-616 in plant size traps. It has also been shown experimentally, that at the high temperature resulting, the carbon will cake together and the bed will plug if addition of C-616 is continued.

Addition of a diluent serves to prevent the temperature rise both by reducing the amount of carbon in a given volume, thus reducing the amount of C-616 which can be absorbed in that section, and by increasing the average heat capacity of the bed. It also serves to separate particles of carbon, thus reducing the probability of getting caking under a given set of conditions.

2. Desired Diluent Characteristics

A list of properties desirable in a diluent is given below:

a. Cheap

b. Non-porous

c. Available in quantity

d. Acceptable to those in charge of reclaiming product

e. High mechanical strength

f. Refractory

g. Non-hygroscopic

h. Hydrogen free

i. No volatile or toxic reaction products on reaction with C-616

j. High heat capacity per unit volume

k. Approximately same density as activated carbon

l. Particle size similar to carbon used

m. Non-caking

The diluent should be refractory since high surface temperatures of the carbon may occur which would melt a non-refractory diluent. Any adsorbed water would produce XO_2F_2 and HF--the solid XO_2F_2 would probably cause caking and the HF would be toxic. In addition, water or any other hydrogen containing compound would be undesirable if the diluent were to be used in the presence of the enriched product.

Unfortunately it proved impossible to satisfy the requirement that the diluent have approximately the same density as the carbon. It was decided to sacrifice ease in mixing and charging, for increased heat capacity.

The effect of particle size in the range 2 to 8 mesh was shown to be relatively small.

3. Tests of Diluent Properties

As a test of the caking properties of diluents alone, and of their mixtures with carbon, a series of experiments were carried out in small reactors operating at relatively constant temperature. The charges were raised to 400°F or 600°F depending on the most severe conditions considered likely to be encountered in practice and C-616 was passed through for six hours (or less if plugging occurred sooner). The reactors were then opened and examined for caking.

In Table III is presented a summary of the results obtained in these experiments, together with the experimental conditions used. The table includes data from experiments on high activity carbon, crystalline alumina, fused silica (crushed quartz), Queen Bee coke, graphite, "zero activity carbon" and pumice.

From these experiments it became evident that SiO_2 and Al_2O_3 were the only diluents tested which would be suitable for use at elevated temperatures. The other materials reacted with C-616, caking and plugging the reactor in the tests.

The experiments showed there was very little difference between SiO_2 and Al_2O_3 at the same temperature. However, the Al_2O_3 has a slightly higher heat capacity per unit volume, so that for a given amount of reaction, a bed diluted with it would not reach as high a temperature as one diluted with SiO_2. In addition alumina is comparatively cheap and readily available in large quantities. The

only difficulty expected from the use of Al_2O_3 is that it has a comparatively high density and may therefore tend to segregate in carbon mixtures to a greater extent. The density difference is, however, so slight (1.3 gm/cc for silica against 1.4 for alumina), that it was decided to use the alumina.

4. Effect of Dilution on Temperature Rise

Mr. A. P. Weber in a memorandum to Dr. M. Benedict has reported some calculations* based on a previous memorandum by Dr. W. I. Thompson**, on the bed temperature attainable in carbon traps on adding C-616. In this memorandum, a general relation for the effect of diluent on the temperature rise is presented. Also, from assumed values for heat capacities and heats of reaction, two graphs indicate for 100% C-616 the relations between temperature rise, C-616 absorption efficiency of the carbon and concentration of diluent. However, when C-616 concentrations of approximately 10% were used instead of 100% C-616, much greater temperature rises were observed in large carbon traps than would be calculated from theory. This is a result of the G-74 flow through the trap. After part of the charge has reacted, the entering gas mixture is heated by passage through the spent portion of the bed, which has been heated from the reaction. The heat absorbed is carried to

* A. P. Weber to Dr. M. Benedict, Maximum Attainable Bed Temperatures in Carbon Traps When Diluted with Inert Material, August 3, 1944, RB:8a

** W. I. Thompson, Temperatures in Charcoal Traps, 4/29/44.

unreacted portions of the bed which are then preheated before they begin to react. This effect is clearly shown by data presented in the section of this report on the performance of charges under pilot plant conditions.

However, the information obtainable from the calculations is reliable qualitatively. In Figure 1 is given a graph showing the temperature rise in carbon-alumina mixtures, derived from theory. Additional lines are drawn showing the effect of diluent concentration on temperature rise at constant capacity per unit volume assuming all charges to have the density of the carbon. It will be noted that the heat capacity of the alumina reduces the attainable temperature by nearly a factor of two. Also noteworthy is the fact that the effectiveness of alumina in reducing temperature rise at constant absorption efficiency per unit volume is confined to the region in which the volume fraction of carbon is greater than 25%. At greater dilutions there is very little decrease in attainable temperature.

C. Selection of Carbon for Use in Traps

On the basis of the data described, it was concluded that activated carbon was the only suitable C-616 absorbent available at the time these experiments were started. This carbon is supplied by the National Carbon Company in a variety of grades, types and sizes. In selecting a charge for carbon traps it became necessary to choose from among these the most satisfactory one for use as a plant absorbent for C-616.

The principal questions which arose in the use of a specific carbon as a C-616 absorbent were:

1. Does it absorb a sufficiently large amount of C-616 to be of use in the draps designed?
2. Will a trap charged with it cake or plug on exposure to C-616 under service conditions?

From the variety of carbons available, a number had been shown previously* to have adequate C-616 absorption. In the actual design of traps it was assumed that the carbon efficiency would be that of the National Carbon Company's medium (50%) activity material, and a safety factor for reduction in this activity was allowed.

1. Heat of Absorption

In the report mentioned above* the heat of absorption of C-616 by high activity carbon was given as 99,000 B.T.U./lb.mole of C-616. Later work by Dr. F. G. Brickwedde at the Bureau of Standards indicated that this figure might be too high by 10-20%, but for safety in the calculations the original figure was retained. When it was found that caking of plant beds of undiluted high activity carbon would result from exposure to C-616 at the temperatures generated by the heat of reaction, it became evident that changes would have to be made in the charge used in the carbon traps. It was assumed in the absence of experimental data, that changes in carbon activity would have little effect on the heat of reaction. Although

* Absorption of C-616 by Charcoal, S. C. Schuman and M. C. Chervenak, III, 2/22/44

this assumption is open to question, it still has not been demonstrated that the reduction in heat of absorption is sufficient to compensate for the advantage in specific heat of the charge obtained by diluting high activity carbon with a denser inert material.

2. Effect of Carbon Activity and C-616 Absorption Efficiency

The rate of chemical reduction of C-616 by carbon at a given temperature should increase with the activity of the carbon. This would imply a more rapid formation of fluorocarbons, and hence, on the theory of caking previously advanced, a more rapid caking of higher activity carbons.

This conclusion is borne out qualitatively in larger scale experiments at elevated temperatures. Data are given in Table IV, Runs 5A and 16A. There, the results of two experiments one made with high, the other with zero activity carbon, are given. The latter was run 200°F hotter than the former, at 600°F instead of 400°F. The run at 600°F lasted only 3.3 hours instead of 6 hours but the higher temperature run is known to be the more severe test of caking properties. In spite of this the zero activity carbon was much less caked at the end of the experiment. It should, however, be noted that the C-616 absorption efficiency of the high activity material was much greater. Comparing Run 125 with, for example, Run 12A it will be seen that for comparable absorption efficiencies the caking of zero activity carbon is much worse.

3. Effect of Dilution

The reduction in temperature due to addition of a denser diluent to the carbon in the bed has already been discussed in the preceeding section of this report. From the calculations given there, it was pointed out that dilution of the carbon beyond a certain point no longer causes appreciable decrease in temperature, assuming that the bed is required to have a fixed C-616 absorption efficiency per unit volume. It is possible in almost all cases in the plant to dilute high activity carbon nearly as much as can advantageously be done and maintain satisfactory trap efficiencies.

Going to a higher carbon activity and more dilution should lead to greater caking, if caking tendencies increase with activity, and at the same time it increases the probable C-616 losses from the trap* when it is operating on C-616, G-74 mixtures.

4. Effect of Other Factors on Caking

In addition to the C-616 absorption efficiency, there are other factors which may influence to some extent the caking of carbon beds. One of these is the size of particles, and another the type of carbon, i.e. - crushed or pelleted. In Table IV are given the data on the influence of these factors. From these rough preliminary experiments there is little choice between pelleted to crushed carbon of comparable activities insofar as caking is concerned. Pelleted carbon was fin-

* Height of Charcoal Bed Required When Diluted With Inert Material, A. P. Weber, 8/2/44

ally selected for use because of its somewhat higher absorption efficiency.

5. Summary

To summarize, then, on the basis of experimental data together with some assumptions and calculations based on them, it was decided to use pelleted high activity carbon in plant traps. This carbon, when diluted with a material of high heat capacity as far as possible within the limits set by the trap size and the required absorption efficiency, should have a minimum temperature rise on C-616 exposure, might show less caking than would be obtained if higher activity carbon more extensively diluted were used, and would more effectively remove traces of C-616 than such a charge.

For many services at the site, a high rate of C-616 absorption proved essential. In these cases 6/8 mesh pellets were used. In Sections 100 and 600, however, it proved desirable to reduce the rate of absorption, spreading the reaction zone out through the trap. For these services, 2/4 mesh pellets were used. A more complete discussion of the selection of charges will be presented later.

D. Selection of Charges for Plant Carbon Traps

1. Basis for Selection

In the selection of charges for use in plant carbon traps tests were performed under conditions of C-616 concentration and mass velocity similar or analogous to those regarded as the most severe to be encountered in practice. These in gen-

eral were the conditions of relief valve blow off, or cold trap failure during cell evacuation, where C-616 had to be absorbed at high velocity and concentrations.* In Sections 100 and 600, however, it was found that charges, suitable for use at high C-616 mass velocities for short times, caked at somewhat lower velocities if the exposure was continued for longer times. In these sections, therefore, the conditions of service finally chosen for testing the charge were those in which the C-616 had to be absorbed over a relatively long time and at about a tenth of the maximum mass velocity.

2. Experimental

In the experiments to be described, the data was obtained by passing C-616 and G-74, or C-616 alone, as required, into Monel cylinders 3" in diameter which were packed with the charge to be tested. The bed depth of the charge was made equal to that to be used in the corresponding service at site. In order to follow the temperature rise as the reaction proceeded a thermocouple well was installed in the center of the reactor. This well was of 3/8" O.D. Constantan tubing, having a wall thickness of 0.010". Insulated iron wires were soldered through this tube and led up the inside to a selector switch to permit

* 1) A. P. Weber and M. Benedict, Carbon Traps in Section 100 and 600, Dec. 28, 1944.
2) M. Benedict and A. P. Weber, Carbon Traps in Section 300, Nov. 1, 1944.
3) Also A. P. Weber, Change Sheet No. 2A for Memorandum "Carbon Traps in Section 300", Jan. 1, 1945.

reading temperatures at various points in the bed. The complementary Constantan lead was a Constantan wire soldered to the outside end of the Constantan tubing. A sketch of a typical reactor is shown in Figure 2.

C-616 flow was measured by means of an orifice and pressure gauges in the line between the source which was a reservoir heated to 100°C, and the reactor. All lines were heat traced electrically to prevent C-616 condensation.

When dilution with G-74 was desired it was introduced at a tee connection on the downstream side of the C-616 orifice. Flow was metered with a rotameter.

This system of testing worked satisfactorily except for occasional plugging at the C-616 orifice. It was necessary to prevent any moist air from reaching this section of the line if plugging was to be avoided. If the system was tight, flushing it with G-74 before shutting down, and cooling down and capping off the leads with a slight flow of dry G-74 passing through was a sufficient precaution.

3. Section 100

The first experiments performed in trying to find a suitable charge for carbon traps plant Section 100 were made at high C-616 mass velocities for short times (7-9 minutes). Some were made in a reactor only two feet long, in order to obtain some preliminary information on the C-616 absorption capacity of various carbon-alumina mixtures and on the probability of their caking under the conditions used. Data on these experiments is

given in Table V. C-616 absorptive capacities are expressed in terms of lb. C-616 absorbed per cubic foot of charge. The experimental conditions are to be compared with conditions at the maximum C-616 flow rate expected in F-110, which are given in the last column of the table.

In the first two experiments 4/6 mesh particles of crushed high (75%) activity carbon were used. From the data obtained in these two cases it became apparent that when such small particles of carbon were used, absorption of C-616 was so rapid that a hot spot developed in the reactor and caking occurred. To avoid caking from this source it was decided to use 3/8" pellets of high activity carbon, which had approximately the same final absorption efficiency as the 4/6 mesh particles, but which absorbed C-616 more slowly, thus spreading out the reaction zone.

From the data in Table V it will be seen that for short exposures to C-616 at high mass velocities, a mixture of one part by volume of 4 mesh crystalline alumina with two parts by volume of 2/4 mesh high activity carbon pellets performed satisfactorily. Any less dilution led to excessive temperature rises.

After performing these tests, runs were made in the same charge under conditions which will also occur in Section 100, namely a C-616 flow rate of about a tenth of the maximum for considerably longer periods of time. Data on these experiments

are given in Table VI. It will be noted that while the temperature rise was no greater than that noted in the other runs at higher mass velocities, the charge caked near the entrance to the reactor. Evidently this charge works satisfactorily at the high flow rates for short times because the rate of absorption of the C-616 by the large carbon particles is low, and because the reduction of the C-616 by carbon at the temperatures reached in 15 minutes is slow. In this time not enough reaction occurs to produce any caking. By lowering the flow rate the length of the reaction zone is decreased but the portion of the bed in which reaction occurs heats up as rapidly as ever. Since a greater time is permitted for reaction to occur, caking results.

In any case, it appeared that the 1:2 mixture would not be suitable for use at a high or medium flow rate over a period of time much longer than 15 minutes. Data on Run XXV in Table VI support this view. It would be satisfactory for services of shorter duration at nearly any possible flow rate, or for services at flow rates low enough to permit the heat of absorption of the C-616 to be balanced by heat losses from the trap. The latter would, however, be at most 1/100 and probably less than 1/1000 of the maximum rate expected.

In view of the caking at intermediate flow rates, it was decided that the 1:2 charge was unsatisfactory for general use in this section. It was calculated that a 2:1 mixture would

have sufficient capacity. Tests on such a mixture were run at the intermediate flow rates, and the charge was found satisfactory both with regard to capacity and to caking tendencies. Data on these experiments is given in Table VI together with the conditions recommended for the test by the Process Engineering group. This 2:1 mixture was the one finally recommended for use in traps at the site.

To prevent condensation of C-616 in the alumina at the entrance to the trap it was found necessary to heat this portion to about 160°F before introducing C-616.

4. Section 600

a. Standard Charge

In Section 600 the duration of flow at the maximum rate of input of C-616 is twice that in Section 100, 14 minutes instead of seven. The maximum C-616 flow rate expected is about 35% greater than in Section 100, but the bed available is 15% deeper, 9 feet instead of 7 feet 8 inches. 100% C-616 is handled in both cases. Evidently the two sets of conditions are quite similar at the maximum rate of C-616 input. This similarity extends to the other, less stringent services in the two sections. Since the conditions are so similar, the charge found suitable for use in Section 100 was tested under Section 600 conditions. Results of the tests are given in Table VII. It will be seen that the 2:1 charge is satisfactory for use in Section 600 as well as in Section 100.

b. 6/8 Mesh High Activity Carbon

Experiments were also performed in which charges other than the 2:1 mixture of alumina and 3/8" high activity carbon pellets were tested. Data on absorption by these mixtures is given in Table VIII. From these experiments it was concluded that 6/8 mesh high activity carbon was not efficient enough for this service when used in dilutions of 3:1 (3 volumes of alumina to one of carbon) or greater, and that overheating and caking occurred if it was used in dilutions of less than 2.5:1. A 2.5:1 mixture (Run XVIII) would perhaps be satisfactory, but even there the temperature rise was rather great, and slight caking occurred. Since heat losses will be less from the large trap than from the ones used in these experiments, it was decided that 6/8 mesh high activity carbon could not be used in these traps.

c. Very High and Low Activity Carbon

As mentioned previously the assumption was made that the heat of absorption of C-616 on activated carbon is independent of the activity of the carbon. To determine qualitatively whether any Heat differences which might exist using carbon of different activities might make unnecessary the use of alumina as a diluent, four experiments were performed. In one a very high activity carbon. In the case of the very high activity carbon the bed was diluted with calcined "zero" activity carbon, to

an extent such that the overall absorptive capacity of the bed was practically unchanged from that of the 1.5:1 mixture taken as the standard charge in Section 300. By using calcined carbon as a diluent the amount of segregation occurring on charging should be greatly reduced, since in this case the activated carbon and the diluent would have practically the same densities. The experiments were performed under conditions approximating those in Section 600 because it was felt that under these conditions laboratory results were most readily interpretable in terms of plant performance. Data on the runs is given in Table IX.

It is apparent that caking was too severe in the 4:1 dilution of special high activity carbon for this charge to be at all acceptable. The plugging in the low activity runs was apparently not caused by reduction of C-616, because there was very little caking. If this plugging had not occurred, the charge would have been fairly satisfactory. The appearance of large amounts of HF and C-616 in the exit gas at the start of the run makes this charge undesirable, however.

These data indicated that a satisfactory mixture of activated carbon and calcined carbon could not be readily developed for use under the conditions of operation required. Of the two possibilities tested the one involving use of low activity carbon appears more promising, if means can be found for avoiding the plugging met in the experi-

ments reported.

5. Section 300

A variety of sizes and shapes of carbon traps are used in this section of the plant. The main feature which all have in common which differentiates them from the traps in Sections 100 and 600 is the fact that they operate on relatively dilute C-616, G-74 mixtures. The conditions expected in Sections +1 and -1 were the most severe with respect to required absorption efficiency and were taken as the basis for charge selection.

Because of the short beds in the traps in this section of the plant, and the high C-616 linear velocity through the trap, C-616 came through the bed too soon when the 3/8" high activity carbon pellets employed in Sections 100 and 600 were used here. Instead, the 6/8 mesh pellets, avoided in these sections because of their high reaction rate, were used.

Before starting experiments to find a charge for use in these carbon traps, calculations were made on the basis of the volume of the trap, and an assumed C-616 absorption efficiency of 2.0 for the carbon which led to the conclusion that a dilution of one part of carbon with three parts of alumina by volume would have a satisfactory absorptive capacity. The temperature rise in this case would only be about 270°F according to the curve of temperature vs. dilution given in Figure 1. To give a margin of safety in efficiency, experiments were started at a 2:1 dilution, and later reduced to 1.5:1 to further increase the

capacity of the bed. In these cases temperature rises of 370°F and 460°F are predicted from the curve.

In addition to experiments under Section 300 ± 1 conditions, data on which are given in Table [illegible], a few experiments were made under the conditions expected in Sections 300 ± 3 (Table XI) and 300 ± 2, (Table XII).

These show that the charges selected had satisfactory absorptive capacity, and did not cake or plug. A much lower temperature rise was observed than had been calculated. It was feared that this last was because of the low rate of exhaustion and high heat losses from the small trap. If so, one might expect a much higher temperature in operating a large trap under the same conditions. It was also noted that the reaction zone under these conditions was quite short, and that it moved steadily up the trap as the reaction proceeded. As this zone moved up the trap the temperature of the zone increased, until it finally reached a point where the heat losses balanced the heat input. This was attributed to the G-74 which served to preheat unreacted portions of the bed with heat from the reaction zone; it could lead to extremely high temperatures in large traps. Correlation of the temperatures observed with those which could be produced as a result of this effect was hampered by the non-adiabaticity of the experimental set up.

6. <u>Mobile Carbon Trap</u>

This trap will be required to absorb G-616 at 0.00185 lb./sec./ft.^{2} for at least 1-1/4 hours. This rate is very low com-

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pared to those obtaining in Sections 100, 300, and 600 and the total absorption required is also comparatively small, considering the fact that a 3' 0" depth of charge is available.

In the first experiment performed under these conditions, a charge was used containing 4 parts of 6/8 mesh alumina to 1 part of 6/8 mesh high activity carbon by volume. Experimental conditions and measurements are given in Table XIII under Run M-1. The reaction was about half way through the bed in 1-1/4 hours; the maximum temperature was only 150°F.

Since the temperature rise in Run M-1 was so small, the dilution was decreased to 2:1 in Run M-2 to increase the total absorptive capacity of the trap. In this run the reaction had just started to enter the second quarter of the bed at the end of the 1-1/4 hours. The temperature only reached 180°F.

From these experiments it was concluded that use of a charge consisting of 2 parts 4 mesh or 6/8 mesh alumina to one part 6/8 mesh high activity carbon will give satisfactory performance in the mobile carbon traps to be used at the site, and that there will be a considerable margin of safety in absorptive capacity if used under the conditions specified.

7. Summary of Conclusions

From the above experiments it was decided that in the traps in Sections 100 and 600, a charge consisting of two parts by volume of 2/4 mesh crystalline alumina to one part by volume of 2/4 mesh high (75%) activity carbon pellets (National Carbon Company) would be satisfactory, if the entrance to the trap was kept above

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150°F.

In Section 300, a 1.5:1 mixture using 6/8 mesh instead of 2/4 mesh high activity carbon was selected.

For the mobile carbon trap a 2:1 mixture of 6/8 mesh high activity carbon and alumina should be more than adequate.

In Section 300 and to a lesser extent in Sections 100 and 600 the heat losses from the traps used in the experiments were so much greater than those expected from the traps at site that experiments in a larger carbon trap were evidently necessary. Such a series of experiments is reported in Section G.

E. Protective Diluents

1. Possibilities

In a memorandum on the total protective diluent requirements of a carbon trap by A. P. Weber,* non-reactive boron or cadmium compounds are suggested for use as protective diluents in carbon traps intended to absorb enriched C-616. An equation for calculating the amount of either of these elements necessary in a given service is presented.

Any of the compounds of boron or cadmium which contain no hydrogen and do not form volatile boron or cadmium compounds on exposure to C-616 would be satisfactory. The attention of this group was focussed primarily on two possibilities, one of them boron carbide, B_4C, and the other cadmium oxide, CdO.

2. Boron Carbide

a. Reaction with C-616

* "Total Protective Diluent Requirement of a Carbon Trap," A. P. Weber to Dr. M. Benedict, August 2, 1944, RB:8a

Since the existence of the volatile BF_3 and other boron fluorides is well known, the possibility that one of these might be formed on exposure of B_4C to C-616 was examined.

10% C-616 in G-74 was passed through a mixture of B_4C and high activity carbon, and the gases issuing from the reactor were tested with a gas-oxygen flame. The flame, after the absorptive capacity of the bed was nearly exhausted, showed the typical bright green of the boron test. That BF_3 would give the test under these conditions was proved experimentally. The bor n compound formed passed through the usual water wash bottle with the formation of a smoke. Very little was absorbed. However, it could be absorbed in concentrated sulfuric acid. The compound is condensable at dry ice temperatures. Since the boiling point of BF_3 is -101°C, this compound is presumably of higher molecular weight. Both B_2F_6 and BF_3HF are possibilities.

Two experiments were performed in which boron carbide was exposed to excess C-616 at 200° and 400°F. The weight gain of the reactor was measured as a function of time. Assuming the reaction to be: $8XF_6 + B_4C \rightarrow 8XF_4 + 4BF_3 + CF_4$, the method should be a sensitive one for the determination of the rate of the reaction.

Data obtained are given in Table XIV. No very accurate conclusions concerning the absolute rate of reaction can be

drawn from them both because of the uncertainty as to the reaction mechanism and because of the small weight gains obtained. There is no evidence that reaction proceeds only to some "saturation" value less than that corresponding to complete reaction.

b. Conclusion

Since boron carbide forms one or more volatile boron compounds on reaction with C-616 it would be unsuitable for use as a protective diluent in a carbon trap.

3. Cadmium Oxide

Cadmium is not known to form any volatile compounds with X or C-216. Since its preparation is relatively simple, it was decided to try to develop it as the required protective diluent.

a. Cadmium Oxide Coating of Alumina

Experiments were performed which showed it to be possible to put a fairly adherent coating of the desired amount, 2% by weight, of cadmium oxide on crystalline alumina. This method of introducing the cadmium into the trap avoided the introduction of a third separate component, thus simplifying the mixing problem. It also afforded the most economical utilization of the cadmium, for the permeability of these compounds to neutrons is low.

b. Reaction with C-616

A sample of cadmium oxide coated alumina containing 1.8% CdO was exposed to excess C-616 at 600°F for three hours. During that time the reactor gained a weight equal to 3% of

the total charge. Examination of the charge after the reaction showed little change in appearance and no caking. Apparently some reaction had occurred, leading to the formation of XO_2F_2. The amount of reaction is so small, however, that it would not contribute noticeably to the temperature rise in the bed. There was no evidence for the formation of any volatile reaction products.

From these experiments it was decided to use cadmium oxide in the form of a coating on the alumina as the protective diluent in traps where such a diluent is required. This material is now being made by the Harshaw Chemical Company. Their process involves roasting alumina particles after coating them with $Cd(NO_3)_2$.

c. Testing of Cadmium Content

To satisfy the protective diluent requirement for the worst case in Section 300 a 1.97 weight percent coating of CdO on the alumina charged to the trap is necessary. However, tests in which coated samples were shaken in a Rotap sieve shaker for five and ten minute periods, losses of 0.23% and 0.30% CdO respectively were found. Since this abrasion resistance test is not particularly severe, it was decided to require from Harshaw a CdO coating thicker than was theoretically required.

In Table XV are given data on the analysis of a sample of coated alumina prepared by Harshaw which was accepted for use at the site. Results of analyses performed both by

Harshaw and by Kellex are given. Two methods, one the electrolytic one developed at Harshaw and the other the polarographic one used at Kellex, are employed in analyzing for cadmium. In dissolving the coating from the alumina Harshaw has simply washed the solid alumina particles thoroughly with acid, whereas at Kellex it was found that slightly higher results were obtained on washing the alumina after crushing it.

