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PROGRESS REPORT FOR NOVEMBER

on

DEVELOPMENT OF TEST UNIT FOR PRODUCTION OF OXYGEN BY A REGENERATIVE CHEMICAL

[illegible] affecting the [illegible]

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Arthur D. Little, Inc.

CHEMISTS·ENGINEERS

CAMBRIDGE, MASS.

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Office of Division B
National Defense Research Committee

OEMsr-269
Supplement No. 1

Date: Dec. 19, 1942

Progress Report for November on Development of Test Unit for Production of Oxygen by a Regenerative Chemical

Work during the past month at Arthur D. Little, Inc. on the performance of units with 35-pound powder capacity has been chiefly a continuation of the work described in the Progress Report of December 2, 1942.

The life test on the shallow-bed case loaded with 20-35 mesh Rumford High-High Salcomine has been stopped at 1350 cycles. At this point the powder had 25 per cent of its original saturation capacity and the case had produced 27.6 pounds of oxygen per pound of powder.

The deep-bed Thermek tube case was loaded with 10-20 mesh Rumford High-High Salcomine and run for 950 cycles. At this point the powder had 50 per cent of its original saturation capacity and had produced 21.8 pounds of oxygen per pound of powder. This coarser mesh powder gave a much lower initial pressure drop than the same powder in 20-35 mesh size. After 950 cycles it showed a pressure drop of 21 p.s.i. compared to nearly 60 p.s.i. for 20-35 mesh granules at 800 cycles.

A second shallow-bed case similar to the first one was loaded with 20-35 mesh Rumford High-High Salcomine powder and the performance of the case studied using cooling water at various temperature levels. With cooling water at 80°F. quite satisfactory operation can be expected with yields of 93 percent of that when using 50°F. cooling water. At higher temperatures the yields drop off rapidly.

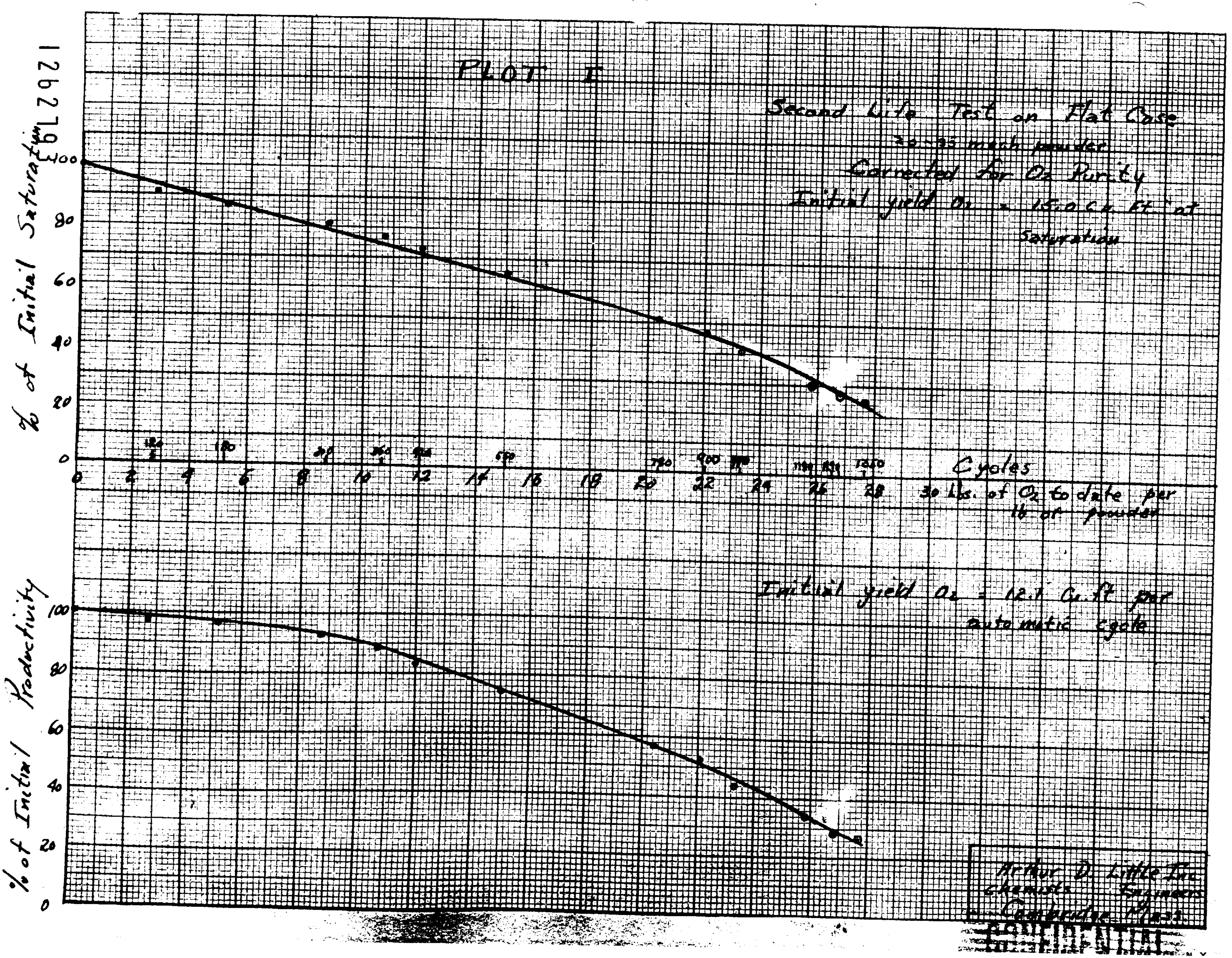
During the course of the early considerations of a design for a small medical unit the performance of Thermek tube cases of various lengths was studied. The results indicate that the oxygen yield at constant pressure and cooling water temperature is a function only of the time of absorption and the air rate per pound. The relative merits of various types of heat transfer surfaces has received consideration. Definite advantages for extended type surfaces have been shown over plain tubes. Pressure drop data for the deep and shallow beds used for the life tests were obtained.

Work has been started on the development of a simple apparatus for control evaluation of the various types of powder being used. An apparatus has been set up and tested in a preliminary way. A detailed description of the equipment will be presented in our next monthly report.

Life Tests

Since the report of December 2, 1942 was prepared two life tests have been in progress. The 20-35 mesh powder test described in our December 2nd report as the second Life Test on Shallow-Bed has been continued. A third Life Test has been carried out on the deep-bed unit using 10-20 mesh powder. A tabulated summary of the results obtained in these two tests are given in Table I and Plots I and II.

	Second Life Test Shallow-Bed	Third Life Test Deep-Bed
Pounds of Fully Absorbed Powder (Rumford High-High Salcomine)	36.1	33.0
Mesh Size	20-35	10-20
Air to unit, cfm.	8.0	7.27
Inlet air pressure, psi.	80.0	-
Outlet " " , psi.	-	80.0
Initial pressure drop, psi.	less than 1	3
Pressure drop after 200 cycles	" " "	4
400 "	" " "	6
600 "	" " "	12
800 "	" " "	16
1000 "	" " "	22 approx.
1200 "	" " "	
Initial Saturation Lbs. of O_2 per 100 lbs. of fully absorbed powder	3.84	3.58
Initial productivity lbs. of O_2 per 100 lbs. of fully absorbed powder.	3.1	2.72
Lbs. of O_2 produced per lb. of powder when powder has lost 40% of its initial saturation capacity (at 60% of initial capacity)	16.7	18



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PLOT I
Second Life Test on Flat Case
20-35 mesh powder
Corrected for O2 Purity
Initial yield O2 = 15.0 cu. ft. at saturation
% of Initial Saturation
100
80
60
40
20
0
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
Cycles
Lbs. of O2 to date per lb of powder
Initial yield O2 = 12.1 Cu. ft. per automatic cycle
% of Initial Productivity
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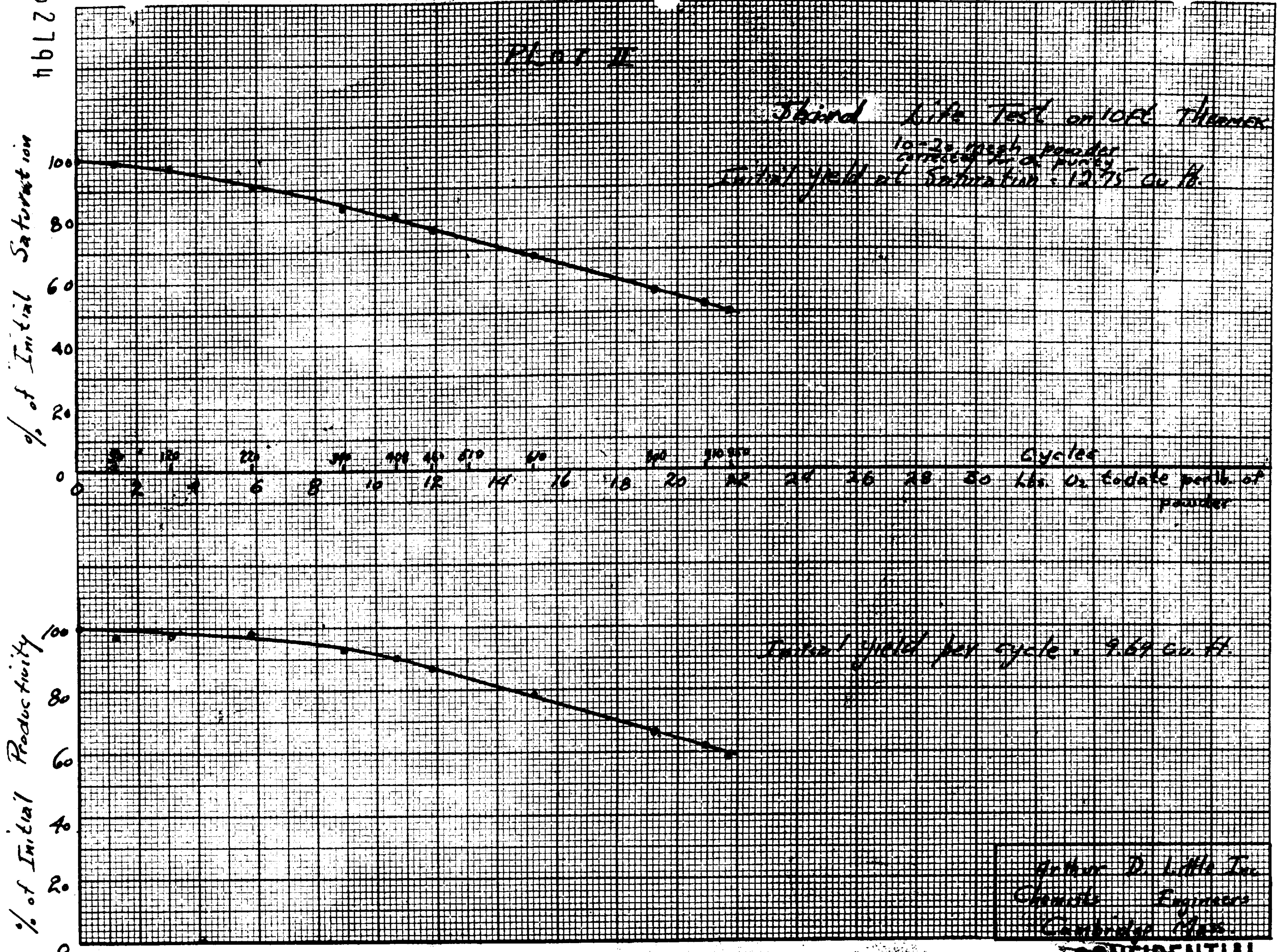
Cambridge, Mass.



