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PROGRESS REPORT FOR DECEMBER

on

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

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Arthur D. Little, Inc.
CHEMISTS·ENGINEERS
CAMBRIDGE, MASS.

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BY AUTH. CG-DAR-1 431
BY David L. Ar ADC DATE 3/20/96
By N E Broderick ADD 3/20/96

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In going over our December report, we note with regret the omission of the titles for the various columns of Table I on page 4. We are enclosing herewith a corrected copy of this table.

Will you please remove the corresponding page of the December Report, replacing it with the enclosed page.

Arthur D. Little, Inc.

ARTHUR D. LITTLE, INC.

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TABLE I

SALCOMINE STUDIES

Key No.	Cycle No.	Saturation Yield C.F. O_2	Percent of init. sat.	Absorption Time in min.	Air pressure psi. ga.	Cooling Water Temp. °F.	Air rate CFM/lb.	Desorp. Pressure	Temp. °F. of water for Desorp.	Total yield O_2 C.F.	Total yield O_2 lbs. per lb. of powder
0	3C	14.6	100	12	80	50	.22	atm.	204	50.4	.13
1	104C	14.2	97.2	12	80	60	.22	"	"	1268.6	3.16
2	212C	12.4	84.9	12	80	80 & 90	.22	"	"	2337.4	5.83
3	281C	12.0	82.2	12	80	100	.22	"	"	2722.2	6.80
4	358C	11.4	78.1	various short cycles						3331.3	8.31
5	671C	10.0	68.5	5.75	80	50	.42	"	"	5652.6	13.85
6	1008C	9.3	63.6	5.75	110	"	"	"	"	8206.8	20.50
7	1191C	9.1	62.3	"	"	"	"	vac	"	9696.2	24.20
8	1487C	8.24	56.4	"	"	"	"	"	"	11943.2	29.80
9	1640C	8.1	55.5	"	"	"	"	"	180	13016.0	32.50
10	1791C	7.45	51.0	"	"	"	"	atm.	204	13922.3	34.80
11	1947C	7.03	48.2	"	"	"	"	vac.	180	14899.1	37.20
12	2127C	6.95	47.6	"	"	"	"	"	204	15989.7	39.90
13	2245C	6.45	44.2	"	"	"	"	"	160	16735.0	41.80
14	2530C	5.54	38.0	"	"	"	"	"	204	18349.2	45.80

Note. Mode of operation between any two successive key numbers was that of the last of the two key numbers.

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Office of Division 11
National Defense Research Committee

OEmsr-269
Supplement No. 1

Date: February 1, 1943

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

The work on this project at Arthur D. Little, Inc. during December has included further studies of Salcomine; a preliminary evaluation of Ethomine in 35 pound powder flat bed cases; a study of the quality of the oxygen produced by these two powders with particular emphasis on carbon containing materials in the gas; an evaluation of the effect of air quality on the productivity of Salcomine; and the designing and construction of an air actuated bellows valve.

In continuing the studies on Salcomine interest has been directed towards obtaining the maximum oxygen yield per hour per pound of powder. Furthermore it was desired to know what factors limit the yield over and beyond the powders own inherent maximum capacity. It now appears that with Rumford High-High Salcomine a yield of 15-17 weight percent per hour from flat bed cases is feasible when using air at 80-100 psi. ga., cooling water at 50°F. for absorption and hot water at 204°F. for desorption with air rate of 0.42 CFM per pound. The yield appears to be chiefly controlled by the heat transfer characteristics of the powder bed. Methods of operation which increase the heat transfer capacity of the bed increase the rate of absorption and consequently the yield.

A life test has been run on Ethomine in the 35 pound shallow bed cases. An air rate of 0.6 CFM per pound with air pressure of 20 psi. ga. was used in conjunction with cooling water at 102°F. for absorption and 15 psi. ga. steam for desorption. An average vacuum of 20" of mercury was maintained during desorption. After the powder had produced 15 pounds of oxygen per pound of powder its saturation level was 37% of its initial saturation and its productivity was 41% of its initial productivity.

A number of tests have been run on the oxygen produced by Salcomine and Ethomine to determine the amount and nature of the carbon containing materials in the oxygen. These tests have included the palladium chloride test, carboxyhemoglobin test, and the use of an absorption train and combustion furnace. Our present data indicate that the oxygen from Rumford High-High Salcomine contains CO_2, but no CO or other carbon containing materials. The CO_2 content is about 1200-1500 ppm. The oxygen from Rumford Ethomine contains less CO_2, namely about 540 ppm., but appears to

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contain about 10-15 ppm. of CO and some other carbon and hydrogen containing material. This latter may be ethoxy-salicyaldehyde.

Tests have been run on the productivity of Salcomine in the 1/2" tube units using in one case air passed through silica gel driers and then further purified by passing through charcoal contrasted with air only passed through silica gel. The data obtained does not appear conclusive although there is an indication that the charcoal treated air is to be preferred. Oil was definitely removed from the inlet air in the charcoal treated air.

A second test on Salcomine in the 1/2" tubes has also been run using air with a dew point of -3.5°C. compared to silica gel dried air with a dew point of at least -30°C. The curves of productivity vs. cycles while displaced one above the other run parallel and do not therefore seem to show any positive advantage for one type of air over the other.

Rumford High-High Salcomine was used on the above two tests.

Some difficulty has been experienced with the air operated cup washer piston type valves used on the pilot plant set up. A new air operated valve has been designed and built and put into cyclic use on steam service. This valve incorporates a Hydron Bellows as the actuating member. Service to date has been excellent.

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Body of the Report

I. Salcomine Studies

As a part of the studies being carried on with Salcomine in connection with its use in a shipboard unit an hourly yield of 10 weight percent was set as a goal. Inspection of absorption curves such as shown in plot II indicated that to get the highest absorption rate the compound should not be allowed to pick up too much oxygen per cycle.

Inspection of the desorption curves such as given in the previous report indicated that 2 minutes was probably quite sufficient time for desorption.

To build up further the hourly yield it seemed desirable to cut down the cooling period as much as possible.

A variety of cycles were accordingly tried and quite complete data taken on each. The various cycles tried are tabulated in Table II and the point in the powder life at which they were run is indicated in Table I (cycles 281-358). The powder used for these cycles had been previously run to determine the effect of cooling water temperature.

In Table II the column labeled "Actual O_2 yield per hr., wt. %" is corrected for oxygen purity while the column labeled "Wt. % O_2 per hour 2 min. desorption" is a calculated value assuming the absorption and cooling time indicated but with two minutes desorption. Plots of some of this data are given in Plots II, III.

For the Shallow Bed cases used at Cambridge and cooling water at 50°F. certain factors appear established. An air rate of over 0.42 CFM per lbs. of powder is without benefit. The use of a cooling period does not increase the yield per cycle sufficiently to warrant its use. The reason for this is seen in plot II.

About 5-6 minutes absorption time appears the best. The use of higher air pressure gives a very real increase in yield per hour. A cycle was chosen as follows and a sufficient number of cycles were run to establish the slope of life test saturation curve: absorption time, 5 1/2 min.; vent, 1/2 min.; desorption time, 2 min. A marked improvement in the saturation curve was noted over the original long cycle used on the previous work with this case. In the first test period with the new cycle 80 psi. ga. air was used at 0.42 CFM air/lb. powder.

Next the air pressure was increased to 110 psi. ga. and the same air rate used. Again the life test saturation curve improved.

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TABLE I

SALCOMINE STUDIES

Key No.											
0	3C	14.6	100	12	80	50	.22	atm.	204	50.4	.13
1	104C	14.2	97.2	12	80	60	.22	"	"	1268.6	3.16
2	212C	12.4	84.9	12	80	80 & 90	.22	"	"	2337.4	5.83
3	281C	12.0	82.2	12	80	100	.22	"	"	2722.2	6.80
4	358C	11.4	78.1	various short cycles						3331.3	8.31
5	671C	10.0	68.5	5.75	80	50	.42	"	"	5652.6	13.85
6	1008C	9.3	63.6	5.75	110	"	"	"	"	8206.8	20.50
7	1191C	9.1	62.3	"	"	"	"	vac	"	9696.2	24.20
8	1487C	8.24	56.4	"	"	"	"	"	"	11943.2	29.80
9	1640C	8.1	55.5	"	"	"	"	"	180	13016.0	32.50
10	1791C	7.45	51.0	"	"	"	"	atm.	204	13922.3	34.80
11	1947C	7.03	48.2	"	"	"	"	vac.	180	14899.1	37.20
12	2127C	6.95	47.6	"	"	"	"	"	204	15989.7	39.90
13	2245C	6.45	44.2	"	"	"	"	"	160	16735.0	41.80
14	2530C	5.54	38.0	"	"	"	"	"	204	18349.2	45.80

Note. Mode of operation between any two key successive key numbers was that of the last of the two key numbers.