A three minute shaking on the Rotap sieve shaking machine has been accepted as the standard abrasion resistance test. It is estimated that this is equivalent to the worst treatment the material will receive in practice.

F. Segregation of Carbon-Alumina Mixtures

Because of the great difference in the densities of carbon and alumina, a mechanical mixture of particles of the two would tend, to segregate into its components, with the alumina falling to the bottom of a container and the carbon rising to the top. It was observed after pouring that there was a definite increase in the concentration of carbon around the outside walls of the cylinder. On the basis of these observations a preliminary series of experiments was performed to determine whether segregation represented a serious problem and to establish qualitatively the best method of charging containers with mixtures of carbon and alumina. For these preliminary experiments, a cylindrical glass bowl, 12" x 12" was covered around the sides and on the bottom with masks in which rectangular holes were cut. The details of the masking are shown in Figure 3.

Mixtures of carbon and alumina were introduced into this bowl by several different methods. The number of particles of alumina visible through the cut out areas of the mask were then counted.

Knowing the number of alumina or carbon particles visible when present alone in a measured area, a calculation can be made to determine the fraction of the visible area occupied by alumina or carbon. Assuming that a volume one layer of particles thick is concerned rather than simply an area, the efficiency of the mixing method may be estimated by comparing the fraction obtained by the calculation outlined above with the ratio of total volume of alumina to total volume of carbon in the mixture as prepared.

Besides the usual random experimental errors, there exists in this method the possibility that certain systematic errors will appear. This is particularly true when particles of widely different sizes are present. It was believed, however, that with the particles being studied this difficulty would not lead to a great error. This assumption was confirmed by one experiment in which both carbon and alumina particles were counted and the sum of the calculated fractions of the total area were determined. This result should be 100% in each case. Data are presented in Table XVI.

Experiments were performed using:

1. 1:2 mixture of 4 mesh Al_2O_3 to 2/4 mesh carbon
   Data in Table XVII
2. 1:1 mixture of 4 mesh Al_2O_3 to 6/8 mesh carbon
   Data in Table XVIII

3. 1:1 mixture of 6/8 mesh Al_2O_3 to 6/8 mesh carbon

Data in Table XIX

In the tables, the letters indicating the "Method of Mixing" correspond to the following methods:

A. Charge mixed in barrel then poured into bowl.

B. Charge mixed in barrel then poured into bowl and there mixed by hand.

C. Charge poured by the scoopful into a funnel held with its lower end 2' above the top of bowl.

D. Charge placed in bowl one scoopful at a time, distributing deposits evenly around bottom of bowl.

E. Filled as in (C) but with the funnel held just above the surface of the charge and describing a uniform pattern around the bottom of the bowl.

All data are presented as percentages of total area occupied by Al_2O_3, except in the one case given in Table XVI where both Al_2O_3 and carbon areas were measured.

In evaluating the data, the greatest significance should be attached to the side view results. These give an idea of how far off the radial distribution is; those at the top and bottom, from their location, will be less sensitive to this type of segregation. It would have been desirable to obtain the sample from the center of the bowl, in order to obtain the other extreme of segregation, but this appeared to be impractical with the apparatus used here.

It will be seen that the worst segregation is obtained when the mixed charge is poured continuously into the bowl. When the charge is introduced in this way and then mixed, very little improvement is

observed, except on top, but this undoubtedly would be rectified by using a more efficient method of mixing. Pouring the charge by the scoopful through a stationary funnel into the bowl helps somewhat, but especially with the smaller particles, it is not very effective. If the funnel is moved during the charging, a considerable improvement is apparent.

The best method of charging seems, however, to be one in which thoroughly mixed portions of the charge are transferred to the bowl and deposited there with as little disturbance as possible.

As a result of these measurements and other considerations it was decided to charge the traps at the site as follows:*

The proportioned streams of carbon and alumina from the adjustable feeders (Syntron Vibra-Flow Dry Volumetric Feeders) would be routed to a common spigot at the end of which was a flexible hose leading into a bag held in a bagholder. The bag would be sized to hold approximately 1/20th of the inventory of a carbon trap and so that they could be easily maneuvered into the carbon traps through the top manhead. The operator when filling a bag would weave a pattern with the flexible hose to avoid, as much as possible, the segregation resulting from pouring. The hose length would be as short as is practicable. The bags would then be closed by running a tie around the top of the bag, which the operator would shirr into a neck. The bags would also be provided with three eyelets, two diametrically opposed at the top and one at the bottom. The

* From: " Memorandum on Meeting on Formulation, Mixing and Loading of Carbon Trap Charges," October 7, 1944, by A. P. Weber, dated October 11, 1944.

mixture in each bag could be kept dry by storing the bags in the containers in which the carbon and alumina are shipped to the site. When a trap is to be filled with absorbent mixture, the tie around the neck is broken, the bag is lowered into the trap from the two top eyelets, and when the bag is just above the surface on which the charge is to be laid down it is overturned by slackening on the two top eyelets and pulling on the bottom eyelet. In this fashion the contents are dumped with as gentle a fall as possible to avoid breakage, dusting and segregation.

Experiments were performed investigating segregation of carbon-alumina mixtures under conditions duplicating as nearly as possible in the laboratory those described above. The apparatus to be described was constructed for this investigation. A 30 gallon drum, 18" in diameter, was cut in two 12" from the bottom and the bottom section fitted with a wooden cover in which were drilled 9 holes, each 2" in diameter. The holes were arranged in the form of a cross, with a hole in the center of the cover, four with centers on a circle 7" in diameter, and four with centers on a circle 13½" in diameter. The entire unit was bolted to a carboy tilter so that it could be inverted with a minimum of difficulty.

Mixtures of carbon and alumina were mixed by hand in a barrel. (This is the usual procedure employed in mixing charges for use in the laboratory experiments on carbon traps). When the mixtures appeared to be homogenous they were poured by the scoopful through a funnel describing a pattern over a container 14" high and 6" in diameter, open at both ends, which rested in the drum. The smaller cylinder, when filled, was lifted out, allowing the mixture to flow

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gently into the drum.

After charging the drum, the cover was bolted on and glass sampling tubes inserted through the holes were forced through the charge to the bottom of the drum. The tubes were then packed with paper and closed with rubber stoppers. The drum was inverted and in this position the sampling tubes were removed.

Inspection of the parts of the charge collected in the tube showed any vertical inhomogeneity or stratification in the mixture. The radial distribution of carbon and alumina was obtained by separating the components in each of the sample tubes and comparing volumes of each found with the original proportions in the mixture.

In Tables XX, XXI, and XXII are given data obtained in experiments with different mixtures of the two components. Table XX presents results on a mixture of 1.5 parts by volume of 4 mesh crystalline alumina with 1 part by volume of 6/8 mesh high activity carbon pellets. In Table XXI are the data on a 2:1 mixture of 4 mesh alumina and 2/4 mesh carbon, while in Table XXII are data on the same mixture in which 2 mesh alumina was used instead of 4 mesh.

The data in Table XX indicate that the mixtures containing 6/8 mesh carbon scheduled to be used in the traps in Section 300 at the site, does not segregate badly when charged by the method used. There is evidence of an increased concentration of carbon at the center and a reduction away from the center but this does not appear to be enough to cause serious difficulty.

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The mixtures in which 2/4 mesh carbon were used did show considerable segregation. The data on the mixture containing 4 mesh alumina show particularly bad segregation. Not only is the tendency of the carbon to concentrate in the center more marked in this case than with the 6/8 mesh carbon, but the deviation of the separate values obtained on different tubes in the same experiment is greater. The latter shows a tendency toward the formation of clumps of carbon or alumina. The data indicate the problem to be serious for plant traps. The concentration of carbon at the center, where the heat losses would be least, introduces the possibility of caking under severe conditions, while the depletion in the carbon near the outside may result in channeling of C-616 through the bed without its being absorbed.

Because of these possibilities, 2 mesh alumina was used to see if any reduction in segregation could be effected by using it. Results show that some improvement may have been obtained, but the improvement is so slight that it is probably not worth making the changes necessary to obtain and use the larger particle size material. The 2:1 charge containing 4 mesh alumina used in these experiments is the one specified for use in Sections 100 and 600 at the site.

In view of segregation observed in these experiments it became imperative that a large scale test be made on a charge mixed and added to the trap by the method outlined.

G. Performance of Charges Under Pilot Plant Conditions

1. Apparatus

The carbon trap around which the unit was built is shown in Kellex Dwg. No. 300-F-75-Q-B. To make this trap more suit-

able for the experimental work desired, certain changes were made.

In this carbon trap, the bed is supported on a screen resting on a ledge ten inches above a large blind flange which forms the bottom. By removing the screen, the available bed depth was increased to 31". In order to discharge the trap, a hole was cut in the bottom flange and a cone was welded on. This cone was 12" in diameter at the weld, 8" deep, and tapered to a 4" opening covered with a small blind flange. By leaving one bolt on this flange in place and removing the rest, it could be swung out of the way, thus permitting the contents of the trap to pour out. During experiments the conical portion of the trap was filled with pure alumina.

With the change in the bed depth, the connection originally designed as a C-616 inlet was closed off. In its place, a 1½" copper tube was brazed into the conical section 4" from the bottom flange. To avoid channeling, the end of the tube was closed off and the gas forced to enter the bed through holes in the bottom of the tube.

The inside diameter of the upper flange of the trap was increased to 8". This made possible charging the trap with buckets of mixed charge.

Since laboratory experience showed that caking and plugging were functions of the temperature, the temperature in the bed during operation was measured. To accomplish this, six thermocouples were installed along the center line of the bed. One was in the cone filled with alumina, and the others were spaced 6½", 11", 16", 20" and 24" from the start of the absorbent charge.

The end result of these changes was a cylindrical trap, 15-1/4" in diameter and allowing a 31" maximum bed depth. The conical section is typical of many of the standard types of plant traps. Charging and discharging by means analogous to those recommended for plant traps was possible, and temperatures in the bed could be followed closely.

To meter C-616 flow, a branched line was used with orifices in the two branches covering different ranges of flow. Pressure drop through the orifices was indicated on a differential pressure transmitter. The source of C-616 was a standard Z cylinder having a capacity of 400 lb. of C-616. This was mounted in an oil bath heated to 100°C, and the entire unit mounted on a scale. The flow rate indicated on the orifice could be substantiated by the change in weight of the cylinder, since the connection between the cylinder and the system was flexible.

G-74 was obtained from a manifold of five commercial cylinders. Flow was measured on a standard Fisher-Porter rotameter.

To remove C-616 or C-216 from the system after a run or after stabilization , an evacuation system consisting of a NaCL trap, another trap containing half high activity carbon and half NaF, a cold trap in liquid G-74 and a two stage Welch Duo Seal pump was added.

For stabilization, a cylinder of 5% C-216 was attached. A high pressure gauge on this lead served to read tank pressure.

Lines through which C-616 was to flow were heat traced if

if they were not enclosed in the oven surrounding most of this piping.

Auxiliary traps, the first containing a 1.5:1 mixture of 2/4 mesh alumina and 6/8 mesh carbon, the second, 4/8 mesh soda lime, were installed to remove HF and unabsorbed C-616 from the gases issuing from the carbon trap. Pressure gages were mounted in the line between the large trap and the auxiliary carbon trap and between the auxiliary carbon trap and the soda lime trap. These served to indicate plugging in these traps. Between the main carbon trap and the auxiliary traps a connection was made for taking gas samples by the method used for the laboratory carbon traps.

2. Technique

To prevent plugging the lines in the oven, the equipment was stabilized with 5% C-216 at 250°F and 5" vacuum for a total of one hour. Stabilization was performed in three shots to avoid difficulties due to C-216 consumption. The stabilization pressure was below atmospheric to prevent C-216 from leaking into the carbon trap. After stabilization, C-216 was pumped out and the system was kept under a positive pressure of "dry" C-74 until ready for use.

In the first few runs, before introducing C-616, the system was cleaned up with C-216 at 220°F for an hour. Later this was shown to be an unnecessary precaution if the system was kept filled with C-74 when not in use.

As previously mentioned, the test trap was charged by a pro-

cedure which duplicated as closely as possible that recommended for use at the site. The automatic mixing by proportioned flows of the alumina and carbon could not be obtained. Instead, mixtures were made up in barrels and mixed by hand as was done in preparing charges for the laboratory traps. The mixtures were then poured by the scoopful through a funnel describing a pattern over a bucket 6" in diameter and 12" high. The bucket was lowered into the trap and, when it reached the surface of the charge, inverted, depositing the contents with as little disturbance as possible.

3. Experimental

In general, the mass velocities of C-616 and C-74 were calculated to correspond to those specified for carbon trap service at the site. Since the bed depth available for these experiments was somewhat smaller than that in the actual traps to be used, it was not expected that C-616 gas flow could be continued for the required time. It was, therefore, the practice to run until the samples taken of the exit gases showed relatively large amounts of C-616 present.

The results of these experiments are compared with the corresponding service at site by means of the C-616 capacity of the beds expressed in lb. C-616 absorbed per cubic foot of charge. In the case of the capacities determined experimentally, the amount of C-616 absorbed was taken to be that present when C-616 begins to appear in such concentration in the exit gas that the solution obtained by introducing 50 ml of water into the one liter sampling bulb was unmistakably yellow in color. It should be noted that in experiments where the dilutions are comparable,

it is to be expected that a somewhat higher carbon efficiency can be obtained in the plant traps than in this experimental trap. This follows from the fact that this trap has a shorter bed depth than the plant traps. At the time when large amounts of C-616 begin to appear in the exit gas, the layers of charge near the exit of the bed will only have started reacting. This zone of unreacted carbon will be at least approximately the same depth in any trap operated under these conditions; thus a smaller fraction of the carbon in the longer beds will go unused. The reaction zone may safely be assumed to correspond to several inches of unused carbon, so the efficiency found in practice may be as much as 20-25% higher than that found in these experiments.

a. Section 600

The experiments were performed under approximately the conditions of traps F-606 and 606A during relief valve service. Data are presented in Table XXIII.

The results obtained indicate that the traps at the site should have little difficulty in taking care of this service. In the short bed available, C-616 was absorbed completely at the required mass velocity for a 60% longer time than would be required at site. The capacity of the trap is therefore ample.

Slight caking occurred in both of the two tests. The caked portions were not particularly hard and were small enough to pass freely through the dumping gate on the usual plant trap. Examination showed they contained an unduly high fraction of carbon. Therefore, caking probably resu ted from

SECRET

local overheating, as was feared would be the case if appreciable segregation occurred. This experience served to emphasize the need for care in charging.

The only difficulties experienced in these experiments occurred during discharging. In one run, the heater on the alumina cone was shut off after the gas flow started. In spite of the fact that during the run the thermocouple in the cone continued to read about 200°F, it was found on opening the flange for discharging that large quantities of C-616 had condensed on the cooler walls. In the other experiments reported, the heating was continued and the outside temperature of the cone kept about 140°F. No condensation occurred.

b. Section 300

Three experiments were performed under conditions comparable to those scheduled for use in the traps in Section 300 ± 1 during discharge of the contents of one cell when the cold traps are not in operation. Data on these runs and all others made in the investigation of charges for traps in this section are given in Table XXIV.

It was found in these experiments that while absorptive capacity of the charge was adequate, some caking occurred. This proably was not due to segregation because the cakes formed were all large and quite homogeneous. They could be broken up comparatively readily by poking with a metal rod, but until this was done they were too large to discharge through the dumping gate of the traps with conical bottoms.

The caking was apparently due to uniform overheating of the bed. In two cases the temperature maximum appeared to have

SECRET

occurred between the first two of the three original thermocouples, preventing an accurate estimate; but the temperature may have reached 600°F in all cases.

Since this caking would be troublesome in discharging traps, attempts were made to change conditions of the experiment and thus avoid it.

Run #5 was made at half the C-616 mass velocity scheduled, and, to compensate for this change, for a longer time. The efficiency increased somewhat, but the caking was worse. This is undoubtedly because the bed is so thick and such a good heat insulator that the reaction, even when carried out over a period of several hours, is practically adiabatic.

In Run #7, half the scheduled C-616 mass velocity was again tried, and this time the run was stopped at the time which would correspond to the absorption of half the contents of one cell in a given trap. This simulates the conditions obtaining if the two available traps were used in parallel rather than in series. Again caking occurred, almost to same extent that it had in the preceding runs.

In Run #8, an increase in the dilution of the C-616 with G-74 was tried. The C-616 content was reduced to 2.7%. A bed which had not caked was found at the end of the run, even though the temperature had gone as high as 585°F. during the time C-616 was flowing. The increased amount of G-74 caused the temperatures at each junction to fall off much more rapidly once the maximum temperature was reached. Also the envelope of the successive maxima appeared to have stopped rising and levelled off at about 600°F instead of continuing to rise

as was the case in other experiments. C-616 absorption was not as complete as was the case where lower gas velocities were used.

Run #11 was made using 3.5% C-616 to see if the amount of G-74 required to prevent caking by this method could be reduced. The maximum temperature reached in this case was 800°F and the temperature maxima were not quite as sharp as in Run #8 where more G-74 was used. In spite of this, there was very little caking; the caked bed was easily broken up. Absorption of C-616 appeared to be more complete than in Run #8.

The objections to this means of preventing caking are two. First, the G-74 consumption is extremely high, and second, larger amounts of C-616 are carried through the bed unabsorbed because of the reduced contact time. Undoubtedly this difficulty would be overcome to some extent in the plant by the longer beds and by using two traps in series.

Run #10 was made to determine the effect of increasing the ratio of alumina to carbon to 2½:1 on the caking under the original operating conditions. The maximum bed temperature, although still increasing when the C-616 was shut off, was lower than in the preceding runs. No caking occurred, but the absorptive capacity was reduced. As in the case where the higher G-74 dilution was tried, the C-616 content of the exit gases was somewhat higher than it was when the original 1½:1 charge was used.

4. Conclusions

It can be stated definitely that the rate of heat loss from beds of the size used in these experiments or larger is extremely small. Under conditions obtaining in Section 300 ± 1 the heat loss is so low that reducing the mass velocity by a factor of two had little or no effect on the maximum temperature reached. It is doubtful that reducing it by a factor of ten would have had a great effect.

From cooling curves on the bed after stopping the gas flow, it appears that the rate of cooling at the center varies from 0.1 to 1.0°F per minute as the temperature varies from 250° to 500°F.

Run #8 shows the effect of diluting the gas with large amounts of G-74. When this is compared with results in a case where the C-616 was four times as concentrated (Run #5), the increased sharpness of the temperature maxima is clearly seen. This must be a result of removal of heat from reacted portions of the bed by the cooler G-74. In addition, although this point is not so clearly shown in the graphs, it appears that the use of larger proportions of G-74 reduces the maximum temperature obtainable.

Using an increased proportion of alumina in the charge (Run #10), the rate of increase of maximum temperature as the reaction proceeds along the bed is reduced, but it is still possible that in a long bed a maximum temperature almost as high as that found with lower alumina dilutions may be obtained.

With respect to charging of the traps, in order to prevent segregation on dumping buckets into the trap, it is necessary

to preserve a nearly level charge surface. If any appreciable amount of rolling of particles occurs on dumping the bucket, it will result in segregation of the alumina from the carbon.

Discharging the spent charges has been a source of some annoyance. Invariably, large amounts of HF are given off during discharging; blowing the trap out, after discharging, with air which passes over the spent charge is not effective in removing it. This leads to difficulty when the trap is opened at the top to add a new charge. Gas masks and rubber gloves are required for this operation as is adequate protective clothing. This hazard can be avoided, at least to some extent, by blowing out the trap with G-74 after discharging and closing the bottom flange.

H. Long Exposure of Carbon to C-616

In some cases, carbon traps at the site will be called upon to absorb C-616 from a stream in which the C-616 mass velocity is very low. Since the rate of supply of C-616 to these traps is so low, their life expectancy, based on the absorption efficiency of the carbon, as determined in tests over shorter times, is quite long. The question then arises of how long the time of exposure affects the carbon absorption efficiency. Also it would be desirable to find how effective carbon traps are in removing C-616 from gas streams of very low C-616 concentration.

A few experiments bearing on this problem were performed. In one, 10% C-616 was passed at room temperature with a C-616 mass velocity of about 4.6×10^{-5} lb./ft.^{2}/sec. through a 1:2 mixture of alumina and 2/4 mesh high activity carbon pellets, for a period of 6.6 days. At the end of that time the average absorption efficiency of the carbon in the sections of the bed, reached by C-616 was 2.7 gm.

C-616/gm. carbon. This figure is believed to be slightly high due to hydrolysis of C-616 in the bed.

Two other experiments were performed to evaluate the probable performance of carbon traps under the conditions specified for continuous purge operation in Sections 3a and 3b. Experimental conditions in the two runs made are compared with the suggested conditions of operation in Table XXV. It will be noted that the laboratory trap operated at a somewhat lower C-616 concentration than expected in the first run, and that CdO coated alumina was not used. Also, in the second run, the bed depth was greater than desired and the carbon content of the charge was too high. Of these, only the last mentioned makes for serious difficulty in interpretation of the results.

Analytical data on the entrance and exit gas in the first run are given in Table XXVI. The C-616 concentration in the exit gas was 0.1 mg/liter (0.001 mole %) or less up to the point where the bed was exhausted.

Because of the high proportion of carbon in the charge used in the second run, data on the C-616 concentration in the exit gas may not be significant, but in this case, too, it was less than 0.1 mg/liter. The ultimate efficiency of the carbon is probably correct. This was 2.2 gm. C-616 absorbed/gm. of carbon.

The efficiencies found in these long term experiments are slightly (30%) above those found under comparable conditions in shorter runs. They are, however, still well below what would be found by operating at 200°-300°F.

I. <u>Analysis of Exit Gases from Carbon Traps</u>

In order to determine some of the personnel hazards involved

in the use of carbon traps, and also the amount of product lost in discharging to carbon traps it was necessary to find what came through the trap during operation and how much of it.

It was found qualitatively that HF, C-616 and some XO_2F_2 dust were present in the gases issuing from the trap, the amounts of each varying with the service to which the trap was put and the amount of C-616 previously absorbed by the bed. No effort was ever made to find the amount of XO_2F_2 coming through, but the techniques described below were used in determining C-616 and HF in the exit gases.

To determine C-616, a large flask of known volume with two leads which can be closed off by stopcocks is attached to the line through which the exit gas is passing. The bulb is evacuated and then the stopcock to the line is opened. The pressure in the bulb is allowed to rise to about 5" Hg vacuum and then the stopcock is closed. The bulb is removed from the line and by opening a stopcock under the surface of a beaker of distilled water, water is sucked into the bulb and the C-616 is hydrolyzed. The water solution is then analyzed for X^{+6}. From the volume of the bulb and the pressure it is then possible to calculate by the gas laws the concentration of C-616 in the exit gases.

Since HF appears in the exit gas before C-616 in cases where the entering gas is diluted with G-74, it proved possible to use the following method to determine its concentration. At the end of the line through which the exit gases passed was attached a tee connection with valves in both arms. To these valves were attached two tubes filled with anhydrous NaF in which HF, and also, to some extent, C-616 are absorbed. By opening one of the valves only, for

a measured time, it was possible, knowing also the G-74 flow rate, to calculate from the weight gain the HF concentration in the exit gases. When C-616 came through too, direct weighing would not distinguish between the two, but an analysis for T^{+6} in the NaF served to give a method of estimating, roughly at least, the concentrations of the two present in the gas.

In both determinations it was found necessary to insert a plug of brass or nickel wool before the collecting device to prevent the XO_2F_2 from interfering with the analyses.

By these methods the data presented in Tables XXVII and XXVIII were obtained. The conditions under which the Section 300 type runs were made is given in Table XXIX; those for the Section 600 type runs in Table XXX. If the capacity of the trap is taken to be the point at which the C-616 concentration in the exit gases passes 10^{-4} lb. C-616 per cubic foot of G-74 it will be seen from the curves that this is quite a sharply defined point, with respect to fraction of the bed utilized. Evidently HF appears sooner than C-616 in the exit gases under Section 300 conditions, in which G-74 is present as a diluent in the gas stream, but not under Section 600 conditions where no G-74 is used.

Under Section 300 ± 1 conditions it appears that when C-616 first enters a cool carbon trap, it passes through to the extent of 5-10 mg. per liter of G-74. After the bed has warmed up as a result of the absorption of C-616 this concentration falls below 0.5 mg. per liter until the trap is nearly saturated. At this point the concentration in the exit gases begins to rise sharply and continues to do so until absorption stops completely. Under these conditions the data indicate that a carbon trap is not exhausted until it has absorbed

about 160% of the required amount in this section.

In Section 600 even a sharper break is observed at the point of saturation of the bed. Here no initial surge of C-616 comes through at the start, probably because of the lack of a carrier gas stream. Here, too, saturation is obtained only long after the required amount of C-616 has been absorbed; actually about 200% of this quantity.

J. Absorption of C-616 by Activated Carbon

The absorption of C-616 by activated carbon is a process, the mechanism and kinetics of which are complicated by the fact that adsorption is accompanied by chemical reaction between adsorbent and adsorbate. In view of the importance of the process in plant operations, it was felt that a detailed knowledge of the mechanism and products would be of interest and might prove valuable. A program of research has therefore been conducted to determine the influence upon this phenomenon of the following factors:

1. Activity of carbon
2. Size of carbon pellets
3. Type of carbon pellets
4. Moisture content of the carbon
5. Temperature of carbon
6. Partial pressure of C-616
7. Mass velocity of C-616

In addition to the investigation of the effect of these physical variables, a study of the reaction products was made to help in reaching an understanding of the chemistry involved in the reactions which occurred.

SECRET

1. Experimental

The general technique employed was one of passing a stream of C-616 and G-74, in the desired proportions, through a bed of carbon kept at constant temperature, noting the gain in weight of the carbon at intervals during the exposure. The apparatus used in most of these experiments is shown in Figure 4. The parts of this apparatus are detailed in the following drawings, Figures 5 - 7.

