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DATE: November 5, 1952

SUBJECT: DESIGN OF THE DISSOLVER OFF-GAS SYSTEM FOR THE IDAHO CHEMICAL PROCESSING PLANT

TO: F. L. Steahly

FROM: John M. Holmes, Foster Wheeler Corporation

AEC RESEARCH AND DEVELOPMENT REPORT

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DESIGN OF THE DISSOLVER OFF-GAS SYSTEM FOR THE IDAHO CHEMICAL PROCESSING PLANT

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OAK RIDGE NATIONAL LABORATORY

TO: F. L. Steahly DATE: November 5, 1952

FROM: John M. Holmes, FOSTER WHEELER CORPORATION

SUBJECT: DESIGN OF THE DISSOLVER OFF-GAS SYSTEM FOR THE IDAHO CHEMICAL PROCESSING PLANT

1.0 CORRELATION OF ADSORPTION DATA

The data used for designing the WN System adsorbers are divided into two groups: the static equilibrium data and the dynamic adsorption data involving mass transfer considerations.

1.1 Static Adsorption Data

These data are presented in figures #1, 2, 3, and 4. These data were determined experimentally by the Linde Air Products Laboratory and from other sources as listed in the bibliography.

In order to estimate the degree of desorption of the adsorbed gases during reactivation, it was necessary to extrapolate many of the curves to higher pressures. An examination of the experimental curves indicated that for low temperatures, krypton, xenon, carbon monoxide and nitrogen follow the Freundlich equation in the low pressure range and the Langmuir equation in the high pressure range. Since the extrapolations were done in the high pressure range, the Langmuir equation was used. It is believed that this method was sufficiently accurate for the reactivation calculations. Extrapolated data are shown as dashed lines on all curves.

Figure #4 for the adsorption of nitrogen on charcoal indicates discrepancies between the high and low pressure data, especially in the low temperature range. This is undoubtedly due to the differences in charcoal used by the various investigators and to experimental errors. However, again the data appear accurate enough for reactivation calculation.

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1.2 Dynamic Adsorption Data

The dynamic adsorption data were obtained from two sources; the Linde Air Products Laboratory, (Linde C.F. No. 51-11-5), and Oak Ridge National Laboratory, (ORNL 52-3-151). The Linde data include runs made at -183°C, -170°C and -150°C on krypton-nitrogen mixtures and krypton-xenon-nitrogen mixtures. The Oak Ridge data were obtained at -170°C with krypton-xenon-nitrogen mixtures. A brief description of the apparatus used by each laboratory follows:

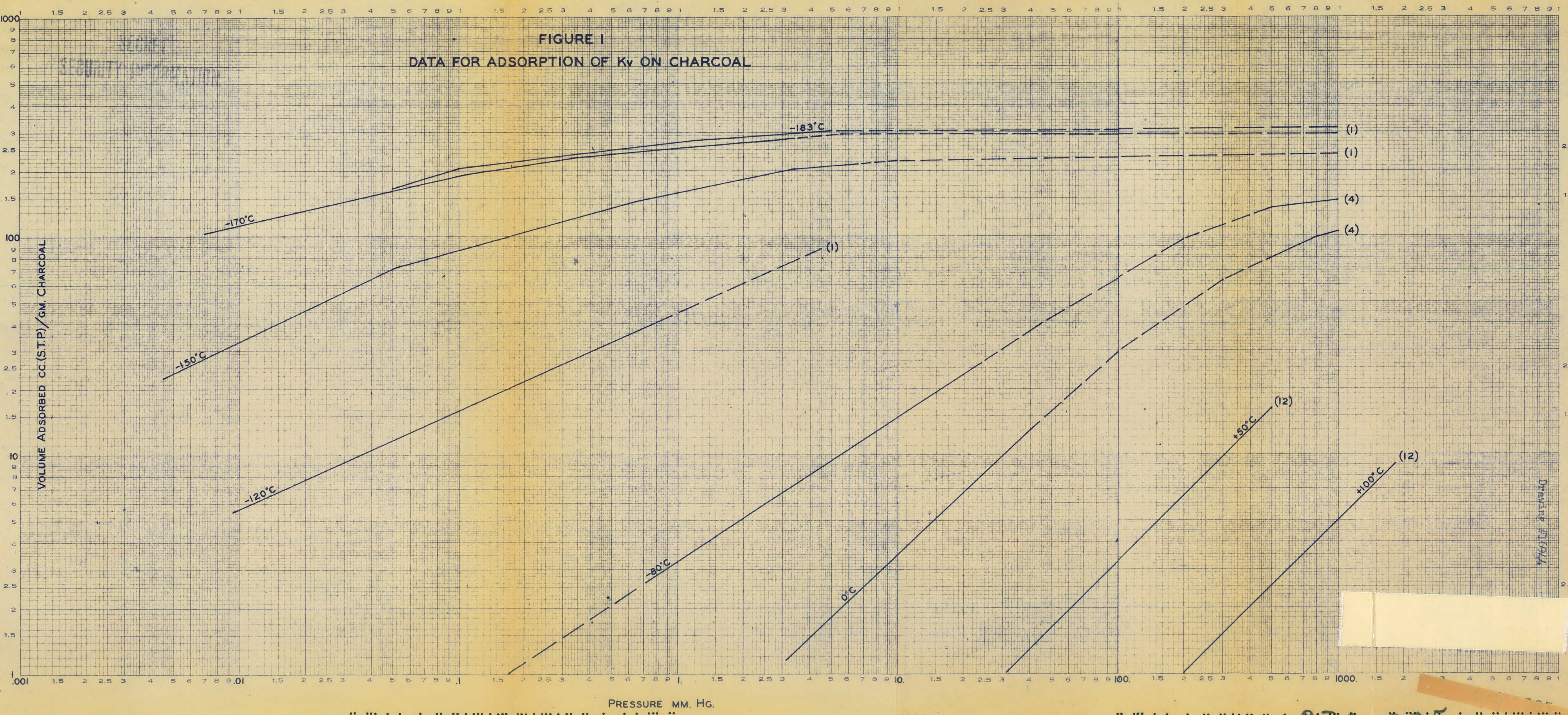

FIGURE I

DATA FOR ADSORPTION OF Kv ON CHARCOAL

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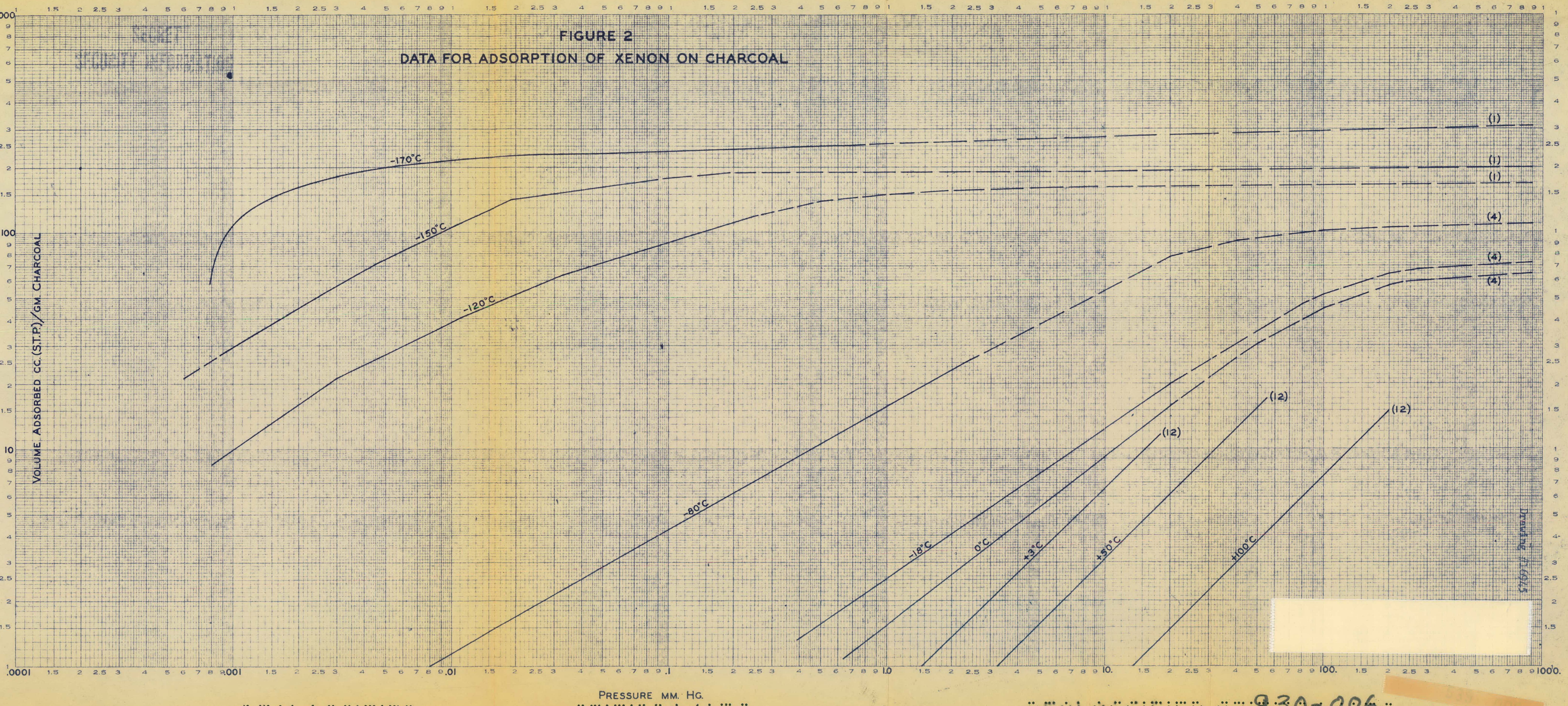