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TABLE II

PRODUCTIVITY OF SHALLOW BED ON VARIOUS CYCLES

1/4 minute to vent high pressure air
at end of absorption

Air rate CFM per lb.	Inlet air pressure psi.	Absorp. Time	Desorp. Time	Cooling Time	Total Cycle Time	Actual Yield/cycle-C.F.	Wt. % per hour ass. yield 100% O_2	Actual O_2 yield per hr. wt.%	Wt.% O_2 per hour 2 min. desorption	Remarks
.22	80	12	4	4 1/2	20 3/4	11.14	8.05	7.37	8.00	Basic cycle used on Life Tests
.22	80	12	4	2	18 1/4	11.10	9.11	8.34	9.21	
.22	80	12	4	1	17 1/4	11.06	9.60	8.80	9.78	
.22	80	12	4	0	16 1/4	10.76	9.92	9.06	10.20	
.22	80	12	4	2	16 1/4	10.60	9.77	8.92	10.02	
.22	80	10	4	1	15 1/4	10.46	10.28	9.37	10.62	
.22	80	10	4	0	14 1/4	10.10	10.62	9.65	11.10	
.22	80	8	4	1	13 1/4	9.30	10.51	9.50	11.08	
.22	80	8	4	0	12 1/4	8.85	10.80	9.69	11.50	
.22	80	7	4	0	11 1/4	7.81	10.40	9.20	11.10	
.22	80	7	4	1	12 1/4	8.60	10.51	9.38	11.15	
.22	80	9	4	0	13 1/4	9.53	10.78	9.75	11.35	
.42	80	5	4	0	9 1/4	8.05	13.03	11.56	14.67	Increased air rate
.42	80	4	4	0	8 1/4	6.40	11.62	9.98	13.10	
.62	80	3	2	0	5 1/4	4.76	13.60	11.03	11.03	Increased air rate
.62	80	4	2	0	6 1/4	6.45	15.48	13.30	13.30	
.75	80	2	2	0	4 1/4	2.38	8.40	5.22	5.22	" " "
.75	80	3	2	0	5 1/4	4.75	13.60	11.03	11.03	
.42	110	5	2	0	7 1/4	9.0	18.56	16.70	16.70	Increased air pressure.

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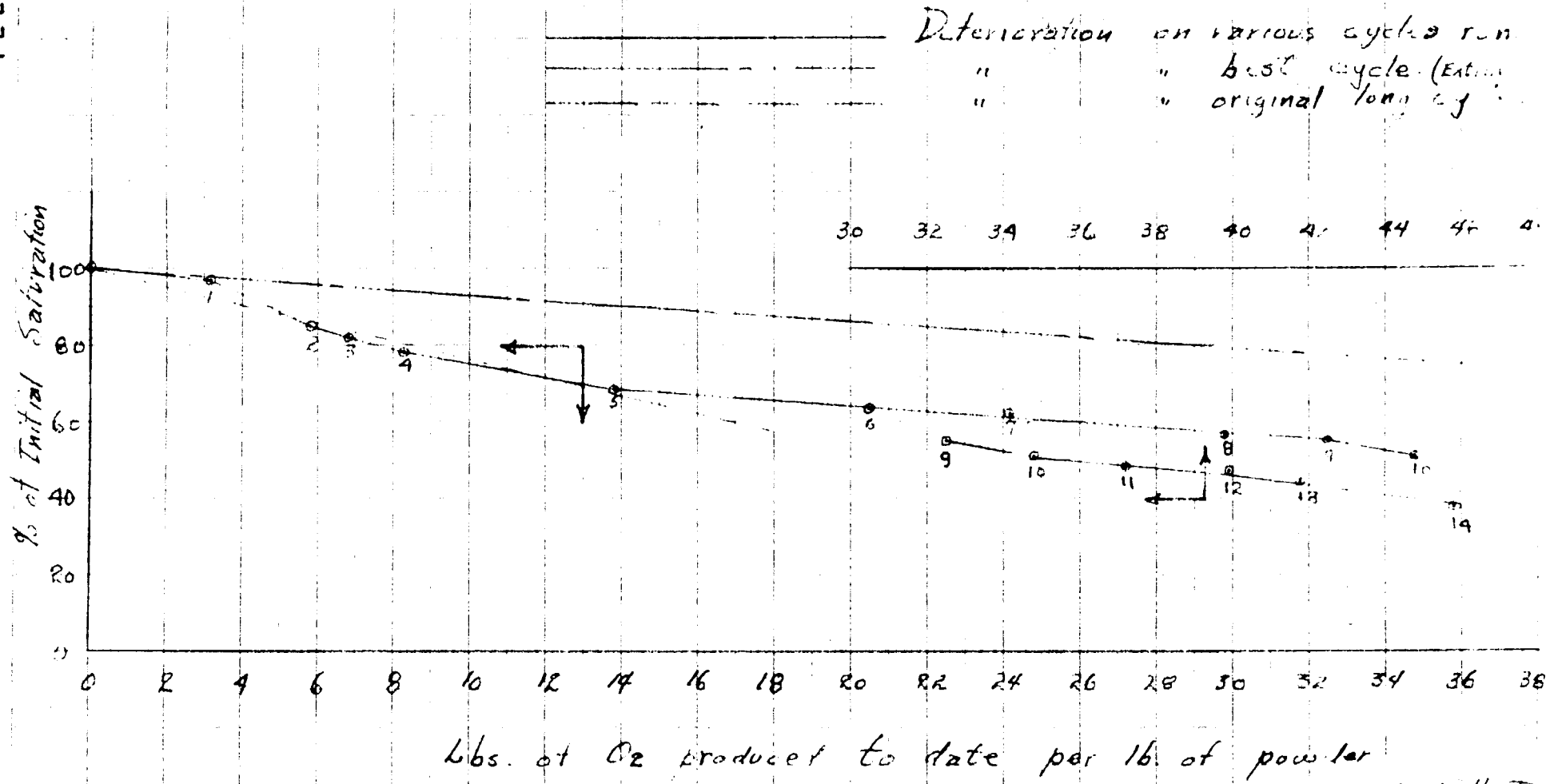
PLOT I

CYCLIC OPERATION of

RUMFORD High High SALCOMINE

See Table I for Key to numbers on main curve



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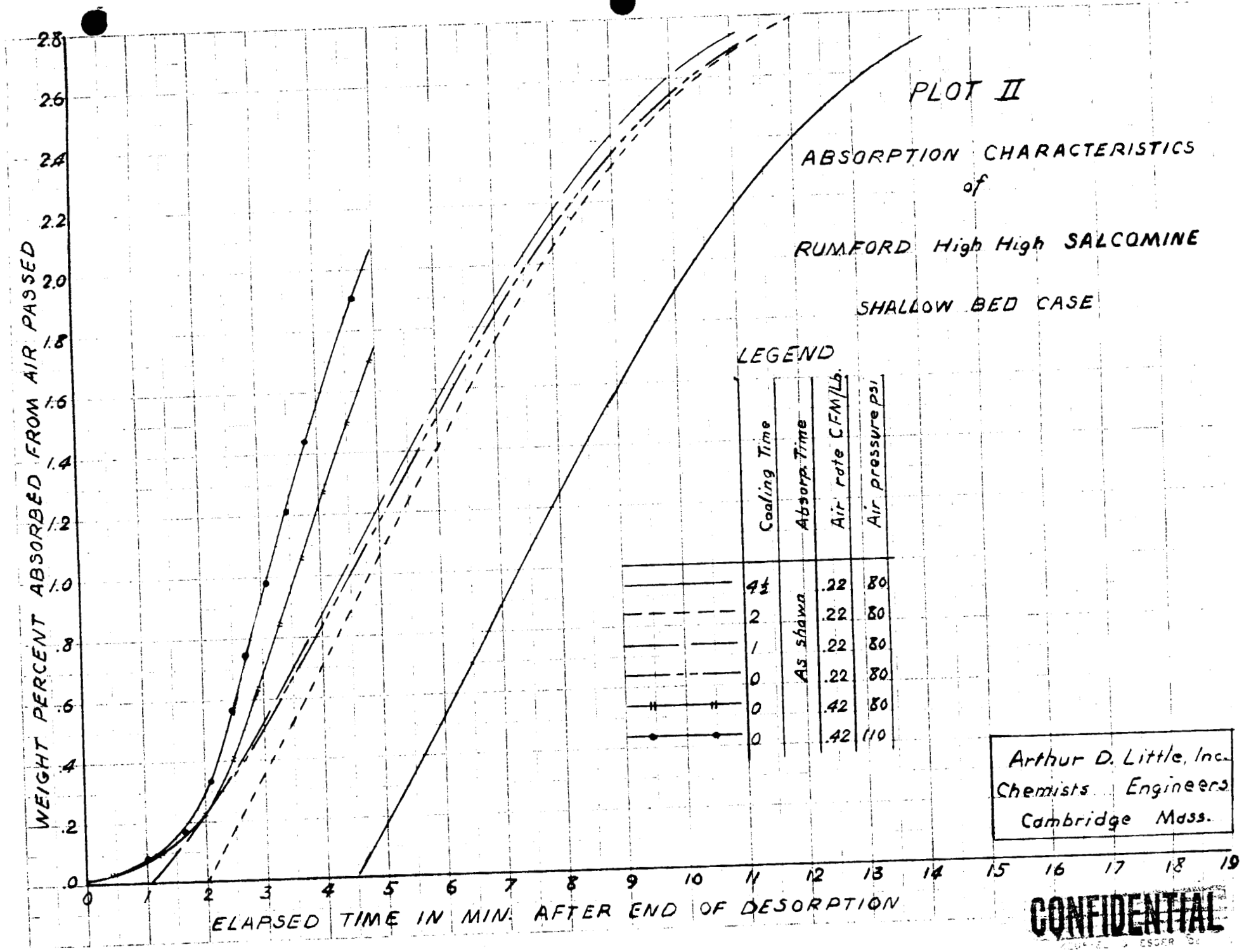