In order to obtain measurements under as nearly isothermal conditions as possible, the C-616 flow rate was kept very low. The flow rate used was about 5 gm/hr. through a 2 gm. sample of activated carbon.

In measuring activities by CCl_4 absorption the apparatus designed by the National Carbon Company for this purpose and described in a memorandum issued by the Carbide and Carbon Chemicals Corporation* was used, with slight modifications. Results of tests made with the apparatus constructed here were in general in good agreement with the values reported by the National Carbon Company.

2. Materials Used

a. Carbon

This was obtained from the National Carbon Company in the following types, grades, and sizes.

(1) Type

Pelleted

Crushed

---

* "Special Laboratory Methods of Testing Activated Carbon" by the Process Division of Carbide and Carbon Chemicals Corporation, January 5, 1939.

(2) Grade

High Activity (70-75%)

Medium Activity (50%)

Low Activity (25%)

(3) Size

2/4 mesh

4/6 mesh

6/8 mesh

The activity rating given by the National Carbon Company is based upon the absorption of CCl_4 by the carbon at 25°C.

On heating the carbon for six hours at 150°C, in general, it lost about 1% of its weight. This weight loss is called the moisture content by the National Carbon Company. Chemical analyses by combustion, as given in Table XXXI, showed, however, that the hydrogen content of material dried in this manner corresponded to about 4% by weight of water. This may be chemically bound to the carbon and therefore not removable by heating to 150°C.

b. C-616

This material was used as received from the Harshaw Chemical Company.

c. G-74

The commercial "dry" product was used without treatment. It is supposed to have a dew point lower than -30°C.

3. Results of Experiments

a. Activity of Carbon

At a particular temperature and partial pressure of

C-616 the efficiency after a given period of exposure is greater, the greater the activity of a given type of carbon.

This behavior can be seen from a comparison of the data obtained at 200°F for the absorption of C-616 from a gas stream containing 1.0 mole percent of C-616 by high, medium, low and zero activity carbon. The results are given in Table XXXII and plotted in Figure 8. (See also Figure 10).

b. Size of Carbon Particles

In Table XXXIII and Figure 9 are given the results of experiments in which 2/4 and 6/8 mesh high and low activity carbon pellets were exposed to 10% C-616 in G-74 at 200° and 500°F. The data show that differences in efficiency exist between particles of the same activity but different sizes at 500°F. At the lower temperature there is no measurable difference after saturation is reached.

At 200°F, experiments designed to estimate the rate of absorption show that a distinct difference exists between the rates of absorption of C-616 on 2/4 and 6/8 mesh activated carbon. The absorption on the larger particles reaches saturation much more slowly than that on the smaller particles. These experiments will be described in detail in the section on the rate of absorption. A comparison of the rapid portion of the absorption of C-616 by the two sizes of carbon is given in Figure 10.

c. Type of Carbon Particles

Although pelleted and crushed carbon of a given activity and mesh size display the same efficiency under comparable conditions, when the adsorbate is CCl_4, the crushed carbon shows a much lower adsorptive capacity for C-616 than does the pelleted carbon. This effect may be seen from the data presented in Table XXXIV giving a comparison of the adsorption of CCl_4 and C-616 on carbon.

d. Moisture Content of Carbon

A sample of 6/8 mesh high activity carbon, diluted with half its volume of alumina, was divided in half. The two halves were used as charges in small reactors. One was exposed immediately at 80°F to a fairly low flow of C-616 while the other was first dried at 500°F by passing through it a stream of dry C-74. (The moisture so removed was approximately 1% of the weight of carbon in the charge). This reactor was then exposed to 10% C-616 under conditions as nearly identical as possible to those used with the first sample. At the end of the runs a sample of charge from the entrance end of each reactor was removed and analyzed for T^{+4} and T^{+6}. The data given in Table XXXV show that, within the analytical error, there is no significant effect of pre-drying the carbon upon either the total absorption or upon the relative amounts of T^{+4} and T^{+6}. This may be because the treatment given does not remove a large enough fraction of some material present which will react with C-616 without reducing the X^{+6}. That all the moisture is not removed by this method is shown by the analyses in Table XXXI,

but the moisture remaining according to those analyses is not sufficient to account for all of the X^{+6} present in the carbon after exposure to C-616, if the hydrolysis proceeds according to the equation usually written for the reaction.

e. Temperature of Carbon

In Table XXXVI and Figure 11 are presented the data obtained on the effect of temperature on the absorption efficiency of high, low and zero activity carbon pellets. From Figure 11 it will be seen that the absorption efficiency depends both on the temperature and on the period of exposure. For exposures of one to six hours the absorption of C-616 increases with temperature up to 300°F or somewhat above, then decreases to a minimum around 400°F, then rises sharply. With longer periods of exposure the efficiency increases continuously with temperature. This behavior is less marked in low than in high activity carbon, and, within the accuracy of the measurements, no break is observed in the data on zero activity carbon.

f. Partial Pressure of C-616

The effect of partial pressure of C-616 is most marked under those conditions of temperature and period of exposure where the extent of chemical reaction between the adsorbent and adsorbate is small - namely low temperature and short exposure. In Table XXXVII are given data for the adsorption efficiency of 6/8 mesh high activity carbon at the end of one half hour exposure at 200°F at various partial pressures

of C-616. The straight line obtained in Figure 12 by plotting log (efficiency) vs. log (partial pressure C-616) indicates that under these conditions the absorption obeys the absorption isotherm:

$$E = k\,p^{1/n}$$

where E = grams C-616/gram of carbon

p = partial pressure of C-616

k&n = constants

g. Mass Velocity of C-616

The exothermic nature of the absorption of C-616 on activated carbon, coupled with the low thermal conductivity of the latter, causes a marked rise in carbon temperature if the absorption is allowed to proceed rapidly. From a comparison of the results obtained in these small scale experiments, made under almost isothermal conditions, with those obtained on the large scale laboratory and plant carbon traps made under almost adiabatic conditions it appears that the C-616 mass velocity through the bed in the latter case affects the efficiency of the absorption because of this temperature rise at the C-616 flow rate used.

The C-616 mass velocity will also affect the rate at which the limiting adsorption efficiency is reached. If the mass velocity is rapid enough so that the gas phase concentration remains essentially constant, the rate of adsorption at any time will depend solely upon the pressure of C-616, the temperature, the nature of the surface

and the fraction of the surface covered by C-616 at that time. If the mass velocity is slow compared to the rate of adsorption measured under the above conditions then that will be the factor which will determine the rate at which the "limiting" or "equilibrium" adsorption is reached. Only in the latter case is it possible to maintain nearly constant carbon temperatures

h. Rate of C-616 Absorption

To determine the rate of C-616 absorption on carbon by noting the weight gain of the adsorbent at periodic intervals it is necessary that the carbon be exposed continuously to a constant pressure of adsorbate. If the absorption rate is rapid and the experiment is performed in a flow system it is necessary that the flow of C-616 be so rapid that its concentration in the gas phase remains essentially constant during the entire experiment.

For this reason the experiments performed to investigate the other aspects of the absorption could not be used for rate determinations because of the low C-616 mass velocity required to maintain isothermal conditions. A separate series of experiments was therefore performed in a special unit in which six copper tubes, 10 inches long and 1.2 inch I.D. were installed in parallel in an oven with valves on both ends of each tube so each one could be completely shut off independently of the rest. For the experiment the six tubes were opened and flooded with pure gaseous C-616 at 1 atm. pressure and at a mass velocity of 0.15 to 0.45

lbs/ft^2-sec., the excess passing through being collected in a large carbon trap. The tubes were then shut off one after the other at measured time intervals and the weight gains noted.

In spite of the use of the small copper tubes for reactors and use of crystalline alumina as a diluent in 1:1 ratio by volume with the carbon, there was, in all cases where high activity carbon was used, a considerable temperature rise at first even on the outside surface of the reactors. For this reason it is probable that at least in those cases both the rate and the total amount of reaction is somewhat higher than would be experienced if the carbon had been kept at the stated temperature. The error should be relatively small if the temperature coefficient measured in some of the experiments is correct.

It should be noted in connection with this error that, since the carbon cools off rapidly in these runs after the major part of the reaction is completed, the reaction rate falls off with time for two reasons. First, the amount of reaction which has taken place reduces the effective surface. Second, there is a decrease in the temperature of the carbon. If it were only the first, some reasonably simple rate equation might be expected to apply but under these conditions it seems doubtful that one will.

The data obtained in these experiments are given in Table XXXVIII and plotted in Figure 10 in which are shown the effects of temperature upon the absorption rate for

high and low activity carbons, and the effect of carbon activity and particle size at a given temperature. The data for 2/4 mesh high activity can be plotted as log (efficiency) vs. log (time) in order to compare the reaction rates at 200°F and 400°F. The almost straight lines so obtained indicate that the rate at 400°F is about three times as great as that at 200°F. Assuming the Arrhenius equation to apply over this temperature range, a value of ~ 3 kcal per mole can be obtained for the activation energy of the process, a figure which might indicate physical adsorption to be the rate determining factor.

The efficiency v. time curves obtained on the runs made with 10% C-616 cannot be used to obtain reaction rate values for reasons previously given. They do, however, indicate that the rapid adsorption observed initially is followed by a slower process in which the rate of absorption is probably determined largely by the surface changes which the carbon undergoes by virtue of chemical reaction between it and the C-616 already adsorbed. Evidence for such chemical reaction has been found in the presence of K^{+4} on the carbon after exposure to C-616 under these conditions. This will be discussed more fully in a later section. The data for such a series of runs with 6/8 high activity carbon are given in Table XXXIX and plotted in Figure 13. The steady rise in the curves may be taken as an indication of the existence of chemical reaction, and as is to be ex-

pected, the slopes of these curves increase with increasing temperature.

It thus appears that the absorption consists of an initial rapid adsorption at all temperatures followed by a period in which the rate is dependent upon the rate of chemical reaction between adsorbent and adsorbate.

i. Reversibility of C-616 Absorption

Since activated carbon is known as an excellent adsorbent, and since the matter was of considerable interest in the operation of carbon traps, experiments were performed to determine whether or not the absorption of C-616 by activated carbon was reversible. The results show it to be only very slightly reversible under the conditions tried. These included absorption of C-616 at room temperature or higher followed by pumping at the temperature of absorption, or above, or blowing out with dry C-74 at the same temperatures. The greatest weight loss found amounted to 5% of the total amount absorbed by the carbon, an amount so small in that case as to be very near the limit of accuracy of the method. This behavior is difficult to explain in view of the dependence of efficiency on pressure found in experiments previously described. It may be that at the low pressures and short times used in the pressure dependence experiments the rate of absorption is reduced, and that the pressure dependence found is only transitory.

j. Fluorocarbon Production During C-616 Absorption by Activated Carbon

Several types of experiments were performed to determine the nature of the fluorocarbons producted in the reaction. In the first, a stream of 100% C-616 at 1 atm. pressure was sent into a bed (12" x 1 1/2") of 6/8 mesh high activity carbon. The exit gases were passed through a washing tower containing standard sodium hydroxide designed to absorb any HF then through a tube containing Ascarite and Dr erite to remove H_2O, and finally through a dry ice and a liquid G-74 trap, after which any residual gases were vented to the atmosphere. It was expected that higher fluorocarbons would collect in the dry ice trap and only those with three carbon atoms or less would be collected in liquid G-74. After collecting the samples they were transferred to bulbs with break off seals and sent out for mass spectrographic analysis.

In the first experiment, in which the carbon temperature was 400-500°F, nothing was collected in the dry ice trap, and back titration of the standard sodium hydroxide solution indicated no HF, within the experimental accuracy. This was not unexpected since the reaction zone proceeded through only about 3/4 of the bed depth, and the Section 600 experiments described previously indicated that when using 100% C-616 appreciable quantities of HF do not appear until the bed is almost completely exhausted. Approximately 3 cc of liquid were found in the liquid G-74 trap.

Dr. Nier's analysis of the gas samples may be summarized as follows:

1) The bulk of the sample consisted of saturated fluoro-

carbons of molecular weight 153 or less, with C_3F_8 accounting for a good part of the mixture.

2) Traces of higher fluorocarbons up to C-616 were found.

3) Traces of hydrocarbons appeared to be present.

4) About 5% of the gas was G-74 and oxygen.

Another experiment in which the reactor was kept at a lower temperature, about 150°F maximum, was performed, but the samples collected were not large enough for satisfactory analysis even by the mass spectrograph. Again, very little was collected in the dry ice trap. The samples from the liquid G-74 trap appeared to contain unsaturated fluorocarbons, in contrast to the high temperature run in which the fluorocarbons were predominantly saturated.

In the light of these results another series of experiments was performed with the object of producing larger quantities of fluorocarbons so that a more reliable identification of products could be made. The set-up constructed for this purpose is shown in Figure 14.

After the reactor was charged, C-616 was rapidly introduced, keeping a valve between the reactor and the collection train closed. When a pressure of 40 psig was reached the C-616 supply was shut off. The pressure first fell rapidly and then began to rise slowly again as the reaction proceeded. By opening and closing the valve the reaction products were slowly bled out through the absorption train and collected in the cold trap.

By this method, using reactor temperatures of 500-900°F about 100 cc. of fluorocarbons, liquid at -190°C, were collected. Operation at lower temperatures failed to produce an appreciable amount of fluorocarbons, and was therefore abandoned. An attempt to produce the fluorocarbons by running experiments under section 600 conditions failed for the same reason.

The liquid produced in the experiments, after a short pumping to remove G-74 and O_2, was sent to the M. W. Kellogg Co.'s analytical laboratory where a distillation curve on the material was obtained using a Podbielniak low temperature fractionating column. Although mechanical difficulties in the distillation introduced some error, the results of the analysis given in Table XL are believed to be correct to within a few percent. For CF_4 a good flat was observed at -182°F (cf. -198°F, Handbook of Chemistry and Physics). The next fraction boiled in the range -120° to -98°F, with possibly a small amount coming over at -75°F. Both C_2F_6 and C_2F_4 are reported to boil at -108°F. Data on the higher members of the series were not given in the readily available reference books, but interpolation between the data on C-716 and those reported should lead to fairly accurate estimates. On this basis, the boiling point of C_3F_8 should be -40°F. The flat in this region is at -25°F. The residue undoubtedly consisted

of compounds containing four or more carbon atoms per molecule.

In addition to the highly volatile compounds mentioned above, a certain amount of material having the consistence of C-2144, and some hard wax was found in trap I just outside the reactor after runs at 900°F. It is quite probable that these materials were formed in the other experiments also, but the temperature was not high enough to drive them out of the reactor. This material, which could cement carbon particles together, may be the explanation for the plugging and caking of carbon traps when operated at 600°F or higher. A vacuum distillation (at 4 mm pressure) was carried out on the material and two fractions taken, one boiling at about 200°C, the other between 230° and 250°C. Molecular weights of each fraction were determined by measuring the boiling point elevation of C-716 solutions of the fluorocarbons, and values of about 500 and 800 respectively obtained.

To determine the extent of heavy fluorocarbon production as a function of the carbon temperature, a series of flow runs was made at temperatures from 200° to 700°F. Runs were made with 1 atm. of 100% C-616 flowing at 2.15×10^{-2} lbs./ft^2-sec. for 1000 seconds into a 12" x 1 1/2" reactor filled with 2/4 mesh high activity carbon. At the end of this time the bottom six inches of the bed were removed, ground to a powder, and extracted with C-816 for 7 hours. The transmission of the extract was determined at 525 mμ and compared with the transmission of pure C-816. The C-816

was then distilled off from the extract and the residue weighed.

The results are plotted in Figure 15 as grams of heavy fluorocarbon extracted from 60 gm. of sample vs. initial and final carbon bed temperatures. A better correlation is obtained in the former plot. In Figure 16 the log % transmission (525 mμ) is plotted against the grams of fluorocarbon extracted from 60 grams of sample. The nearly linear relationship so obtained implies a closely related structure in the products of the reaction at different temperatures. Table XLI gives, in addition to this, the X^{+4}/gm. of sample for each of the runs. It will be noted that this does not rise nearly as rapidly with temperature as does the heavy fluorocarbon production.

Besides the heavy fluorocarbons mentioned above it was observed in at least one case when carbon was exposed to 10% C-616 at 400°F for 24 hours that a considerable quantity of clear needle-like crystals of a material answering the description of $(CF)_x$ were formed.

k. <u>X Analysis on Carbon after Exposure to C-616</u>

A large number of analyses were performed by the analytical group on spent carbon from the experiments described above with the object of obtaining a clearer picture of the mechanism and chemistry of the absorption process. The analytical method developed for the purpose has been described in their reports. It is believed that the data obtained are fairly reliable, but at very high and

very low efficiencies, because of the large effect small errors would have on the calculated functions, the accuracy of the reported values may not be good.

The data are summarized in Table XLII and the average values from the table plotted in Figure 17. The top portion of Figure 17 gives the relation between temperature of exposure and the ratio, weight of X^{+6} divided by weight of carbon, as calculated from the analyses for X^{+4} and X^{+6} and the measured absorption efficiency. In these calculations it was necessary to assume that no loss of volatile products occurred on C-616 absorption, an assumption which was found to lead to calculated values of the efficiencies more nearly on accord with the measured efficiencies than the other possible assumptions - i.e. - that the X^{+4} is present as XF_4 and the X^{+6} as XF_6 or XO_2F_2. The calculated values are open to some question quantitatively but it is believed that the general shape of the curves, which show in nearly all cases a maximum X^{+6}/C ratio at 400°F, is correct.

The fact that it is necessary to assume little or no loss of volatile products as a result of C-616 absorption on carbon makes it appear probable that the reduction of the C-616 on absorption results principally in non volatile fluorocarbons or partially fluorinated carbon compounds. This is not necessarily in disagreement with observations mentioned previously in which low boiling fluorocarbons were isolated from the exit gases, since in those experi-

ments the reaction temperature was considerably higher and even there not nearly enough volatile fluorocarbons were collected to account for all the C-616 absorbed. That an appreciable fraction of the absorbed C-616 is reduced even at the lower temperatures is shown in the lower portion of Figure 17.

The X^{+4}/X ratios in Figure 17 were all measured after 24 hours C-616 exposure. In Table XLII it will be seen that after 1 hour exposure the ratio is in general slightly less than that reported after 24 hours exposure, at 200° and 300°F.

4. Interpretation of Results

The interpretation of the analytical data is made uncertain not only because of the possible errors in the calculations but also because of the complexity of the phenomenon of absorption. It is evident, both from the analyses, and from the fact that fluorocarbons are formed, that besides absorption of C-616 there is also reduction of C-616 to XF_4. XF_4, in the presence of C-616 at the temperatures and pressures used in these experiments, can combine with it to form XF_5 or one of the other mixed compounds reported. In addition carbon hydrogen analyses on high activity carbon by Dr. W. F. Libby's group at SAM indicate that there is some water present, actually about 4% by weight on high activity carbon after drying six hours at 150°C, and probably 5 - 6% by weight on the undried material as received*. C-616 will probably react with this moisture to form XO_2F_2. An attempt will, how-

* See Table XXXI

ever, be made to rationalize the observations reported.

The presence of moisture on the carbon complicates matters to some extent, but the quantity of moisture present should only contribute 0.4 - 0.6 units to the efficiency, an amount which is relatively small in comparison to the minimum total efficiency measured for high activity carbon. The remainder of the measured efficiency must be explainable in terms of adsorption, chemical reaction, or a combination of the two.

In an attempt to determine experimentally the relative amounts of C-616 and XO_2F_2 on carbon after C-616 exposure, two samples of 6/8 mesh high activity carbon pellets were exposed to 10% C-616 at 200°F for 2 hours. At the end of this time, one of the two was submitted for analysis. The other, without removing it from the original reactor, was flushed with dry C-74 for a few minutes, and then heated in a muffle furnace to 650°F with the ends of the reactor closed off. After 4 hours at 650°F this sample, too, was removed and analyzed. It was hoped that the change in the proportions of T^{+6} and T^{+4} on the carbon would be a measure of the amount of C-616 present originally.

Analytical data on the two samples are given in Table XLIII. It will be seen that some reduction of C-616 occurred from the increase in the X^{+4}/X ratio. However, the value of X^{+6}/carbon, while reduced by the heating, is much higher than would be predicted from the measured moisture content. There are numerous possible explanations of this, and, since this information was obtained from a single experiment only, speculation as to the reason for this behavior does not appear profitable.

Returning to the discussion of the other absorption and analytical data, it is relatively easy to explain the observations at 500°F. There reduction of C-616 is undoubtedly the predominant factor as shown by the sharp rise in X^{+4}/X at that temperature. Evidently XF_5 either is not stable or does not have a chance to form there since the ratio becomes greater than 0.5. This would account for the decrease in the X^{+6}/C ratio and for the steady rise in efficiency with time.

Between 300°F and 500°F reaction is appreciable as shown by the change in efficiency with time in that region (Figure 13). The decrease in efficiency with increasing temperature for periods of exposure too small to permit any great amount of reaction to occur is typical of adsorption phenomena, so it appears that the first step in the absorption at these temperatures is a result of a relatively rapid adsorption. Further "absorption" is due to the slow reduction of C-616 by the carbon. The data in Table XLII indicate that an increase in both X^{+4}/X and X^{+6}/C occurs with time at 300°F which may be taken to imply that a compound of X^{+4} and X^{+6} is being formed on the carbon. Otherwise the increase in X^{+6}/C would not occur. From the X^{+4}/X ratios the compound could be XF_5, allowing for some uncertainty in the data and a certain amount of XO_2F_2 present as a result of hydrolysis. Actually a material answering the visual description of XF_5 has been found on carbon after reaction with C-616 under these conditions.

The most difficult temperature region to interpret is that between room temperature and 300°F, in which reaction is extremely slow, and there is an increase in absorption efficiency

with temperature over any time interval tried. Such behavior is not characteristic of a normal physical adsorption. It is, however, found, in cases where both physical adsorption and chemisorption are possible, in the region in which the chemisorption begins to occur at an observable rate. In view of the fact that reaction occurs in this case at temperatures only slightly higher than the ones under discussion and since the observed adsorption appears to be irreversible, another characteristic of many cases of chemisorption, it is not impossible that this is the explanation for the anomolous absorption here. Since the analyses show an appreciable amount of XF_4 to be present when C-616 is absorbed under these conditions the picture is undoubtedly influenced by reaction of C-616 with carbon in this region, too. The usual theory of chemisorption, involving the assumption of a surface whose activity varies from place to place, could be used to account for this, if it is assumed that the reactivity and activity of the surface are directly related.

As a tentative explanation for the observed facts the following is postulated:

a. Hydrolysis or some other non-reductive reaction of C-616 sufficient to account for at least 0.4 - 0.6 efficiency units.

b. Adsorption, perhaps chemical in nature, which increases with temperature up to about 300°F and then falls off with any further increase in temperature.

c. Reduction of C-616 to XF_4, with the formation principally of non-volatile fluorocarbons. This reaction

occurs very slowly at 200°F and quite rapidly at 500°F. The types and proportions of fluorocarbons formed probably change somewhat with temperature.

d. Formation of XF_5 at 400°F and perhaps at lower temperatures also due to reaction of C-616 with XF_4.

e. Formation principally of XF_4 at 500°F.

TABLE I

PROPERTIES OF SOLID C-616 ABSORBENTS

	Carbon			Carbon[a] Impregnated Alumina	Florite Dessicant	Activated Alumina	Silica Gel		Sodium Fluoride
	High Activity	Low Activity	C-716 Impregnated				Davco	Santocel	
Absorptive Capacity[b] gm C-616/cc. of absorbent	1.3	0.4	1.3	0.3[c]	0.5[d]	0.2	0.1	<0.8[e]	0.13
Density, gm./cc.	0.48	0.48	0.48	0.5-0.7	0.79	0.6-0.8	~0.5	0.1	~1.0
Temperature Rise,[f] °F	>1000	400	-	300	300	-	-	800	-
Caking and Plugging	Bad	above	400°F	Fair	None	Bad[g]	None	Bad	Occasional
Reversibility	Very slight		Very slight	Very slight		-	-	-	-
HF Evolution	1% by weight of C-616 absorbed				None	-	-	Bad	None
Rate of C-616 Absorption	Very Rapid				Rapid Initially	Rapid	-	Rapid	Rapid

a Carbon coated alumina, prepared in these laboratories was also tested. In one case it proved satisfactory, but development of the National Carbon Co.'s product led to the abandonment of further work.

b At 200°F for the carbon and fluorite. In runs starting at room temperature in the other cases.

c Mixture containing 19% carbon by weight.

d After 12 hrs. After 1/2 hr. the efficiency is only 0.1

e This was measured on very finely powdered material. Another less finely divided Santocel had a capacity of 0.1 under analogous conditions.

f Observed on adding 100% C-616 at about 100 gm/min. to the absorbent in a 3" diameter reactor.

g Due to hydrolysis products.

h Bad in the presence of HF. In this case fusion of the charge has occasionally been observed.