FIGURE 2

DATA FOR ADSORPTION OF XENON ON CHARCOAL

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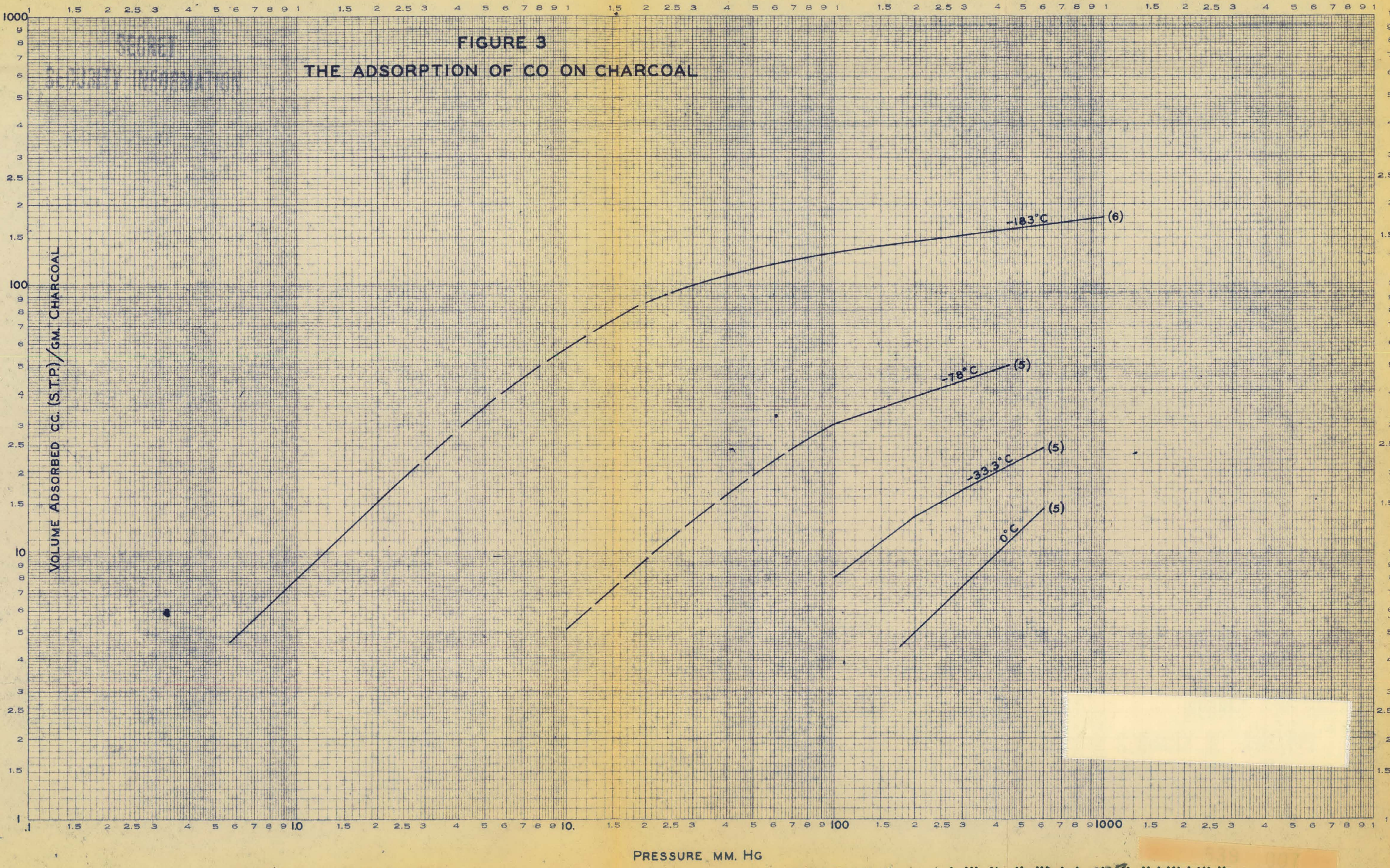

FIGURE 3
THE ADSORPTION OF CO ON CHARCOAL

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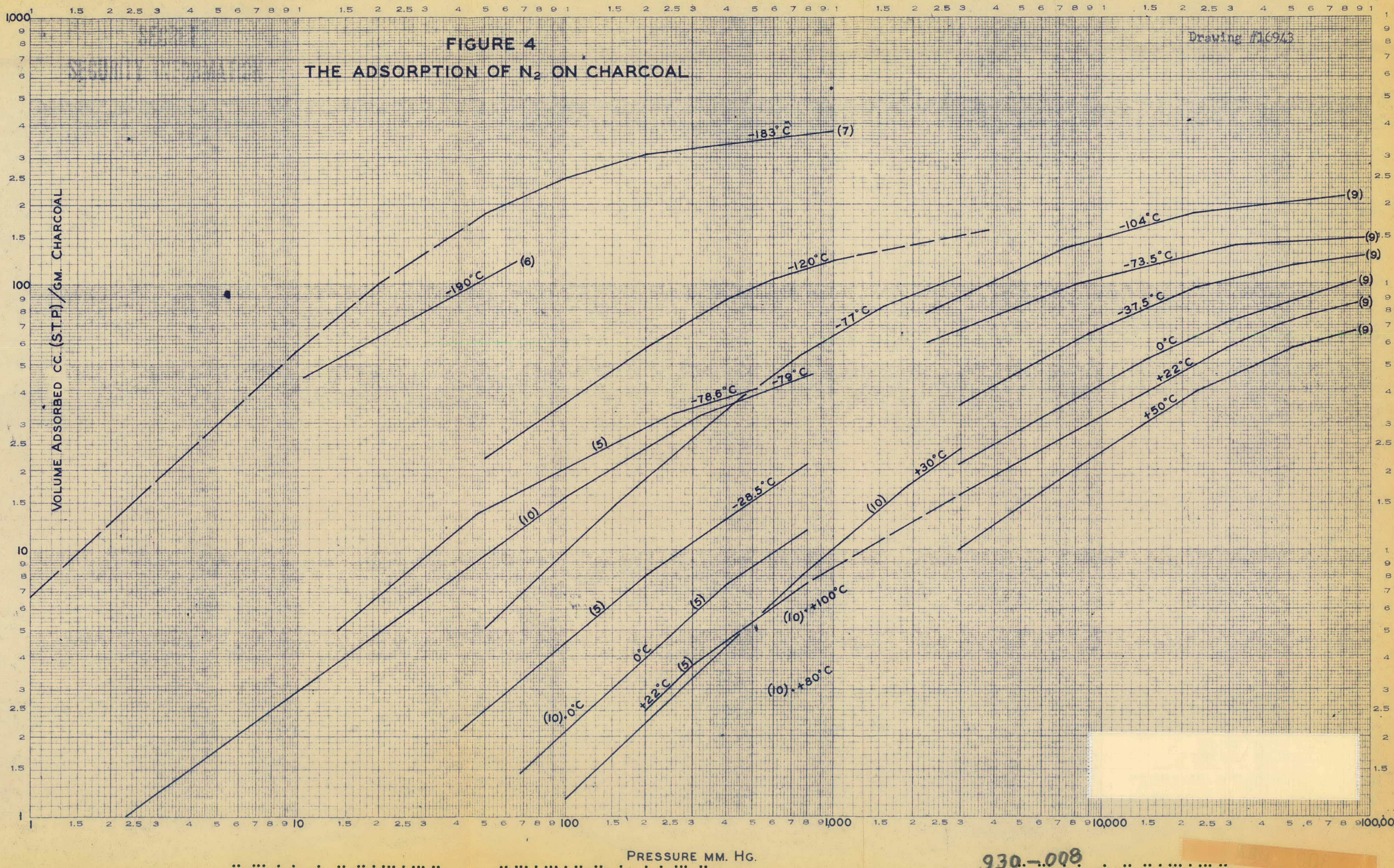
FIGURE 4

THE ADSORPTION OF N_2 ON CHARCOAL

Drawing #16943



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Linde Air Products Laboratory. The adsorption trap consisted of a pyrex glass "U" trap 1.65 cm. in diameter, with a bed of 10 gms. Columbia CXA activated carbon, 11.0 cm. in depth. Flow rates and temperatures were varied. The rare gas concentrations of the effluent gas were measured with a mass spectrometer capable of detecting a concentration as low as 10 ppm krypton or xenon.

Oak Ridge National Laboratory. The adsorption trap consisted of a 35 gram bed of 4 to 6 mesh CXA Columbia carbon, 6 inches in length and 1.04 inches in diameter. Flow rates were varied at a constant temperature of -170°C. The krypton concentration of the effluent was determined by adding a small amount of radioactive krypton to the stable krypton and xenon mixture entering the bed and then counting the inlet and outlet gas streams. The ratio of the inlet and outlet counts per minute were then equal to the ratio of the inlet and outlet krypton concentrations.

The first data from the Linde Air Products Laboratory were correlated employing the equations of Hougen and Marshall, Chemical Engineering Progress 43, 197, 1947, and are presented in ORNL 51-5-193. Later data from the Linde laboratory and Oak Ridge National Laboratory are correlated in this report using a simplified solution to these equations presented by S. H. Jury, (ORNL C.F. 51-7-41.) The simplified equation became:

$$2\,Y/Y_0 - 1 = \phi\left[\sqrt{ax} - \sqrt{b\tau}\right]$$

Where Y = concentration of rare gas in effluent

Where Y_0 = concentration of rare gas in inlet stream

ϕ = indicates the error function of the terms enclosed by brackets

a = 1/Hog

Hog = Height of a transfer unit for gas adsorption, ρm

x = length of bed, ρm

b = $\dfrac{CG.}{\rho \text{Hog}}$

c = equilibrium constant $\dfrac{\text{gms adsorbent}}{\text{gms } N_2 \text{ gas}}$

G_1 = Mass flow rate, $\dfrac{\text{gms}}{\text{hr.cm}^2\text{ bed}}$

ρ = density adsorbent gm/cc of bed

τ = time bed has run, hrs.

Probability paper has been prepared by plotting $\phi(\xi)$ vs. ξ on the ordinate and with a linear coordinate on the abscissa as suggested by Dr. Jury. Therefore, if $2\,Y/Y_o - 1$ is plotted vs. $t^{1/2}$ a straight line should be obtained with $\sqrt{ax}$ as the intercept and $\sqrt{b}$ as its slope. A simple method for determining the intercept is to extrapolate the curves to $2\,Y/Y_o - 1 = 0$. Then $\sqrt{ax} = \sqrt{b\tau}$ and $\sqrt{ax}$ can be determined from the slope $= \sqrt{b}$ and $\sqrt{\tau}$ when $2\,Y/Y_o - 1 = 0$.

Dr. Jury in his report, "Design of Percolators," ORNL 51-7-41, suggests that an adsorption bed should be designed for each component separately. However, in correlating the Oak Ridge National Laboratory adsorption data, this would be impossible since the xenon concentrations throughout the bed are not known. Further, the lengths of bed for xenon and krypton adsorption at any one time vary, causing the intercept term $\sqrt{ax}$ to change. Therefore, it has been decided to correlate the data for xenon and krypton as one component. This may be done without introducing appreciable errors because the two terms which vary with the components, c, the equilibrium constant and Hog, the height of a transfer unit are of the same order of magnitude. The following calculation is included to prove this point:

1. Hog - The height of a transfer unit will vary as the 2/3 power of the Schmidt number according to the correlation of Gamson, Thodos, and Hougen.