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These tests have been completed and the units are currently being unloaded for inspection of the degraded powder.

It will be noted that the initial oxygen saturation values according to the table for the shallow-bed differ from the values given in the December 2nd report on this bed. A more detailed study of oxygen purity has been carried out in an attempt to correlate better the life test data on the larger cases with that obtained on the 1/2" tube units at M.I.T. It was found that the oxygen produced was not as pure as originally believed. This is due in part to the physical design of the beds and also in part to leakage of air past the inlet air values into the cases during desorption. This amount of air was sufficient to reduce the oxygen purity in the case of the shallow-bed to 86% for the entire amount of oxygen taken off during desorption. Corrections for oxygen purity have therefore been made on the data included in this report except where noted. In Plots III and IV no correction for oxygen purity has been made as it is not yet known just how the purity varies with the amount of oxygen desorbed.

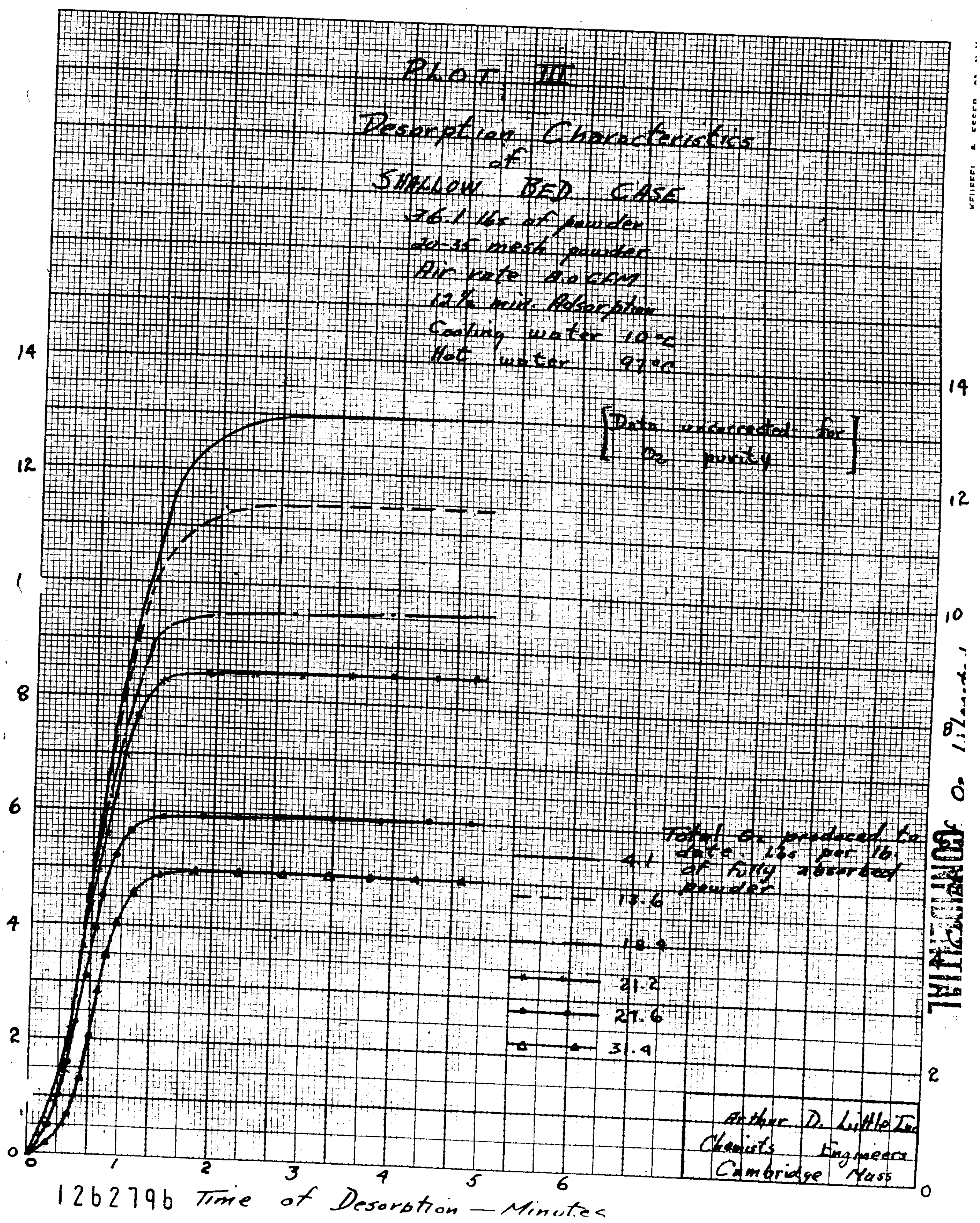
The shallow-bed shows with fresh powder an average oxygen purity of 92-94%. This purity drops off gradually with use of the powder since the amount of air introduced per cycle is a constant, and oxygen produced per cycle decreases.

Pauling meter data and desorption curves have been taken frequently to show the changes in performance characteristics as the beds aged. This data is given in Plots III, IV, V, and VI.

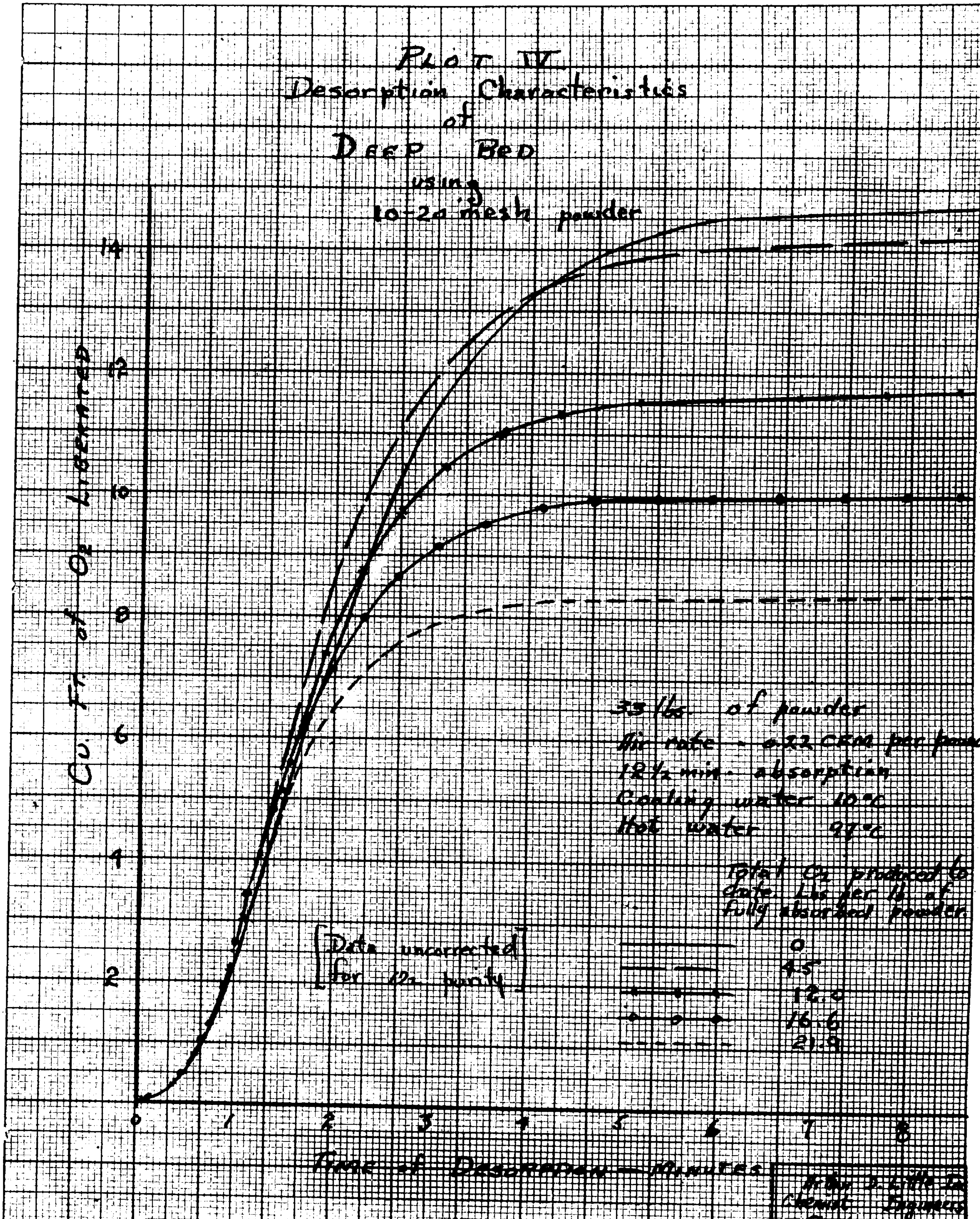
With the shallow-bed it is clearly seen that even after extended use of the powder 2 minutes is ample for desorption. Considerably slower desorption is shown for the deep bed case. To obtain over 90% desorption at least three minutes are required.