TABLE II

COMPARISON OF HEATING EFFECTS

Charges of High Activity Carbon Diluted with Alumina
vs.
Charges of Low Activity Carbon

Run No.	XXX	XXXI	L-2	L-3
Carbon Used Form Mesh Size	High Activity Pellets 2/4	High Activity Pellets 2/4	Low Activity Pellets 2/4	Low Activity Pellets 2/4
Diluent Used* Form	Alumina Cryst. 4	Alumina Cryst. 4	None	None
Diluent Bulk Density $lb.ft^3$	90	90	----	----
Dilution Ratio**	2:1	2:1	----	----
Bed Dimensions, in. Diameter Depth	3	3	3	3
Bed Temperature, °F Initial Maximum	70 323	80 307	100 425	80 515
Mole % C-616 in Feed	100	100	100	100
C-616 Mass Velocity, $lb./ft^2/sec.$	.131	.113	.151	.131
Linear Velocity ft./sec.	.144	.125	.166	.144
Duration of Run, hours	.23	.34	.17	.25
Time Before KOH Yellowed, hrs.	None	None	Initial Surge	HF Initially
Capacity, lb. C-616 abs/ft^3 of charge	13.8	17.5	9.6	12.5
Plugging	None	None	In line	In line
Remarks	Slight cake	----	----	Slight Cake

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon

TABLE III

TESTS ON DILUENTS

Run No.	12T	9A	16A	12B	13B
Diluent Used Form Mesh size	None	None	None	Alumina cryst. 4/6	Alumina cryst. 4/6
Carbon Used Form Mesh size	High Activity Pellets 6/8	High Activity crushed 4/6	High Activity Pellets 2/4	None	High Activity crushed 4/6
Diluent Bulk Density, lb/ft^3	----	----	----	90	90
Diluent Ratio*	----	----	----	----	8:1
Bed Dimensions, in. Diameter Depth	1.4 14	1.4 14	1.4 14	1.4 14	1.4 14
Bed Temperature, °F. Initial Maximum	400 490	400 430	400 530	600 665	400 435
Mole % C-616 in Feed	100	100	100	100	100
C-616 Mass Velocity, $lb/ft^2/sec.$	4.9×10^{-3}	1.3×10^{-3}	4.4×10^{-3}	4.0×10^{-3}	4.5×10^{-3}
Linear Velocity, ft/sec.	4.6×10^{-3}	1.2×10^{-3}	3.9×10^{-3}	3.6×10^{-3}	3.2×10^{-3}
Duration of Run, hrs.	6	6	6.5	6	6
Time Before KOH Yellowed, hrs.	----	----	1.4	0	1.7
Capacity, lb. C-616 abs./ft^3 of charge	----	----	----	0	----
Plugging	None	None	None	None	None
Remarks	Bad Cake	Bad Cake	Bad Cake	No Cake Charge Slightly green	Caked

* Expressed as volume of diluent : volume of carbon

TABLE III (continued)

Run No.	117	10A	11A	129	84	126
Diluent Used	Silica	Silica	Silica	Queen Bee Coke	Graphite	Zero Activity
Form	Fused	Fused	Fused	Crushed	---	Crushed
Mesh size	4	4	4	2/4	4	4/6
Carbon Used	None	High Activity	High Activity	None	None	None
Form		Crushed	Pellets			
Mesh size		4/6	6/8			
Diluent Bulk Density, lb/ft^3	80	80	80	20	40	30
Dilution Ratio*	---	8:1	8:1	---	---	---
Bed Dimensions, in.						
Diameter	1.4	1.4	1.4	1.4	1.4	1.4
Depth	14	14	14	14	14	14
Bed Temperature, °F						
Initial	600	400	400	600	600	600
Maximum	600	425	445	655	630	655
Mole % C-616 in Feed	100	100	100	100	100	100
C-616 Mass Velocity, lb/ft^2/sec.	3.2×10^{-3}	1.6×10^{-3}		3.8×10^{-3}	5.5×10^{-3}	4.3×10^{-3}
Linear Velocity, ft/sec.	2.9×10^{-3}	1.3×10^{-3}		3.4×10^{-3}	5.0×10^{-3}	4.0×10^{-3}
Duration of Run, hrs.	0.5	3.3	6	1.4	1.7	6
Time Before KOH Yellowed, hrs.	0	---	1.4	0.2	---	---
Capacity, lb. C-616 abs/ft^3 of charge	0	---	---	---	---	---
Plugging	None	None	None	Bad	Bad	None
Remarks	No reaction	Slight Cake	Slight Cake	Bad Cake	Bad Cake	Bad Cake

* Expressed as volume of diluent : volume of carbon

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TABLE III (continued)

Run No.	5A	4A
Diluent Used	Zero Activity	Pumice
Form	Pellets	Crushed
Mesh size	2/4	2/8
Carbon Used	None	None
Diluent Bulk Density, lb/ft^3	30	---
Dilution Ratio*	---	---
Bed Dimensions, in.		
Diameter	1.4	1.4
Depth	14	14
Bed Temperature, °F		
Initial	600	600
Maximum	685	650
Mole % C-616 in Feed	100	100
C-616 Mass Velocity, $lb/ft^2/sec.$	3.9×10^{-3}	5.0×10^{-3}
Linear Velocity, ft/sec.	3.5×10^{-3}	4.7×10^{-3}
Duration of Run, hrs.	3.3	0.5
Capacity, lb. C-616 abs./ft^3 of charge	---	---
Plugging	Bad	Bad
Remarks	Bed Caks	Caked

* Expressed as volume of diluent : volume of carbon

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TABLE IV

TESTS ON DIFFERENT TYPES OF CARBON

Run No.	16A	5A	125	130	133	15A
Carbon Used						
Form	High Activity Pellets	Zero Activity Pellets	Zero Activity Crushed**	High Activity Crushed	High Activity Crushed	High Activity Pellets
Mesh size	2/4	2/4	6/8	4/6	4/6	6/8
Diluent Used						
Form	None	None	None	Alumina Cryst.	Alumina Cryst.	Alumina Cryst.
Mesh size				4/6	4/6	6/8
Diluent Bulk Density, lb/ft^3.	---	---	---	90	90	90
Dilution Ratio*	---	---	---	8:1	8:1	8:1
Bed Dimensions, in.						
Diamter	1.4	1.4	1.4	1.4	1.4	1.4
Depth	14	14	14	14	14	14
Bed Temperature, °F						
Initial	400	600	600	400	400	400
Maximum	530	685	610	485	435	420
Mole % C-616 in Feed	100	100	100	100	100	100
C-616 Mass Velocity, $lb./ft^2/sec.$	4.4×10^{-3}	3.9×10^{-3}	3.2×10^{-3}	x	4.5×10^{-3}	4.2×10^{-3}
Linear Velocity, ft/sec.	3.9×10^{-3}	3.5×10^{-3}	2.8×10^{-3}	x	3.2×10^{-3}	3.8×10^{-3}
Duration of Run, hrs.	6.5	3.3	6	6	6	4.2
Time before KOH Yellowed, hrs.	1.4	0.4	3.7	1.6	1.7	1.4
Total Absorption, gm/gm of carbon	5.8	0.42	1.00	6.3	9.3	≥4.0
Caking	Badly Caked	Caked	Badly Caked	Slightly Caked	Badly Caked	Badly Caked

* Expressed as volume of diluent : volume of carbon

** Obtained by crushing 2/4 mesh zero activity carbon pellets.

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TABLE IV (continued)

Run No.	13B	13A	11A	12A	14A
Carbon Used	High Activity	High Activity	High Activity	High Activity	High Activity
Form	Crushed	Crushed	Pellets	Pellets	Pellets
Mesh Size	6/8	6/8	6/8	6/8	6/8
Diluent	Silica	Silica	Silica	Silica	Silica
Form	Fused	Fused	Fused	Fused	Fused
Mesh size	4	4	4	4	4
Diluent Bulk Density, lb/ft^3	80	80	80	80	80
Dilution Ratio*	8:1	4:1	8:1	4:1	4:1
Bed Dimensions, in.					
Diameter	1.4	1.4	1.4	1.4	1.4
Depth	14	14	14	14	14
Bed Temperature					
Initial	400	400	400	400	400
Maximum	465	420	445	425	460
Mole % C-616 in Feed	100	100	100	100	100
C-616 Mass Velocity, $lb./ft^2/sec.$	4.0×10^{-3}	2.3×10^{-3}	x	x	2.6×10^{-3}
Linear Velocity, ft/sec.	3.5×10^{-3}	2.0×10^{-3}	x	x	2.4×10^{-3}
Duration of Run, hrs.	6	6	6	6	6
Time before KOH Yellowed, hrs.	x	5.3	1.4	3.6	2.8
Total Absorption, gm/gm of carbon	7.8	7.5	4.0	3.6	6.4
Caking	Caked	Caked	Slightly Caked	None	Badly Caked

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* Expressed as volume of diluent : volume of carbon
x Information not available

TABLE X

PRELIMINARY TESTS - SECTION 100 CONDITIONS

Run No.	I	II	III	IV	V
Carbon Used Form Mesh Size	High Act. Crushed 4/6	High Act. Crushed 4/6	High Act. Pellets 2/4	High Act. Pellets 2/4	High Act. Pellets 2/4
Diluent Used* Form Mesh Size	Alumina Cryst. 4/6	Alumina Cryst. 4/6	None	Alumina Cryst. 4/6	Alumina Cryst. 4/6
Diluent Bulk Density, lb./ft.^{3}	90	90		90	90
Dilution Ratio**	4:1	1:1		1:1	1:4
Bed Dimensions, in. Diameter Depth	3 23	3 23	3 23	3 23	3 23
Bed Temperature, °F. Initial Maximum	80 240	80 250	80 1045	80 350	80 345
Mole % C-616 in Feed	100	100	100	100	100
C-616 Mass Velocity, lb./ft^2/sec.	0.41	0.113	0.165	0.174	0.153
Linear Velocity, ft./sec.	0.168	0.139	0.198	0.208	0.182
Duration of Run, hrs.	0.15	0.03 & 0.05	0.08	0.13	0.10
Time before KOH Yellowed, hrs.	___ a	___ a	___ a	___ a	___ a
Capacity, lb. C-616 abs./ft^3 of charge	9.1	18.4	15.8	3.1	21.4
Plugging	None	Condensed C-616	Plugged	None	None
Remarks	___	Moderate Cake	Moderate Cake	___	___

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon
[a] Due to the short bed, C-616 came through immediately

TABLE V (continued)

Run No.	VI	VII	IX	Required Conditions F-110
Carbon Used	High Act.	High Act.	High Act.	
Form	Pellets	Pellets	Pellets	
Mesh Size	2/4	2/4	2/4	
Diluent Used*	Alumina	Alumina	Alumina	
Form	Cryst.	Cryst.	Cryst.	
Mesh Size	4/6	4/6	4	
Diluent Bulk Density, lb/ft^3	90	90	90	
Dilution Ratio*	1:4	1:1	1:2	
Bed Dimensions, in.				
Diamter	3	3	3	30
Depth	23	23	108	46
Bed Temperature, °F				
Initial	200	200	97	165
Maximum	655	350	322	—
Mole % C-616 in Feed	100	100	100	100
C-616 Mass Velocity, $lb/ft^2/sec.$	0.086	0.152	0.186	0.073
Linear Velocity, ft/sec.	0.102	0.182	0.254	0.093
Duration of Run, hrs.	0.13	0.13	0.13	0.12
Time before KOH Yellowed, hrs.	—[a]	—[a]	—	—
Capacity, lb. C-616 abs./ft^3 of charge	20.6	6.0	10.7	7.3
Plugging	None	None	None	None
Remarks	—	—	—	—

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon

TABLE VI

C-616 ABSORPTION - SECTION 100 CONDITIONS

Run No.	XXIII	XXIV	XXV	XVI
Carbon Used Form Mesh Size	High Act. Pellets 2/4	High Act. Pellets 2/4	High Act. Pellets 2/4	High Act. Pellets 2/4
Diluent Used* Form Mesh Size	Alumina Cryst. 4	Alumina Cryst. 4	Alumina Cryst. 4	Alumina Cryst. 4
Diluent Bulk Density, lb./ft^3	90	90	90	90
Dilution Ratio	1:2	1:2	1:2	2:1
Bed Dimensions, in. Diameter Depth	3 90	3 90	3 90	3 92
Bed Temperature, °F Initial Maximum	80 345$^{\pi}$	100 470$^{\pi}$	50 640$^{\pi}$	30 295$^{\pi}$
Mole % C-616 in Feed	100	100	100	100
C-616 Mass Velocity, lb./ft^2/sec.	2.16×10^{-2}	1.9×10^{-2}	9.1×10^{-2}	1.68×10^{-2}
Linear Velocity, ft./sec.	2.96×10^{-2}	2.89×10^{-2}	0.149	1.85×10^{-2}
Duration of Run, hrs.	2.0	2.0	0.47	2.22
Time before KOH Yellowed, hrs.	——	——	No reaction	No reaction
Capacity, lb. C-616 abs./ft^3 of charge	22	20	22	17
Caking	Bad in first quarter	Bad in first third	Bad at entrance	None
Remarks	$^{\pi}$5000 sec, 28" from entrance	$^{\pi}$1000 sec, 7" from entrance	$^{\pi}$1500 sec, 12" from entrance	$^{\pi}$1500 sec, 12" from entrance

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon

TABLE VI (continued)

Run No.	XVII	Required Conditions Section 100
Carbon Used	High Act.	———
Form	Pellets	———
Mesh Size	2/4	———
Diluent Used*	Alumina	———
Form	Cryst.	———
Mesh Size	4	———
Diluent Bulk Density, lb./ft.^{3}	90	———
Dilution Ratio**	2:1	———
Bed Dimensions, in.		
Diamter	3	3
Depth	92	92
Bed Temperature, °F.		
Initial	60	Room Temp.
Maximum	350[π]	———
Mole % C-616 in Feed	100	100
C-616 Mass Velocity, lb./ft^2/sec.	1.43×10^{-2}	1.42×10^{-2}
Linear Velocity, ft./sec.	1.58×10^{-2}	1.56×10^{-2}
Duration of Run, hrs.	2.33	2.33
Time before KOH Yellowed, hrs.	No Reaction	No reaction
Capacity, lb. C-616 abs./ft^3 of charge	15	15
Caking	None	None
Remarks	[π]2600 sec, 25" from entrance. C-616 condensed in bed	———

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon

TABLE VII

C-616 ABSORPTION - SECTION 600 CONDITIONS

Run No.	XXX	XXXI	Required Conditions F-605
Carbon Used	High Act.	High Act.	———
Form			
Mesh Size	2/4	2/4	———
Diluent Used*	Alumina	Alumina	———
Form	Cryst.	Cryst.	———
Mesh Size	4	4	———
Diluent Bulk Density, lb./ft^3	90	90	———
Dilution Ratio*	2:1	2:1	———
Bed Dimensions, in.			
Diameter	3	3	36
Depth	90	90	60
Bed Temperature, °F			
Initial	70	80	180
Maximum	323	307	———
Mole % C-616 in Feed	100	100	100
C-616 Mass Velocity, lb./ft^2/sec.	0.131	0.113	0.099
Linear Velocity, ft./sec.	0.144	0.125	0.131
Duration of Run, hrs.	0.23	0.34	0.23
Time before KOH Yellowed, hrs.	———	———	———
Capacity, lb. C-616 abs./ft^3 of charge	13.8	17.5	8.3
Caking	Slight	None	None
Remarks	3" from entrance 160°F at start	3" from entrance 160°F at start	———

* 2" Alumina at entrance

** Expressed as volume of alumina : volume of carbon

TABLE VIII

C-616 ABSORPTION IN CHARGES CONTAINING 6/8 MESH HIGH ACTIVITY CARBON - SECTION 600 CONDITIONS

Run No.	XXI	XIII	XIV	XV
Carbon Used				
Form	High Act. Pellets	High Act. Pellets	High Act. Pellets	High Act. Pellets
Mesh Size	6/8	6/8	6/8	6/8
Diluent Used*				
Form	Alumina Cryst.	Alumina Cryst.	Alumina Cryst.	Alumina Cryst.
Mesh Size	4	4	4	4
Diluent Bulk Density, lb./ft^3	90	90	90	90
Dilution Ratio*	1:2	1.5:1	1.5:1	1.5:1
Bed Dimensions, in.				
Diameter	3	3	3	3
Depth	90	90	90	90
Bed Temperature, °F				
Initial	80	80	80	75
Maximum	1200+	580+	610+	745+
Mole % C-616 in Feed	100	100	100	100
C-616 Mass Velocity, lb./ft^2/sec.	0.153	0.057	0.182	0.187
Linear Velocity, ft./sec.	0.344	0.092	0.298	0.338
Duration of Run, hrs.	0.20	0.11	0.20	0.20
Time before KOH Yellowed, hrs.	——	——	——	——
Capacity, lb. C-616 abs./ft^3 of charge	16.1	3.1	18.5	19.2
Caking	Bad in first quarter	None	Bad where hottest	Bad where hottest
Remarks	+at 680 sec, 12" from entrance	+at 380 sec, 3" from entrance	+at 720 sec, 28" from entrance	+at 720 sec, 12" from entrance

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon

TABLE VIII (continued)

Run No.	XVIII	XVII	XVI	Required Conditions F-605
Carbon Used	High Act.	High Act.	High Act.	———
Form	Pellets	Pellets	Pellets	———
Mesh Size	6/8	6/8	6/8	———
Diluent Used*	Alumina	Alumina	Alumina	———
Form	Cryst.	Cryst.	Cryst.	———
Mesh Size	4	4	4	———
Diluent Bulk Density, lb./ft^3	90	90	90	———
Dilution Ratio**	2.5:1	3:1	4:1	———
Bed Dimensions, in.				
Diameter	3	3	3	36
Depth	90	90	90	60
Bed Temperature, °F				
Initial	92	90	60	180
Maximum	505$^{\pi}$	310$^{\pi}$	250$^{\pi}$	———
Mole % C-616 in Feed	100	100	100	100
C-616 Mass Velocity, lb./ft^2/sec.	0.118	0.177	0.159	0.099
Linear Velocity, ft/sec.	0.182	0.275	0.214	0.131
Duration of Run, hrs.	0.20	0.20	0.18	0.23
Time before KOH Yellowed, hrs.	———	x$^{\pi\pi}$	x$^{\pi\pi}$	———
Capacity, lb. C-616 abs./ft^3 of charge	12.2	14.1	13.4	2.86^a
Caking	Slight	None	Due to Segregation	
Remarks	$^{\pi}$at 300 sec, 12" from entrance	$^{\pi}$at 660 sec, 3" & 46" from entrance $^{\pi\pi}$ C-616 absorption incomplete	$^{\pi}$ at 720 sec, 3" from entrance. $^{\pi\pi}$ C-616 absorption incomplete	

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon
[a] F-605 and F-606 in series

TABLE IX

C-616 ABSORPTION BY LOW AND VERY HIGH ACTIVITY CARBON

Run No.	H-1	L-1	L-2	L-3
Carbon Used	Very High Act.	Low Act.	Low Act.	Low Act.
Form	Pellets	Pellets	Pellets	Pellets
Mesh Size	6/8	2/4	2/4	2/4
Diluent Used*	Zero Act. C	None	None	None
Form	Pellets			
Mesh Size	2/4			
Diluent Bulk Density, lb./ft^3	30	—	—	—
Dilution Ratio**	4:1	—	—	—
Bed Dimensions, in.				
Diameter	3	3	3	3
Depth	46	46	106	106
Bed Temperature, °F				
Initial	75	100	100	80
Maximum	615$^{\pi}$	460$^{\pi}$	425$^{\pi}$	515$^{\pi}$
Mole % C-616 in Feed	100	100	100	100
C-616 Mass Velocity, lb./ft^2/sec.	0.108	0.089	0.151	0.131
Linear Velocity, ft/sec.	0.119	0.098	0.166	0.144
Duration of Run, hrs.	0.30	0.14	0.17	0.25
Time before KOH Yellowed, hrs.	HF	0.02	Initial Surge	HF Initially
Capacity, lb. C-616 abs./ft^3 of charge	28	11	9.5	12
Caking	Bad	None	None	Slight
Remarks	4.5 psi plug $^{\pi}$1000 sec, 17" from entrance	1 psi plug $^{\pi}$500 sec, 7" from entrance	6 psi plug $^{\pi}$600 sec, 7" from entrance	9 psi plug $^{\pi}$900 sec, 28" from entrance

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon

TABLE X

C-616 ABSORPTION - SECTION 300 1 RECOVERY SYSTEM

Run No.	300-26	300-27	300-35	300-30
Carbon Used	High Act.	High Act.	High Act.	High Act.
Form	Pellets	Pellets	Pellets	Pellets
Mesh Size	6/8	6/8	6/8	6/8
Diluent Used*	Alumina	Alumina	Alumina	Alumina
Form	Cryst.	Cryst.	Cryst.	Cryst.
Mesh Size	6/8	6/8	4	6/8
Diluent Bulk Density, lb./ft^3	90	90	90	90
Dilution Ratio**	2:1	1.5:1	1.5:1	1:1
Bed Dimensions, in.				
Diameter	3	3	3	3
Depth	42	42	42	42
Bed Temperature, °F				
Initial	70	50	85	50
Maximum	200"	210"	200"	405"
Mole % C-616 in Feed	8.2	6.5	8.3	9.4
C-616 Mass Velocity, lb./ft^2/sec.	7.7×10^{-3}	7.8×10^{-3}	1.21×10^{-2}	1.17×10^{-2}
Linear Velocity, ft./sec.	0.109	0.150	0.160	0.145
Duration of Run, hrs.	1.78	2.0	2.0	2.0
Time before KOH Yellowed, hrs.	———	No reaction	———	———
Capacity, lb. C-616 abs./ft^3 of charge	15.3	17.5	18.0	26.3
Plugging	Due to XO_2F_2	None	None	None
Remarks	Trap nearly exhausted ~5000 sec. 27" from entrance	Trap nearly exhausted ~300 sec. 7" from entrance	50 gm. through trap ~4500 sec. 27" from entrance	Trap nearly exhausted ~5500 sec, 27" from entrance

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon

TABLE X (continued)

		Required Conditions FR-303,304
Run No.	300-31	
Carbon Used	High Act.	———
Form	Pellets	———
Mesh Size	6/8	———
Diluent Used*	Alumina	———
Form	Cryst.	———
Mesh Size	4	
Diluent Bulk Density, lb./ft^3	90	———
Dilution Ratio**	1:1	———
Bed Dimensions, in.		
Diameter	3	28
Depth	42	42
Bed Temperature, °C.		
Initial	70	140
Maximum	430π	———
Mole % C-616 in Feed	10.3	11.1
C-616 Mass Velocity, lb./ft^2/sec.	1.31×10^{-2}	1.09×10^{-2}
Linear Velocity, ft./sec.	0.146	0.122[a]
Duration of Run, hrs.	1.81	1.35
Time before NO$_2$ Yellowed, hrs.	———	———
Capacity, lb. C-616 abs./ft^3 of charge	26.5	15.3
Plugging	XO_2F_2 in exit line	
Remarks	π600 sec, 27" from entrance	

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon
[a] Calculated from temperature, C-616 mass velocity, and mole % C-616 given. This does not agree with the figure given in the corrected table Z-1257, change sheet 2A for Memorandum "Carbon Traps in Section 300," A. P. Weber, Jan.1, 1945.

TABLE XI

C-616 ABSORPTION - SECTION 300 - 2 RECOVERY SYSTEM

			Required Conditions FR-302
Run No.	300-33	300-34	
Carbon Used	High Act.	High Act.	———
Form	Pellets	Pellets	———
Mesh Size	6/8	6/8	———
Diluent Used*	Alumina	Alumina	———
Form	Cryst.	Cryst.	———
Mesh Size	4	4	———
Diluent Bulk Density, lb./ft^3	90	90	———
Dilution Ratio**	1.5:1	1.5:1	———
Bed Dimensions, in.			
Diameter	3	3	23
Depth	39	42	39
Bed Temperature, °F			
Initial	65	70	140
Maximum	245[π]	340[π]	———
Mole % C-616 in Feed	9.8	11.2	9.1
C-616 Mass Velocity, lb./ft^2/sec.	1.03×10^{-2}	1.20×10^{-2}	0.86×10^{-2}
Linear Velocity, ft/sec.	0.116	0.118	0.107[a]
Duration of Run, hrs.	1.75	1.53	1.40
Time before KOH Yellowed, hrs.	———	———	———
Capacity, lb. C-616 abs./ft^3 of charge	19.2	18.0	13.3
Plugging	None	None	None
Remarks	[π]5500 sec, 27" from entrance	[π]400 sec. 17" from entrance	

* 2" Alumina at entrance
** Expressed as volume of alumina : volume of carbon
[a]Note: (Same as Table VIII)

SECRET

TABLE XII

C-616 ABSORPTION - SECTION 300-1 RECOVERY SYSTEM

Run No.	300-11	300-16	300-19	300-25	Required Conditions FR-301
Carbon Used	High Act.	High Act.	High Act.	High Act.	———
Form	Pellets	Pellets	Pellets	Pellets	———
Mesh Size	6/8	6/8	6/8	6/8	———
Diluent Used*	Alumina	Alumina	Alumina	Alumina	———
Form	Cryst.	Cryst.	Cryst.	Cryst.	———
Mesh Size	4	4	4	6/8	———
Diluent Bulk Density, lb./ft^3	90	90	90	90	———
Dilution Ratio**	3:1	2.5:1	2:1	2:1	———
Bed Dimensions, in.					
Diameter	3	3	3	3	23
Depth	18	18	18	18	18
Bed Temperature °F					
Initial	80	70	70	55	140
Maximum	170"	190"	190"	205"	———
Mole % C-616 in Feed	9.2	9.0	8.5	8.5	9.8
C-616 Mass Velocity, lb./ft^2/sec.	3.6×10^{-3}	3.5×10^{-3}	3.3×10^{-3}	3.3×10^{-3}	3.73×10^{-3}
Linear Velocity, ft./sec.	4.5×10^{-2}	4.5×10^{-2}	4.5×10^{-2}	4.5×10^{-2}	4.64×10^{-2} [a]
Duration of Runs, hrs.	1.22	1.22	1.42	1.42	1.25
Time before KOH Yellowed, hrs.	1.00	1.22	———	No reaction	———
Efficiency, lb. C-616 abs./ft^3 of charge	11.2	11.0	12.0	12.0	11.3
Plugging	None	In line	None	None	None
Remarks	"3500 sec, 8" from entrance	"3500 sec, 9" from entrance	"4000 sec, 9" from entrance	"5100 sec 13" from entrance	

* 2" Alumina at entrance

** Expressed as volume of alumina : volume of carbon

a Same as Table X

SECRET

TABLE XIII

C-616 ABSORPTION - MOBILE CARBON TRAP CONDITIONS

Run No.	M-1	M-2	Required Conditions
Carbon Used			
Form	High Act. Pellets	High Act. Pellets	High Act. Pellets
Mesh Size	6/8	6/8	6/8
Diluent Used*			
Form	Alumina Cryst.	Alumina Cryst.	Alumina Cryst.
Mesh Size	6/8	6/8	6/8
Diluent Bulk Density, lb./ft^3	90	90	90
Dilution Ratio**	4:1	2:1	———
Bed Dimensions, in.			
Diameter	3	3	3
Depth	36	36	36
Bed Temperature, °F			
Initial	75	70	Room Temp.
Maximum	150	180	<500
Mole % C-616 in Feed	9.2	9.9	9.2
C-616 Mass Velocity, lb./ft^2/sec.	1.67×10^{-3}	1.84×10^{-3}	1.85×10^{-3}
Linear Velocity, ft/sec.	2.12×10^{-2}	2.14×10^{-2}	2.15×10^{-2}
Duration of Run, hrs.	1.31	1.31	1.31
Time before KOH Yellowed, hrs.	No reaction	No reaction	———
Capacity, lb. C-616 abs./ft^3 of charge			
Plugging	None	None	None
Remarks	Reaction in second quarter	Reaction entering second quarter	———

*Expressed as volume of diluent : volume of carbon

TABLE XIV

C-616 - B_4C REACTION RATES

Run #	B_4C Type	Mesh	Temp. °F. Start	Temp. °F. Max.	Time to reach Max, Minutes	g C-616 / g B_4C	Time Min.
12	cryst.	6/8	175	190	10	0.045	2
						0.000	5
						0.001	10
						0.023	15
						0.048	30
						0.033	60
13	cryst.	6/8	370	390	3.3	0.105	2
						0.126	5
						0.175	10
						--	15
						0.126	15
						0.234	15

TABLE XV

ANALYSES OF CdO COATED ALUMINA

Sample	% CdO	Remarks
Polarographic Harshaw Solution Method	2.38	
Polarographic Kellex Solution Method	2.54	
Harshaw Electrolytic	2.6	Reported by K. E. Long
Polarographic Kellex Solution Method 5 min. Rotapping	1.94	
Harshaw Electrolytic 5 min. Rotapping	1.91	Reported by K. E. Long
Polarographic Kellex Solution Method 3 min. Rotapping	2.06	

TABLE XVI

CHECK ON METHOD OF COMPUTATION OF MIXING RESULTS*
1:2 MIXTURE, 4 MESH Al_2O_3 : 2/4 MESH HIGH ACTIVITY CARBON

Method of Mixing	Fraction of Total Area Occupied by Components								
	Side			Top			Bottom		
	C	Al_2O_3	$C+Al_2O_3$	C	Al_2O_3	$C+Al_2O_3$	C	Al_2O_3	$C+Al_2O_3$
A	89.6±9.1	4.8±2.1	94.4±11.2	114.2±5.9	5.6±1.0	119.8±6.3	70.9±9.9	9.7±4.0	80.6±13.9
B	80.6±6.5	10.1±3.7	90.7±10.2	104.3±11.8	10.5±5.6	114.8±17.4	57.1±7.9	33.9±2.4	91.0±10.3
C	72.7±5.2	14.9±8.0	87.6±13.2	116.1±7.9	9.7±4.0	125.8±11.9	44.0±8.0	16.1±15.3	60.1±23.3

* Fraction of total area calculated by formula given in Table XVII for Al_2O_3, by analogous formula for carbon. Under $C+Al_2O_3$ is given the simple sum of the areas for C and Al_2O_3. This last should equal 100 if the method of computation is correct. Deviations given are mean deviations of the four counts made in each case. Deviation on the total is the sum of the deviations of the separate parts.

