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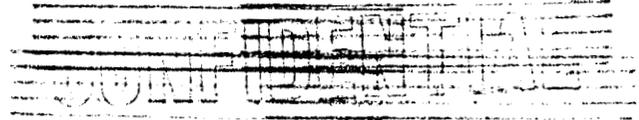
IRVIN STEWART, Executive Secretary

1530 P STREET NW.
WASHINGTON, D. C.

April 10, 1942

R

Dr. Melvin Calvin
Department of Chemistry
University of California
Berkeley, California



Dear Dr. Calvin:

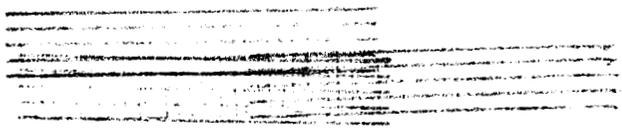
I am sending you herewith a copy of
a letter from Dr. E. R. Gilliland, addressed
to Dr. Furnas, together with its ten attachments.

Sincerely yours,

S. S. Prentiss
Technical Aide
Section B-7

Enclosure

This document contains information affecting the national
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BY *D. B. Owen* DATE *4/1/96*
by the President's ADD 3/11/96

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CONFIDENTIAL

April 1, 1942

Prof. C. C. Furnas
National Defense Research Committee
1530 P Street NW
Washington, D. C.

Dear Prof. Furnas:

A number of desorption runs have been carried out at 55, 66, and 77°C. in our usual 1/2-inch tube units. Desorption pressures of 1/4, 1/2, and 1 atmosphere were employed. These tests were made by saturating the compound using air at cooling water temperature, and then carrying out the desorption in the usual way employing hot water in the jacket around the tube. The residual air was removed from the voids by evacuation prior to regeneration.

The results of these tests are summarized in Figures 1, 2, and 3. Figure 1 indicates the results obtained at 55°C. At this temperature the per cent of the oxygen desorbed for a given time is a very definite function of the desorption pressure. Apparently, at this temperature, this pressure region must correspond to a rather rapid change in the saturation with pressure. In fact, the desorption curve at atmospheric pressure appears to be almost asymptotic and would suggest that a saturation of 90 to 95 per cent might be in equilibrium with oxygen at atmospheric pressure. Figure 2 is a similar plot of the results obtained at 66°C. In this case there is very little difference in the rate of desorption at 1/4 and 1/2 atmospheres. However, 1 atmosphere is still somewhat slower. This may mean that the equilibrium-saturation curve has shifted with the increase in temperature such that both 1/4 and 1/2 atmospheres are in the lower portion of the saturation pressure curve, i.e., in the region where the degree of saturation is relatively independent of the pressure. On the other hand, 1 atmosphere of oxygen appears to be sufficient to give an appreciable equilibrium saturation at this temperature. It may be that heat transfer is a controlling factor and that greater differences in the rate of desorption as a function of pressure will be obtained in an adiabatic operation. Of course, these curves represent rate of desorption and not equilibrium, but it appears that the changes in rate with temperature and pressure fit in well with the expected shifts in equilibrium.

The third Figure shows similar curves for a desorption temperature of 77°C. The lower pressures still give the highest rates of desorption, but the difference is becoming less. In this latter case, the rate at 1/4 atmosphere appears to be about 1-1/2 times as fast as the rate of 1 atmosphere.

These curves indicate that practical desorption rates can be obtained in the temperature region 55° to 77°C. At 55°C. the rate of desorption at 1 atmosphere is undoubtedly too slow and in fact it

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appears doubtful whether over 5 to 10 per cent of the saturation value could be desorbed. At a desorption pressure of 1/2 atmosphere, the rate still appears to be relatively slow, and even with 1/4 atmosphere it would appear that relatively long cycles would be required. Thus, a desorption period of 20 minutes would be required to remove 50 per cent of the oxygen. Lower pressures would presumably give still faster rates of desorption and with a pressure of 1/10 atmosphere, it might be possible to obtain a 50 to 60 per cent desorption in 10 minutes. This rate of desorption is probably getting into the practical range. Engineering calculations should indicate whether it is preferable to pump this oxygen through such a high compression ratio rather than compress the larger volume of air through a somewhat increased compression ratio and employ an operating temperature sufficient to give rapid desorption at higher pressures.

At 66°C. it appears that desorption at pressures from 1/2 to 1 atmosphere are probably the most desirable. The rates obtained with pressures in this region appear to be sufficient to allow a desorption cycle of not over 10 minutes. The rates at 77°C. are better than at lower temperatures, particularly with a desorption pressure of 1 atmosphere, but it would appear doubtful that the improvement over 66°C. is sufficient to justify the greater difficulty during absorption and purging.

We feel that these data indicate that an isothermal unit, or an adiabatic in which sufficient heat capacity is introduced such that the temperature variation during the cycle is of the order of 5°, can be designed and constructed. In such a unit there will be certain problems of which the most difficult would appear to be the purging of the air. It may be possible to accomplish this by rapid evacuation of the unit. Thus, from results given in Figure 2, it might be possible to obtain a rather complete evacuation and still not lose an amount of oxygen corresponding to more than 5 per cent of the saturation value.

A number of absorption runs have been carried out at 65°C. due to the fact that this temperature appeared to be about the most desirable on the basis of the desorption experiment. These absorption experiments were carried out in the usual way but with the heated water circulating through the jacket. The inlet air did not pass through a preheater as such but it did pass through about 6 inches of the jacketed section of the 1/2-inch tube before contacting the absorbent. In addition, it passed through portions of the piping before this jacketed section that were heated by conduction and were above room temperature but lower than 65°C. The results of these runs are shown in Figures 4 to 10, and summarized in Table 1.