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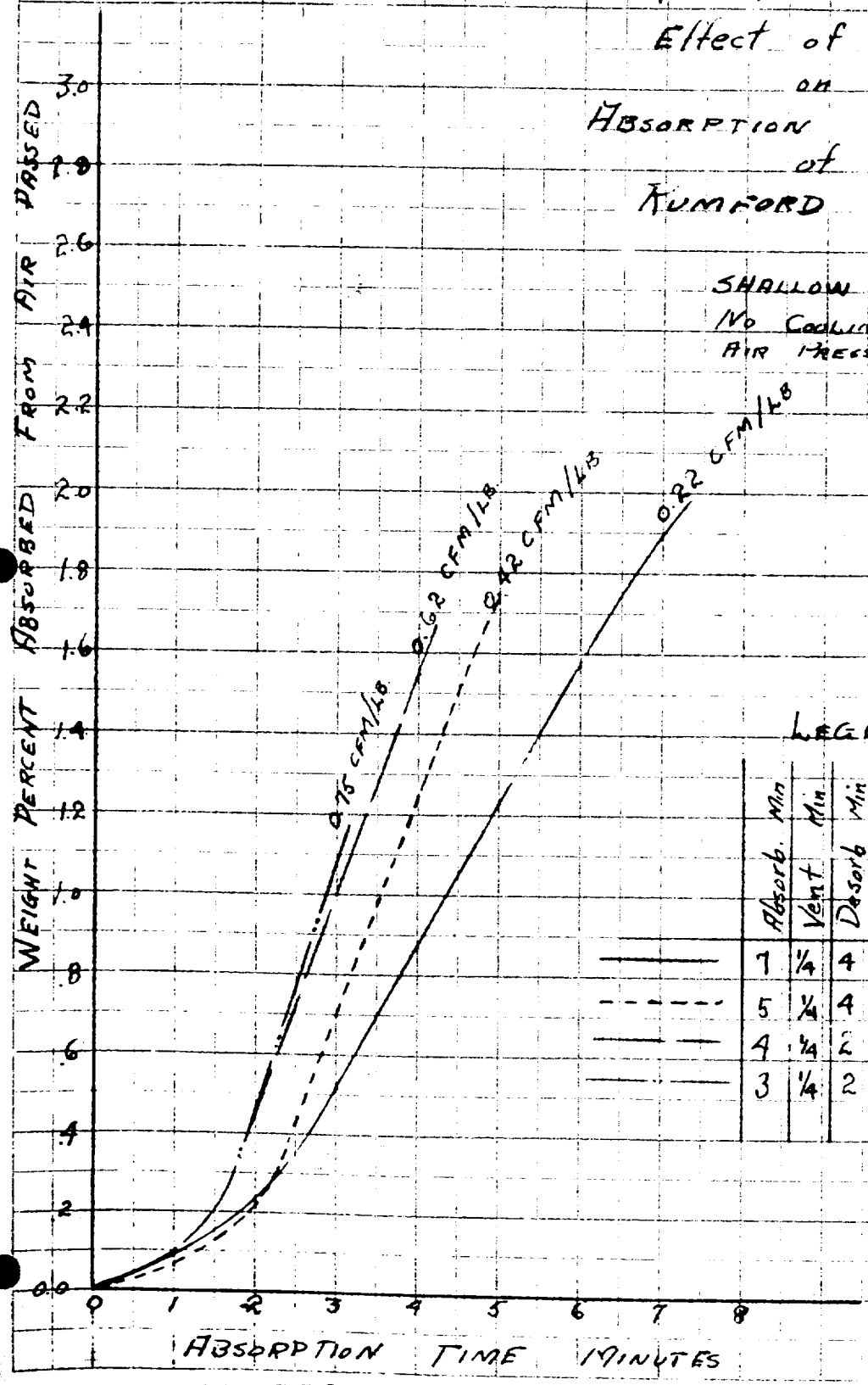
Plot [illegible]

Effect of Air Rate on Absorption Characteristics of Rumford High High Salcomine

SHALLOW BED CASE
No Cooling Period
Air Pressure 80 psi



LEGEND

	Absorb. Min	Vent Min	Desorb Min	Air rate CFM/LB	Slope of St. portion of Absorption Curve
———	7	¼	4	.22	.355
- - - - -	5	¼	4	.42	.676
—— —	4	¼	2	.62	.545
—— · ——	3	¼	2	.75	.620

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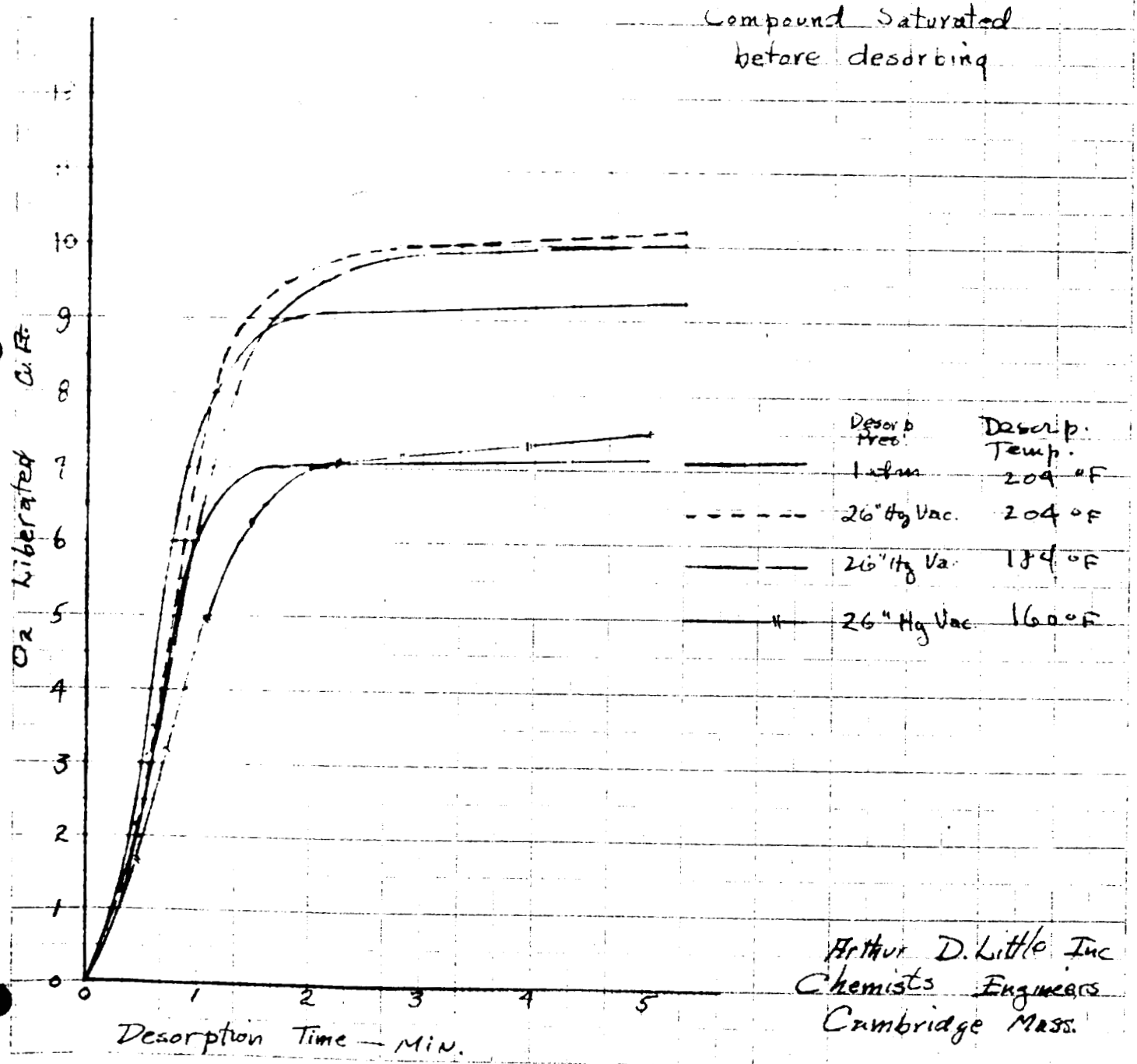
PLOT III

DESORPTION CHARACTERISTICS of RUMFORD High High SALCOMINE

SHALLOW BED CASE
Compound Saturated before desorbing



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Work at M.I.T. indicated that the use of vacuum during desorption would improve the life of the compound. To check this on our units a Nash Hytor pump used as an evacuator was connected to the case and an average vacuum of 20 Hg used during desorption. No improvement in the saturation life curve was noted.