** By hand in the bowl used for counting.

TABLE XVII

1:2 MIXTURE, 4 MESH Al_2O_3 : 2/4 MESH HIGH ACTIVITY CARBON PELLETS

For perfectly homogeneous mixing the % of the total area occupied by Al_2O_3 should be 33%.

Method of Mixing	% of Total Area Occupied by Al_2O_3*		
	Side	Top	Bottom
A	4.8 ± 2.1	5.6 ± 1.0	9.7 ± 4.0
B	10.1 ± 3.7	10.5 ± 5.6	33.9 ± 2.4
C	37.1 ± 7.2	16.0 ± 2.7	
D	24.2 ± 12.1	23.9 ± 4.3	27.4 ± 5.6
E	11.3 ± 3.3	15.4 ± 3.2	44.4 ± 4.0

* % of Total Area Occupied by Al_2O_3

$$\frac{\text{\# particles } Al_2O_3 \text{ in area (when } Al_2O_3 \text{ is diluted with carbon)}}{\text{\# particles in area when area is all } Al_2O_3} \times 100$$

TABLE XVIII

1:1 MIXTURE, 4 MESH Al_2O_3 : 6/8 MESH HIGH ACTIVITY CARBON PELLETS

For perfectly homogeneous mixing the % of the total area occupied by Al_2O_3 should be 50%.

Method of Mixing	% of Total Area Occupied by Al_2O_3*		
	Side	Top	Bottom
A	4.8 ± 3.2	61.4 ± 5.5	34.9 ± 7.0
B	14.9 ± 5.9	54.8 ± 7.1	40.5 ± 11.6
C	11.7 ± 2.7	47.6 ± 7.9	21.4 ± 1.6
D	38.8 ± 2.0	50.0 ± 4.0	26.9 ± 3.2
E	9.8 ± 1.0	35.5 ± 5.8	32.7 ± 3.7

* See Table XVII

TABLE XIX

1:1 MIXTURE, 6/8 MESH Al_2O_3 : 6/8 MESH HIGH ACTIVITY CARBON PELLETS

For perfectly homogeneous mixing the % of the total area occupied by Al_2O_3 should be 50%.

Method of Mixing	% of Total Area Occupied by Al_2O_3*		
	Side	Top	Bottom
A	4.4 ± 1.4	42.1 ± 6.6	24.4 ± 2.5
B	17.2 ± 6.8	46.7 ± 1.7	21.9 ± 3.9
C	11.7 ± 2.2	40.5 ± 5.0	**
D	25.9 ± 2.5	29.8 ± 7.0	21.1 ± 4.6
E	9.8 ± 1.0	35.5 ± 5.8	32.7 ± 3.7

* See Table XVII

** No count made - bottom view obscured by charcoal dust.

TABLE XX

SEGREGATION IN A 1.5:1 MIXTURE OF 4 MESH ALUMINA AND 6/8 MESH HIGH ACTIVITY CARBON PELLETS

Ratio*, $\frac{\text{Volume } Al_2O_3}{\text{Volume C}}$	Distance of Sample from Center of Drum, inches		
Expt. No.	0" (1 tube)	3½" (4 tubes)	6 3/4" (4 tubes)
I	1.38	1.55 ± 0.08	1.75 ± 0.15
II	1.40	1.57 ± 0.13	1.80 ± 0.18
Average	1.39 ± 0.01	1.56 ± 0.01	1.78 ± 0.03
% Deviations**	-7 ± 1	4 ± 1	19 ± 2

* The ± quantity on the ratios in the body of the table is the mean deviation of results on the 4 tubes in a given ring around the center in a single experiment. In the averages it is the mean deviation of the averages for the separate experiments from the grand average.

** % Deviation = $\frac{(\text{measured ratio} - \text{original ratio})}{\text{original ratio}} \times 100$

SECRET

TABLE XXI

SEGREGATION IN A 2:1 MIXTURE OF 4 MESH ALUMINA AND 2/4 MESH HIGH ACTIVITY CARBON PELLETS

Ratio*, $\frac{\text{Volume } Al_2O_3}{\text{Volume } C}$	Distance of Sample from Center of Drum, inches		
Expt. No.	0" (1 tube)	3⅛" (4 tubes)	6 3/4" (4 tubes)
I	1.96	1.92 ± 0.16	2.90 ± 0.17
II	1.19	1.76 ± 0.49	2.48 ± 0.12
III	2.50	2.53 ± 0.37	2.57 ± 0.17
Average	1.88 ± 0.46	2.07 ± 0.31	2.65 ± 0.17
% Deviation	-6 ± 23	4 ± 16	33 ± 8

* The ± quantity on the ratios in the body of the table is the mean deviation of results on the 4 tubes in a given ring around the center in a single experiment. In the averages it is the mean deviation of the averages for the separate experiments from the grand average.

** % Deviation = $\frac{(\text{measured ratio} - \text{original ratio}) \times 100}{\text{original Ratio}}$

TABLE XXII

SEGREGATION IN A 2:1 MIXTURE OF 2 MESH ALUMINA AND 2/4 MESH HIGH ACTIVITY CARBON PELLETS

Ratio*, $\frac{\text{Volume } Al_2O_3}{\text{Volume C}}$	Distance of Sample from Center of Drum, inches		
Expt. No.	0"	3⅛"	6 3/4"
	(1 tube)	(4 tubes)	(4 tubes)
I	1.93	2.14 ± 0.56	2.63 ± 0.08
II	2.14	2.34 ± 0.16	2.35 ± 0.33
Average	2.01 ± 0.13	2.26 ± 0.08	2.49 ± 0.14
% Deviation**	1 ± 7	13 ± 4	25 ± 7

* The ± quantity on the ratios in the body in the table is the mean deviation of results on the 4 tubes in a given ring around the center in a single experiment. In the averages it is the mean deviation of the averages for the separate experiments from the grand average.

** % Deviation = $\frac{(\text{measured ratio} - \text{original ratio})}{\text{original Ratio}} \times 100$

TABLE XXIII

C-616 ABSORPTION PILOT PLANT RUNS
UNDER SECTION 600 CONDITIONS

Run No.	2	9	Required Cond., F-606
Carbon Used			
Form	High Activity Pellets	High Activity Pellets	High Activity Pellets
Mesh Size	2/4	2/4	2/4
Diluent Used	Al_2O_3	Al_2O_3	Al_2O_3
Form	cryst.	cryst.	cryst.
Mesh Size	4 + 2/4	2/4	4
Dilution Ratio	2:1	2:1	2:1
Bed Dimensions			
Diameter, in.	15-1/4	15-1/4	60
Depth, in.	31	31	51
Bed Temperature, °F			
Initial	80	80	-
Maximum	290	325	-
Mole % C-616	100	100	100
C-616 Mass Velocity, lb/ft^2/sec.	0.034	0.035	0.036
Linear Velocity, ft/sec.	0.037	0.038	0.040
Duration of Run, hrs.	0.43	0.42	0.23
Time Before Exit Gas Sample Yellowed, hrs.	0.42	-	-
Time before Plugging, hrs.	None	None	-
Estimated Capacity, lbs. C-616 abs./ft^3 of charge	20.4	20.4	3.5
Caking	Slight	Slight	None

Remarks :

#2 - Some C-616 condensed in alumina cone

#9 - No gas samples taken. Run continued until temperature in the auxiliary carbon trap rose sharply.

Notes:

1. Linear velocity calculated from gas volumes measured at 70°F and 14.7 psia.
2. In all runs, entrance of gas was through an 8" cone of pure alumina, pre-heated to 150°-200°F.
3. G-74 was blown through the bed at 7.9 cfm. for 15 minutes before starting any of the runs.
4. Dilution ratios are given as volume of alumina : volume of carbon

TABLE XXIV

C-616 ABSORPTION - PILOT PLANT RUNS UNDER SECTION 300 CONDITIONS

Run No.	1	3	4	5	7
Carbon Used	High Act.	High Act.	High Act.	High Act.	High Act.
Form	Pellets	Pellets	Pellets	Pellets	Pellets
Mesh Size	6/8	6/8	6/8	6/8	6/8
Diluent Used	Al_2O_3	Al_2O_3	Al_2O_3	Al_2O_3	Al_2O_3
Form	cryst.	cryst.	cryst.	cryst.7	cryst.
Mesh Size	4	2/4	2/4	4	2/4
Dilution Ratio	1.5:1	1.5:1	1.5:1	1.5:1	1.5:1
Bed Dimensions					
Diameter, in.	15 1/4	15 1/4	15 1/4	15 1/4	15 1/4
Depth, in.	31	31	31	31	31
Bed Temperature, °F.					
Initial	100	80	80	75	100
Maximum	>566	>330	>425	625	>205
Mole % C-616	10.07	11.12	11.03	8.01	8.5
C-616 Mass Velocity lb/ft^2/sec.	1.05×10^{-2}	1.18×10^{-2}	1.17×10^{-2}	5.96×10^{-3}	5.68×10^{-3}
Linear Velocity, ft./sec.	0.115	0.117	0.117	0.074	0.073
Duration of Run, hr.	1.30	0.86	1.00	2.87	1.5
Time before Exit Gas Sample Yellowed, hrs.	1.25	-	0.63	2.61	-
Time before Plugging, hrs.	1.06	0.75	0.58	2.28	None
Estimated Capacity, lbs. C-616 abs/ft^3 of charge	19.2	14.2	16.3	23.9	11.9
Caking	slight	bad	slight	Moderate	Bad

TABLE XXIV (continued)

Run No.	8	10	11	Req'd Cond. FR-303
Carbon Used				
Form	High Act. Pellets	High Act. Pellets	High Act. Pellets	High Act. Pellets
Mesh Size	6/8	6/8	6/8	6/8
Diluent Used				
Form	Al_2O_3 cryst.	Al_2O_3 cryst.	Al_2O_3 cryst.	Al_2O_3 cryst.
Mesh Size	2/4	2/4	2/4	4
Dilution Ratio	1.5:1	2.5:1	15:1	1.5:1
Bed Dimensions				
Diameter, in.	15 1/4	15 1/4	15 1/4	28
Depth, in.	31	31	31	42
Bed Temperature, °F.				
Initial	80	80	75	
Maximum	585	>410	800	
Mole % C-616	2.7	10.84	3.2-3.6	11.1
C-616 Mass Velocity lb/ft^2/sec.	6.44×10^{-3}	1.15×10^{-2}	5.9×10^{-3}	1.09×10^{-2}
Linear Velocity, ft./sec.	0.267	0.116	0.202	0.132
Duration of Run, hr.	2.14	0.83	3.0	1.35
Time before Exit Gas Sample Yellowed, hrs.	1.42	0.5	2.6	-
Time before Plugging, hrs.	None	0.66	None	-
Estimated Capacity, lbs. C-616 abs/ft^3 of charge	19.2	13.3	24.6	15.3
Caking	None	None	None	None

Remarks:

#1 Cake easily broken up by prodding with steel rod. 1½# plug.
#3 C-616 came through in initial purge 3½# plug. Bad cake apparently between couples 4 and 5.
#4 2½# plug.
#5 ½# plug.
#7 Worst cake of all
#10 2½# plug

Notes: 1. Linear velocity calculated from gas volumes measured at 70°F and 14.7 psia.
2. In all runs, entrance of gas was through an 8" cone of pure alumina, preheated to 150°-200°F.
3. G-74 was blown through the bed at 7.9 cfm for 15 min. before starting any of the runs.
4. Dilution ratios are given as volume of alumina: volume of carbon.

* Stated plugging probably occurred in auxiliary traps rather than in the main carbon trap

TABLE XXV

ABSORPTION OF C-616 UNDER CONTINUOUS PURGE UNIT CONDITIONS

Run No.	1	2	Required Conditions
Carbon Used			
Form	High Activity Pellets	High Activity Pellets	High Activity Pellets
Mesh Size	6/8	6/8	6/8
Diluent Used			
Form	Alumina Cryst.	Alumina Cryst.	Alumina Cryst.
Mesh Size	2/4	2/4	2/4
Diluent Bulk Density, $lb./ft^3$	90	90	90
Dilution Ratio*	3.25:1	1.75:1	3.25:1
Bed Dimensions, in.			
Diameter	1.5	1.5	1.5
Depth	11.5	14.0	11.5
Temperature at Entrance, °F.	110	110	110
Mole % C-616 in Feed (max.)	0.03	0.2	0.2
C-616 Mass Velocity, $lb/ft^2/sec.$	7.8×10^{-6}	4×10^{-5}**	3.9×10^{-5}
Linear Velocity, ft./sec.	0.033	0.033	0.026
Duration of Run, hrs.	62	186	----
Estimated Efficiency, $\frac{lb.\ C\text{-}616}{lb.\ carbon}$	----	2.2	----
Remarks	C-616 concentration high in last 6 hrs.	C-616 Concentration low in first 80 hrs. Run continued at least six hours after C-616 came through.	

* Expressed as volume of diluent : volume of carbon
** For last 102 hours. Calculated from weight gain of trap over this period.

TABLE XXVI

C-616 CONCENTRATIONS - ENTRANCE AND EXIT GASES
UNDER CONTINUOUS PURGE UNIT CONDITIONS

Run #1

Time, hrs.	C-616 Concentration, mg/liter Entrance*	Exit**	Exit / Entrance
0	0.6	---	---
0.7	---	0.08	---
2.7	---	0.12	---
4.0	---	0.06	---
14.5	2.7	0.06	0.022
25.0	---	0.05	---
40.0	4.6	0.03	0.006
62.0	4.1	---	---

* By Kellex bulb sampling method
** By spot test as developed by Dr. J. Greenspan

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TABLE XXVII

EXIT GAS ANALYSIS AS FUNCTION OF C-616 ABSORBED
SECTION 300 & 1

C-616 300-42			C-616 300-44			HF 300-46		
Estimated Wt. C-616 Added, gm.	% of* Required Absorption	X in Exit Gases lb/ft^3	Estimated Wt. C-616 Added, gm.	% of* Required Absorption	X in Exit Gases lb./ft.^{3}	Estimated Wt. C-616 Added, gm.	% of* Required Absorption	X in Exit Gases lb./ft.^{3}
6	0.5	17×10^{-5}	16	1.4	1×10^{-5}	153	13.5	0.0×10^{-3}
36	3.2	45	80	7.1	0	341	30.5	0.0
1600	143	1	1235	110	1	449	40.0	0.0
1655	148	0	1370	122	0	575	51.5	0.0
1715	153	1	1465	131	1	710	63.5	0.0
1775	158	0	1575	140	0	842	75.0	0.3
1835	164	4	1665	149	1	970	92.0	0.9
1865	167	2	1720	153	0	1078	96.0	0.9
1930	172	21	1800	160	29	1176	105.0	2.0
1990	178	110	1880	168	830			
2050	183	340	1920	172	1600			
2110	190	720						

* Required absorption is 229 lb./15 ft^3, or, in these 0.16 ft^3 reactors, 215 lb. or 1120 gm.

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TABLE XXVIII

EXIT GAS ANALYSIS AS FUNCTION OF C-616 ABSORBED
SECTION 600, TRAP F-605

Estimated Wt. C-616 Added gm.	% of Required Absorption*	HF in Exit Gas lb./sec.ft.^{2}	C-616 in Exit Gas lb./sec.ft.^{2}
532	41	0.0×10^{-5}	0.0×10^{-5}
1330	102	0.3	0.0
1861	143	0.1	0.0
2317	178	4.5	0.0(?)
2588	199	170	131

* Required absorption, F-605 = 813 lb./ft.^{3}. In this 0.35 ft.^{3} trap, a total absorption of 2.9 lb. or 1300 gm. is required.

TABLE XXIX

EXPERIMENTAL CONDITIONS - SECTION 300 & 1 RUNS FOR EXIT GAS ANALYSIS

Run No.	300-42	300-44	300-40
Carbon Used	High Act.	High Act.	High Act.
Form	Pellets	Pellets	Pellets
Mesh Size	6/8	6/8	6/8
Diluent Used*	Alumina	Alumina	Alumina
Form	Cryst.	Cryst.	Cryst.
Mesh Size	4	4	4
Dilution Ratio**	1.5:1	1.5:1	1.5:1
Bed Dimensions, in.			
Diameter	3	3	3
Depth	42	42	42
Bed Temperature, °F.			
Initial	70	80	70
Maximum	365	340	250
Mole % C-616	6.3	8.2	8.8
C-616 Mass Velocity, lb./ft.^{2}/sec.	1.03×10^{-2}	1.37×10^{-2}	0.92×10^{-2}
Linear Velocity, ft./sec.	0.180	0.184	0.115
Duration of Run, hrs.	2.92	1.98	1.89
Capacity at Saturation, lb. C-616/ft.^{3} of charge	28.7	26.2	16.7
Caking or Plugging	None	None	NaF Plugged

* 2" Alumina at entrance

** Expressed as volume of alumina: volume of carbon

TABLE XXX

EXPERIMENTAL CONDITIONS - SECTION 600 RUNS FOR EXIT GAS ANALYSIS

Run No.	XXXI
Carbon Used	High Act.
Form	Pellets
Mesh Size	2/4
Diluent Used*	Alumina
Form	Cryst.
Mesh Size	4
Diluent Bulk Density, lb./ft.^{3}	90
Dilution Ratio**	2:1
Bed Dimensions, in.	
Diameter	3
Depth	90
Bed Temperature, °F.	
Initial	80
Maximum	307
Mole % C-616 in Feed	100
C-616 Mass Velocity, lb./ft.^{2}/sec.	0.113
Linear Velocity, ft./sec.	0.125
Duration of Run, hrs.	0.34
Time Before KOH Yellowed, hrs.	0.34
Capacity, lb. C-616 abs./ft.^{3} of charge.	17.4
Plugging	None
Remarks	———

* 2" Alumina at entrance

** Expressed as volume of alumina: volume of carbon

TABLE XXXI

CARBON AND HYDROGEN ANALYSIS* ON ACTIVATED CARBON

Dried Sample**	Low (25%) Activity	High (25%) Activity
Weight % Carbon	92.5	86.2
" " Hydrogen	0.46	0.53
" " Ash	5.1	8.3
" " Water***	4.2	4.8
" " Total	101.8	99.3

* Analyses by Dr. W. F. Libby's group at SAM

** Samples both lost about 1% by weight on drying six hours at 150°C. Approximately the same weight loss was observed in a different sample after drying at 500°F in a current of dry G-74.

*** Assuming hydrogen to be present as chemisorbed water.

TABLE XXXII

EFFECT OF CARBON ACTIVITY ON ABSORPTION OF C-616

Temp. = 200° F.
Partial press. C-616 = 0.1 atm.

Carbon Used	Run #	Mass Vel. lbs/ft^2-sec	Time of Exposure hrs.	Efficiency
6/8 High Activity Pellets	A-52	1.56×10^{-3}	1	1.89
			2	2.40
			3 1/2	2.38
			4	2.49
			6 1/2	2.48
			9 1/2	2.55
			12 1/2	2.62
			24 1/2	2.71
6/8 Medium Activity Pellets	27		4 3/4	1.78
	31		4 1/2	1.76
6/8 Low Activity Pellets	A-55	1×10^{-3}	1	0.48
			2 1/2	0.74
			6	0.81
			12	0.83
2.4 Zero Activity Pellets	A-26		3	0.07*
			6	0.09
			12	0.11

* Run made with 44% C-616 instead of 10%

TABLE XXXIII

EFFECT OF CARBON PARTICLE SIZE ON ABSORPTION OF C-616

Partial pres. C-616 = 0.1 atm.

Carbon Used	Run #	Temperature	Mass vel. lbs/ft^2-sec.	Time of Exposure	Efficiency
6/8 High Activity Pellets	A-38	500°F	8.03×10^{-4}	1	1.24
				3	3.86
				6	5.97
				9	7.73
				12	8.96
				15	9.74
				18	10.66
				21	10.80
				24	10.96
2/4 High Activity Pellets	A-39	500°F	9.26×10^{-4}	3	4.75
				6	6.94
				9 1/4	10.11
				12	11.50
				15	12.24
				18	12.51
				21 1/2	12.85
				24	12.82
6/8 Low Activity Pellets	A-42	500°F	9.5×10^{-4}	1	1.52
				3 1/2	3.78
				6 1/2	4.60
				9	4.95
				12	5.38
				15 1/4	5.21
				18 1/4	5.42
				21 1/4	5.48
				24	5.83
2/4 Low Activity Pellets	A-43	500°F	9.5×10^{-4}	3	3.40
				6	5.13
				9 1/4	5.93
				12 1/4	6.33
				15 1/4	6.66
				18	6.98
				21	7.34
				24	7.45

TABLE XXXIII (continued)

Carbon Used	Run #	Temperature	Mass. vel. lbs/ft^2-sec.	Time of Exposure	Efficiency
6/8 High Activity Pellets	A-52	200° F	1.56×10^{-3}	1	1.89
				2	2.40
				3 1/2	2.38
				4	2.49
				6 1/2	2.48
				9 1/2	2.55
				12 1/2	2.62
				24 1/2	2.71
2/4 High Activity Pellets	A-53	200° F	1.02×10^{-3}	1	1.42
				3 1/2	1.99
				6 1/2	2.43
				9 1/2	2.59
				12 1/2	2.65
				24 1/2	2.81
6/8 Low Activity Pellets	A-55	200° F	1×10^{-3}	1	0.48
				2 1/2	0.74
				6	0.81
				12	0.83
2/4 Low Activity Pellets	A-54	200° F		1	0.38
				4 1/2	0.65
				7 1/2	0.82
				9 1/2	0.82
				22	0.88

TABLE XXXIV

COMPARISON OF C-616 AND CCl_4 ABSORPTION ON ACTIVATED CARBON SHOWING EFFECT OF TYPE OF CARBON ON THE ABSORPTION OF C-616

Partial press. C-616 = 0.1 atm.
Exposure = 3 hrs.

Carbon Used	Temperature	Efficiency*(C-616) Pellets	Efficiency*(C-616) Crushed	Activity*(CCl_4) Pellets	Activity*(CCl_4) Crushed
6/8 High Activity	75°F	1.62	.99		
		1.64	1.14		
6/8 High Activity	200°F	2.50	1.55	0.526	0.490
		2.41	1.49	0.530	0.567
		2.55		0.523	0.496
		2.48			
6/8 Medium Activity	200°F	1.78	0.425	0.394	0.358
		1.76	0.581	0.399	0.341
			0.634		

* Both efficiency and activity are here expressed as: $\frac{\text{weight of adsorbate}}{\text{weight of adsorbent}}$

The activity is used here to refer to CCl_4 absorption in general rather than the absorption under the conditions specified by the National Carbon Company.