The continued degradation of the powder in the shallow-bed is seen clearly from the Pauling Meter Data for this case shown on Plot V. Absorption characteristics of the deep-bed using 10-20 mesh powder are shown in Plot VI.

It is interesting to note from both the absorption and desorption curves the further evidence of better action of the powder after some use. On the deep-bed desorption rates are higher after some use. On both beds the degree of stripping at 2 minutes is greater after some use of the powder.



PLOT III
Desorption Characteristics of SHALLOW BED CASE
36.1 lbs of powder
20-35 mesh powder
Air rate 8.0 CFM
12% min. Adsorption
Cooling water 10°C
Hot water 97°C
[Data uncorrected for O2 purity]
Total O2 produced to date. lbs per lb. of fully absorbed powder
4.1
13.6
18.9
21.2
27.6
31.4
0
2
4
6
8
10
12
14
0
1
2
3
4
5
6
Arthur D. Little Inc
Chemists Engineers
Cambridge Mass
12b2796 Time of Desorption — Minutes



PLOT IV
Desorption Characteristics
of
DEEP BED
using
10-20 mesh powder
14
12
10
8
6
4
2
0
1
2
3
4
5
6
7
8
CU. FT. of O2 LIBERATED
TIME of DESORPTION — MINUTES
35 lbs. of powder
Air rate - 0.22 CFM per pound
12½ min. absorption
Cooling water 10°C
Hot water 97°C
Total O2 produced to date. Lbs per lb. of fully absorbed powder
0
4.5
12.0
16.6
21.9
Data uncorrected for O2 purity
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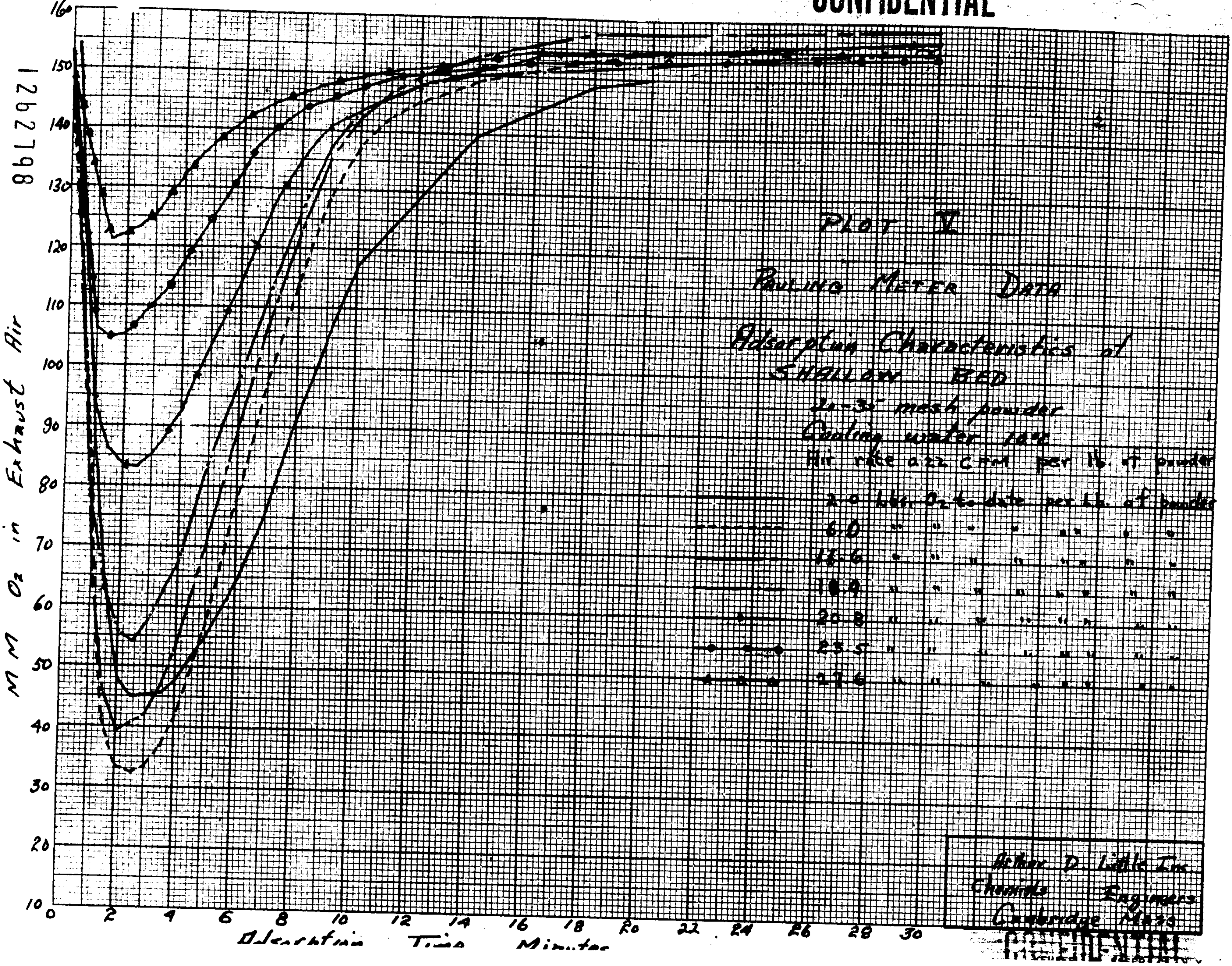

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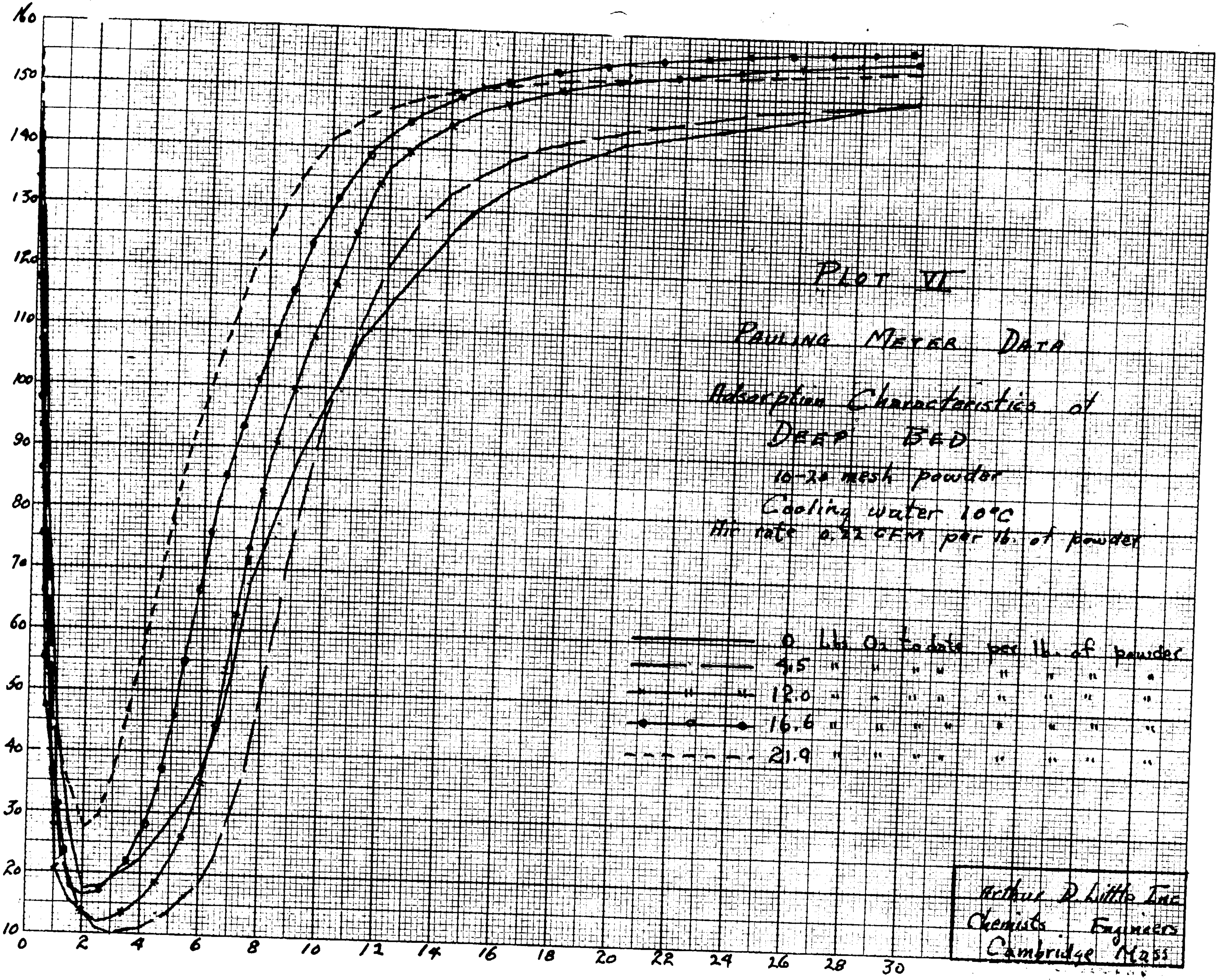
PLOT VI
PAULING METER DATA
Adsorption Characteristics of
DEEP BED
10-20 mesh powder
Cooling water 10°C
Air rate 0.22 CFM per lb. of powder
0 Lbs. O2 to date per lb. of powder
4.5 " " " " " " " "
12.0 " " " " " " " "
16.6 " " " " " " " "
21.9 " " " " " " " "
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160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30

1262799

On the basis of the work done to date with these cases it is felt now that the deep-bed does not offer sufficient advantages to warrant further work at present. The case shows high pressure drop build-up with use, poorer overall thermal action, awkward physical configuration, difficulty in loading and unloading. No further work is proposed on this type bed for the present.