In considering possible reasons for the noted improvement in the life of Salcomine certain factors seemed indicated. First the powder was being so used as to produce oxygen at a much higher rate. Secondly, the powder was never heated as hot during desorption as on the previous long cycle. The question arose as to whether using cooler water during desorption and vacuum might not further improve the life without materially reducing the desorption rate.

The effect on desorption rate and life is shown in plots IV and I.

Reducing the water temperature from 204° to 180°F. caused slight if any reduction in desorption rate. A further reduction to 160°F. however did show some definite lowering of this rate. However, a definitely poorer life curve was obtained. It is not clear just why this should be unless either moisture gradually accumulates reducing the activity of the material or possibly oil known to be in the air accumulates on the powder and at the reduced temperature is not burned out as completely as when 204°F. water is used.

Thus at the air pressures and cooling water and heating water temperatures available we seemed to have reached a fairly good figure as to maximum yield per hour that could be hoped for. This lies between 15 to 17 wt. % per hour. The question that naturally arises is what is limiting this yield.

Two rates are limiting, namely absorption and desorption. The absorption rate is a function of the degree of saturation of the compound and the heat transfer characteristics of the bed. That this is so can be seen from Plot V. It is noted that as the wt. % absorbed increases the instantaneous stripping of the air falls off. Again it is seen that when the air rate is increased from 0.22 CFM to 0.42 CFM with air pressure maintained at 80 p.s.i. ga. the instantaneous volumetric efficienty (I.V.E.) falls off. Yet when the pressure is increased to 110 psi. ga. the I.V.E. curve is the same as for 80 psi. air and 0.22 air rate. It is known that with 110 psi. the bed can operate hotter and still absorb. This gives a higher T or in other words a higher heat transfer rate so that more heat can be removed. The lower I.V.E. with the 0.42 cfm./lb. air rate and 80 psi. appears to be due to insufficient heat transfer capacity. With the greater amount of air and higher heat release the bed becomes heated to such a degree that the absorption is slowed down.

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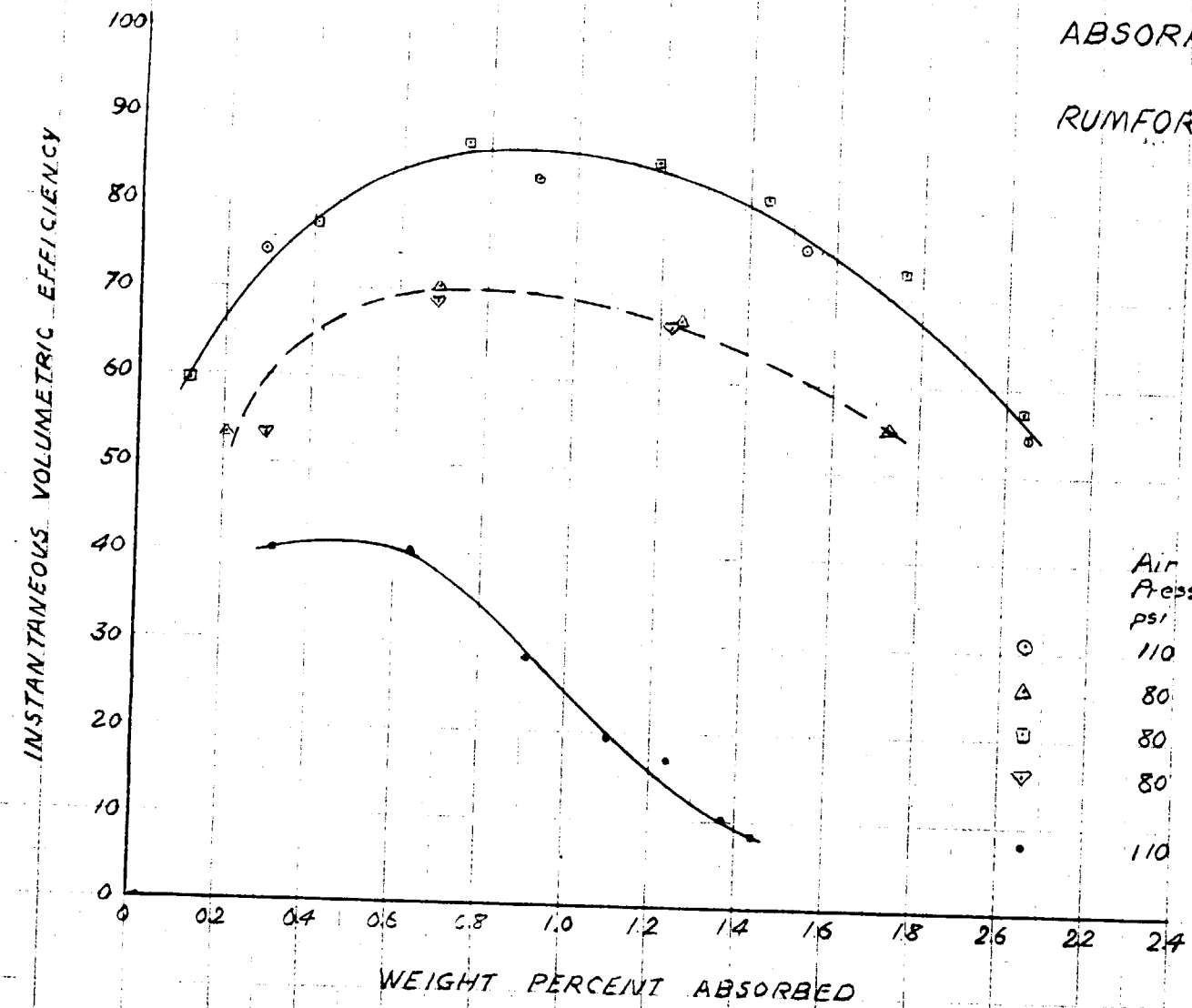
PLOT V

ABSORPTION CHARACTERISTICS of RUMFORD High High SALCOMINE

Shallow Bed Case
No Cooling Period



LEGEND

	Air Pressure psi	Air Rate CFM/Lb.	Desorp. Time	Sat. Level % of Inst. Sat.
⊙	110	0.42	2	79.2
△	80	0.42	4	
⊡	80	0.22	4	
◁	80	0.42	4	
•	110	0.42	2	43.2

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The desorption rate again is a function of the amount of oxygen on the compound and the heat transfer characteristics of the bed. Unlike absorption, highest desorption rates occur when the compound has considerable oxygen on it.

Thus to get high yields per hour the high rate periods of both absorption and desorption should be used, and the compound should be not completely desorbed nor completely saturated. Doubtless the ideal cycle would work in the range of about 10% saturated to 60-70% saturated.

Practically this is somewhat difficult to do. If one establishes a cycle, air rate and pressure, and cooling and heating media temperatures for any point in the powder life such that the 10-60% saturated range is used as the powder ages, one will absorb less and less per cycle and desorb more and more of the base 10% until the compound is completely desorbed each cycle. Thus, as the powder ages the range will deviate more and more from the ranges of maximum absorption and desorption rates.

A practical solution which appears feasible is to set up a cycle so that the powder is used at the highest rates of absorption and desorption during its useful life. This would be done by establishing a fixed cycle with fixed temperature controlling medias and then to vary the air pressure during the life of the powder. A low pressure would be used when the powder was fresh and then as the powder degraded the pressure would be increased.

II. Studies on Ethomine

A series of preliminary runs was made on Ethomine to determine the proper operating conditions for the flat bed-type of case. M.I.T. data indicated 100°F. as being the preferred cooling water temperature and 130°C. steam as satisfactory for desorption. An air pressure of 20 psi. ga. was used and the air rate varied from 0.22 to 0.8 CFM. per lb. of powder. No benefit was obtained by increasing the air rate above 0.6 CFM.