TABLE XXXV

EFFECT OF DRYING CARBON ON F-616 ABSORPTION

Sample	g X^{+4}/g C	g X^{+6}/g C	X^{+4}/X^{+6}	g Total X/g C
6/8 High Activity Pellets (dried)	0.97	1.58	0.61	2.55
6/8 High Activity Pellets (undried)	1.15	1.34	0.86	2.49

TABLE XXXVI

EFFECT OF CARBON TEMPERATURE ON ABSORPTION OF C-616

Partial pressure C-616 = 0.1 atm.

A. 6/8 High Activity Carbon

Period of Exposure	Temperature	Efficiency
1 hour	200°F	1.89
	300°F	2.66
	400	1.60
	500	1.24
3	200	2.40
	300	3.50
	400	2.90
	500	3.86
6	75	1.63
	200	2.48
	300	3.73
	400	3.95
	500	6.10
9	200	2.51
	300	3.95
	400	4.55
	500	7.72
12	200	2.56
	300	4.12
	400	5.18
	500	8.98
15	200	2.61
	300	4.29
	400	5.81
	500	9.91
18	200	2.67
	300	4.47
	400	6.40
	500	10.53
21	200	2.68
	300	4.59
	400	7.00
	500	10.87

TABLEX XXVI (cont)

A. 6/8 High Activity Carbon

Period of Exposure	Temperature	Efficiency
24	200	2.70
	300	4.77
	400	7.61
	500	10.99

B 2/4 Low Activity Carbon

1	200	0.33
	300	0.68
	400	0.76
	500	1.52
3	200	0.67
	300	1.24
	400	1.63
	500	3.37
6	200	0.81
	300	1.63
	400	2.39
	500	4.97
9	200	0.81
	300	1.88
	400	2.83
	500	5.88
24	200	0.90
	300	2.29
	400	3.30
	500	7.31

C. 2/4 Zero Activity Carbon

6	200	0.09
	250	0.14
	350	0.21
	500	0.72

TABLE XXXVII

EFFECT OF C-616 PARTIAL PRESSURE ON ABSORPTION EFFICIENCY OF CARBON

Temp. = 200°F
Time of exposure = 1/2 hour
Carbon used = 6/8 high activity

Run #	Partial Pressure C-616	Efficiency	
E-1	1 atm.	2.99 2.92	2.96
E-13		3.37 3.23	3.30
E-17		3.44	
E-8	0.32	3.04 2.74	2.89
E-4	0.13	1.40 (.3 hrs.)	
E-2	0.12	2.55 2.10	2.33
E-5	0.08	1.79 2.01	2.90
E-6	0.05	2.32 2.04	2.18(1½ hrs.)
E-7	0.009	1.74 2.04	1.89 (5 hrs.)
E-3	0.002	1.45 1.67	1.56

TABLE XXXVIII

RATE OF C-616 ABSORPTION BY ACTIVATED CARBON

Carbon Used (Pellets)	Run #	Temperature	Time of Exposure Minutes	Efficiency
2/4 High Activity	1	200°F	4	0.83
			12	2.07
			13.7	1.81
			17	2.30
2/4 High Activity	2	200	1	0.27
			3	1.00
			5	1.19
			10	1.84
			15	1.79
			30	2.10
2/4 High Activity	3	400	1	0.62
			3	2.28
			5	3.48
			10	6.01
			15	6.15
			30	7.13
2/4 High Activity	4	400	1	1.35
			3	2.25
			5	3.75
			10	3.88
			15	5.12
			30	5.62
6/8 Low Activity	6	200	1	0.32
			3	0.45
			5	0.39
			10	0.41
			10	0.41
6/8 Low Activity	10	400	1	0.91
			5	1.89
			15	2.03
			30	1.61
			60	2.05
2/4 Zero Activity	5	200	1	0.07
			3	0.11
			10	0.09
			15	0.10
			30	0.13
6/8 Medium Activity	9	200	1	0.98
			5	0.93
			15	0.85
6/8 High Activity	7	200	2	2.75
			10	3.12
			20	3.07

TABLE XXXIX

EFFECT OF TIME ON C-616 ABSORPTION BY ACTIVATED CARBON

Partial pressure C-616 = 0.1 atm.
Carbon used = 6/8 Mesh High Activity Carbon Pellets

Temperature	Run #	Mass velocity lbs/ft^2-sec.	Time of Exposure Hours	Efficiency
200°F	A-52	1.6×10^{-3}	3½	2.38
			6½	2.48
			9½	2.55
			12½	2.62
			24½	2.71
300	A-50	1.2×10^{-3}	3	3.34
			6	3.73
			9	4.01
			12	4.20
			15½	4.42
			18	4.53
			21	4.66
			24	4.78
400	A-44	8.1×10^{-4}	3 1/4	2.95
			6	3.90
			9	4.68
			12	5.25
			15	6.00
			18	6.45
			21	7.16
			24	7.44
500	A-38	8.0×10^{-4}	1	1.24
			3	3.86
			6	5.97
			9	7.73
			12	8.96
			15	9.74
			18	10.66
			21	10.80
			24	10.96

TABLE XL

DISTILLATION OF 100 CC. OF FLUOROCARBONS FROM THE C-616, CARBON REACTION

Boiling Range °F	Probable Compounds	Volume of Gas, Liters	Mole % of Gas Collected
-200° to -150°	CF_4	32	82
-150° to -75°	C_2F_x	5.5	14
-30° to +30°	C_3F_x	1.5	4
Residue (liq.)	C_4-C_n	2.5 ml (liq.)	-

TABLE XLI

PRODUCT OF HEAVY FLUOROCARBONS IN THE C-616, CARBON REACTION

Run No.	Average Temp. °C.	Heavy Fluorocarbon Extracted mg/gm. of sample	X^{+4} mg/gm of sample
32	300	0.25	54
35	320	0.48	130
37	480	3.0	136
34	705	5.8	179
36	850	10.8	210

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TABLE XLII

X ANALYSES ON CARBON AFTER C-616 EXPOSURE

Run No.	Temp. °F.	Time of C-616 Exposure, hrs.	Wt. X^{+4}/Wt. X	Av.	Wt. X^{+6}/Wt. C	Av	Average Time, hrs.
6/8 Mesh High Activity Carbon Pellets							
52	200	24	.38		1.13		
58		24*	.42		1.31		
59		15*	.26	.35	1.39	1.28	20
60		1	.41		.76		
61		1*	.25		2.05		
68		1*	.12	.26	.98	.82	1
50	300	24	.42		.89		
78		21	.21	.31	1.77	1.83	24
70		1	.32		1.23		
73		1	.27	.30	.97	1.07	1
44	400	24	.44		2.83		
83		23	.49	.47	2.09	2.46	24
38	500	24	.77		1.74		
88		21	.76	.77	1.05	1.40	24
2/4 Mesh High Activity Carbon Pellets							
53	200	24	.42		1.10		
57		24*	.35	.39	1.20	1.15	24
62		1*	.043		.82		
65		1	.18	.11	.78	.80	1
80	300	58	.28	.28	3.44	3.44	58
51		24	.37		1.92		
79		19	.15	.27	1.12	1.52	22
69		1	.15		.77		
75		1	.21	.18	.50	.64	1

SECRET

TABLE XLII (continued)

Run No.	Temp. °F.	Time of C-616 Exposure, hrs.	$Wt.X^{+4}/Wt.X$	Av.	$Wt.X^{+6}/Wt.C$	Av.	Average Time, hrs.
6/8 Mesh Low Activity Carbon Pellets							
55	200	12	.42	.42	.33	.33	12
66		1*	.35		.21		
67		1	.12	.24	.98(?)	.21	1
48	300	24	.51		.48		
81		15	.53	.52	.36	.42	20
72		1	.51	.51	.26	.26	1
46	400	21	.42		1.56		
86		22	.53	.48	1.10	1.33	22
42	500	24	.63		1.44		
89		24	.74	.69	.84	1.14	24
2/4 Mesh Low Activity Carbon Pellets							
54	200	22	.41	.41	.35	.35	22
63		1	.22		.20		
64		1*	.25	.24	.21	.21	1
49	300	24	.54	.54	.68	.68	24
71		1	.48		.20		
74		1	.42	.45	.21	.21	1
47	400	24	.49		1.12		
85		20	.54	.52	.87	1.00	22
43	500	24	.68		1.63		
70		24	.73	.71	1.02	1.33	24

* 2% C-616 was used in these experiments instead of 10%. Since no definite effect was noted these data are averaged in with the rest.

TABLE XLIII

X ANALYSES ON CARBON - SPECIAL TEST

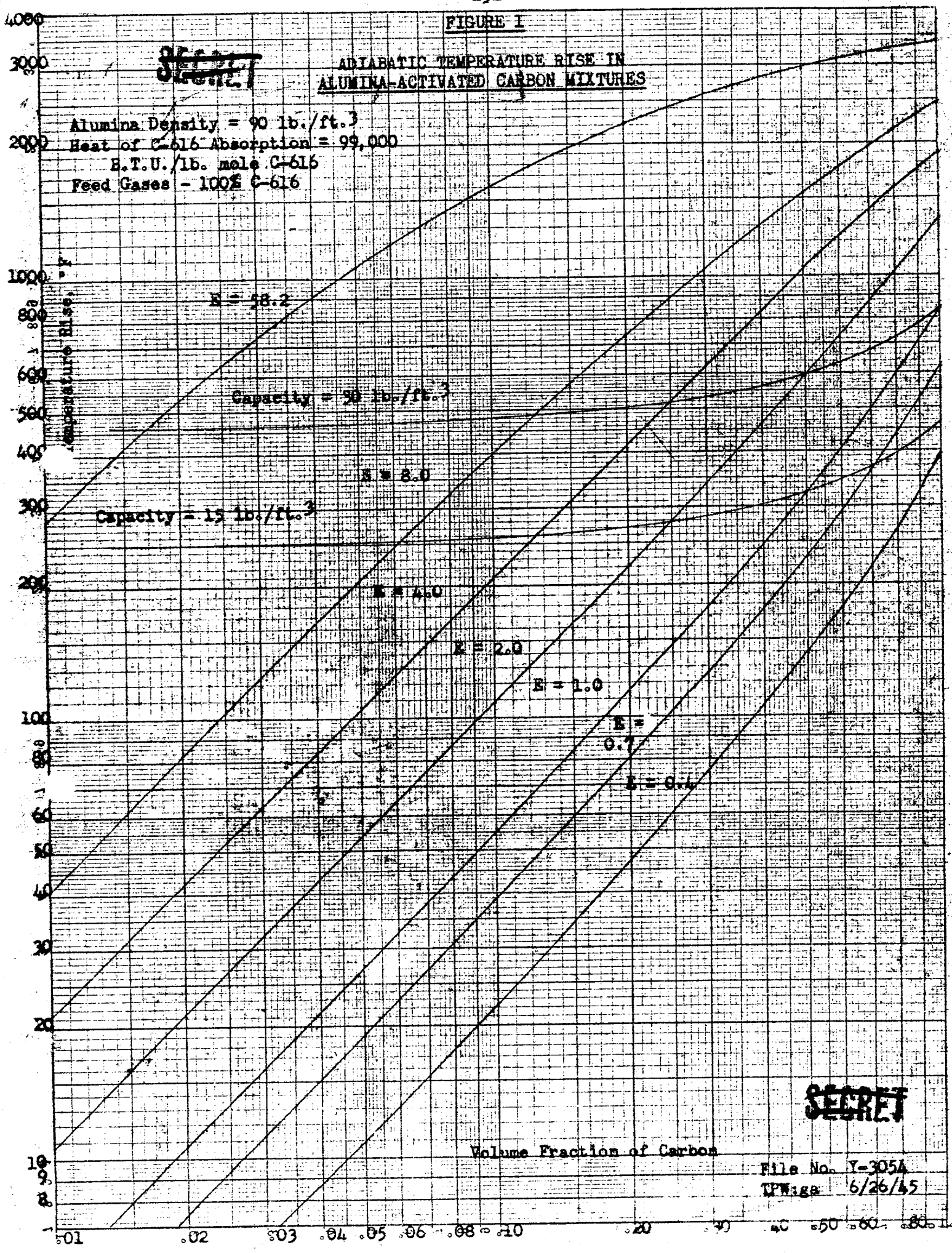
Carbon	Sample A 6/8 Mesh High Activity	Sample B* 6/8 Mesh High Activity	
C-616 Exposure			
C-616 Partial Pressure, atm. Temper	0.10	0.10	0 (static)
Temperature °F	200	200	650
Time, hrs.	2	2	4
Wt. X^{+4}/Wt.X	0.22	0.39	
Wt. X^{+6}/Wt. Carbon	1.33	1.18	

* Sample B was first exposed to C-616 under conditions identical with those used for Sample A. It was then sealed up under dry G-74 and heated to 650°F as indicated.

FIGURE I

SECRET

ADIABATIC TEMPERATURE RISE IN
ALUMINA-ACTIVATED CARBON MIXTURES



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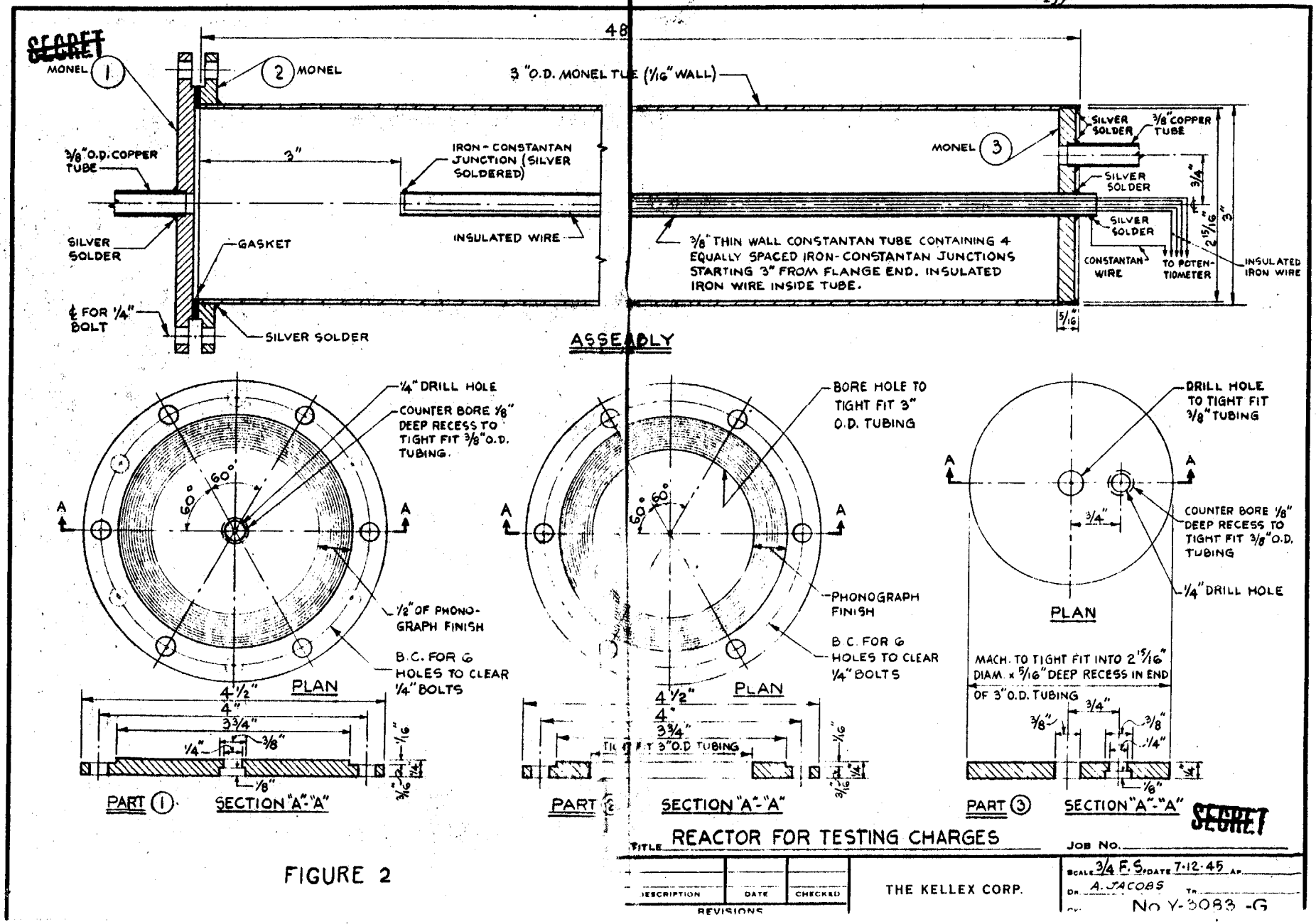

SECRET
48
MONEL (1)
(2) MONEL
3" O.D. MONEL TUBE (1/16" WALL)
3/8" O.D. COPPER TUBE
3"
IRON-CONSTANTAN JUNCTION (SILVER SOLDERED)
SILVER SOLDER
GASKET
INSULATED WIRE
℄ FOR 1/4" BOLT
SILVER SOLDER
MONEL (3)
SILVER SOLDER
3/8" COPPER TUBE
SILVER SOLDER
SILVER SOLDER
3/8" THIN WALL CONSTANTAN TUBE CONTAINING 4 EQUALLY SPACED IRON-CONSTANTAN JUNCTIONS STARTING 3" FROM FLANGE END. INSULATED IRON WIRE INSIDE TUBE.
CONSTANTAN WIRE
TO POTENTIOMETER
INSULATED IRON WIRE
3/4"
2 15/16"
3"
5/16"
ASSEMBLY
1/4" DRILL HOLE
COUNTER BORE 1/8" DEEP RECESS TO TIGHT FIT 3/8" O.D. TUBING.
60°
A
1/2" OF PHONOGRAPH FINISH
B.C. FOR 6 HOLES TO CLEAR 1/4" BOLTS
PLAN
4 1/2"
4"
3 3/4"
1/4"
3/8"
1/8"
1/16"
3/16"
1/4"
PART (1)
SECTION "A"-"A"
BORE HOLE TO TIGHT FIT 3" O.D. TUBING
PHONOGRAPH FINISH
B.C. FOR 6 HOLES TO CLEAR 1/4" BOLTS
TIGHT FIT 3" O.D. TUBING
PART (2)
DRILL HOLE TO TIGHT FIT 3/8" TUBING
COUNTER BORE 1/8" DEEP RECESS TO TIGHT FIT 3/8" O.D. TUBING
1/4" DRILL HOLE
MACH. TO TIGHT FIT INTO 2 15/16" DIAM. x 5/16" DEEP RECESS IN END OF 3" O.D. TUBING
3/8"
PART (3)
SECRET
TITLE REACTOR FOR TESTING CHARGES
JOB NO.
DESCRIPTION
DATE
CHECKED
REVISIONS
THE KELLEX CORP.
SCALE 3/4 F.S. DATE 7-12-45
DR. A. JACOBS
No Y-3083-G

FIGURE 2

SECRET

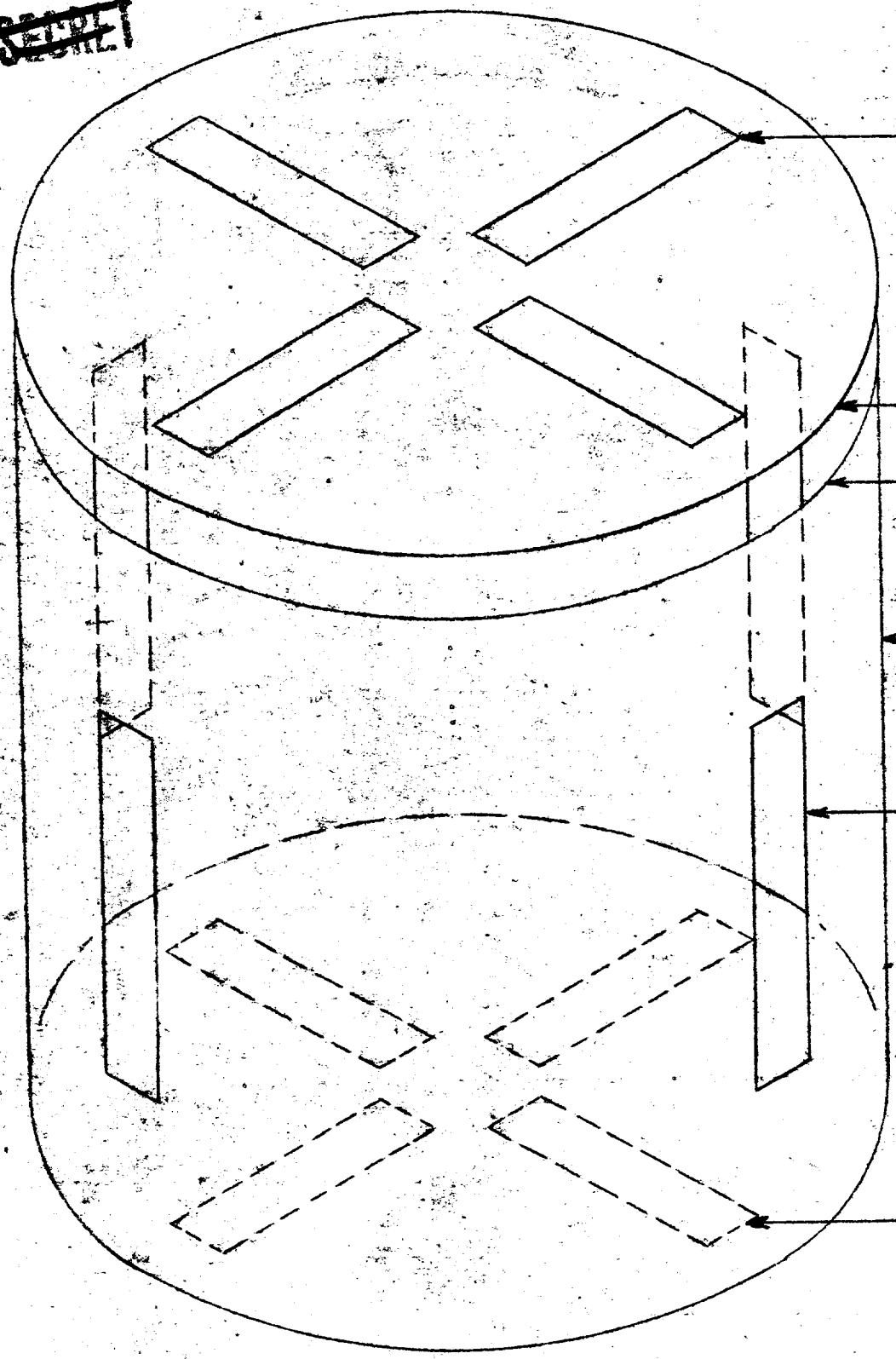


FIGURE 3

SECRET

TITLE GLASS JAR FOR PARTICLE COUNT JOB NO.

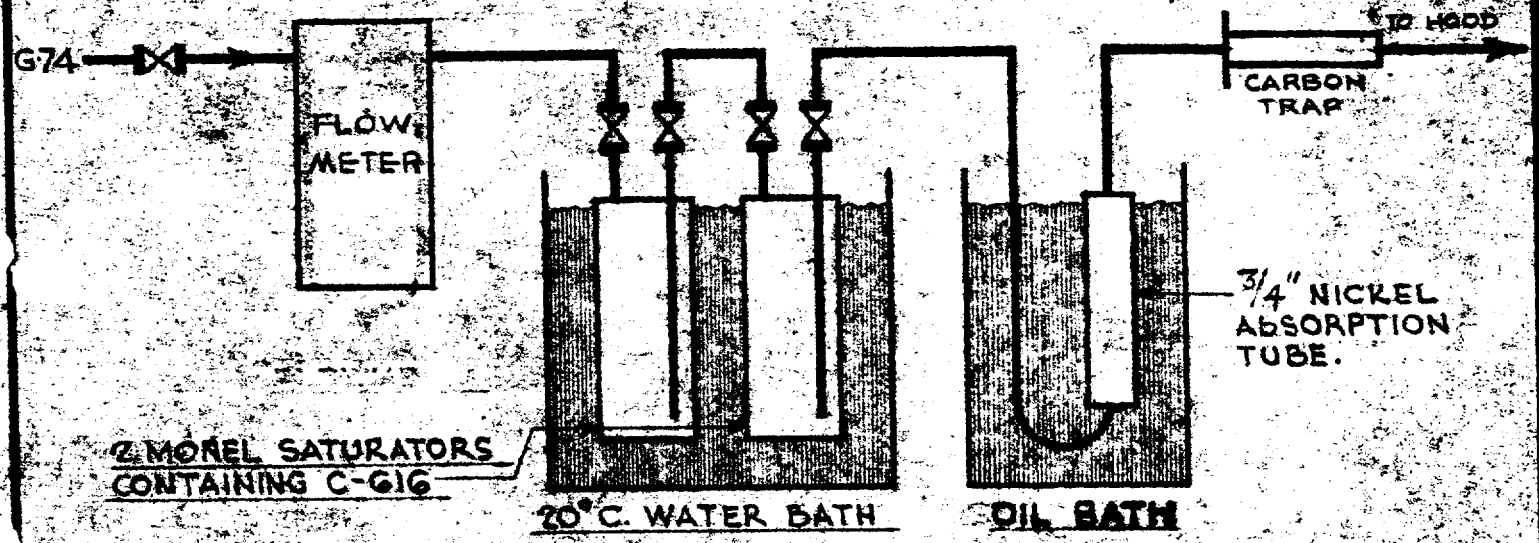
DESCRIPTION	DATE	CHECKED
REVISIONS		

THE KELLEX CORP.

SCALE DATE AP.
DR. A. JACOBS TR.
CKD No. Y-1747 -D

K & E CO., N.Y.