Rumford Screening and Activity Tests

At the end of the Second Life Test on the deep-bed (800 cycles) the contents of tube #5 were fractionated into five portions from Top to Bottom. These portions plus some dusty powder in the very top of Tube #2 were submitted to Rumford for their screening and activity analysis.

The results of their tests are given in Table II and in the plot following the table.

The degree of segregation of fines seems to be less marked at this stage of powder use than after 244 cycles when similar tests were run before.

Their activity tests show some variation in degree of powder degradation from top to bottom with a maximum occurring part way up the tube rather than at the inlet end as might be expected.

The high activity of the fine dust from the top of tube #2 was quite unexpected. Probably this occurred because the powder at this point is not lying as close to the spined Thermek heating and cooling surface. Doubtless the powder therefore did not handle as much oxygen per pound during the 800 cycles as that in the body of the tube where the powder temperature was more nearly equal to the cooling water temperature during absorption.

TABLE II

Report on Recycled Salcomine from Thermek Units

At Arthur D. Little, Inc.

Six samples, designated as follows, were received:

1. "Top, Tube #5"
2. "Next to Top, Tube #5"
3. "Center, Tube #5"
4. "Next to Bottom, Tube #5"
5. "Bottom, Tube #5"
6. "Top, Tube #2"

All samples thru 800 cycles

Approximately 250 grams of samples 1-5 were used for screen analysis. Only 28 grams of #6 were available. The samples were screened on the Rotap for 25 minutes.

Screen Analysis

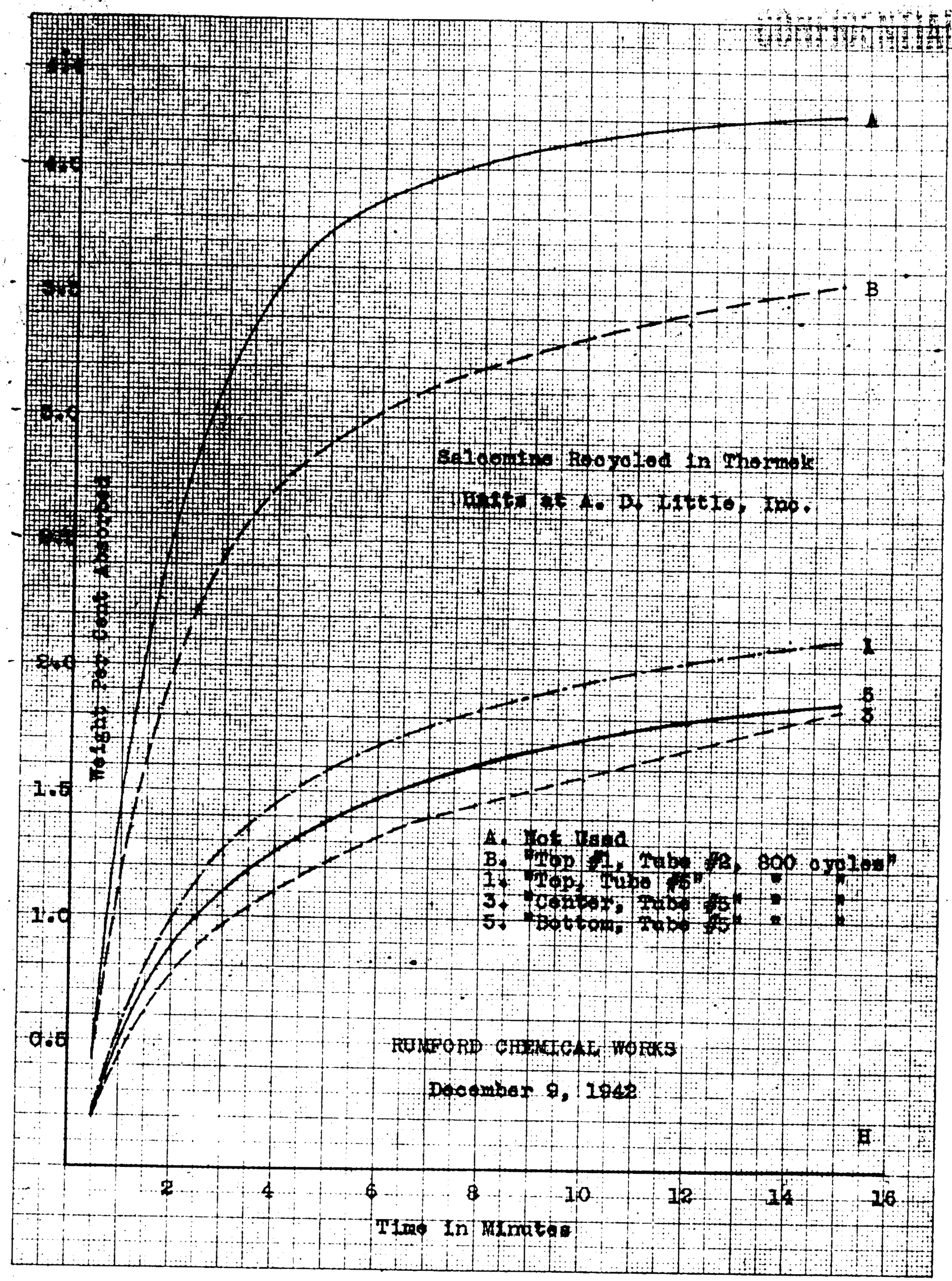
Mesh Sample:	1	2	3	4	5	6
28	35.72%	39.52%	37.87%	32.15%	36.48%	12.9%
28-35	34.63	37.35	39.78	40.48	38.68	16.4
35-48	12.74	11.05	10.16	13.49	11.38	12.9
48-65	4.95	3.68	4.01	4.62	3.98	12.5
65-100	4.05	2.66	2.79	3.70	3.08	13.1
100-150	2.70	1.87	2.80	1.71	2.47	13.1
150-200	3.65	2.53	1.66	2.11	2.58	11.0
200-325	0.50	0.70	0.37	1.18	1.07	4.6
-325	0.17	0.16	0.05	0.17	0.10	1.0
Mechanical Loss	0.89	0.48	0.51	0.39	0.18	2.5

Rates of Absorption

Determined in Differential Manometric
Apparatus at Room Temperature Using Oxygen at 35 psi. ga.

Weight Per Cent Absorbed

Time Min.	Time Sec.	Sample: 1	2	3	4	5	6
0	0						
	5						
	10						
	15						
	20	0.10		0.12			0.20
	25	0.15	0.12	0.17	0.20	0.14	0.33
	30	0.21	0.18	0.23	0.26	0.20	0.47
	35	0.31	0.20	0.26	0.31	0.23	0.62
	40	0.36	0.25	0.31	0.33	0.30	0.73
	45	0.40	0.29	0.35	0.40	0.36	0.84
	50	0.46	0.34	0.38	0.43	0.42	0.98
	55	0.50	0.37	0.42	0.46	0.46	1.09
1		0.54	0.39	0.45	0.50	0.50	1.20
	15	0.67	0.47	0.54	0.60	0.62	1.35
	30	0.80	0.56	0.61	0.66	0.72	1.64
	45	0.90	0.63	0.70	0.75	0.78	1.82
2		0.98	0.69	0.76	0.80	0.86	1.96
	15	1.07	0.75	0.82	0.85	0.93	2.09
	30	1.13	0.79	0.87	0.90	0.99	2.22
	45	1.21	0.85	0.92	0.94	1.05	2.36
3		1.24	0.91	0.96	0.99	1.09	2.44
	30	1.34	0.97	1.03	1.04	1.17	2.58
4		1.43	1.06	1.09	1.11	1.27	2.71
	30	1.53	1.11	1.16	1.18	1.33	2.80
5		1.59	1.17	1.22	1.23	1.38	2.89
6		1.68	1.27	1.32	1.31	1.47	3.02
7		1.76	1.36	1.41	1.39	1.55	3.12
8		1.84	1.45	1.46	1.46	1.61	3.20
10		1.93	1.59	1.56	1.57	1.71	3.29
12		2.03	1.73	1.68	1.69	1.79	3.38
15		2.12	1.89	1.84	1.84	1.87	3.55
Av. Total Capacity		2.46	2.10	1.98	1.95	2.12	3.59


Salcomine Recycled in Thermek
Units at A. D. Little, Inc.
Weight Per Cent Absorbed
Time in Minutes
0.5
1.0
1.5
2.0
2
4
6
8
10
12
14
16
A
B
1
3
5
H
A. Not Used
B. "Top #1, Tube #2, 800 cycles"
1. "Top, Tube #5" " "
3. "Center, Tube #5" " "
5. "Bottom, Tube #5" " "
RUMFORD CHEMICAL WORKS
December 9, 1942

1262803

Effect of Cooling Water Temperature

The design of a satisfactory regenerative chemical unit based on the use of Rumford "High-High" Salcomine necessitated a knowledge of the marked importance of the temperature of the cooling water that may be used. A shallow-bed case similar to that used for the life test was loaded with 20-35 mesh - Rumford "High-High" Salcomine and operated with cooling water at different temperatures. The same cycle used for the life tests was employed for these tests.

The data is given in Plots VII, VIII and IX. Plot VII gives the change in productivity and Plot VIII shows the change in saturation level with cooling water temperature. With water at 80°F. yields (on automatic cycles) of 90% of that possible with 50°F. water can be expected. With increasing water temperature, oxygen productivity falls off rapidly reaching zero at about 108°F.