Therefore:

$$\frac{\text{Hog}(Xe)}{\text{Hog}(Kr)} = \left[\frac{\mu Xe}{\mu Kr}\right]^{2/3} \times \left[\frac{\rho Kr}{\rho Xe}\right]^{2/3} \times \left[\frac{D_v(Kr)}{D_v(Xe)}\right]^{2/3}$$

where:

μ = gas viscosity

ρ = gas density

D_v = diffusivity of vapor in carrier gas

Then from handbook data:

at approximately - 180°C

$$\left[\frac{\mu Xe}{\mu Kr}\right]^{2/3} = \left[\frac{210.7}{233.4}\right]^{2/3} = 0.932$$

$$\left[\frac{\rho Kr}{\rho Xe}\right]^{2/3} = \left[\frac{83.7}{131.3}\right]^{2/3} = 0.741$$ (the densities will vary as the molecular weights)

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Drawing #16947

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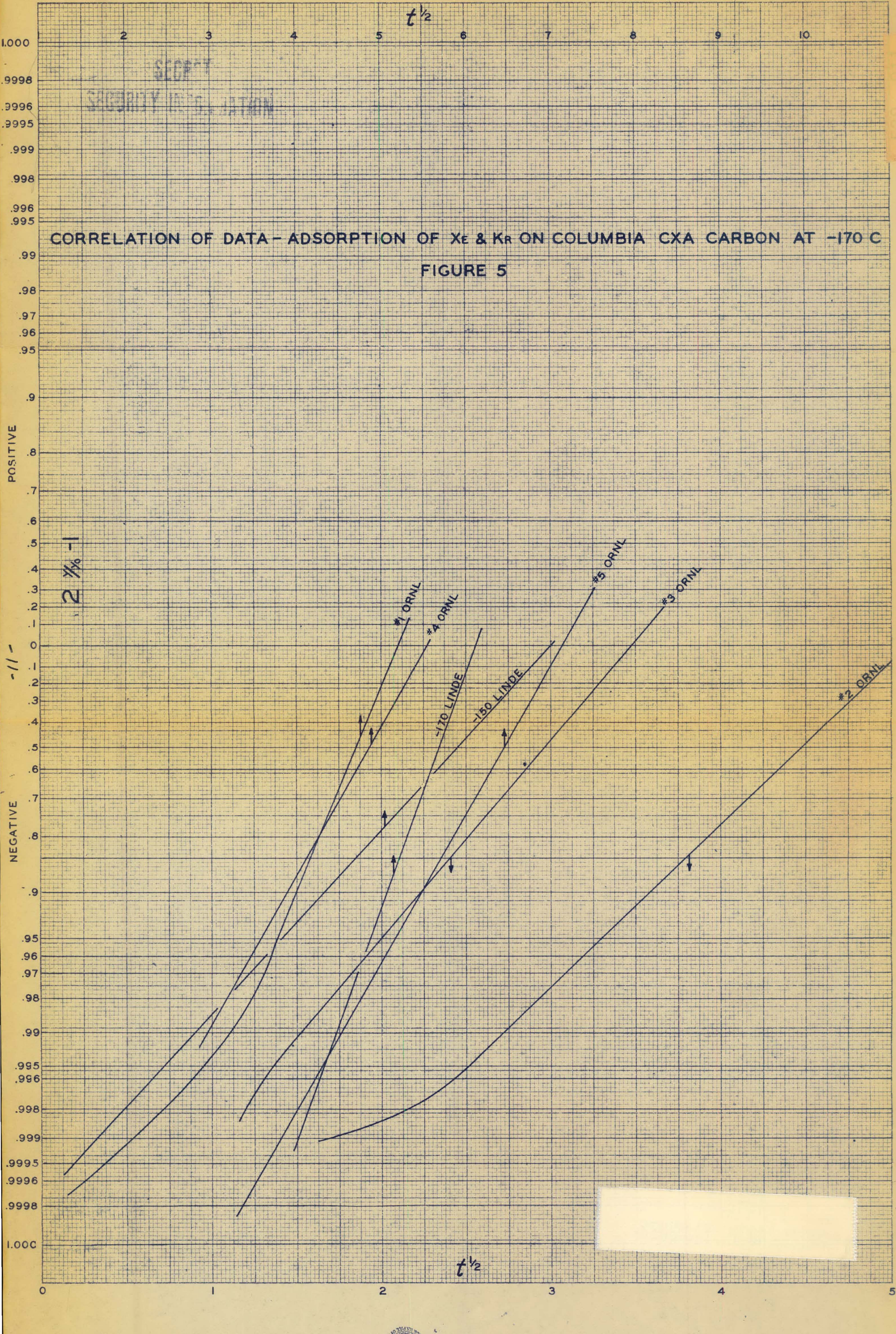

CORRELATION OF DATA - ADSORPTION OF X_E & K_R ON COLUMBIA CXA CARBON AT -170 C

FIGURE 5

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The diffusivities may be calculated from the equation given by Sherwood excluding the terms which are constant for each gas, (temperature, total pressure, and the constant.)

$$\frac{D_v\ (Kr)}{D_v\ (Xe)} = \frac{\sqrt{1/M_{Kr} + 1/M_{N_2}}}{\left[V_{Kr}^{1/3} + V_{N_2}^{1/3}\right]^2} \quad \frac{\sqrt{1/M_{Xe} + 1/M_{N2}}}{\left[V_{Xe}^{1/3} + V_{N2}^{1/3}\right]^2}$$

Where M = molecular weights

V = molecular volume at the normal boiling point. CC/gm mole.

From Perry:

$V_{N_2} = 31.2$

From Handbook data:

$$Kr = \frac{83.7 \text{ gm/mole}}{2.818 \text{ gm/cc}} = 29.7 \text{ cc/gm mole}$$

$$Xe = \frac{131.3 \text{ gm/mole}}{3.06 \text{ gm/cc}} = 43.0 \text{ cc/gm mole}$$

Therefore:

$$\frac{D_v\ (Kr)}{D_v\ (Xe)} = 1.13 \qquad \frac{D_v\ (Kr)^{2/3}}{D_v\ (Xe)} = 1.085$$

Therefore:

$$\frac{Hog(Xe)}{Hog(Kr)} = 0.932 \times 0.741 \times 1.085 = 0.748$$

2. The Equilibrium constant.

The adsorption equations used for correlations assume a linear equilbirium curve through the origin such that $y^* = CW$ where y^* = concentration of adsorbable vapor in equilibrium with the adsorbate in the carbon W. This is not the case for rare gas adsorption, for the equilibrium equations are actually of the exponential type,

$y^* = CW^n$. However, values for the equilibrium constant c have been calculated from the adsorption data and are plotted in Figure (6) as c versus the total concentration of rare gas in ppm. Further it has been determined that these values are of the same order of magnitude as the c values calculated by assuming a linear curve through the origin and the inlet concentration of rare gas, an example of which follows:

C for Krypton

$$C = \frac{\text{gm Kr/gm gas}}{\text{gm Kr/gm carbon}}$$

$$= \frac{\text{Mol fraction Kr} \times 83.7/28}{\boxed{\frac{\text{cc Kr}}{\text{gm carbon}}}\ \boxed{\frac{\text{mole}}{22{,}400\ \text{cc}}}\ \boxed{\frac{83.7\ \text{gm}}{\text{mole}}}}$$

$$= \frac{800 \times \text{Mol Fraction Kr}}{\text{cc Kr/gm carbon}}$$

Inlet Kr = 4000 ppm

partial pressure = $4000 \times 10^{-6} \times 760 = 3.04$ mm.
cc/gm from Figure (1) = 277 cc/gm

Therefore:

$$C = \frac{800 \times 0.004}{277} = 0.0116$$

C for Xenon

Inlet Xe = 4000 ppm. partial pressure = 3.04 mm
cc/gm from Figure (2) = 314 cc/gm

Therefore:

$$C = \frac{800 \times .004}{314} = 0.0102$$

Average C = 0.0109

C determined experimentally from Figure (6) = 0.0170.

The calculated values are reasonably close to the experimental and very close to each other.

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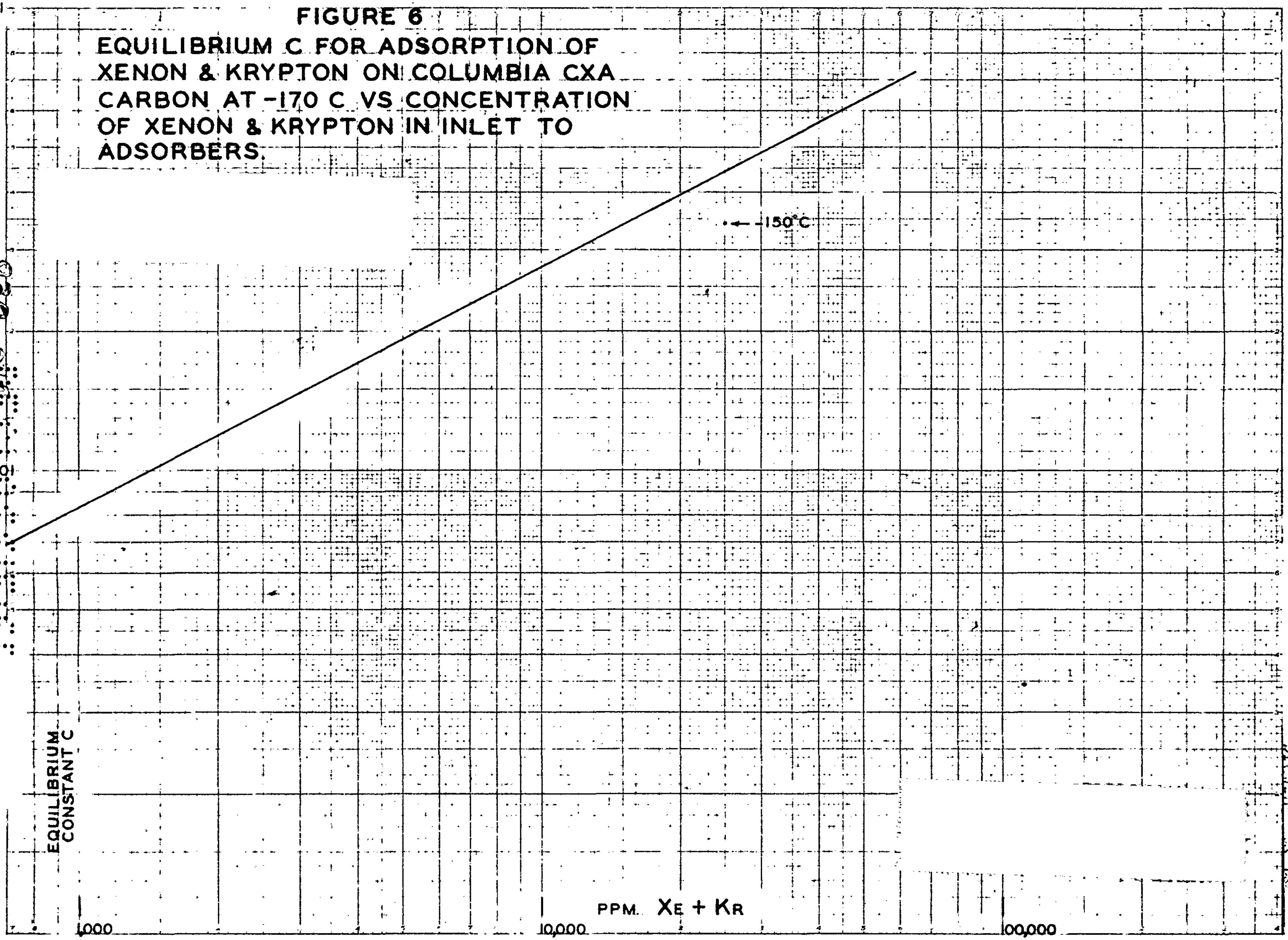

FIGURE 6

EQUILIBRIUM C FOR ADSORPTION OF XENON & KRYPTON ON COLUMBIA CXA CARBON AT -170 C VS CONCENTRATION OF XENON & KRYPTON IN INLET TO ADSORBERS.