Figure 4



NOTES

⋈ - KEROTEST VALVES

ALL TUBING TO BE 3/8" COPPER

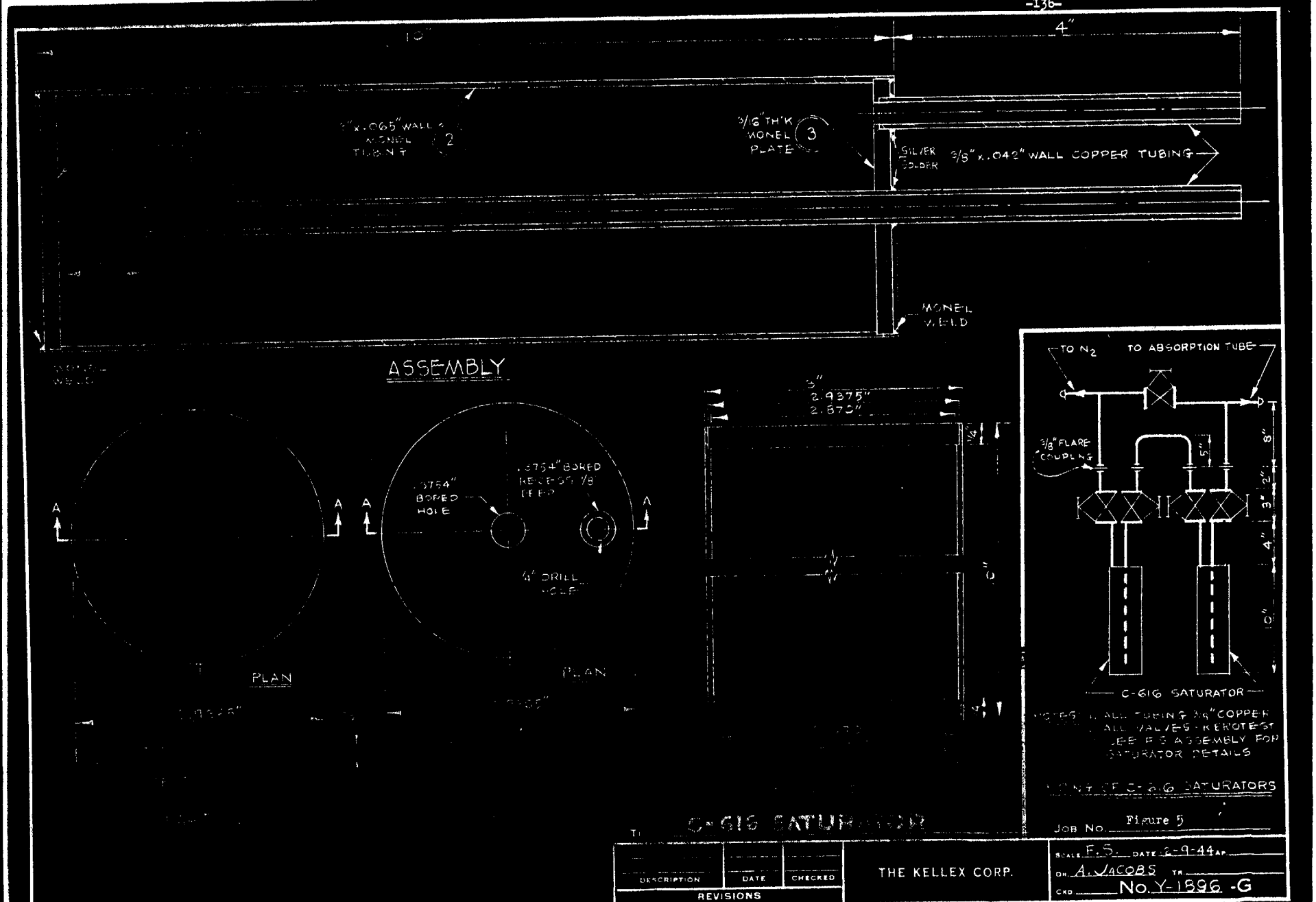
APPARATUS FOR DETERMINING EFFICIENCY OF CARBON AS ABSORBENT FOR C-616

TITLE

JOB NO.

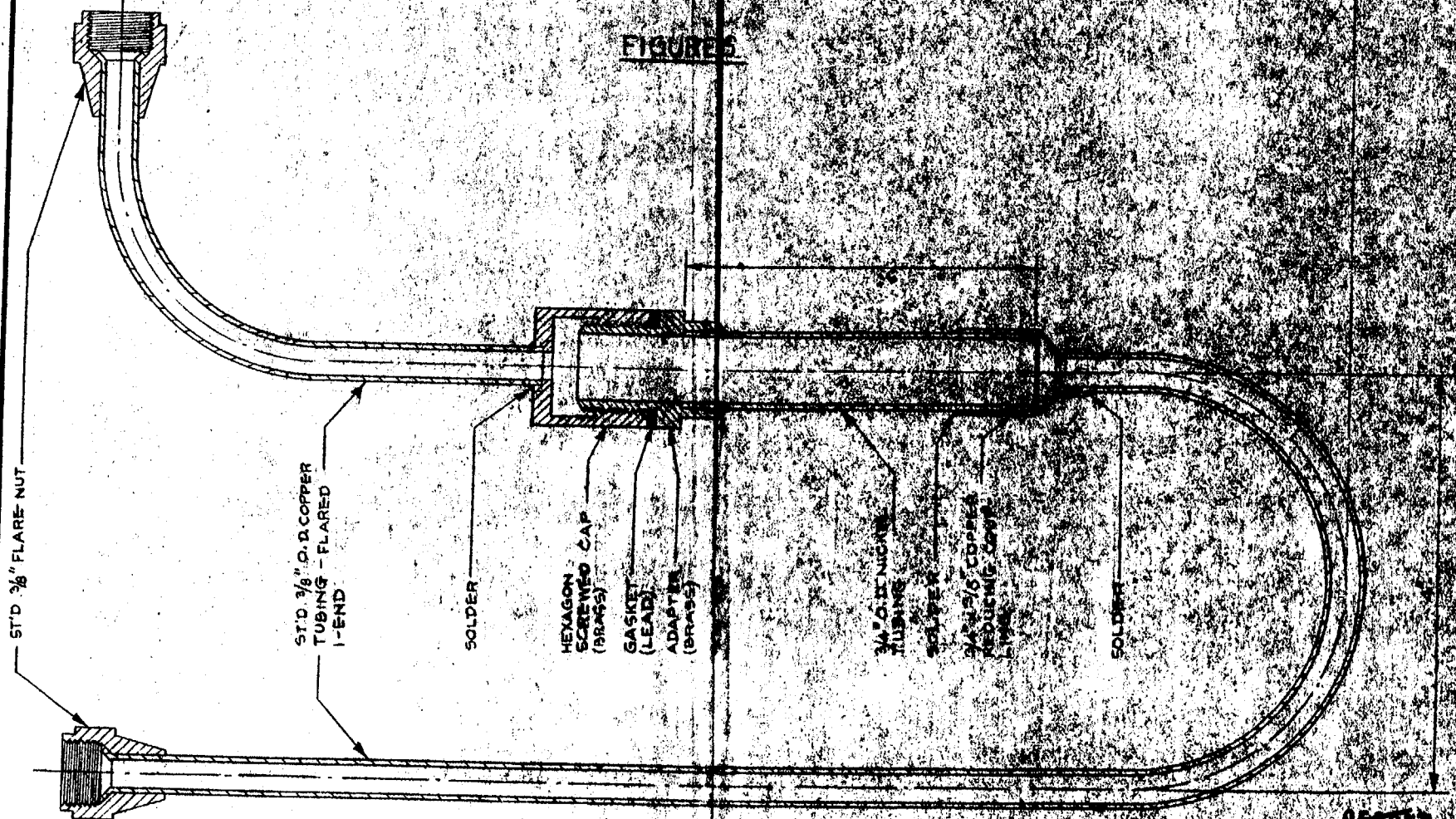
WATER BATH TO OIL BATH	7/12/45	T.P.W.	THE KELLEX CORP.	SCALE NONE DATE 12-2-44 AP.
DESCRIPTION	DATE	CHECKED		DR. S. BANKS TR.
REVISIONS				CKD. No.-Y-1878-D

X & E CO., N.Y.


10"
4"
3/16" TH'K MONEL PLATE 3
SILVER SOLDER
3/8" x .042" WALL COPPER TUBING
MONEL WELD
ASSEMBLY
.3754" BORED HOLE
.3754" BORED RECESS 1/8 DEEP
4" DRILL HOLE
PLAN
PLAN
3"
2.9375"
2.870"
TO N_2
TO ABSORPTION TUBE
3/8" FLARE COUPLING
C-616 SATURATOR
C-616 SATURATOR
JOB NO. Figure 5
DESCRIPTION
DATE
CHECKED
REVISIONS
THE KELLEX CORP.
SCALE F.S.
DATE 2-9-44
DR. A. JACOBS
CKD
No. Y-1896 -G

SECRET

FIGURE 5



SECRET

TITLE 3/4" O.D. CARBON ABSORPTION TUBE FOR C-61G

JOB NO.

DESCRIPTION	DATE	CHECKED
REVISIONS		

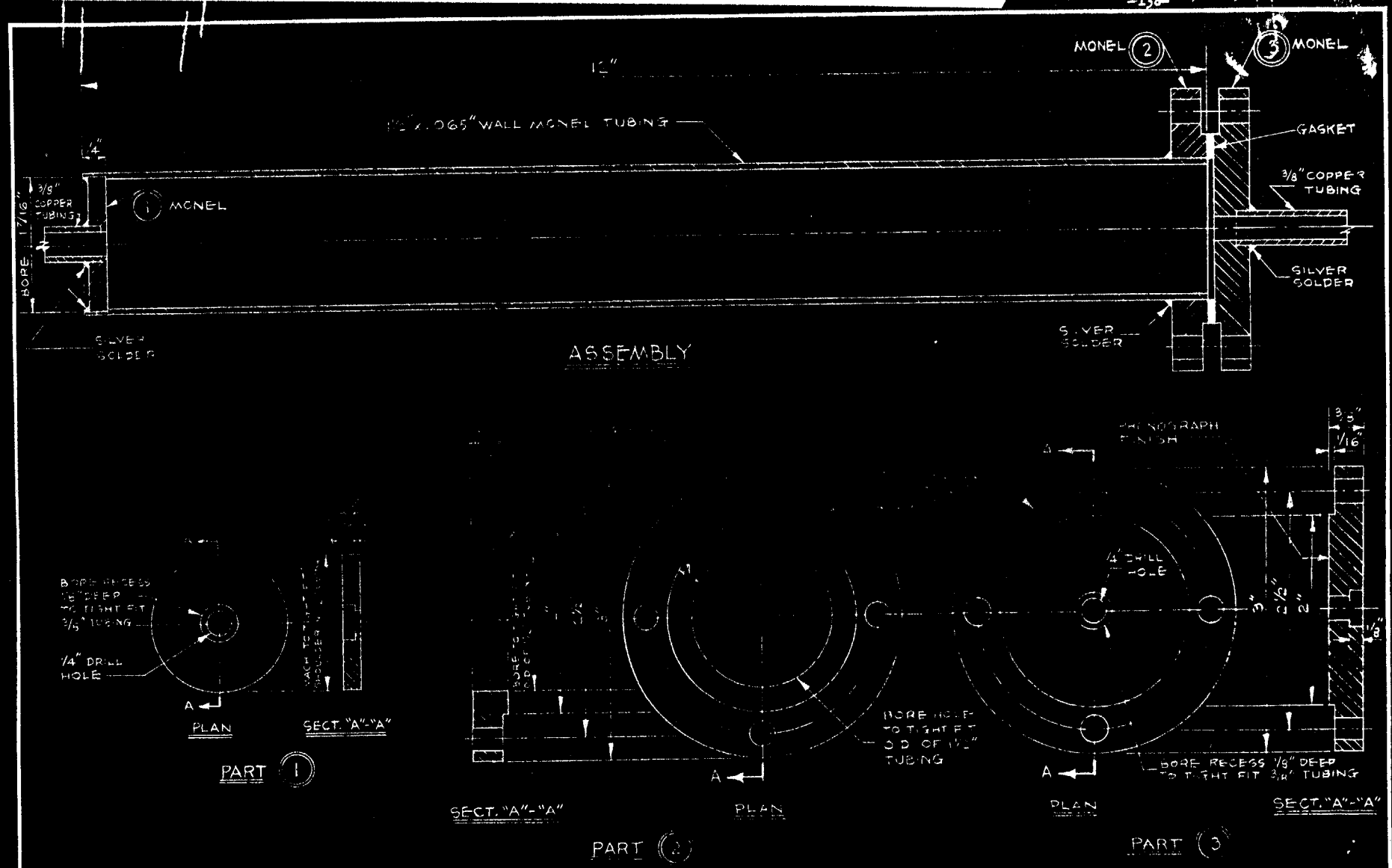
THE KELLEX CORP.

SCALE F.S. DATE 1-3-44

DR. A. JACOBS TR.

ORD. No. Y-1929 -G

K & E CO., N.Y.



TITLE DETAIL OF EXIT CARBON TRAP

Figure 7

JOB NO.

DESCRIPTION	DATE	CHECKED
REVISIONS		

THE KELLEX CORP.

SCALE F.S. DATE 6-12-44 AP.

DR. A. JACOBS TR.

CKD. No. Y-1897 -G

FIGURE [redacted]

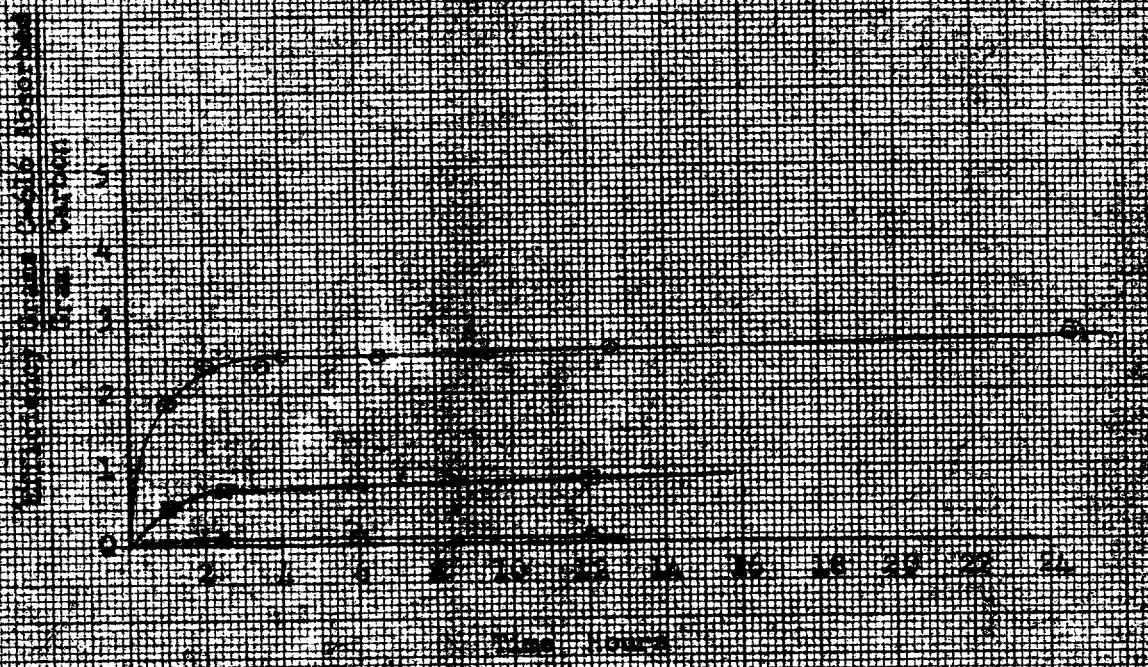
EFFECT OF CARBON ACTIVITY
ON
ABSORPTION OF Cs^{136}

SECRET

6/8 Mesh High Activity Carbon

6/8 Mesh Low Activity Carbon

2/4 Mesh Zero Activity Carbon



[redacted]

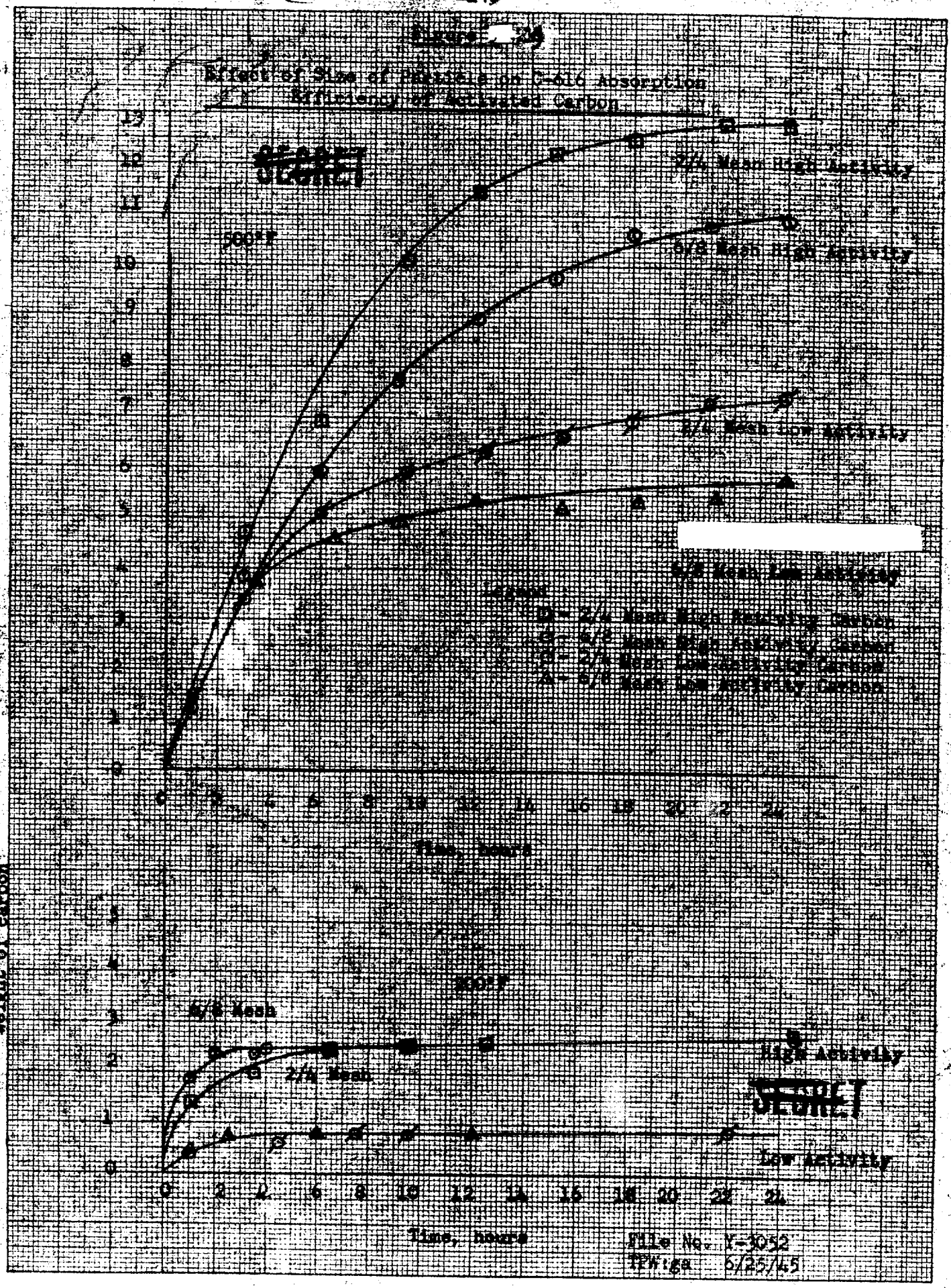
7/12/45

SECRET

KEUFFEL & ESSER CO., N. Y. NO. 359-14
Millimeters, 5 mm. lines accent 1, lines heavy.
MADE IN U.S.A.

Figure 24

Effect of Size of Particle on C-616 Absorption Efficiency of Activated Carbon



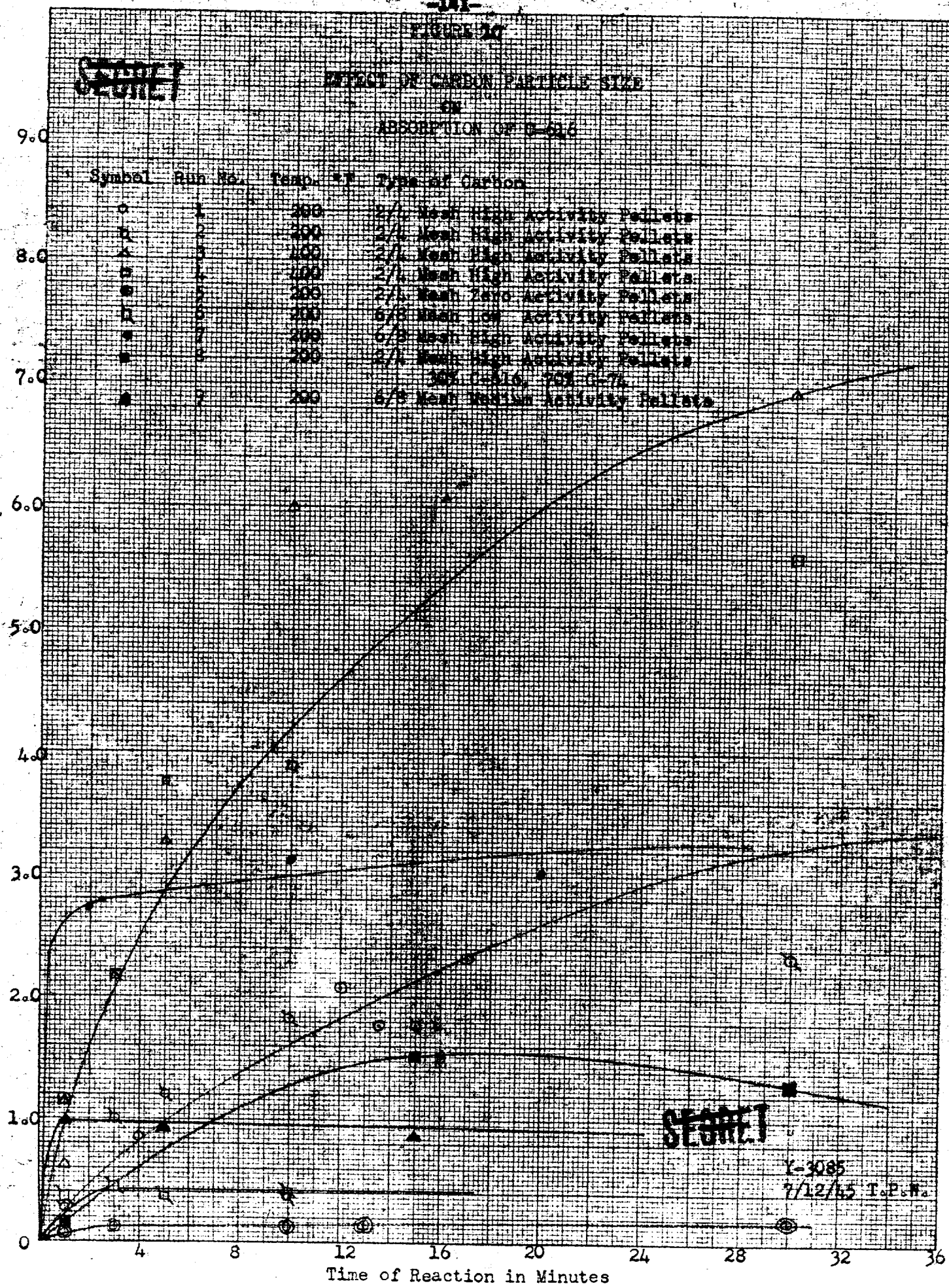
File No. Y-3052
TPW:ga 6/25/45

FIGURE 20

SECRET

EFFECT OF CARBON PARTICLE SIZE
ON
ABSORPTION OF C-616

Symbol	Run No.	Temp. °F	Type of Carbon
○	1	200	2/4 Mesh High Activity Pellets
◐	2	200	2/4 Mesh High Activity Pellets
△	3	400	2/4 Mesh High Activity Pellets
◑	4	400	2/4 Mesh High Activity Pellets
◐	5	200	2/4 Mesh Zero Activity Pellets
◐	6	200	6/8 Mesh Low Activity Pellets
●	7	200	6/8 Mesh High Activity Pellets
■	8	200	2/4 Mesh High Activity Pellets 30% C-616, 70% C-74
▲	7	200	6/8 Mesh Medium Activity Pellets



SECRET

Y-3085
7/12/45 T.P.W.

KEUFFEL & ESSER CO., N. Y. NO. 359-14
Millimeters, 5 mm. lines accented, 1mm heavy.
MADE IN U.S.A.