The case during these preliminary tests became accidentally filled with water and stood overnight in this condition. The case was drained and then heated for half an hour with 40 psi. steam with a vacuum on the powder. It recovered nearly all of its original activity after this drastic treatment. Its saturation level was about 2.9 Wt. %.

The case was reloaded with fresh Rumford Ethomine and put on the following cycle for life test: Absorption,

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5 min.; venting, 1 min.; desorption, 2 min.; cooling, 2 min. An air rate of 0.6 CFM per lb. was used and an air pressure of 20 psi. ga. Cooling water for absorption was at 100-104°F. and steam for desorption at 15 psi. ga. An average vacuum of 20" Hg was used for desorption.

The life test data is given in plot VI. The data checks well with the M.I.T. data on 1/2" tubes.

No attempt has been made to date to determine the cycle and mode of operation which will give the maximum yield per hour for Ethomine. This work will be done in the near future.

Typical absorption and desorption curves for Ethomine are given in plots VII and IX. Plot VIII shows absorption and desorption on the same plot. As with Salcomine, desorption is at a higher rate than absorption. Why this should be true is not certain unless it is simply that the heat transfer capacity of the unit is less during absorption than desorption. If this be true lowering the cooling water temperature should increase the absorption rate. This is to be investigated.

Readings were taken of the variation in vacuum as desorption progressed. These are tabulated below:

Time secs.	Run 689 Vacuum outlet side of case	Run 947D Vacuum inlet side of case	Run 946D Vacuum Outlet side of case
15	21 in. Hg.	22 5/8 in. Hg.	23 3/8 in. Hg
30	17 1/4	21 3/4	23 1/8
45	18 1/4	22 1/8	23 3/8
60	21 1/4	23 3/8	24 3/8
75	25	24 5/8	25 1/4
90	26	25 1/2	26
105	26 1/2	26 1/4	26 3/8
120	26 1/2	26 5/8	26 3/4
135		25 7/8	27 1/4

It is noted that only slight variation in vacuum exists across the powder fed.

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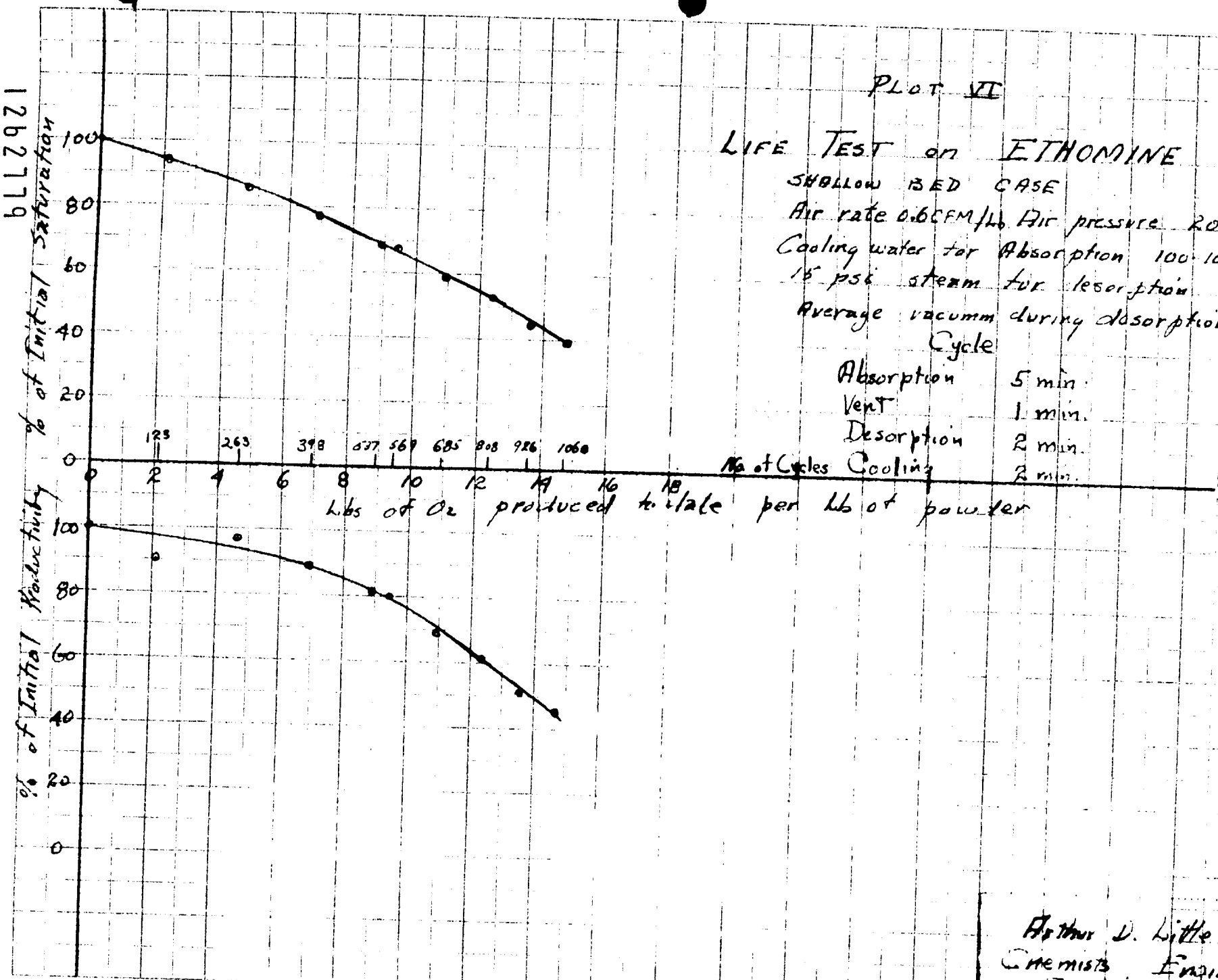

PLOT VI

LIFE TEST on ETHOMINE

SHALLOW BED CASE

Air rate 0.6 CFM/lb Air pressure 20 psi

Cooling water for Absorption 100-104°F

15 psi steam for desorption

Average vacumm during desorption 20" Hg.

Cycle

Absorption	5 min.
Vent	1 min.
Desorption	2 min.
Cooling	2 min.

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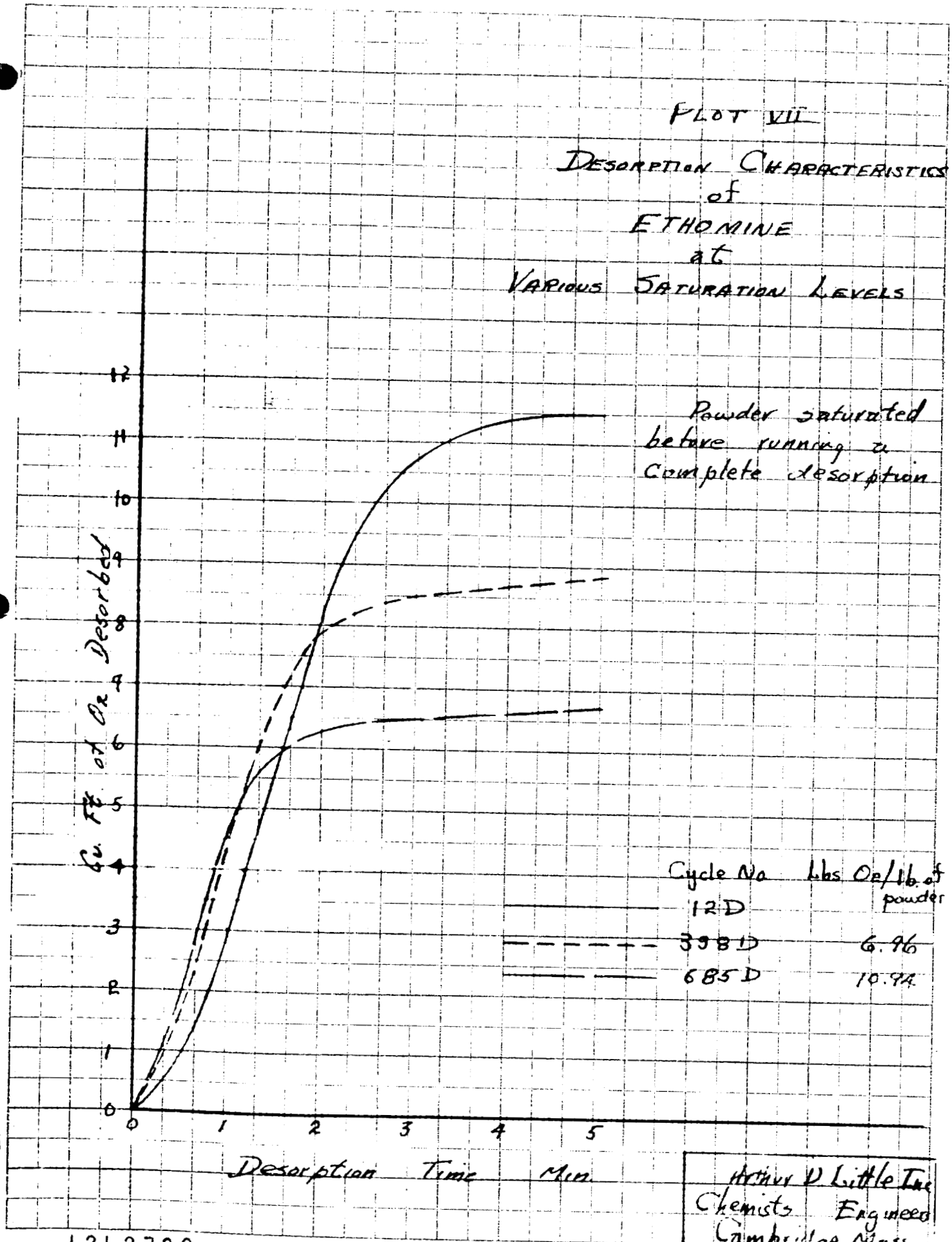