The data for the five ORNL experimental runs and two Linde runs are plotted in Figure (5) according to the theory. For most runs, excellent straight lines were obtained for values of Y/Y_o above 0.01.

The values for the height of a transfer unit, Hog, and the equilibrium constant, C, are given in the following figures. Hog values are plotted versus velocity in Figure (7). Contrary to the correlation by Gamson, Thodos, and Hougen, the value for the slope of the Hog versus velocity plot in Figure (7) is 0.61 instead of 0.51. Equilibrium data calculated from the experimental data are given in Figure (6) as C versus ppm of total rare gas concentration.

These data are used to determine the length of time the designed adsorption bed for the Arco, Idaho Plant will run before "break through" of radioactive krypton.

2.0 WN SYSTEM ADSORPTION EQUIPMENT

2.1 Description of WN Adsorption Equipment

Two complete and independent adsorption units are provided in order that one may be operated while the other is being regenerated. Each unit consists of an Insulation box (WN 131 or WN 132), Gas Pre-Cooler, (WN 324 or WN 326), Adsorption Unit, (WN 114 or WN 116) and an adsorption Clean-Up unit, (WN 115 or WN 117).

The flow of gas through the vessels in WN 131, (flows through WN 132 are identical), are as follows: Gases enter the Gas Pre-Cooler, (WN 324), at room temperature, are cooled to -159°C by the gases leaving the Adsorption Clean-Up Unit, (WN 115), and during this cooling the remaining N_2O and H_2O are condensed out in WN 324. They are then sent to the Adsorption Unit, (WN 114), where they are cooled to -180°C and the rare gases adsorbed. The stream is then sampled for rare gas concentration, and passed through the Adsorption Clean-Up Unit, (WN 115), and Pre-Cooler, (WN 324.) Gases leaving WN 324 are near room temperature, free of all radioactive material and are sent to the stack after being filtered in WN 116.

The WN Adsorption System is equipped with a pressure vessel, WN 130, which has sufficient volume to keep the pressure of the adsorption system below 300 psig. should a breakdown occur in the liquid nitrogen system. Therefore, the system could be isolated and the rare gases confined even if the temperature of the adsorption beds was at room temperature.

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Drawing 16949

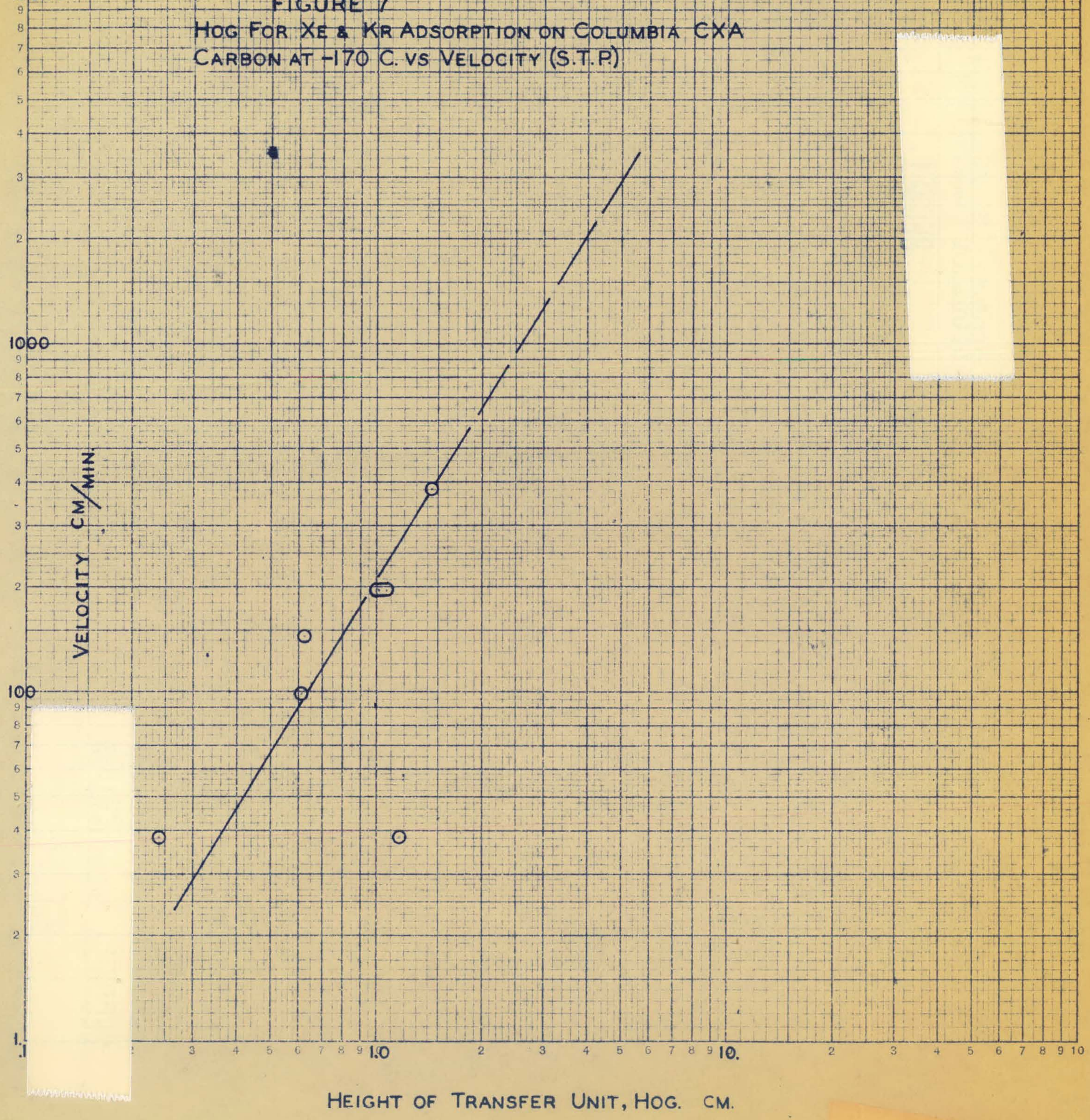

FIGURE 7

HOG FOR XE & KR ADSORPTION ON COLUMBIA CXA CARBON AT -170 C. VS VELOCITY (S.T.P)

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2.2 Reference Drawings of WN Adsorption Equipment

Details of the WN System Adsorption equipment are given on the following Alloy Fabricators drawings:

WN 114 & WN 116	A-1390-A-15 A-1390-A-17
WN 115 & WN 117	A-1390-A-16 A-1390-A-17
WN 324 & WN 326	A-1390-A-18
WN 131 & WN 132	A-1390-A-10 A-1390-A-11 A-1390-A-12 A-1390-A-13 A-1390-A-14 A-1390-A-21
WN 130	A-1390-A-9

Details of the flow system are given on WN Flowsheet 542-41-P1341C and details of the Cold Box piping layout are given on 542-41-P1388 and 542-41-P1389.

3.0 HEAT TRANSFER FROM REFRIGERANT TO TUBES IN WN 114 & WN 116

3.1 Calculation Procedure

Calculations on WN 324 and WN 326 show that the temperature of the process gases entering WN 114 or WN 116 will be approximately -254°F. It will be necessary to cool these gases to -292°F, the operating temperature of the adsorber. The assumption is made that the greater part of this cooling will be done in the upper part of the adsorber tubes since the area for heat transfer from the refrigerant to the gas in the adsorber head is small. The level of liquid nitrogen will be maintained near or below the center line of the vessel so it is assumed that the heat will be transferred across two gas films - from the vaporized nitrogen to the tube and from the tube to the carbon granules and the flowing gases.

The heat transfer coefficient for the gas film outside the tube is taken from the chart on page 477, Perry's Chemical Engineer's Handbook, third edition. The mass velocity for the N_2 gas is so low that a free convection coefficient is used. The heat transfer coefficient for the gases flowing over the granular bed is calculated using the chart on page 165 in McAdams', "Heat Transmission," second edition for air inside tubes at low velocities. This coefficient is corrected for pressure and the presence of carbon particles using the data of Colburn, A.P., Industrial Engineering Chemistry, 23,910, 1931.

3.2 Heat Transfer Calculations

A. Physical Dimensions

Tubes:

OD = 1 in

ID = 0.870 in

Wall = 0.065 in

Inside Area = 0.00413 ft^2 x 48 = 0.198 ft^2

Area for Heat Transfer =
Inside = 0.2277 ft^2/ft
Outside = 0.2618 ft^2/ft

Charcoal:

Specific Heat = 0.242 Btu/lb°F

OD Dimensions = 3/16 in
Length = 1/8 in to 3/8 in
Average = 1/4 in
Mesh Size = 4 to 6

Density = 30 lbs/ft^3

Thermal Conductivity = 0.326 $\frac{\text{Btu in}}{\text{hr ft}^2\ {}^\circ\text{F}}$

Gases:

Composition = (neglect N_2O and rare gases) N_2 at -254°F (-159°C) to -292°F (-180°C)

Pressure = 8.05 psia.

Temperature (Average) = -273°F

Heat Capacity = 0.25

Density = $\frac{28}{359} \times \frac{8.05}{14.7} \times \frac{492}{187} = 0.112$ lb/ft^3

Flow Rate = 3.69 SCFM

Velocity (Standard Conditions) = $\frac{3.69}{48 \times 0.00413} = 18.6$ ft/min

Velocity (Actual Conditions) = $18.6 \times \frac{.078}{0.112} = 12.95$ ft/min

Viscosity = 0.0056 cp. = 0.0136 lb/hr ft

Mass Flow Rate = $28 \times 0.589 + 2 \times 0.0285 + 2 = 16.6$ lbs/hr

Thermal Conductivity = 0.0037 Btu ft/hr ft^2 °F

B. Heat Transfer Coefficient Inside Tubes

(From Chart, Page 165 McAdams, Heat Transmission, Second Edition)

$h = 1.5$ for $G < 800$

Corrected for Pressure

$$h_1 = 1.5 \times \left[\frac{(0.55)}{(1.07)}\right]^{2/3} = 0.97$$

Diameter Carbon = 0.188

Diameter Tube = 0.870

$D_p/D_I = 0.216$ $h/h_I = 7.5$ (From Colburn IEC 23, 910, 1931)

$h = 7.5 \times 0.97 = 7.3$ Btu/hr ft^2

C. Heat Transfer Coefficient Outside Tubes

If Liquid N_2 Level is below the area for transfer, the gas film will control.