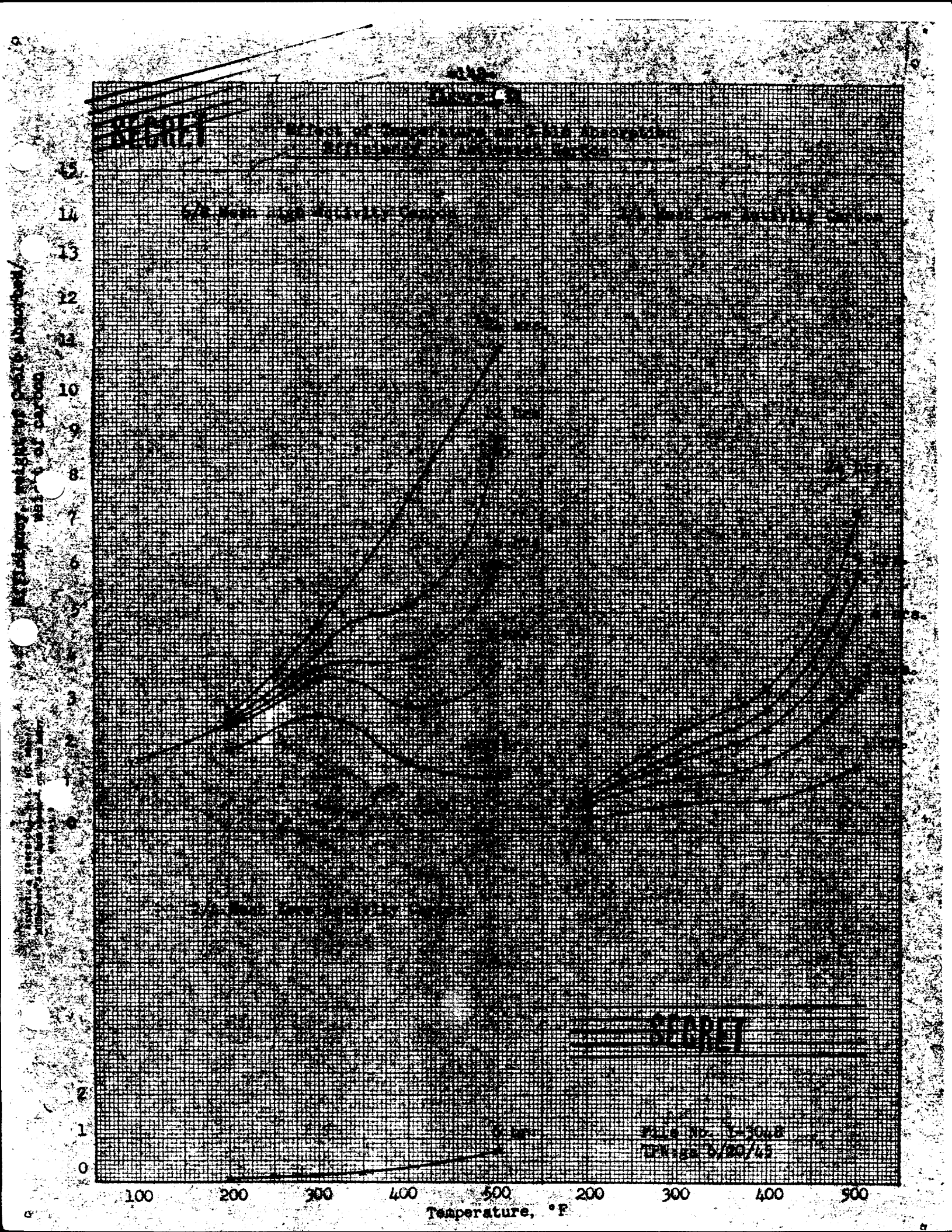
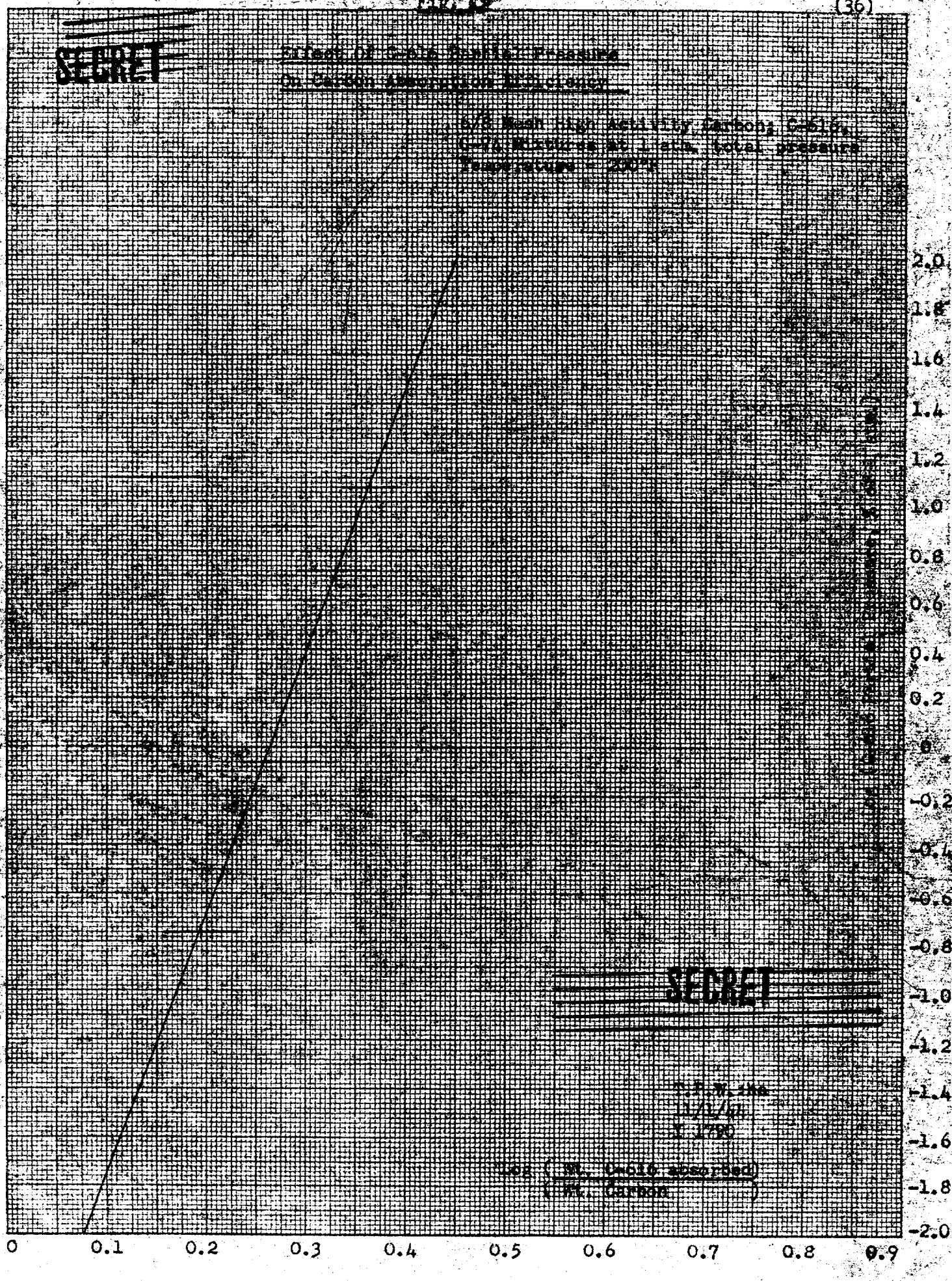

SECRET
Effect of Temperature on C-616 Absorption
Efficiency of Activated Carbon
6/8 Mesh High Activity Carbon
2/4 Mesh Low Activity Carbon
2/4 Mesh Low Activity Carbon
Efficiency, weight of C-616 absorbed/weight of carbon
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
2
1
0
100
200
300
400
500
200
300
400
500
Temperature, °F
SECRET
File No. 1-3048
TPW:ga 6/20/45

Fig. 12

(36)


SECRET
Effect of C-616 Partial Pressure
On Carbon Absorption Efficiency
6/8 Mesh High Activity Carbon; C-616,
C-74 Mixtures at 1 atm. total pressure
Temperature = 200°F
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
-1.4
-1.6
-1.8
-2.0
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
SECRET
T.P.W.:ha
11/1/44
Y 1790
Log (Wt. C-616 absorbed / Wt. Carbon)

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

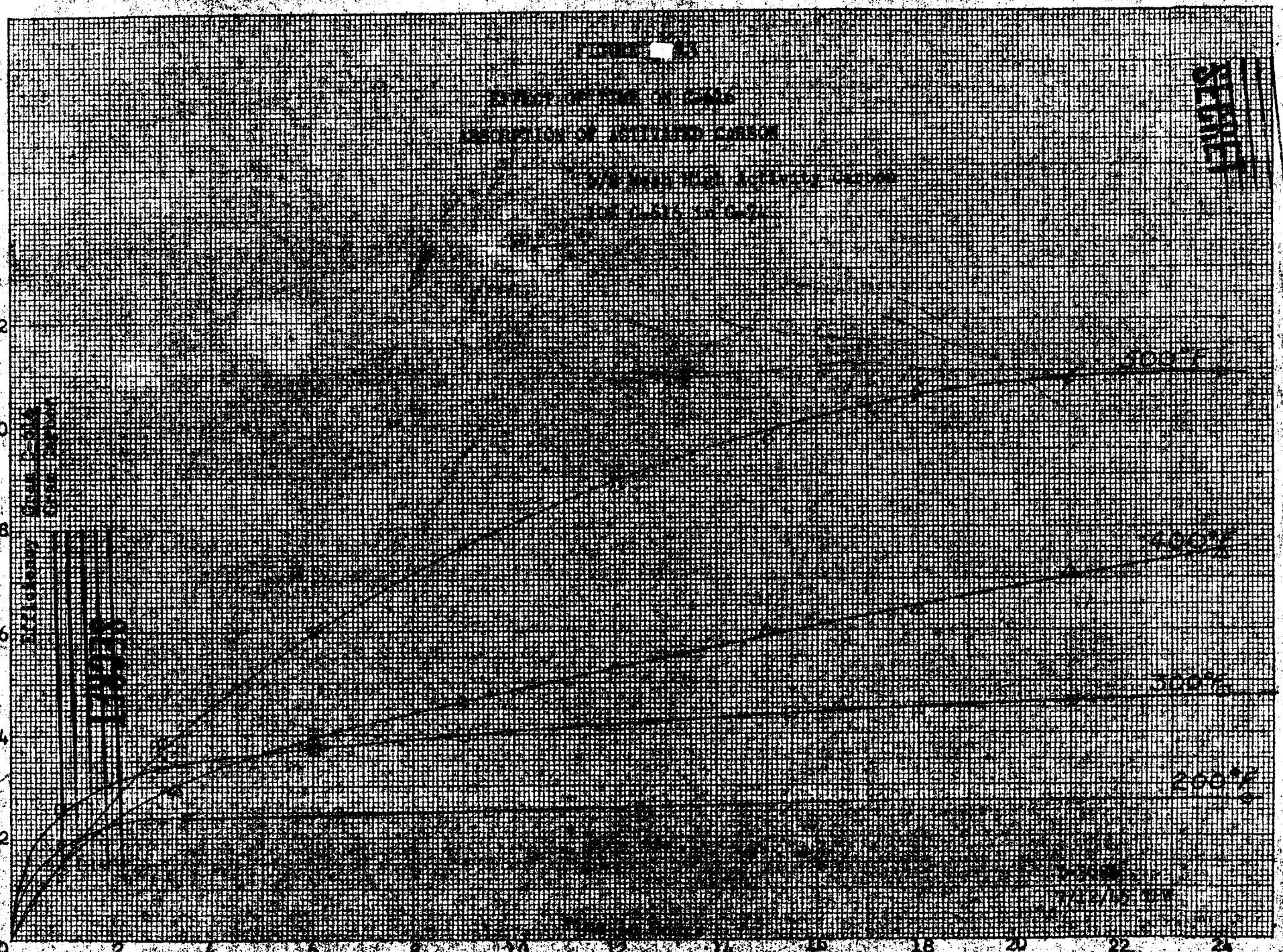
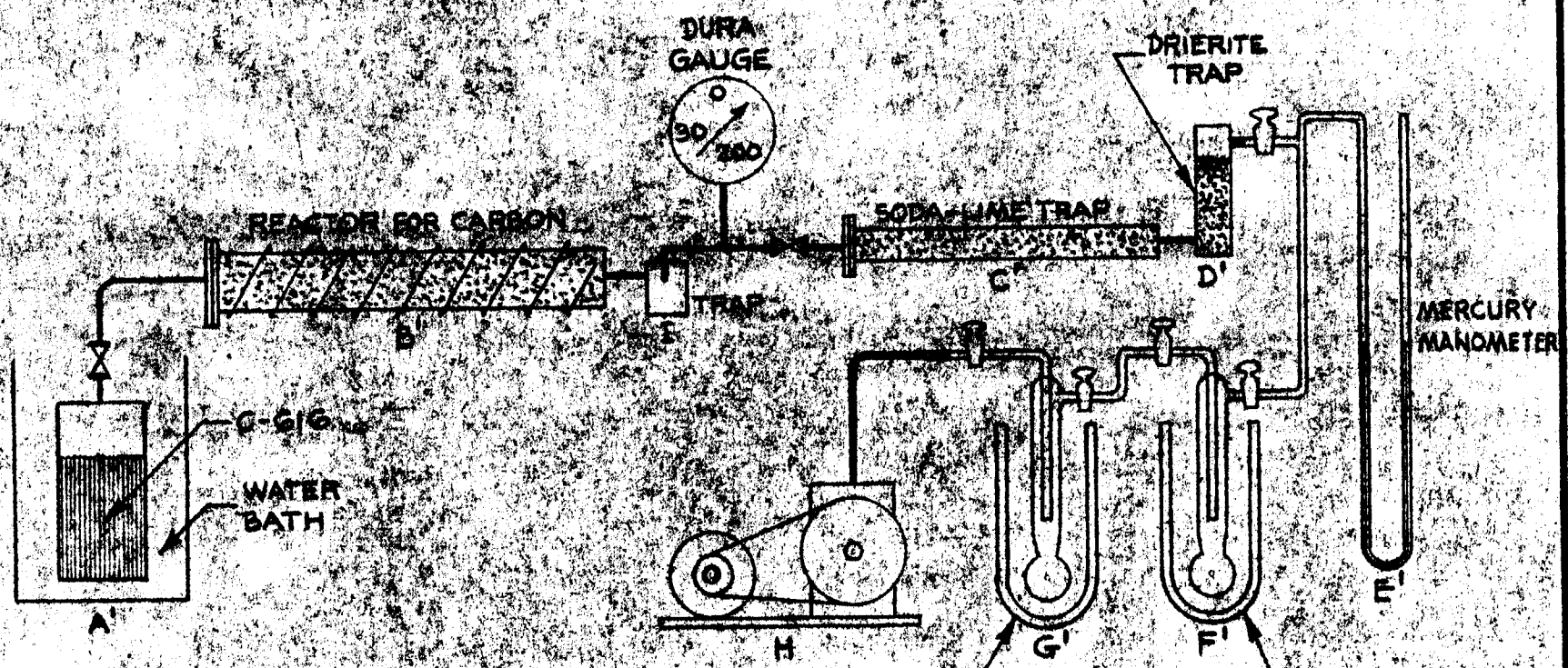


FIGURE 14

SECRET



SECRET

TITLE APPARATUS FOR FLUOROCARBON PRODUCTION

JOB NO.

REVISIONS		
DESCRIPTION	DATE	CHECKED

THE KELLEX CORP.

SCALE — DATE 2-2-45 AP.

DR. A. JACOBS TR.

CKD. NO. Y-1975-D

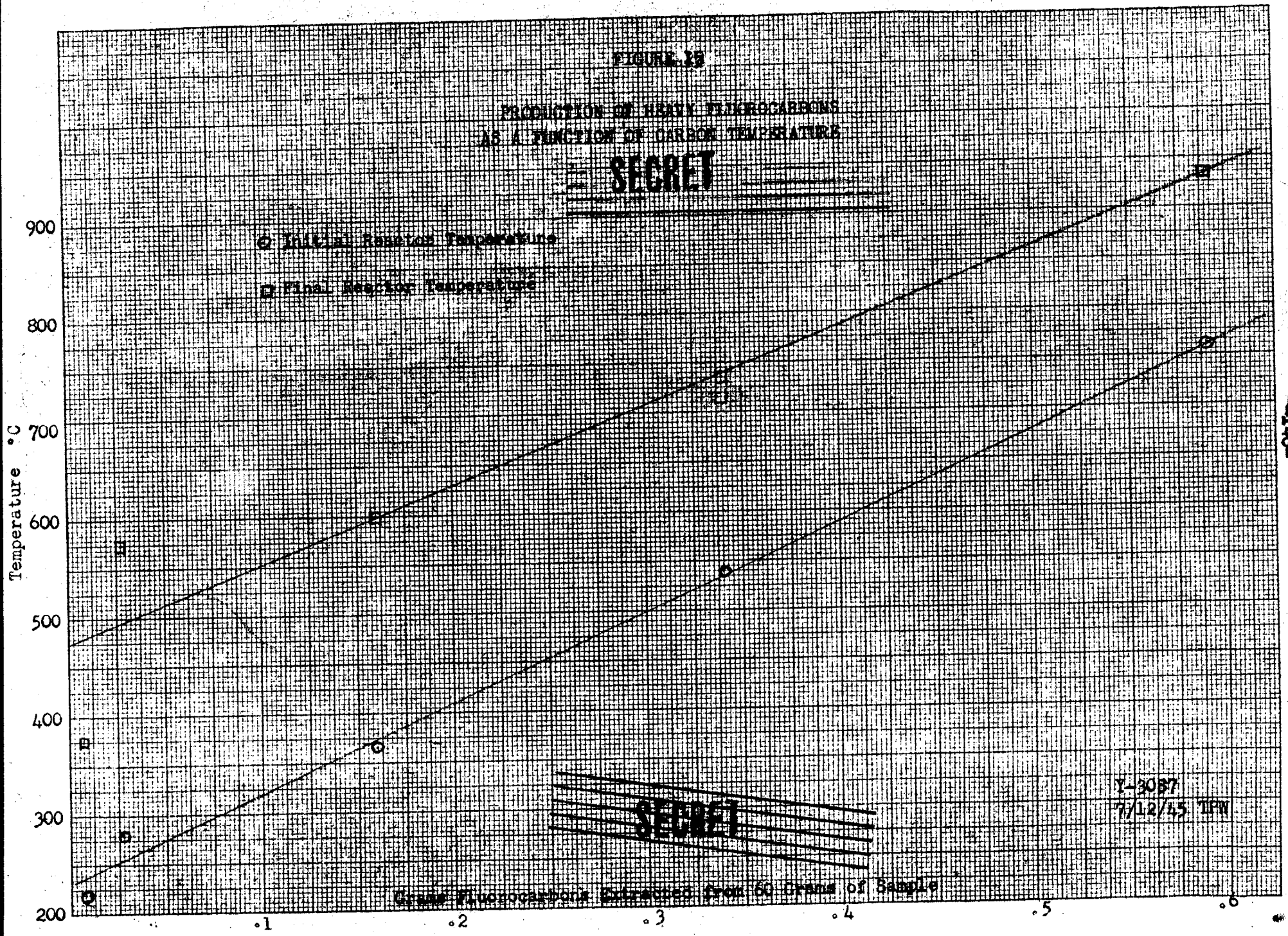
K & E CO., N.Y.

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

FIGURE 25

PRODUCTION OF HEAVY FLUOROCARBONS
AS A FUNCTION OF CARBON TEMPERATURE

SECRET

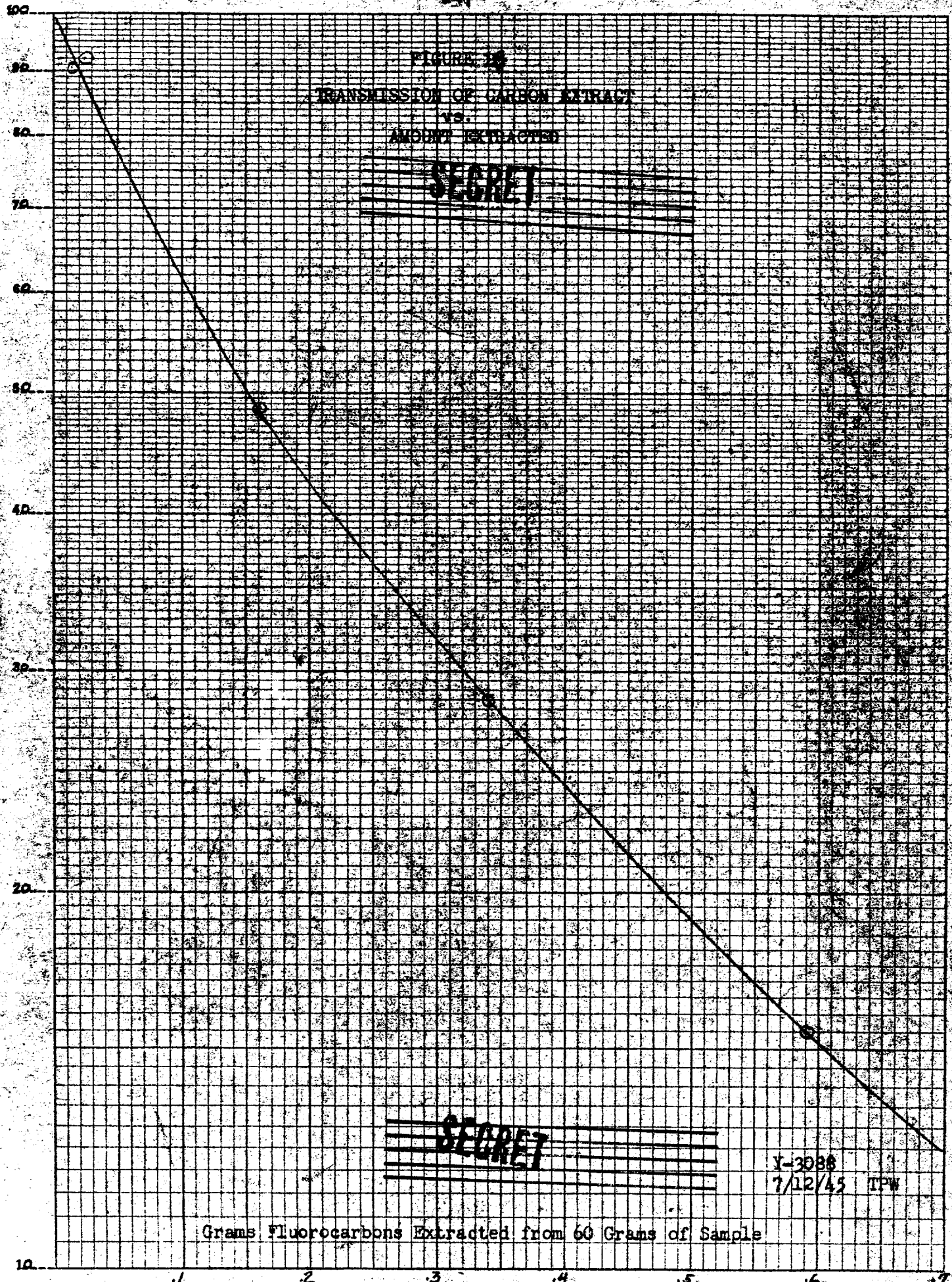


SECRET

FIGURE 14

TRANSMISSION OF CARBON EXTRACT
vs.
AMOUNT EXTRACTED

SECRET

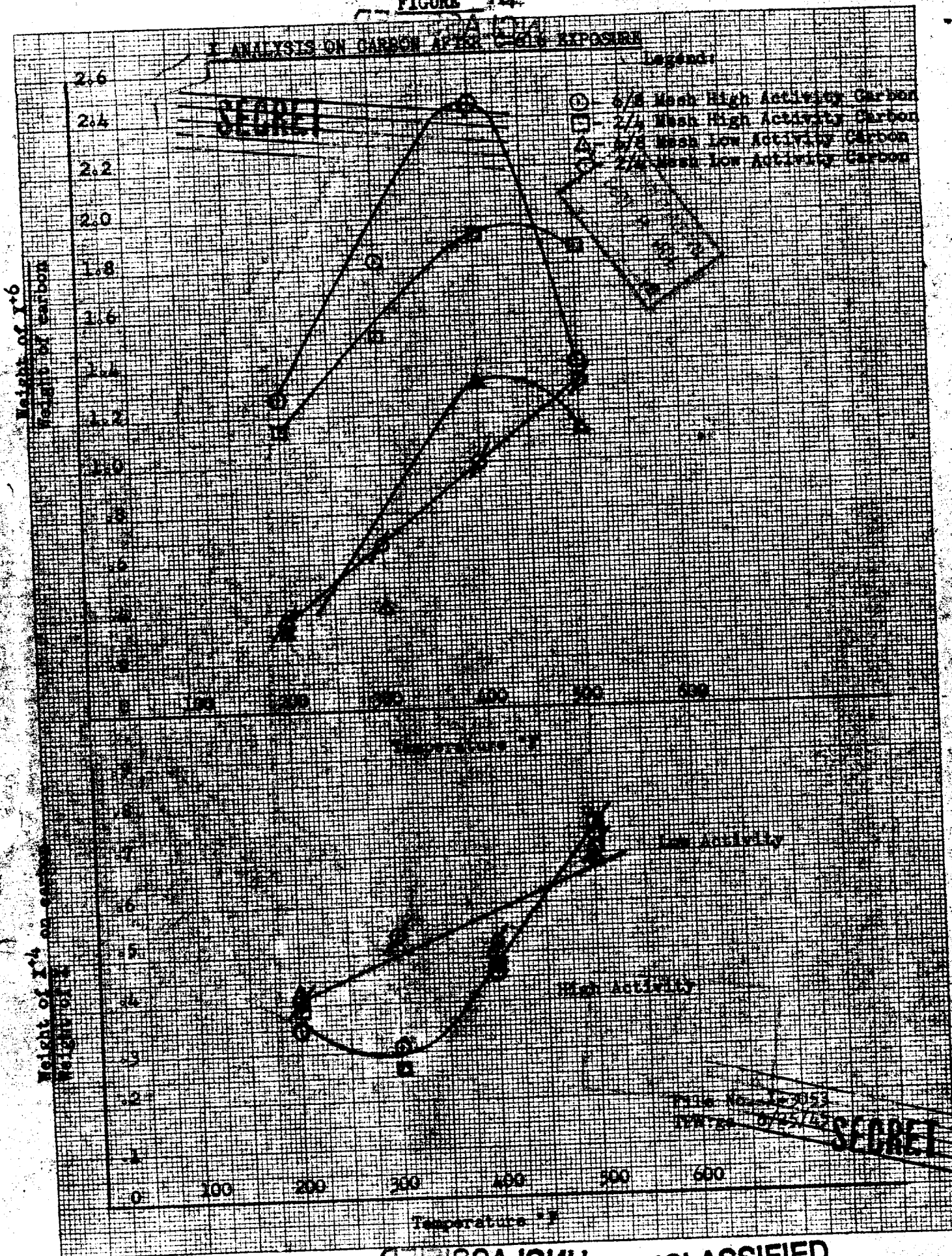


SECRET

Y-3088
7/12/45 TPW

UNCLASSIFIED

FIGURE 17



SECRET

UNCLASSIFIED