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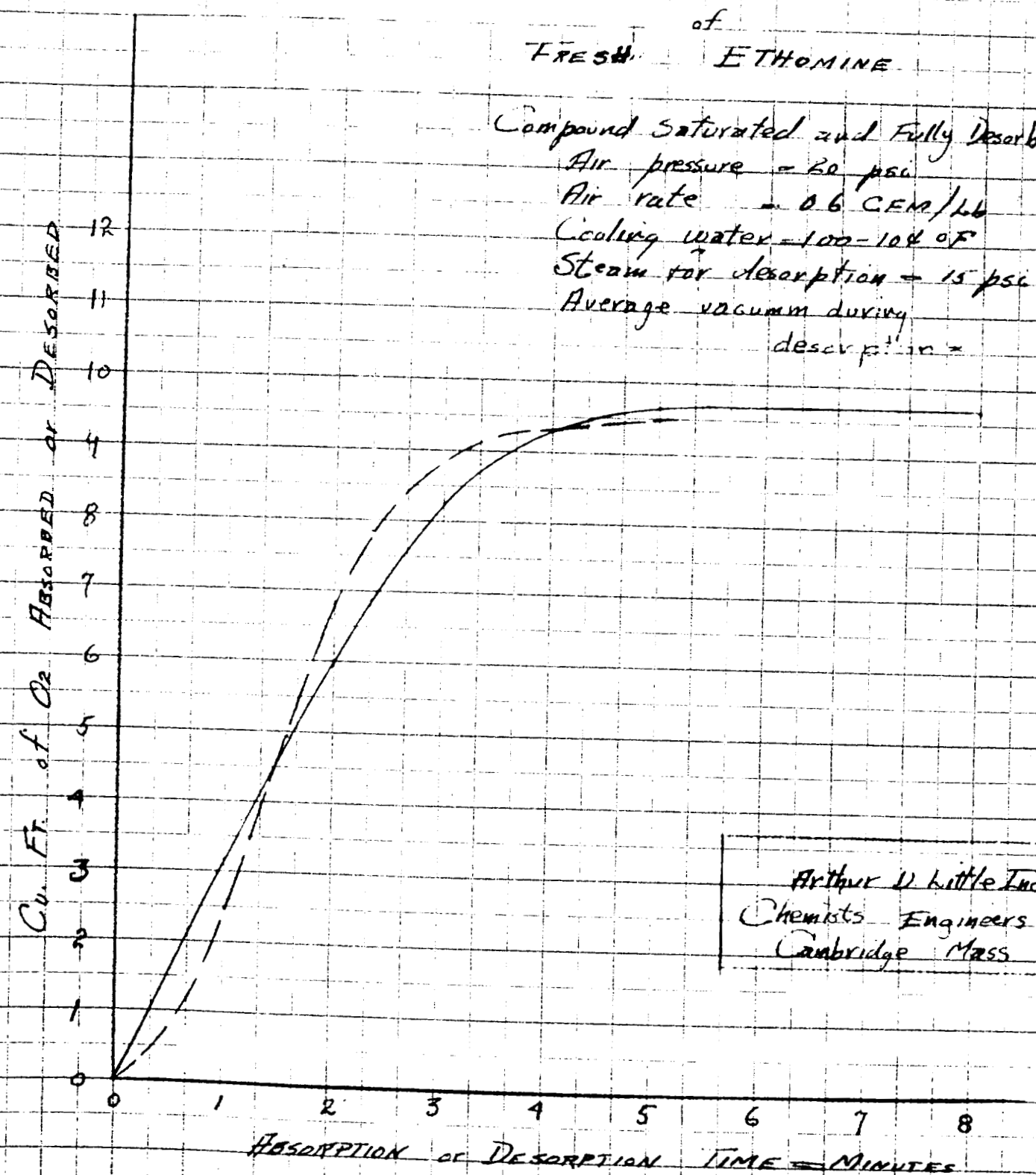
PLOT VIII

ABSORPTION and DESORPTION CHARACTERISTICS of FRESH ETHOMINE

Compound Saturated and Fully Desorbed
Air pressure = 20 psi
Air rate = 0.6 CFM/Lb
Cooling water = 100-104 °F
Steam for desorption = 15 psi
Average vacumm during desorption =



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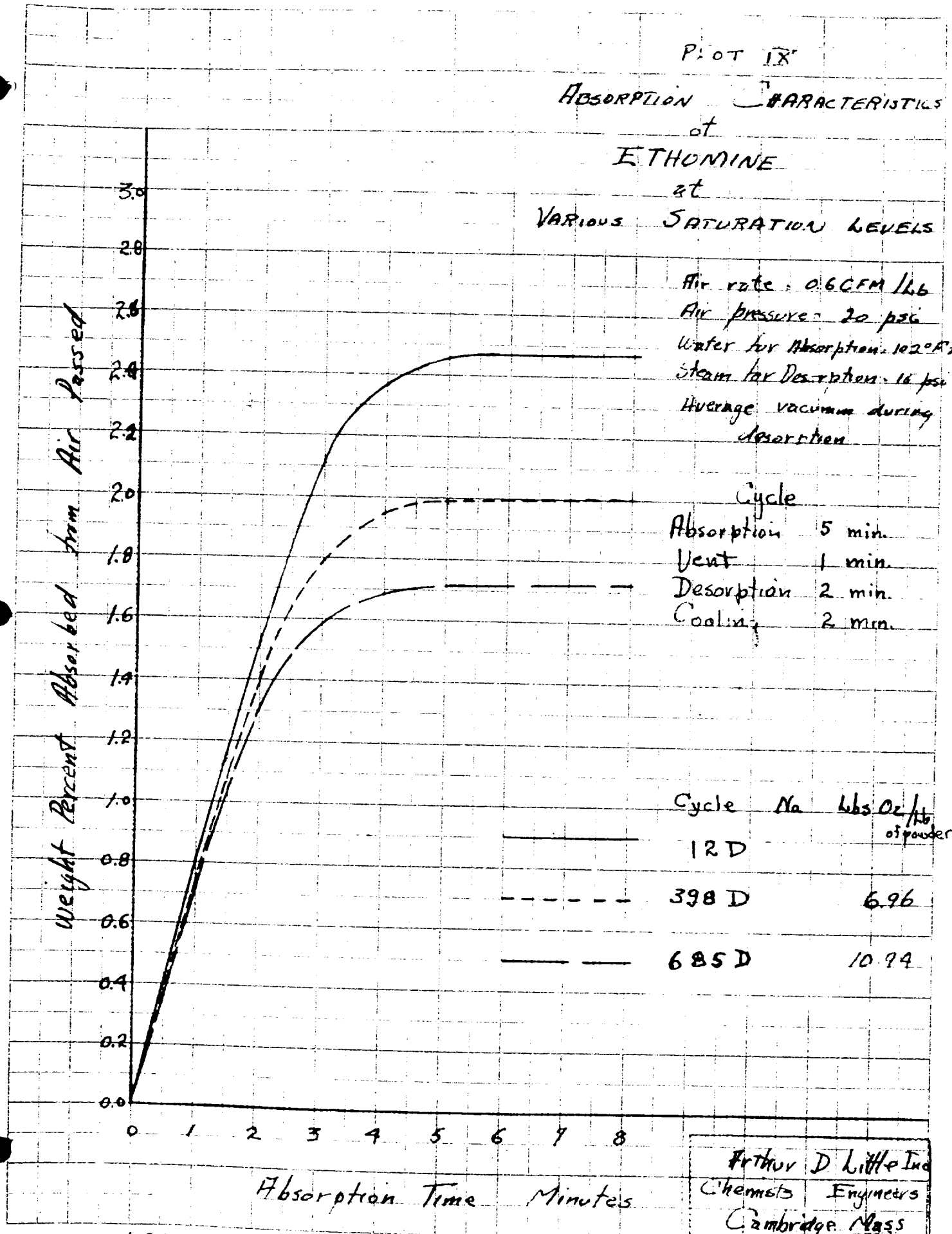

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III. Quality of Oxygen from Salcomine and Ethomine

Early tests by the palladium method at Arthur D. Little, Inc. indicated the presence of CO in the oxygen made with Salcomine, but on the basis of recent work, this early test result is now believed to have been caused by aldehyde in the oxygen. Tests were not made on Ethomine-derived oxygen at that time. Present interest in the use of Ethomine for an air-flown unit, has necessitated a review and extension of this work.