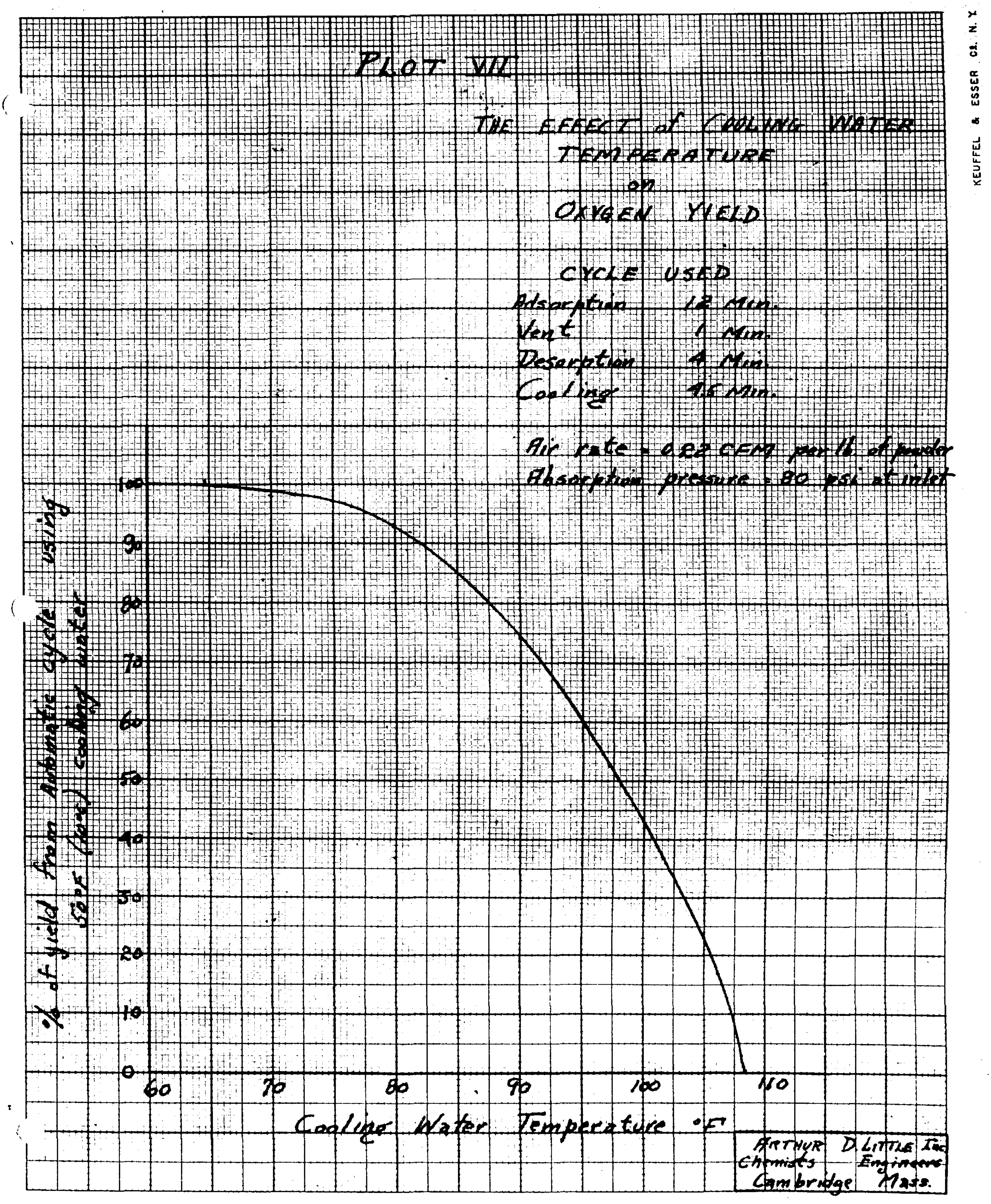
Plot IX shows the absorption curve characteristics when using cooling water at different temperature levels.

Performance of Various Length Thermek Reactors

The performance of various length Thermek reactors has been investigated to get data for the design of a 60-80 CFHr. medical unit. The following tubes were studied.

Tube Length	Spined Diameter
46"	2 7/16"
120"	1 3/4"
32"	1 1/2"
55 1/2"	1 1/2"

Absorption time and air rate through the powder were studied. The cycle used was as follows: Absorption for period desired using: 70-75°F. cooling water; inlet air pressure 80 psi. with flow controlled to a steady value on the outlet side of the tubes; vent to release 80 psi. pressure; desorption using low pressure steam; a purge after desorption consisting of blowing air into the reactors until 80 psi. pressure was built up and then releasing this before cooling; a cooling period to return the bed to approximately the cooling water temperature. Absorption was followed with the Pauling oxygen meter and the oxygen was liberated through a wet test gas meter. Oxygen yields and degree of air stripping were calculated from the Pauling meter data. The total amount of oxygen for the entire absorption period based on the Pauling meter readings checked the wet test meter yields to within at least 10% and in most cases to within 2 to 3%.


PLOT VII
THE EFFECT of COOLING WATER
TEMPERATURE
on
OXYGEN YIELD
CYCLE USED
Adsorption 12 Min.
Vent 1 Min.
Desorption 4 Min.
Cooling 4.5 Min.
Air rate = 0.22 CFM per lb of powder
Absorption pressure = 80 psi at inlet
% of yield from Automatic cycle using
50°F (10°C) cooling water
100
90
80
70
60
50
40
30
20
10
0
60
70
80
90
100
110
Cooling Water Temperature °F
ARTHUR D. LITTLE Inc.
Chemists Engineers
Cambridge Mass.
KEUFFEL & ESSER CO. N. Y.

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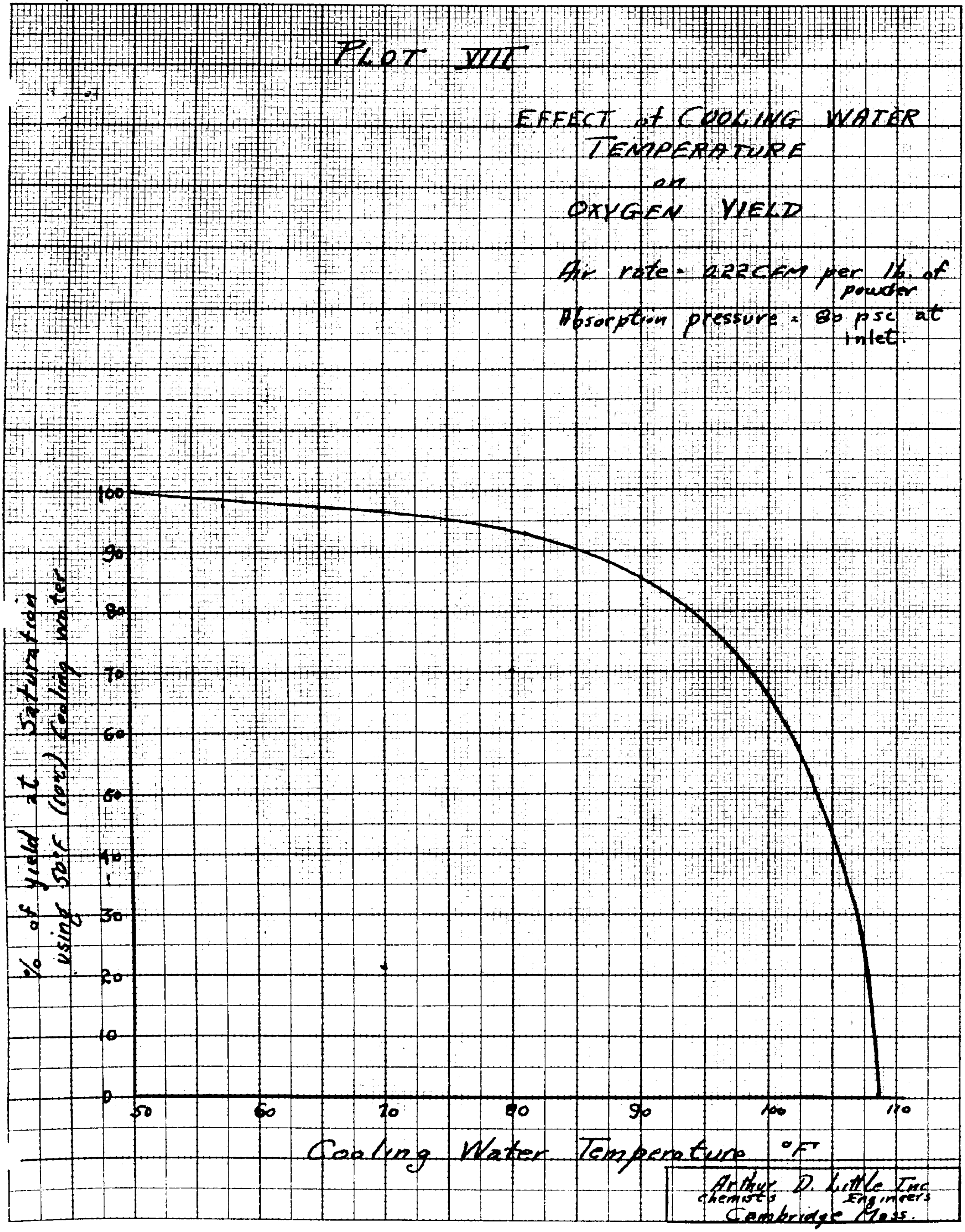
PLOT VIII
EFFECT of COOLING WATER TEMPERATURE on OXYGEN YIELD
Air rate = .222 CFM per lb. of powder
Absorption pressure = 80 psc at inlet.
% of yield at Saturation using 50°F (10°C) Cooling water
Cooling Water Temperature °F
0
10
20
30
40
50
60
70
80
90
100
50
60
70
80
90
100
110
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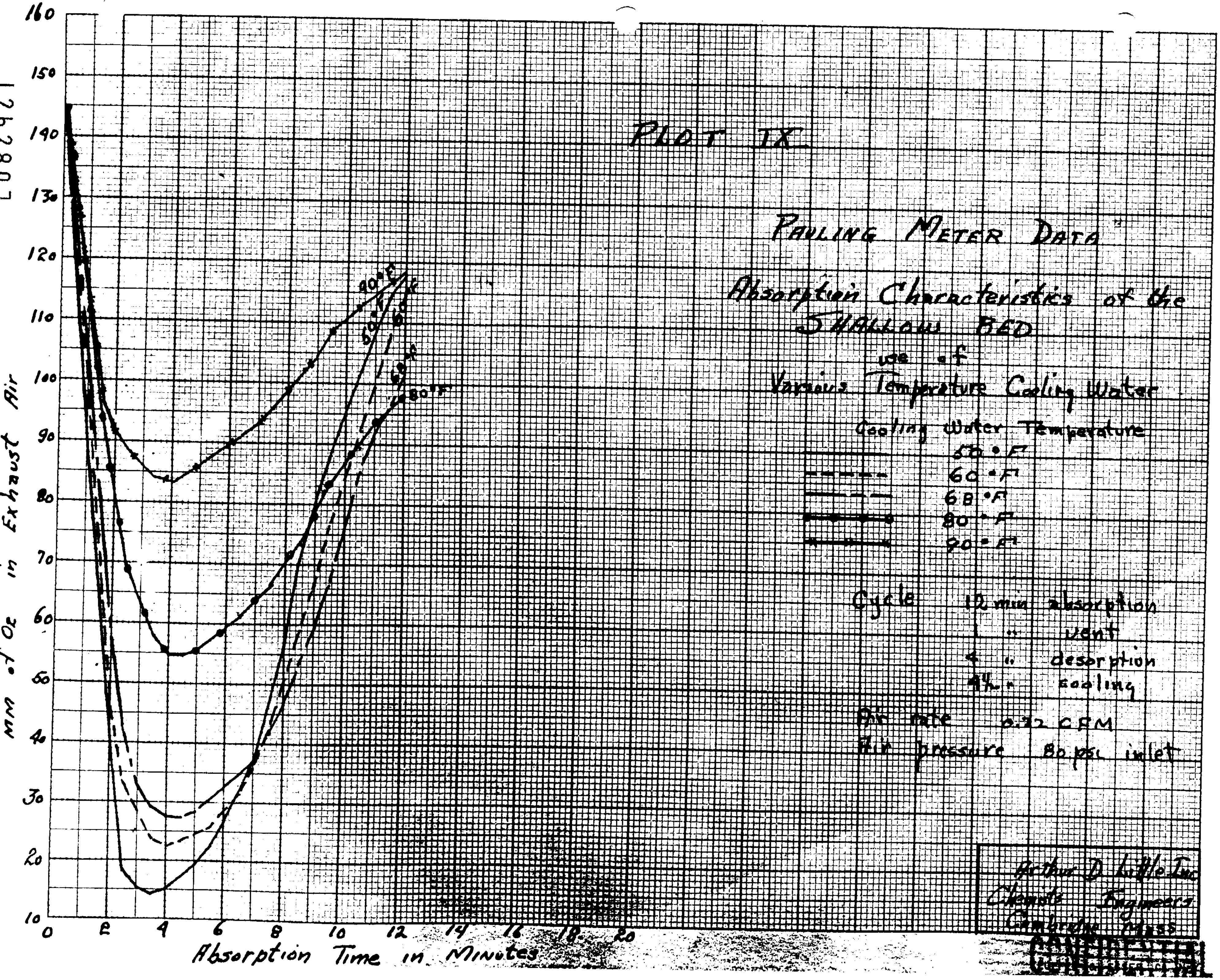
PLOT IX
"PAULING METER DATA"
Absorption Characteristics of the SHALLOW BED
use of Various Temperature Cooling Water
Cooling Water Temperature
50 °F
60 °F
68 °F
80 °F
90 °F
Cycle 12 min absorption
" vent
4 " desorption
4½ " cooling
Air rate 0.22 CFM
Air pressure 80 psi inlet
mm of O2 in Exhaust Air
Absorption Time in Minutes
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
4
6
8
10
12
14
16
18
20
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Plot X gives curves of volume efficiency and weight efficiency vs. air rate per pound of powder for various length reactors using an absorption time of 15 minutes. It is clearly seen that the data for the various tubes fall very well on a single curve. Plot XI shows this data plus that for other absorption periods. This data indicates quite clearly that for a given absorption period the controlling factor is the air rate per pound of powder.