Area for flow $= 0.785 \times \left[\frac{(10)}{(12)}\right]^2 - 48 \times .785 \left[\frac{(1)}{(12)}\right]^2$

$= 0.542 - 0.260 = 0.282$ ft^2

Average Flow N_2 = 20 lbs/hr

Therefore:

$$G = 20/0.282 = 71 \text{ lbs/hr ft}^2$$

$$G^1 = \frac{71}{3600} = 0.0197 \text{ lbs/sec ft}^2$$

Use chart Page 472, Perry, <u>Chemical Engineers Handbook, Third Edition.</u>

Temperature of N_2 gas = Saturation Temperature Liquid N_2 at 62 psia = -294°F(-181.0°C)

Final temperature N_2 gas = $\frac{158}{20 \times 0.25} - 294 = -262.4^\circ\text{F}(-163.5^\circ\text{C})$

$$\text{Average}\Delta t = \frac{(-159 + 163.5) + (-180 + 181)}{2} = 2.75^\circ\text{C}$$

Absolute Pressure of N_2 = 62 psia = 4.22 atomspheres

$$p^2\Delta t_m = 17.7 \times 2.75 = 48.7$$

$$h = 2.7 \frac{\text{Btu}}{\text{hr ft}^2 \,^\circ\text{F}}$$

D. <u>Overall Coefficient</u>

$$\frac{1}{UoA} = \frac{1}{7.3 \times 48 \times 0.2277 \text{ ft}^2/\text{ft}} + \frac{1}{2.7 \times 48 \times 0.2618 \text{ ft}^2/\text{ft}}$$

$$UoA = 23.8 \frac{\text{Btu}}{\text{hr}\,^\circ\text{F}}$$

E. <u>Log Mean Temperature Difference:</u>

$$= 1.8 \times \frac{4.5-1}{\ln 4.5/1} = 4.2^\circ\text{F}$$

$$Q = 16.6 \times 0.25 \times 38 = 158 \frac{\text{Btu}}{\text{hr}}$$

F. <u>Length of Tubes Necessary for Heat Transfer</u>

$$Q = UAL\Delta t_m$$

$$L = \frac{q}{UA\Delta t_m} = \frac{158}{23.8 \times 4.2} = 1.6 \text{ ft}$$

Therefore it will take 1.6 ft to cool the gases from -254°F to -292°F.

4.0 PROCESS CALCULATIONS ON ADSORBERS WN 114, WN 115, WN 116 AND WN 117

4.1 Design Basis

The basis for design will be the material balance streams from the preliminary heat exchangers, WN 324 or WN 326 for MTR processing:

COMPONENT	LB MOLES PER HOUR	MOL %	SCFM (32°F & 1 atm)	pp at TOTAL PRESS = 415 mm	CONDENSATION TEMPERATURE °C
N_2 + A	0.5875	95.11	3.5152	395	-203
H_2	0.0285	4.61	0.1705	19.1	-261
CO	0.0012	0.194	0.0072	0.806	-222
N_2O	8.1×10^{-5}	1.31×10^{-2}	- - -	0.0544	-159
Kr	69.92×10^{-6}	1.13×10^{-2}	- - -	0.0470	-210
Xe	406.2×10^{-6}	6.59×10^{-2}	- - -	0.2738	-174
TOTAL	0.6177		3.6929		

These data show that N_2O and Xe will enter the -180°C adsorption bed with condensation temperatures higher than the bed temperature. However, ORNL and Linde data show that Xenon was adsorbed without condensation on a bed which has a temperature of -170°C while the condensation temperature of the Xenon was greater than -170°C.

4.2 Determination of Length of Time Bed Will Operate Before "Break Through"

4.21 Method Of Calculation

A. Assume a period of 300 hrs or two months MTR operation.

B. Determine quantity of charcoal used up by N_2O gas in first adsorption band of bed.

C. Determine the length of time the bed will run by two methods:

1. Using data from the adsorption bed experiments conducted by the Linde Air Products Laboratory

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(CF 51-11-5), and Oak Ridge National Laboratory (ORNL 52-3-151), along with the Hougan and Marshall Charts, Page 882 in Perry's Chemical Engineers' Handbook, Third Edition.

2. Using a factor determined from the above data for the bed loading at equal contact times, and using equilibrium data, determine the amount of rare gases the bed will hold before "break through."

4.22 Quantity of Charcoal Used by N_2O Adsorption

Data are not available for N_2O adsorption at -180oC. Instead one may use the volume of solid N_2O and the pore volume of charcoal as determined from adsorption of N_2gas.

Total pressure of system = 415 mm.

Volume of liquid N_2 at 90°K = 37.5 cc/gm mole

Formula for capacity of charcoal for N_2 - National Bureau of Standards

Paper R.P. 1496 page 191, 1942

$$p/v = 0.00251\ P + 0.15$$

where p = partial pressure mm
v = capacity cc/gm S.T.P.

$$415/v = 0.00251 \times 415 + 0.15$$

$$v = 348 \text{ cc/gm}$$

Therefore:

$$\text{Liquid volume} = \frac{348 \times 37.5 \text{ cc liq}}{22{,}400 \text{ gm mole}} = 0.583 \text{ cc/gm}$$

Solid volume N_2O = 1.226 gm/cc

Therefore:

$$\text{Capacity for } N_2O = 1.226 \text{ gm/cc} \times 0.583 \text{ cc/gm}$$

$$= \frac{0.714 \text{ lb } N_2O}{\text{lb charcoal}}$$

N_2O accumulated after 300 hrs = $8.1 \times 10^{-5} \times 300 \times 44$ lb/mol = 1.07 lb

$$\text{Charcoal used} = \frac{1.07}{0.714} = 1.50 \text{ lb charcoal}$$

$$\text{Length of Bed} = \frac{1.50}{30} \times \frac{1}{0.198} = 0.25 \text{ ft}$$

Use a safety factor of 3.25, therefore length = 0.25 x 3.25 = 0.82. Therefore N_2O will take up 0.82 ft which is less than the 1.6 ft necessary to cool the bed down to the temperature of the liquid nitrogen. The remainder of the bed will be approximately -170°C to -180°C, nearly the saturation temperature of the refrigerant. The data used for Xenon adsorption are for -170°C so the calculation will be on the conservative side.

4.23 Time Bed Will Operate Before "Break Through" of Krypton

A. Length of Bed Available to Krypton and Xenon.

Pack bottom of bed with quartz to a height of 1 1/2 in and the remainder with CXA carbon. Length = 7 ft - 0.125 - 0.82 = 6.055 ft.

B. Height of Transfer Unit Hog

Velocity at standard temperature but corrected for pressure:

Flow rate = 3.69 SCFM

$$\text{Velocity} = \frac{3.69}{48 \times 0.00413} \times \frac{14.7}{8.05} = 34 \text{ ft/min} = 1037 \text{ cm/min}$$

Use Figure 7 for the value of Hog

Hog = 2.70 cm

Therefore Hog = 0.0885 ft

C. Equilibrium Constant - c

Total ppm of rare gas = 113 + 659 = 772

partial pressure = 772×10^{-6}

$$\text{Therefore } c = \frac{800 \times 0.000772}{245} = 0.00252$$

c from Figure 6 = 0.0072

D. Calculation of b

$B = \frac{CG_1}{\rho_s \text{ Hog}}$

Where:
G_1 = Mass Flow rate lb/hr ft^2
= Density of CXA Charcoal lb/ft^3

Mass Flow rate = 16.6 lb/hr

$G_1 = \frac{16.6}{48 \times 0.00413} = 83.7$ lb/hr ft^2

$\rho_s = 30$ lb/ft^3

Hog = 0.0885 ft

Therefore = $b = \frac{0.0072 \times 83.7}{30 \times 0.0885} = 0.227$

E. Calculation of ax

x = 6.055 ft

a = 1/0.0885

Therefore ax = 68.5

Where:
a = 1/Hog
x = Length of Bed

F. Calculation Of Y/Y_o

Y allowable = 20 ppm

$Y/Y_o = 20/772 = 0.026$

Where:
Y = concentration rare gas leaving bed.
Y_o = concentration rare gas entering bed.

G. Time Bed Will Run For:

$Y/Y_o = 0.026$, ax = 68.5 from chart Page 882 in Perry's *Chemical Engineers' Handbook*, Third Edition.

$b\Upsilon = 48$

$\Upsilon = 48/0.227 = 212$ hrs.

Where:
Υ = time bed has operated.

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4.24 Calculation of Bed from Equilibrium Data and Contact Time

A. Contact time for bed = 6.055/34 x 60 = 10.7 sec.

B. Experiment No. 5 from ORNL 52-3-151 would correspond to this time. The contact time for this run was:

$$\frac{6 \times 2.54 \times 60}{97.5} = \underline{9.4 \text{ sec}}$$

This bed containing 35 gms of CXA charcoal ran 20 hours before "break through" i.e., before effluent rose to 20 ppm.

C. Material balance from Experiment No. 5 ORNL Data

Flow rate = 533 cc/min
Inlet Kr = 2500 ppm
Inlet Xe = 2000 ppm

Krypton = 533 x 60 x 20 hrs x 2500 x 10^{-6} = 1600 cc

Xenon = 533 x 60 x 20 x 2000 x 10^{-6} = 1280 cc

TOTAL = 2880 cc

Bed loading = 2880/35 = 82.4 cc/gm

Bed loading from equilibrium data:

Krypton pp = 0.0025 x 760 = 1.9 mm

Equilibrium conc. from Figure = 265 cc/gm

Xenon pp = 0.002 x 760 = 1.52 mm

Equilibrium concentration from Figure = 260 cc/gm

Weighted loading = (25/45) x 265 + (20/45) x 260 = 263 cc/gm

% of equilibrium loading = 82.4/263 x 100 = 31.4

D. Total weight of charcoal available to Xe and Kr adsorption = 6.055 ft x 0.198 ft^2 x 30 lbs/ft^3 = 36.0 lbs.

Lex X = hours bed will run. Then total lbs. Kr accumulated =

$(69.92 \times 10^{-6}) \times (83.7 \text{ lb/mole}) \times X = (58.4 \times 10^{-4})\, X$

Total lbs Xe = $(406.2 \times 10^{-6}) \times (131.3 \text{ lb/mole}) \times X = (534 \times 10^{-4})\, X$

Partial Pressure Kr = $1.13 \times 10^{-4} \times 415 = 0.0469$ mm

Equilibrium concentration Kr = 160 cc/gm

Partial Pressure Xe = $6.59 \times 10^{-4} \times 415 = 0.274$ mm

Equilibrium concentration Xe = 242 cc/gm

Charcoal that Kr will use for equilibrium adsorption =

$$\frac{58.4 \times 10^{-4}\, X}{160 \times \left(\frac{83.7}{22,400}\right)} \text{ gm/gm} \quad = 0.00975\, X$$

Charcoal Xe will use = $\dfrac{534 \times 10^{-4}\, X}{242 \times \left(\frac{131.3}{22,400}\right)} = 0.0376\, X$

Total Charcoal available = $36.0 \times 0.314 = 11.3$ lbs

Time bed will run = $\dfrac{11.3}{0.0376\, X + 0.00975\, X} = 239$ hours

4.25 <u>Additional Time Bed Will Run After "Break Through" in Main Adsorber (WN 114 or WN 116)</u>

Using Hougan and Marshall Method

Hog = 0.0885 ft

X = 6.055 + 2 - 0.125 = 7.93

ax = 7.93/0.0885 = 89.6

$b\tau$ = 66

τ = 66/0.227 = 291 hours total

Therefore additional time = 291 - 212 = 79 hours

Using equilibrium loading method

Time = 239 x 7.93/6.055 = 312 hours total

Additional time = 312 - 239 = 73 hours

4.3 Basis for Material Balance

The material balance will be figured on a basis of 300 hours of MTR operation. Therefore the amount of gases adsorbed will be as follows:

Total length bed = 9 - 2 x 0.125 = 8.75 ft

Pounds charcoal = 8.75 x 0.198 x 30 = 52.0 lbs

Calculation of N_2 + A and CO:

Assume that N_2 + A and CO are adsorbed in quantities proportional to their inlet concentrations. Total charcoal available to N_2 + A and CO = 52.0-1.50-2.94-11.30 = 36.26 lbs.