The palladium test, depending on the reduction to metal of a palladium salt is not specific to CO but is affected by reducing agents like aldehydes which may be released from imperfectly purified powders. This test has been rerun on oxygen made from Rumford High-High Salcomine, and on oxygen from Ethomine desorbing both under vacuum and atmospheric pressure with results as follows:

	Desorp. Pressure	P.P.M. of CO
Salcomine	1 atm.	0
Ethomine	1 atm.	6-10
"	1/3 "	10-15

It is to be noted that this test showed no CO in the oxygen from Salcomine. The palladium test is known not to be specific for CO but will be affected by any easily oxidized material. The early Salcomine prepared by Rumford was not entirely free of excess aldehyde which came off gradually in the oxygen during desorption. This loss of aldehyde is also quite apparent in the current Rumford Ethomine. Present Rumford High-High Salcomine on the other hand is free of excess aldehyde and yields an oxygen with slight if any odor. It is therefore believed that the early palladium test for CO in the oxygen from this early Salcomine was due to such aldehyde.

A more specific test for CO was sought as well as a means of determining just what carbon containing materials were actually in the oxygen. The literature indicated that the reaction of hemoglobin, CO, and pyrotannic acid to be specific for CO. This test depends on a color change that occurs when pyrotannic acid is added to hemoglobin contrasted to that which occurs with carboxyhemoglobin. With hemoglobin a grey brown color develops while with carboxy hemoglobin a carmine color occurs. Gas to be tested is passed through the blood diluted in the ratio:1 part of blood to 20 parts of water, with a defoaming agent such as octyl alcohol present and then pyrotannic acid added.

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The oxygen from present Rumford High-High Salcomine showed no CO while the oxygen from Ethomine apparently showed some CO. No attempt was made to use this test in a quantitative manner although this can be done.

A combustion train has been assembled. Absorption tubes filled with Ascarite were used to remove CO_2 initially present in the oxygen. Combustion was at 800°C. with CuO as a catalyst. Water and CO_2 formed by combustion were absorbed in $CaCl_2$- and Ascarite-filled tubes, respectively. Results to date on this train do not appear conclusive. In Salcomine oxygen to date no CO_2 has been formed in the combustion furnace while there has been some in Ethomine oxygen.

Thus all the data to date indicates the absence of CO in Salcomine oxygen and 6-15 ppm. of CO in Ethomine oxygen.

As an aid to determining the volumetric throughput in the combustion train a spot CO_2 determination has been made on a 5 gallon sample of the gas under test in the train. A barium hydroxide-oxalic acid titration procedure was used. The oxygen from Salcomine contains 1200 to 1500 ppm. of CO_2 while that from Ethomine contains 510-650 ppm.

IV. Effect of Air Quality on Productivity of Rumford High-High Salcomine

In the normal operation of the 35 lb. flat cases at Cambridge the air driers are used continuously for 8 hours and then regenerated. No variation in oxygen productivity is noted when a freshly regenerated drier is put on stream. Dew points have been determined on the air to the units during the period of use of the two driers in the system.

Time	#1 Drier dew point, °C.	#2 Drier dew point, °C.
11 min.	-70	
25 min.		-60
1 1/4 hr.	-60	-69
2 hr.	Below -63	
2 1/2 hr.	-67	-58
6 hr.	-42	-30
6 1/4 hr.		-28
7 hr.	-35	-28

Thus it would appear that in this range of dew points moisture has little if any effect on the productivity of either Salcomine or Ethomine.

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The compressed air at Cambridge is known to contain some oil mist. The 1/2" tube units were filled with Rumford High-High Salcomine and run both on the regular dried air containing oil mist and on this air further purified by passage through an active charcoal bed.

The data taken on this test is shown in plot XI. While the slope of the life curve for air passed through active charcoal is less than that for the air passed only through silica-gel during the first 500 cycles, beyond 500 cycles the two curves become parallel indicating no advantage of one over the other. Furthermore, the data on life test #3 for the Thermek bed shows as good or better a life than that from the charcoal treated air test during the first 1000 cycles of operation. The test does not seem to be conclusive.

Plot X shows data taken with same 1/2" tubes loaded with fresh Rumford High-High Salcomine. In this test two tubes were run on air containing ± 0.37 grains of moisture per cu. ft. while the other two tubes used the regular silica-gel dried air with an average moisture content of ± 0.04 grains per cu. ft. Again the tests appear inconclusive since their slopes are so nearly the same. The initial differences in productivity and the dips in both curves at 350 cycles would seem to be due to some feature of operation other than moisture content of the air.

V. Bellows Actuated Valve

The valves used on the Cambridge units have been Lunkenheimer Quick Acting valves opened by air pressure on leather cup washers mounted in piston chambers. On lines carrying high temperature media such as hot water and steam difficulty has been experienced with drying out of the leather cup washers. This permits the air acting on the washers to by-pass the washer so that the valve fails to open. Also extended when the units are not operated for periods the leather washers dry out.

Figure 1 shows a new valve constructed at Cambridge incorporating a Hydron metal bellows instead of the cup washer to actuate the valve. No problem of drying out exists with this unit either in operation or upon standing during periods of idleness for the unit. The valve has been in service on both 15 and 80 psi. steam and has proved most satisfactory. The design is compact and can be made more compact since the packing nut may be adjusted without disassembling the valve.