Heat Transfer and Pressure Drop Data

In connection with the design of full scale units it was necessary to consider the relative value of plain tubes, spined Thermek tubes, and finned tubes as heat transfer surfaces for use in the regenerative chemical oxygen units. A general summary of the conclusions included in this report together with a plot of heat transfer coefficients and pressure drops for the various type surfaces. (See plot XII).

The pressure drop on the shallow and deep bed units used for the life tests described in our December 2nd report have also been determined. The data is shown by plots XIII and XIV.

HEAT TRANSFER SURFACES FOR USE IN CATALYST BEDS

Comparison of Weight, Volume and Friction loss of three types of surfaces on the service basis of 1000 BTU/1000 lbs. air/hr./1°F. M.T.D.

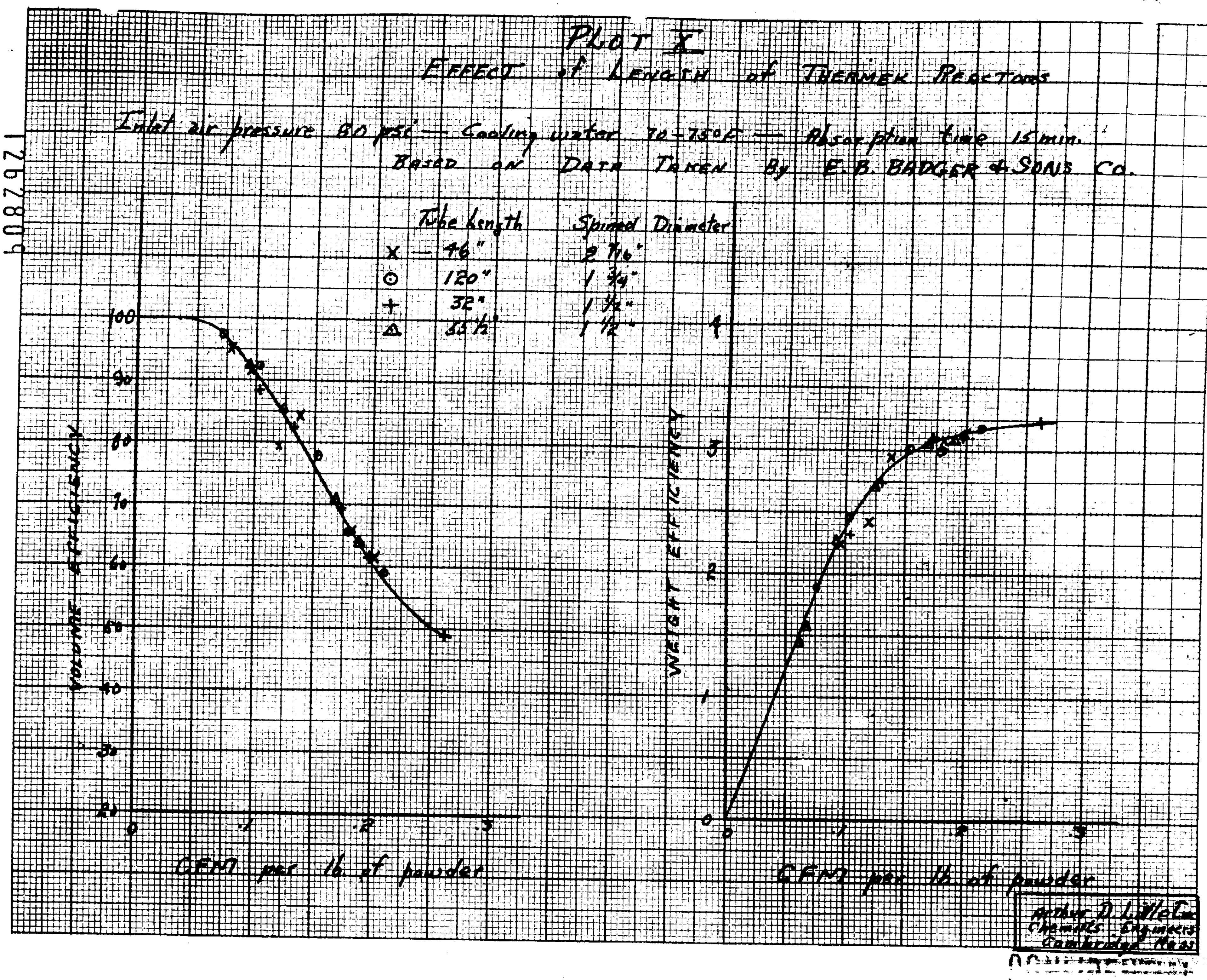
A) 1/2" ID #18 gauge copper tube

B) 1/2" C x 1-5/8 x 1/8 x 40 Thermek

C) #16 Nesbitt finned heating section

The catalyst has a very low heat conductivity and this fact must be considered in evaluating the specific advantages and disadvantages of the various surfaces.