Let X = lb moles N_2 + A adsorbed

Let Y = lb moles CO adsorbed

X/Y = 0.5875/0.0012 = 490

$$\frac{28\ X \times 22{,}400}{335 \times 28} + \frac{28\ Y \times 22{,}400}{6.5 \times 28} = 36.26$$

X = 0.491 lb moles

Y = 0.0010 lb moles

COMPONENT	LB MOLES	LBS	MOL. PERCENT	EQUILIBRIUM CONCENTRATION	LBS OF ADSORBENT USED
N_2 + A	0.4910	13.73	74.5	335 cc/gm	32.77
N_2O	0.0243	1.070	3.6	363 cc/gm	1.50
CO	0.0010	0.028	0.2	6.5 cc/gm	3.49
Kr	0.0210	1.757	3.2	160 cc/gm	2.94
Xe	0.1218	16.01	18.5	242 cc/gm	11.30
TOTAL	0.6591		100.0		52.00

5.0 CALCULATIONS FOR THE REGENERATION OF ADSORPTION BEDS (WN 114, WN 115, WN 116, WN 117)

5.1 Regeneration Procedure

Linde Air Products Laboratory recommended heating the adsorption beds to 100°C while pulling a vacuum in order to completely remove all adsorbed gases. Xenon and N_2O are the most difficult gases to remove, and therefore will come off at the higher temperatures, while the others will be desorbed almost immediately on heating.

The procedure for regenerating the WN adsorption beds will involve the heating of the beds from the shell side of the adsorbers using warm dry air supplied by blower WN 211, air drier WN 134, and heater WN 315. The dry air will be available at a temperature of between room temperature and 300°F and a flow of 60 SCFM. Desorbed gases from the beds will flow to the cold traps WN 118 or WN 119. These traps will be cooled by liquid nitrogen under vacuum, which in turn will cool the condensing vapors to a temperature low enough that a vacuum will be created on the beds. The desorbed gases from the beds will be condensed in the inner vessels of WN 118 or WN 119. After regeneration is complete, the valves to WN 118 or WN 119 will be closed and the vessels allowed to warm up to room temperature, thus vaporizing the desorbed gases and raising the pressure to nearly 1400 psig. WN 118 or WN 119 will then be connected to storage cylinders where the gases will be stored by progressively opening and closing cylinder valves until the pressure in the cold trap is down below 100 psig. Each cylinder will then be removed from the manifold when its pressure has reached the highest pressure obtainable on warming up WN 118 or WN 119.

5.2 Determination of the Heat Required for Regeneration

5.21 Calculation Procedures

The procedure for calculating the heat required for regenerating the adsorption beds in Cold Boxes WN 131 and WN 132 follows. The time for regeneration is arbitrarily set at 10 hours. Flows and temperatures have been specified to meet this condition.

The total quantity of heat required to raise the adsorber temperatures to 212°F from -292°F, is calculated. An approximation is necessary in order to determine the heat that the insulation will absorb during regeneration. This involves calculating the space mean temperatures of the insulation around the adsorbers for different thicknesses until a temperature is reached where the heat load drops off rapidly. The resulting thickness is 6 inches of insulation. An approximate figure is also included to account for the heat conducted through the 6 inches of insulation

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during the 10 hour period.

The time to heat WN 131 or WN 132 is calculated from the heat load and the heat transfer rate employing a graphical integration method. The total heat transferred for an average vessel temperature of T, is plotted versus the reciprocal of the heat transfer rate at T, in Figure 8. The area under the curve is therefore equal to the time. Figure 9 gives the time versus temperature curve for WN 131 or WN 132.

5.22 Conditions

A. Original Temperature = -180°C (-292°F)

B. Final Temperature = 100°C (+212 F)

C. Total Weights of Material

Steel = 2430 lbs
Carbon = 52 lbs

D. Time to Regenerate = 10 hours

5.23 Heat Necessary for Regeneration

Steel = 2430 x 0.12 x (212 + 292) = 147,000 Btu

Carbon = 52 x 0.15 x 504 = 3930 Btu

Insulation:
To calculate an approximate heat load for insulation, assume that temperature at start = -292°F and determine space mean temperatures at different depths until a temperature is reached where the heat load drops off rapidly.

Variables:

$$x = \frac{K\theta}{Pct^2}$$

Therefore:

$$x = \frac{0.0192 \times 10}{14.5 \times 0.26t^2}$$

$$x = \frac{0.0509}{t^2}$$

Where:

K = Conductivity

$$= \frac{0.23 \text{ Btu in}}{\text{hr ft}^2\ ^\circ\text{F} \times 12} = 0.0192$$

θ = 10 hours

C = 0.26 Btu/lb°F

P = 14.5 lb/ft^3

$$Y = \frac{t' - t_a}{t' - t_b} \qquad \frac{212 - t_a}{212 + 292}$$

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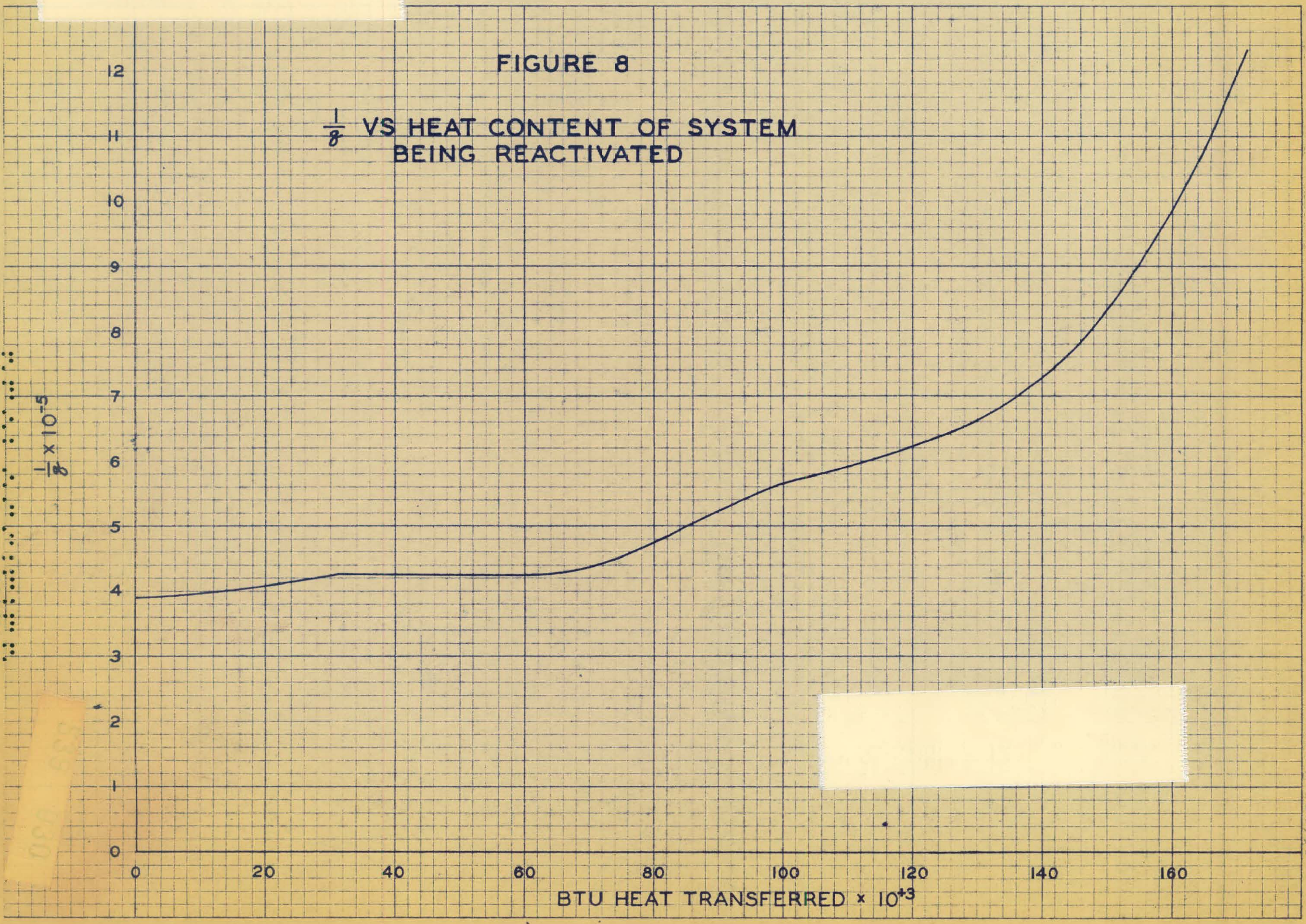