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Plot X

Effect of Moisture in Air on Productivity of Rumford High High Salcomine

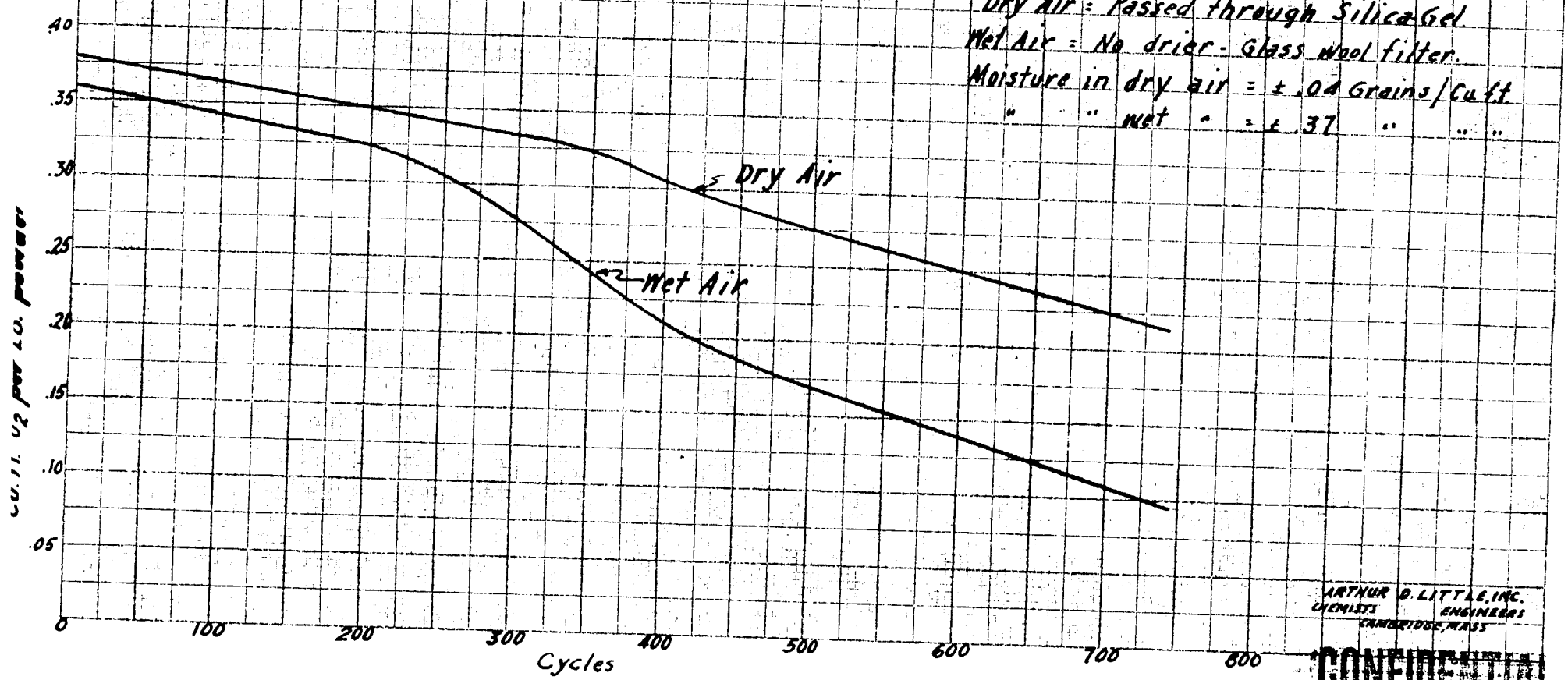

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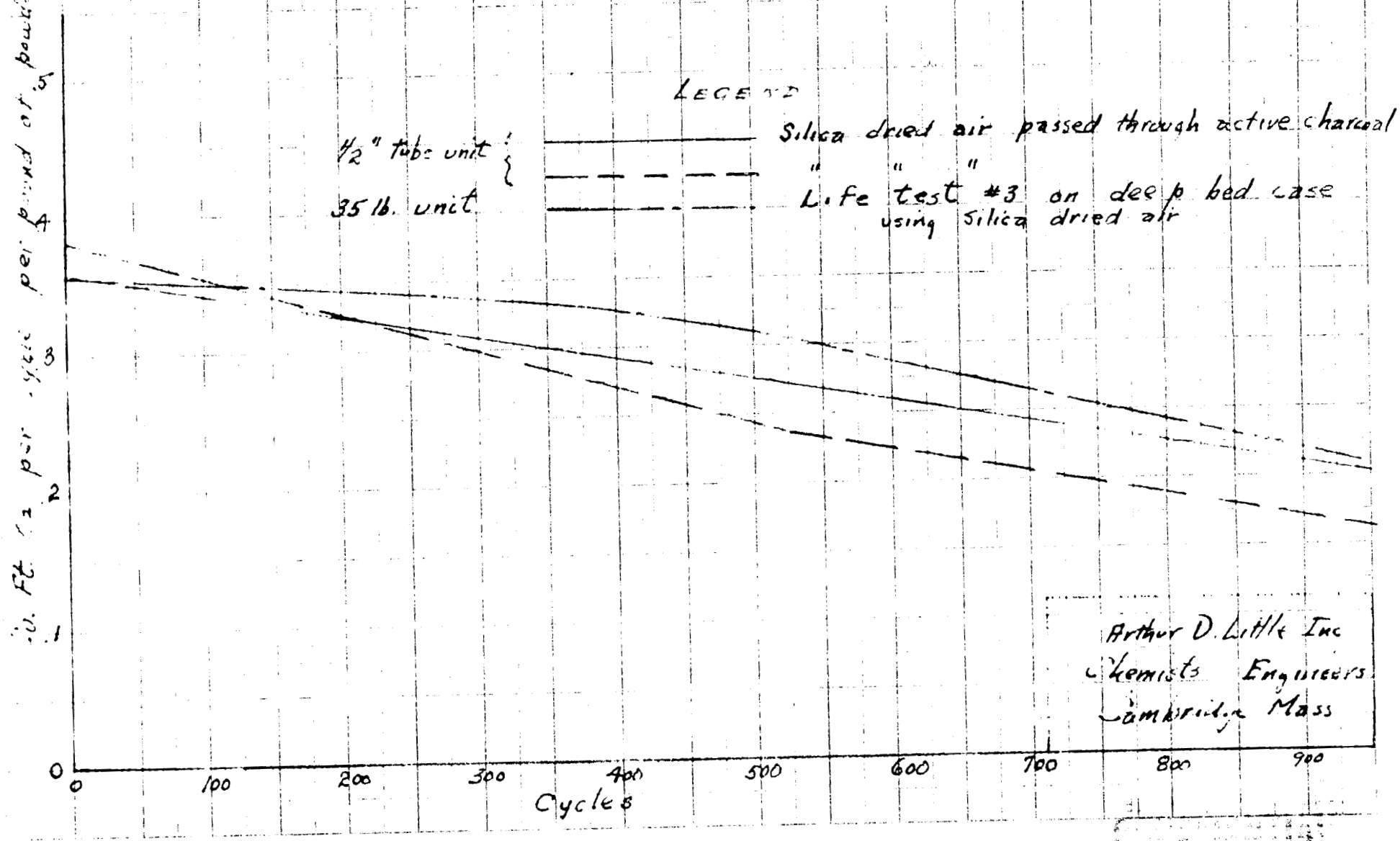
Plate VI

EFFECT of AIR PURITY on PRODUCTIVITY of Rumford High High SANCOMINE



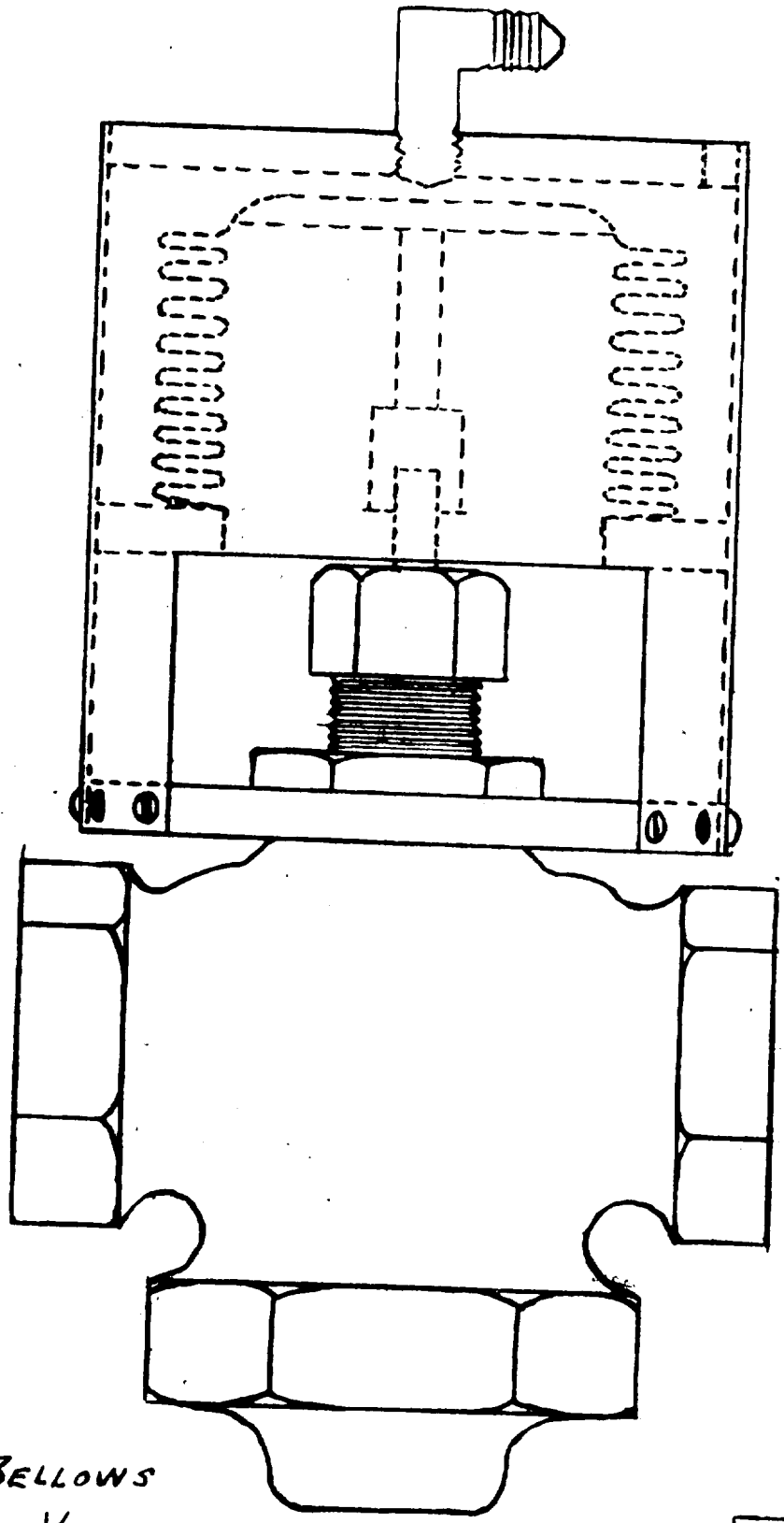


Fig. 1
HYDRON BELLOWS
ACTUATED VALVES
Full scale for 1" valve

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The bellows used in the present unit is hydraulically formed and has according to the manufacturer a life expectancy, as we are using it, of about 15,000 cycles. Bellows are available on the market which could in all probability increase this life expectancy to over 1,000,000 cycles.

Respectfully submitted,

Arthur D. Little, Inc.

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