A) 1/2" ID Copper Tube: The advantage of the low friction drop is likely to disappear when surfaces are compared in a catalyst filled condition. Large weight and volume would seem to relegate this surface to the least desirable ones. The need for filling and discharging the catalyst may however make this surface desirable for actual installations, especially stationary ones where weight is not


PLOT X
EFFECT of LENGTH of THERMEX REACTORS
Inlet air pressure 80 psi — Cooling water 70-75°F — Absorption time 15 min.
BASED ON DATA TAKEN By E. B. BADGER & SONS CO.
Tube length
Spined Diameter
X — 46" 2 7/16"
⊙ 120" 1 3/4"
+ 32" 1 1/2"
Δ 35 1/2" 1 1/2"
VOLUME EFFICIENCY
100
90
80
70
60
50
40
30
20
0
.1
.2
.3
CFM per lb. of powder
WEIGHT EFFICIENCY
4
3
2
1
0
0
.1
.2
.3
CFM per lb. of powder
Arthur D. Little Inc.
Chemists Engineers
Cambridge Mass
KEUFFEL & ESSER CO. N.Y.
1262809

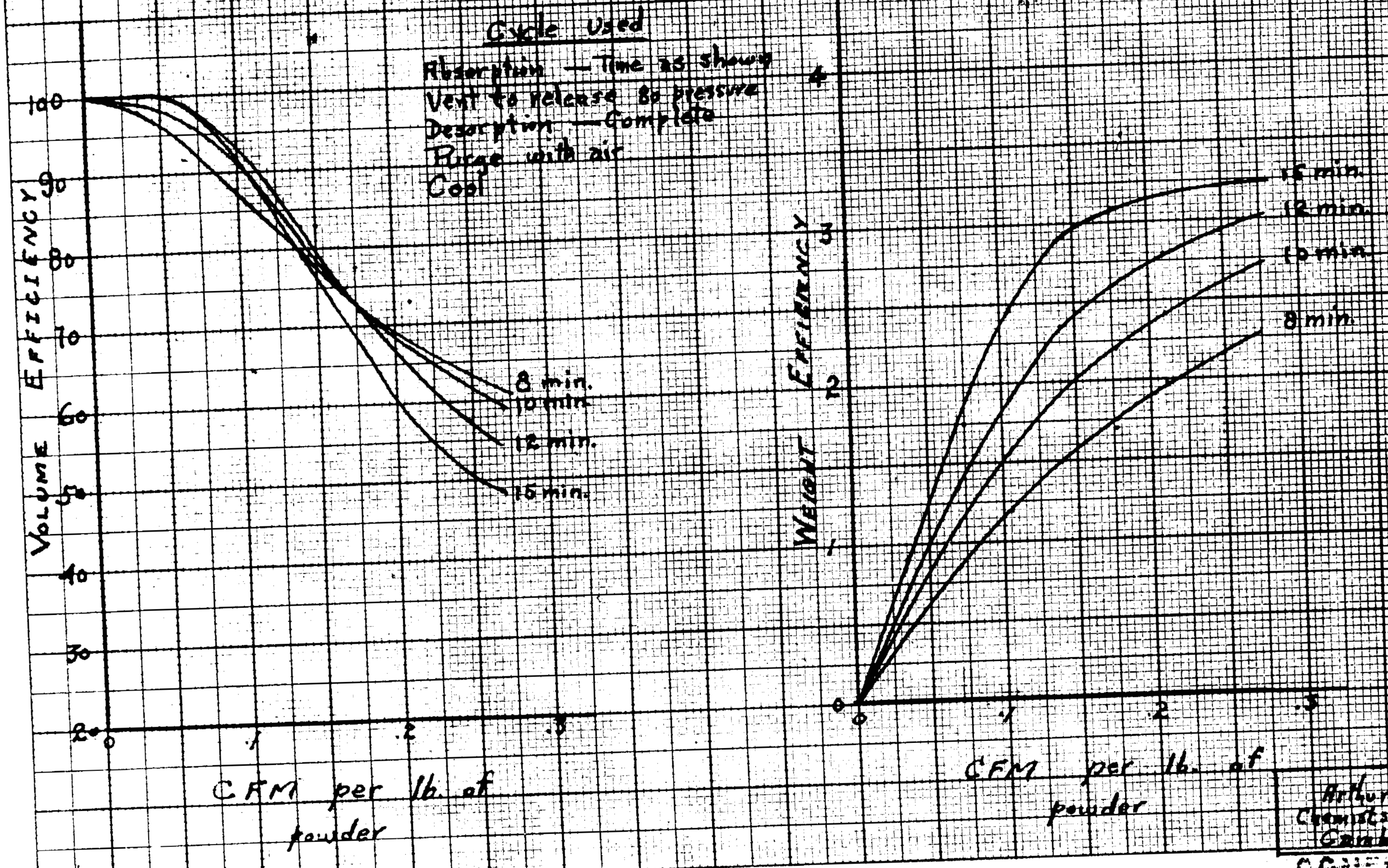
Plot XI

PERFORMANCE of VARIOUS LENGTH THERMEK REACTORS

All data from Thermek Tubes 30" to 120" long fall on curves plotted

Inlet air pressure - 80 psi. —— Cooling water 70-75°F —— Desorption using 1-2 psi steam

Based on Data taken by E. B. Badger & Sons Co.



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PLOT XII

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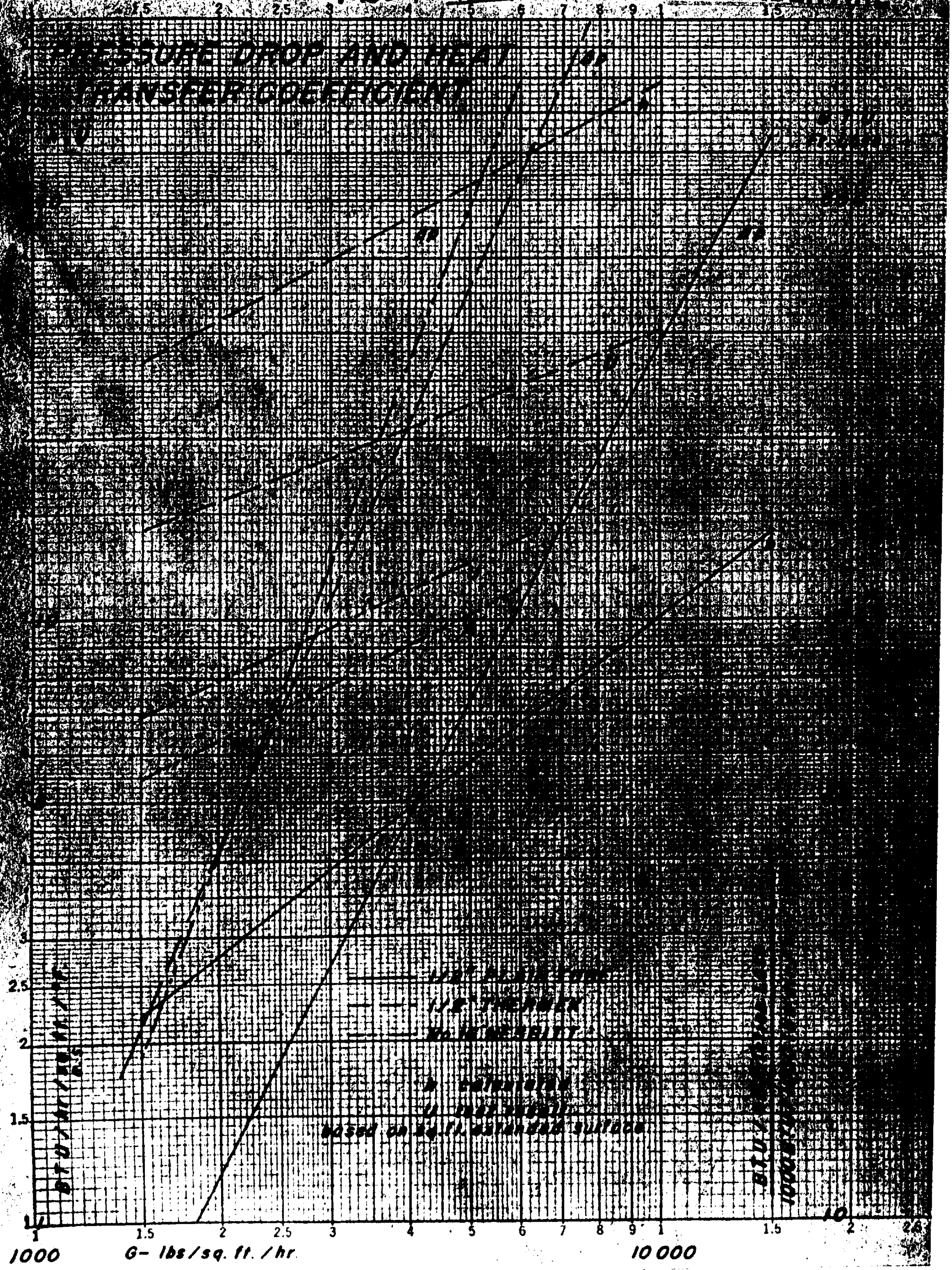