FIGURE 8

$\frac{1}{\delta}$ VS HEAT CONTENT OF SYSTEM BEING REACTIVATED

Drawing #16950

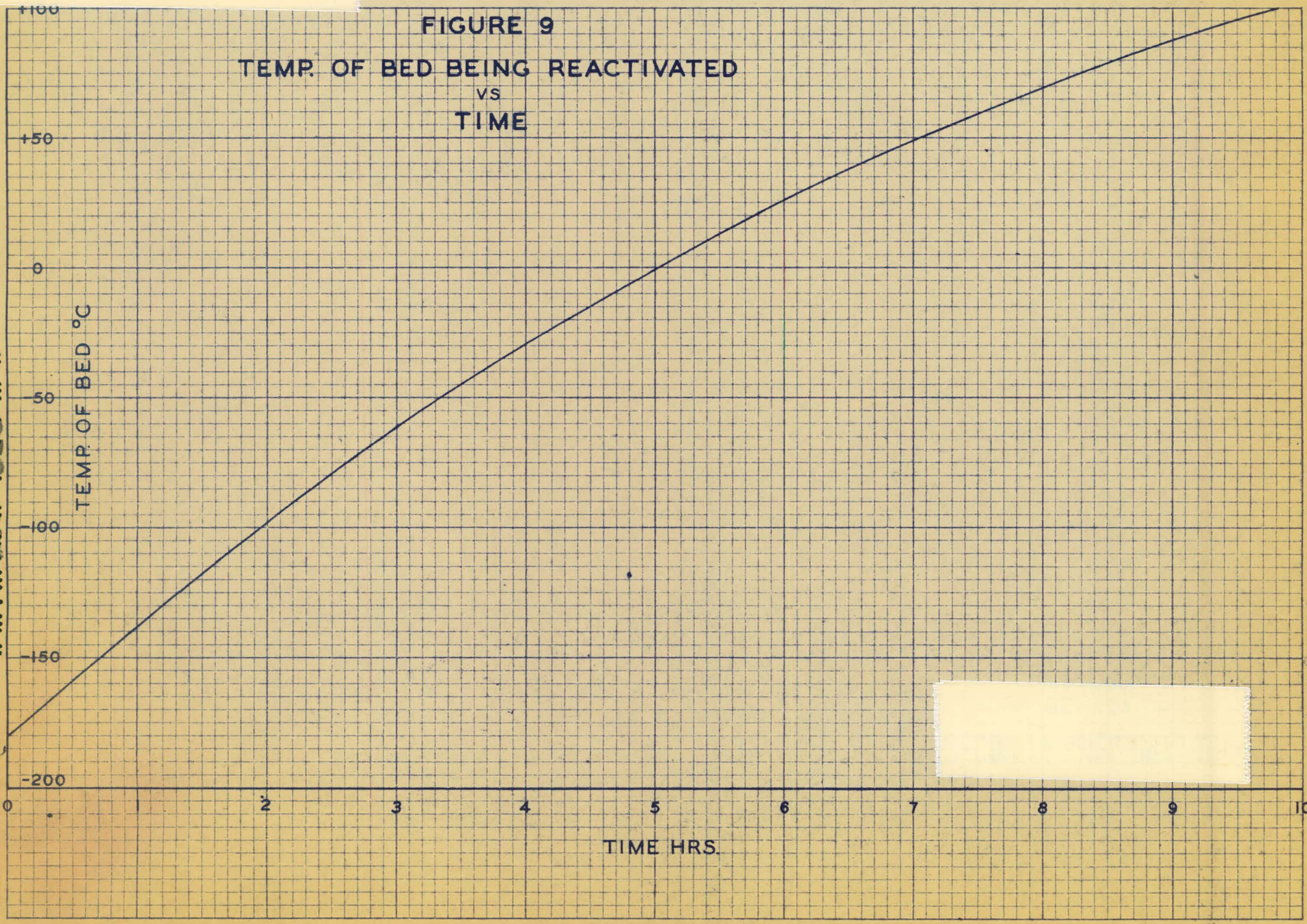

FIGURE 9

TEMP. OF BED BEING REACTIVATED VS TIME

Drawing #16951

Depth		t^2	X	Y	504 Y	t_a 212 - 504 Y
Inches	Feet					
1/4	0.0208	0.000433	117.2	0.01	----	207 space mean temp.
1	0.0833	0.00691	7.36	0.01	----	207 space mean temp.
3	0.25	0.0625	0.814	0.110	55.4	156.6 space mean temp.
5	0.417	0.174	0.292	0.40	201.6	10.4 space mean temp.
6	0.50	0.250	0.204	0.49	246.4	-34.4 space mean temp.
6	0.50	0.250	0.204	0.74	373.2	-160.2 (temp at t = 6")

Therefore Heat Being Transferred

$$q = \frac{0.23\ A_m\ (212 + 160.7)}{6} = 143\ A_m$$

Mean Area for Heat Transfer A_m

$$\text{Inside}\quad 11.9 \times \pi \times 10.75/12 + 4 \times \frac{\pi}{4} \left[\left(\frac{10.75}{12}\right)\right]^2 = 35.9$$

$$\text{Outside}\quad 11.9 \times \pi \times 22.75/12 + 4 \times \frac{\pi}{4} \left[\left(\frac{10.75}{12}\right)\right]^2 = 73.4$$

$$\text{Average} = \frac{73.4 + 35.9}{2} = 54.7 = A_m$$

$$q = 14.3 \times 54.7 = 783 \text{ Btu/hr}$$

Volume of Insulation

$$= 11.9 \times \frac{\pi}{4} \left(\frac{22.75}{12}\right)^2 - 11.9 \times \frac{\pi}{4} \left(\frac{10.75}{12}\right)^2 + 4 \times 1/2 \times \left(\frac{10.75}{12}\right)^2 \frac{\pi}{4}$$

$$= 27.4$$

Heat Load

$$= 27.4 \times 7 \times 0.26 \times (-34.4 + 292) = 12{,}830 \text{ Btu}$$

Therefore total load = 147,000 + 3930 + 12,830 + 783 Btu/hr x 10 hrs =

171,590 Btu

5.25 Time to Heat WN 131 or WN 132 from $-292^{\circ}F$ to $+212^{\circ}F$

Calculate heat transfer rate and then calculate time by a graphical integration. (Figure 8.)

$$\text{Time} = \frac{\text{Heat Content per Temp. rise}}{q(\text{Btu/hr})}$$

a.
$$\begin{array}{lcl} \text{Heat Load Per}^{\circ}\text{F} & = & 171{,}590 \\ \hline \text{Heat per }^{\circ}\text{F} & = & 212 + 292 \end{array} = 341.\ \text{Btu}/^{\circ}\text{F}$$

b. Determination of Area for Heat Transfer

Area Adsorber Tubes = $48 \times 0.262\ \text{ft}^2/\text{ft} \times (7 + 2^*) = 113.0\ \text{ft}^2$

* Secondary Adsorber

Area of Shells

$$9.0\ \text{ft} \times 2.55\ \text{ft}^2/\text{ft} = 23.0\ \text{ft}^2$$

Area Heads

$$4 \times \frac{\pi}{4}\left[\frac{(10)}{(12)}\right]^2 - 4 \times 48 \times \frac{\pi}{4}\left[\frac{(1)}{(12)}\right]^2 = 0.28\ \text{ft}^2$$

There is no area inside the low temperature heat exchangers available for warming operation.

$$\text{Therefore:}\quad \text{Total Area} = \begin{array}{r} 113.0 \\ 23.0 \\ 0.3 \\ \hline 136.3 \end{array}\ \text{ft}^2$$

5.25 Heat Transfer Coefficient for Warming Gases in Adsorber Shell

From nomograph in Clarke, "Manual for Process Engineering Calculations," Page 241, McGraw Hill Book Company, 1947.

Corss Sectional Area of Adsorbers:

$$\frac{\pi}{4}\left[\frac{(10)}{(12)}\right]^2 - 48 \times \frac{\pi}{4}\left[\frac{(1)}{(12)}\right]^2 = 0.544 - 0.261 = 0.283\ \text{ft}^2$$

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Flow = 60 SCFM = 270 lbs/hr

$G = 270/0.283 \times 3600 = 0.265$ lb/Sec. ft^2

do = 1 inch

Cp for N_2 at approximately 300°F = 0.245

$h/Cp = 4.7 \quad h = 4.7 \times 0.245 = 1.15$ Btu/hr ft^2 °F

$q = UADT$

$U = 1.15$ Btu/hr ft^2 °F

$A = 136.3$ ft^2

T = gas entering temperature

T_2= gas exit temperature $= T - q/(270 \times 0.24) = T - q/64.8$

T_1= Average Temperature of vessel

$q = 1.15 \times 136.3 \Delta t = 156.745 \Delta t$

$$\Delta t = \frac{(T-T_1) - (T_2-T_1)}{\ln \dfrac{T - T_1}{T_2 - T_1}} = \frac{q/64.8}{\ln \dfrac{T - T_1}{T - \dfrac{q}{64.8} - T_1}}$$

$$\Delta t = \frac{q/64.8}{\ln \dfrac{T - T_1}{T - T_1 - q/64.8}}$$

$$q = 156.8 \frac{q/64.8}{\ln \dfrac{T - T_1}{T - T_1 - q/64.8}} \qquad \ln \frac{T - T_1}{T - T_1 - q/64.8} = 2.42$$

$$\frac{T - T_1}{T - T_1 - q/64.8} = 11.20$$

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5.26 Calculation of Heat Transferred to WN 131 During Reactivation

$$\frac{T - T_1}{T - T_1 - q/64.8} = 11.2$$

$$\frac{T - T_1}{11.2} = T - T_1 - q/64.8$$

T_1	T	$T-T_1$	$\frac{T-T_1}{11.2}$	q/64.8	q	$\frac{1}{q} \times 10^{-5}$	Total q Trans at T	T_1 °C
-292	+150	442	39.4	402.6	26,100	3.83	0	-180
-250	+175	425	38.9	386.1	25,000	4.00	14,300	-157
-200	+200	400	35.7	364.3	23,600	4.24	31,400	-129
-150	+250	400	35.7	364.3	23,600	4.24	48,400	-101
-100	+300	400	35.7	364.3	23,600	4.24	65,400	- 73.3
- 50	+300	350	31.3	318.7	20,600	4.86	82,900	- 45.6
0	+300	300	26.8	273.2	17,700	5.65	100,000	- 17.8
+ 50	+325	275	24.6	250.4	16,200	6.17	117,000	- 10.0
+100	+350	250	22.3	227.7	14,720	6.79	134,000	37.8
+150	+350	200	17.9	182.1	11,800	8.55	151,000	66
+212	+350	138	12.3	125.7	8,150	12.28	171,500	100

Heat Transfer Increment x 10 + 3	Average $1/q \times 10^{-5}$	Time Hours	Total Hours
0-30	4.00	1.20	1.20
30-60	4.24	1.27	2.47
60-100	4.80	1.92	4.39
100-120	6.20	1.24	5.63
120-140	6.60	1.32	6.95
140-160	8.30	1.66	8.61
160-171	10.90	1.20	9.81
		9.81	

5.3 Estimation of the Desorption vs. Temperature and Time Curves

An attempt is made using the equilibrium curves to estimate the desorption of the gases from the beds at bed temperatures during reactivation. The vapor pressure of the gases condensing in cold traps WN 188 or nitrogen at the cold trap temperature. The other rare gas components will not have appreciable vapor pressures at this temperature. The calculation method is a trial and error procedure over small increments of temperature for which data are available. Bed compositions are assumed for the higher temperatures, a material balance then gives the mole fractions of the components in the desorbed gases and the H. C. mole fractions are compared with the partial pressures of the components left in the bed. This procedure would be exact if very small increments of temperature were used, but data are not available for the calculation.

Figure (10) gives the results of the desorption calculation and Figure (11) gives the total quantity of desorbed gas vs. time curve. The calculations show that for a final temperature of 100°C and 178 mm Hq. total pressure, only a negligible percent of the nitrogen, less than 2.2% of the Krypton, a negligible percent of the CO, 5.2% of the Xenon and 0.1% of the N_2O will remain. The concentration of the Xenon and N_2O remaining should not affect the subsequent operation of the bed.

John M. Holmes
John M. Holmes by W E Stockdale
FOSTER WHEELER CORPORATION

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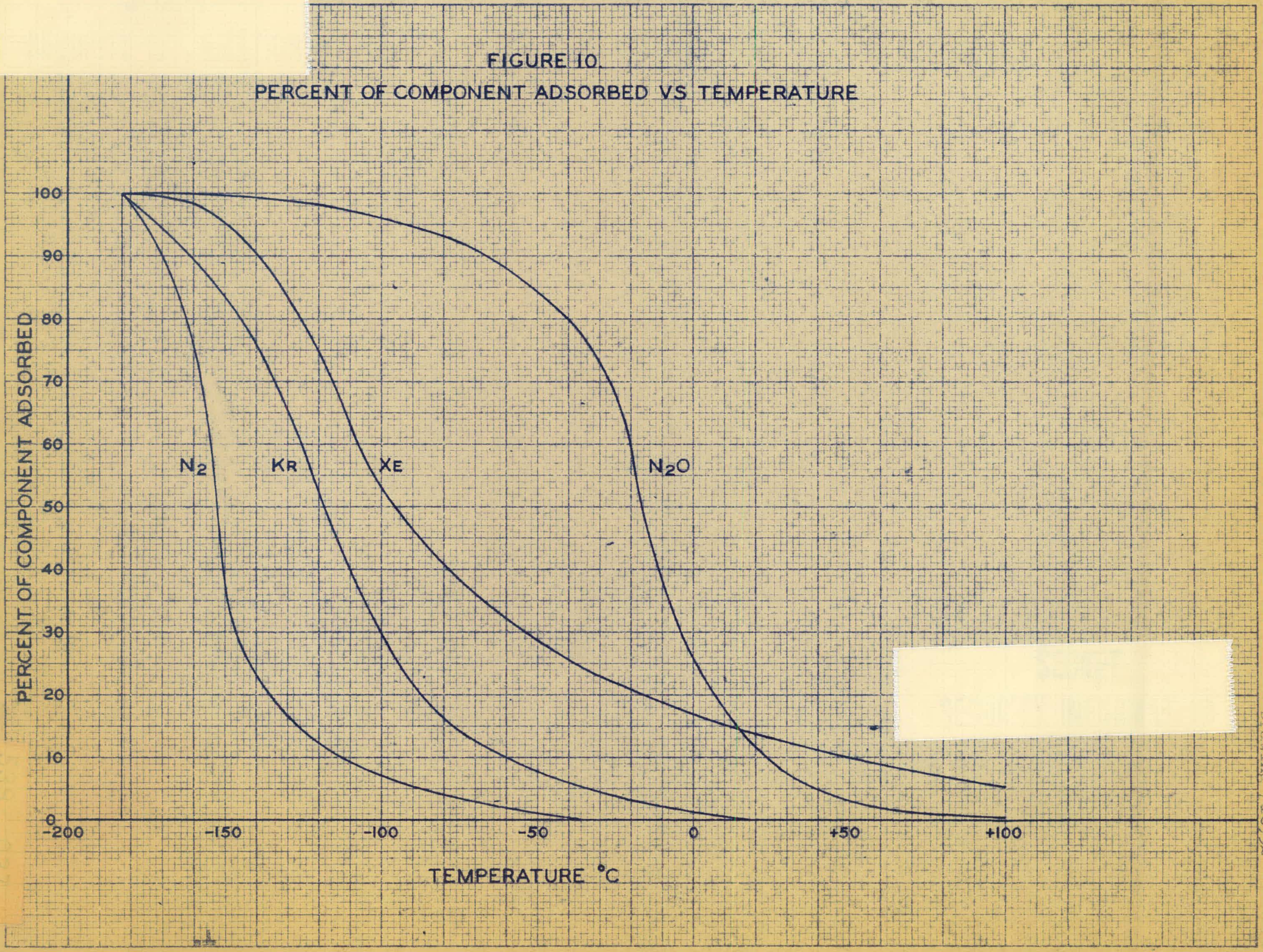

FIGURE 10.
PERCENT OF COMPONENT ADSORBED VS TEMPERATURE

Drawing #16952

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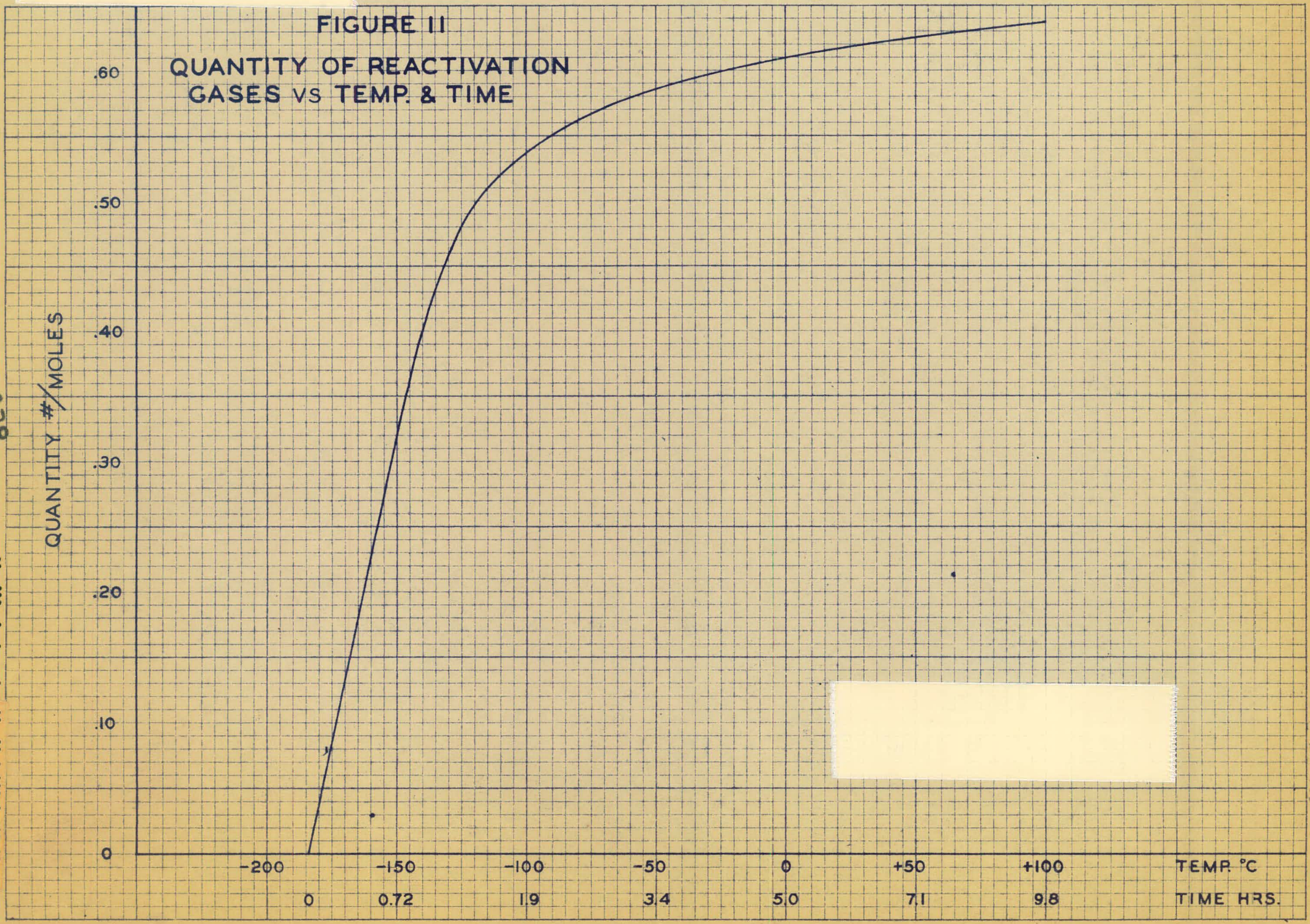

FIGURE II

QUANTITY OF REACTIVATION GASES VS TEMP. & TIME

Drawing #16953

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5.31 Calculation of the Desorption Vs. Temperature Chart

Temp. Range	Comp.	Comp		pp T_1 mm	Assumed Final pp	Comp.		# moles desorbed	mol%	Average Partial	%Comp. Remain	Moles Desorbed
		cc/gm	#moles			cc/gm	#moles					
-183	N_2+ A	335	0.4920	395	173.5	120	0.1755	.3165	97.2	98.0	35.8	
to	Kr	160	0.0210	0.047	0.5	133	0.0183	.0027	0.8	0.3	83.2	
-150	Xe	242	0.1218	0.274	2.0	233	0.1174	.0054	1.7	1.1	96.2	
	CO	6.5	0.0010	0.806	1.0	1	0.0001	.0010	0.3	0.6	15.4	
								.3256				0.3256
-150	N_2+ A	120	0.1755	173.5	127	41	0.0600	.1155	73.7	71.8	12.2	
to	Kr	133	0.0183	0.5	6	100	0.0131	.0043	2.7	3.4	62.5	
-120	Xe	233	0.1174	2	43	160	0.0808	.0369	23.5	24.3	69	
	CO	1	0.0001	1.0	1.0	neg.	neg.	.0001	0.1	0.5	0	
								.1568				0.4824
-120	N_2+ A	41	0.0600	127	82	13.2	0.0194	.0406	49.7	46.4	3.9	
to	Kr	100	0.0131	6	25	27	0.0035	.0096	11.8	14.1	16.9	
-80	Xe	160	0.0808	43	70	0.98	0.0495	.0313	38.4	39.6	40.4	
					177			.0815				0.5639
-80	N_2+ A	13.2	0.0194	82	75	7	0.0103	.0091	36	42	2.1	
to	Kr	27	0.0035	25	15	12	0.0015	.0020	7.9	8.5	7.5	
-50	Xe	98	0.0495	70	90	70	0.0354	.0141	56	51	29	
								.0252				0.5981
-50	N_2+ A	7	0.0103	75	10	0.5	0.0007	.0096	8.9	5.7	neg.	
to	Kr	12	0.0015	15	11	3.5	0.0004	.0011	9.9	6.2	2.2	
-0	Xe	70	0.0354	90	156	52	0.0263	.0091	81.2	88	21.5	
								.0198				0.6089

5.31 Calculation of the Desorption Vs. Temperature Chart (Cont'd)

Temp. Range	Comp	Comp cc/gm	Comp #moles	pp T_1 mm	Assumed Final pp	Comp. cc/gm	Comp. # moles	# moles desorbed	mol %	Average Partial	% Comp Remain	Moles Desorbed
0	Xe	52	0.0263	156	70	27	0.0137	.0126	36	39.6	11.2	
to	N_2O	363	0.0243		107 (13)	27	0.0018	.0225	64	60.4	7.5	
+25								.0351				0.6440
+25	Xe	27	0.0137	70	80	24.9	0.0126	.0011	44	45	10.3	
to	N_2O	27	0.0018	170	97 (13)	6.1	0.0004	.0014	56	55	1.7	
+50								.0025				0.6465
+50	Xe	24.9	0.0126	80	170	12.5	0.0063	.0063	94	96	5.2	
to	N_2O	6.1	0.0004	97	7 (13)	0.4	neg.	.0004	6	4	0.1	
+100								.0067				0.6532

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