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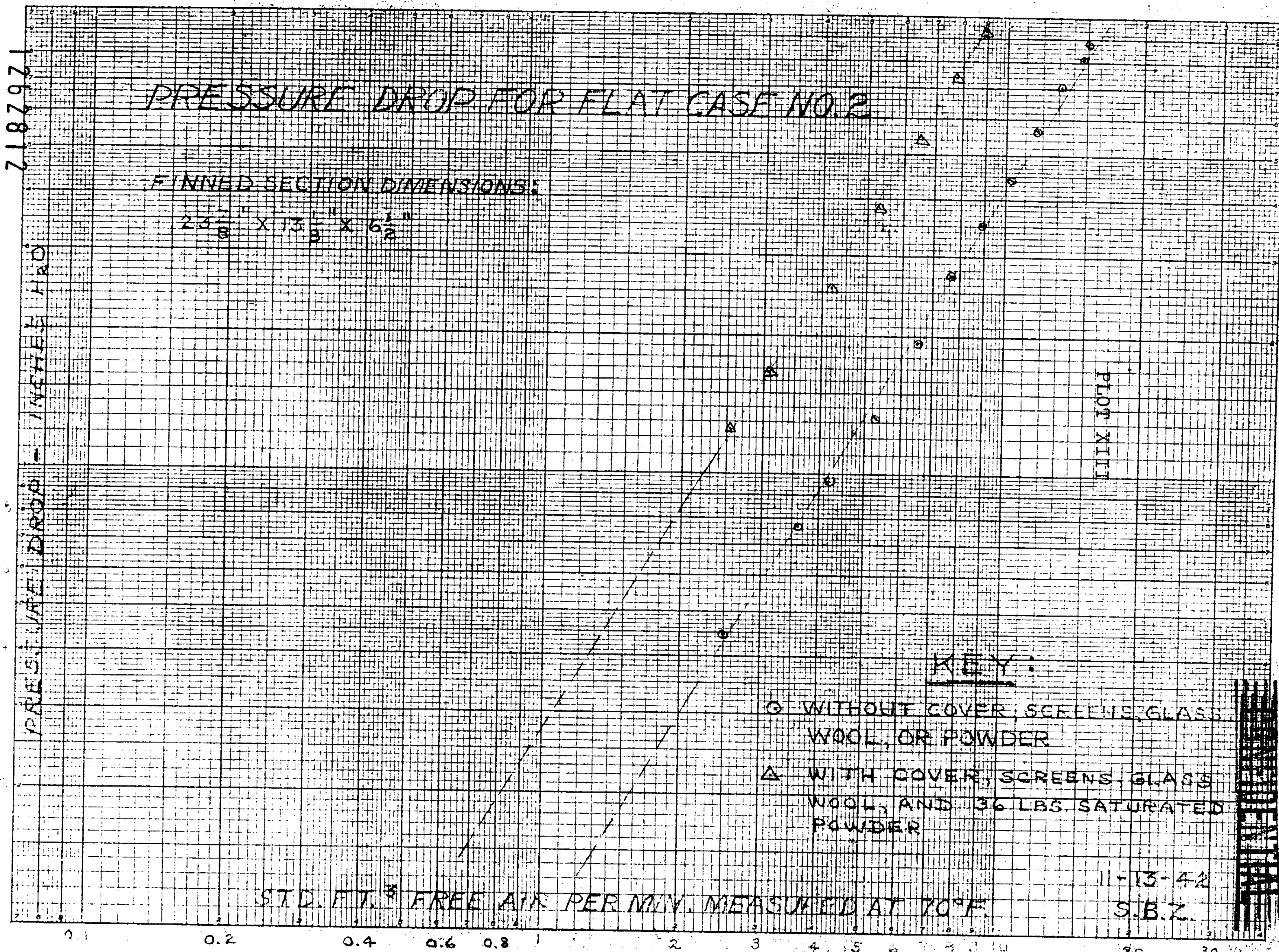
PRESSURE DROP FOR FLAT CASE NO. 2
FINNED SECTION DIMENSIONS:
23 5/8" X 13 1/8" X 6 1/2"
PLOT XIII
KEY:
⊙ WITHOUT COVER, SCREENS, GLASS WOOL, OR POWDER
△ WITH COVER, SCREENS, GLASS WOOL, AND 36 LBS. SATURATED POWDER
11-13-42
S.B.Z.
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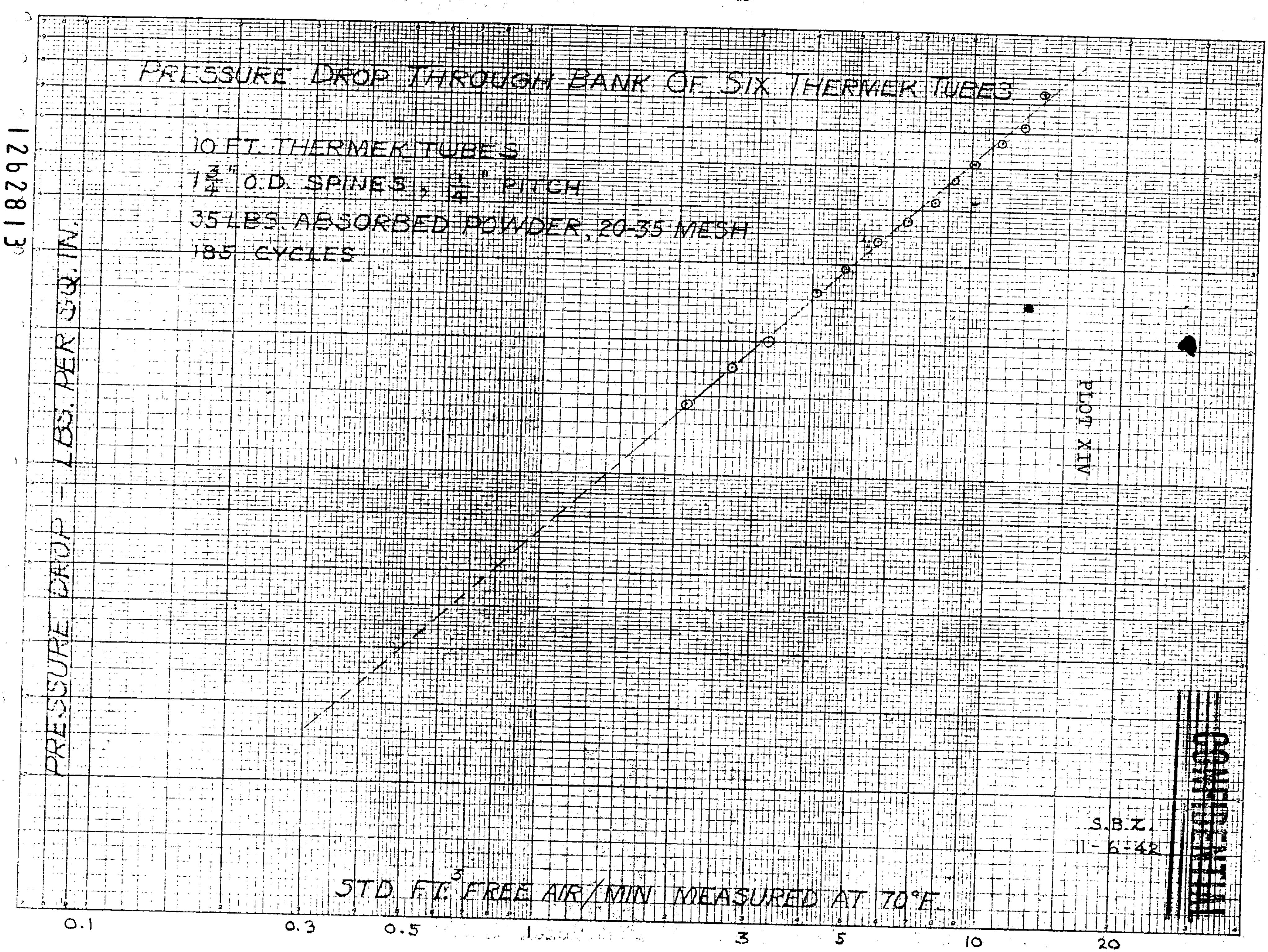
PRESSURE DROP - INCHES H2O
STD. FT.3 FREE AIR PER MIN. MEASURED AT 70°F.
0.1
0.2
0.4
0.6
0.8
1
2
3
4
5
20
30

1262812


PRESSURE DROP THROUGH BANK OF SIX THERMEK TUBES
10 FT. THERMEK TUBES
1 3/4" O.D. SPINES, 1/4" PITCH
35 LBS. ABSORBED POWDER, 20-35 MESH
185 CYCLES
PLOT XIV
PRESSURE DROP - LBS. PER SQ. IN.
STD FT.3 FREE AIR/MIN MEASURED AT 70°F.
0.1
0.3
0.5
1
3
5
10
20
S.B.Z.
11-6-42
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a factor. The disadvantages of fairly thick catalyst layers can possibly be ameliorated by loose metal spirals or ribbons. The low heat density per lb. of catalyst due to large catalyst volume may also prove a help.

B) 1/2" x 1-5/8 x 1/8 x 40 Thermek in 1 1/2" ID shell: Weight is greatly reduced (less than 1/10 of plain tubes). Thermek provides the largest transfer surface per lb. of catalyst with surface very finely divided. Compared with weight and cost saved the power cost is negligible. Calculations have shown that the temperature gradient along the spines is very small. Loading and unloading may prove difficult and spines may suffer badly if this operation has to be repeated in actual use. The small interstices between spines limit the particle size of the catalyst resulting in a high pressure drop and consequently movement and abrasion of catalyst grains.

C) #16 Nesbitt finned tube sections afford almost the same low weight per unit of transfer service as the Thermek tubes. Volume of catalyst and surface are larger than with Thermek resulting in a lower heat density during the operating cycle. The increased heat capacity due to a larger catalyst volume is small and is not expected to be noticeable in actual operation. The uniform fin spacing suggests the use of a larger particle size catalyst resulting in a lower pressure drop and materially lower catalyst disintegration due to less motion and abrasion. Loading and unloading of catalyst will be difficult although fins will possibly stand up better than spines. A shell to provide for individual insertion of finned sections which appears necessary to load the catalyst presents design difficulties, the more so since bypassing of gas around the catalyst must be avoided and the finned sections with their headers will need to be given a possibility for heat expansion. Design difficulties appear to be the most serious drawback for this type of surface.

Comparative data for various heat transfer surfaces on a performance basis of 1000 BTU/1000 lbs. air/hr./1°F M.T.D. *

	1/2"ID #18 Copper Tube		1/2"Cu Thermek in 1 1/2" ID tube		#16 Nesbitt Finned Tubes	
G-Mass vel: lbs./hr/sq.ft.	1500	2500	1500	2500	1500	2500
Net Weight of Active Surface	890	590	32	23	56	43
Approx. Gross Weight of equipment incl. case						
Transfer surface, sq. ft.	448	295	72	52	147	112
Catalyst volume, cb. feet	4.67	3.08	.30	.22	.86	.66
Ratio Surface / catalyst vol.	96	96	240	240	171	171
Net Volume of surface	6.3	4.1	.41	.29	1.07	.82
Gross Volume of Equipment						
Friction loss KW Hrs. 1000 hrs. operation	2.14	4.48	6.3	20.5	7.04	24.3
Yearly power cost $ 8000 hrs./.8@/KW Hr.	.14	.29	.404	1.31	.45	1.56
Net cost of surface $	666	443	32	23	56	43
Approx. cost of equipment incl. case and headers						

* Methods described in McAdams "Heat Transmission" 2nd Ed. were used in arriving at values shown.

Respectfully submitted,

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