The most complete absorption data were obtained at 600 lbs / sq. in. ga. and with a rate of air flow corresponding to 2 cu. ft./hr. (measured under standard conditions). 40.7 grams of absorbent were used in the unit. The results for these conditions are given in Figure 4. The left-hand plot of this Figure indicates the cumulative per cent

TABLE I

	Pressure lb/sq.in.ga.	Temperature °C.	Flow Rate C.F.H. **	Sorption Time Minutes	Wt. % O ₂ in Compound	Cumulative Mol % O ₂ Removed from Air
*	100	12	1	30	4.0	-----
	200	65	3	5	0.04	0.06
	350	65	3	5	0.65	13.8
	400	65	0.87	4	0.4	35
	600	65	0.87	2	0.52	75.5
	"	"	"	4	0.97	76.6
	"	"	2	1	0.32	52.1
	"	"	"	2	0.70	50.2
	"	"	"	4	1.25	45.6
	"	"	"	7	1.79	38.5
	"	"	"	15	2.74	28.1
	"	"	4	1	0.51	38.1
	"	"	"	2	0.90	33.4
	"	"	"	4	1.79	33.7
	"	"	6	1	0.50	26.9
	"	"	"	2	0.99	25.6
	800	"	0.87	4	1.15	84.6
	"	"	"	7	1.78	79.5
	"	"	"	10	2.21	70.7
	"	"	2	4	1.86	65.3
	"	70	0.87	4	1.00	78.4
	"	78	"	"	0.30	26.5

* Saturation Run

** Measured at room temperature and pressure (40.7 grams absorbent)

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oxygen removed from the air as a function of the absorption time. The dotted line is the theoretical curve based on limitation of either complete oxygen removal or the maximum oxygen that could have been removed as limited by saturation of the compound. The solid curve represents the experimental data. It will be noted that initially about half of the oxygen is removed from the air, and that the cumulative oxygen removal falls off such that for a 12-minute absorption period only 30 per cent of the oxygen has been removed. The right-hand Figure presents the weight per cent oxygen in the compound as a function of the absorption time. The dotted curve again gives the stoichiometric maximum amount that could be obtained. It will be noted that the rate of absorption is slower than is obtained with 80 lbs. pressure and an absorption temperature of 20°C. Our results for this latter condition indicate that about 2-1/2 weight per cent saturation would be obtained in 5 to 6 minutes. This decreased rate of absorption is more apparent when it is realized that the adiabatic unit will not operate with complete desorption and therefore the absorption period will probably start with a compound already containing 1 to 2 weight per cent oxygen. The rate of absorption in this region is appreciably less than that initially obtained with a completely regenerated sample.

Figures 5, 6, and 7 give similar results for absorption at 600 lbs./sq. in. ga., 65°C., and different rates of air flow. As would be expected, higher rates of air flow give less oxygen recovery but higher saturation for a given absorption time. It would appear that flow rates corresponding to 4 cu. ft./hr. might be justified but that higher rates would be wasteful of the compressed air.

Figure 8 summarizes two runs carried out with an absorption pressure of 800 lbs./sq. in. These results indicate much better oxygen removal but of course such an operation would involve increased compression power for a given quantity of air although the lower air requirements might make the total power consumption less.

Figure 9 illustrates the effect of temperature for absorptions carried out at 800 lbs./sq. in. ga. and an air flow rate of 0.87 C.F.H. The absorption time in all cases was 4 minutes. The left-hand Figure illustrates the oxygen removal from the air as a function of the absorption temperature. This curve appears to break rather rapidly around 70°C., indicating that higher temperatures are very unfavorable. The right-hand Figures shows the same data plotted as the weight per cent oxygen in the compound, and again shows a rather sharp dropping off about 70°C.

Figure 10 illustrates the effect of pressure on the rate of absorption. These experiments were carried out at 65°C. with a flow rate of 0.87 C.F.H. and with an absorption period of 4 minutes. It will be noted that essentially no absorption is obtained at 200 lbs./sq. in. under these conditions, but that the rate of absorption rises rapidly with an increase in pressure above this level. This rise is particularly striking when it is realized that the higher pressures correspond to a much more complete removal of the oxygen from the air. It seems probably that the falling off of the curves

above 600 lbs./sq. in. is due to the depletion of the oxygen in the air. Higher rates of air flow should make the higher pressures appear still more favorable. These results indicate that absorption pressures of at least 600 lbs. should be used for an operating temperature of 65°C. Still higher pressures will probably give less power consumption due to the decreased air requirements.

We are constructing a small adiabatic unit and hope to make some tests on this within the next few days. In order to make this unit adiabatic we are planning to thermally insulate it from the pressure vessel and have made some calculations indicating that the heat transfer between the bed and the surroundings should be rather small. We are planning to try several types of heat-absorbing systems, such as a mixture of the absorbent with copper or aluminum shot or an arrangement in which strips or rods of these metals are employed. Another method we would like to employ would be the actual incorporation of the finely divided metal into the compressed cake. Thus, we are planning to make pressed cakes in which aluminum or copper powder is incorporated. This latter type of construction should give very intimate heat transfer, but may interfere with the absorption properties. We are planning to incorporate an amount of metal such that the temperature variation during the cycle will not exceed 5°C.

We have done a little figuring on the change of state idea suggested by Dr. Prentiss. While we have found several systems that will operate in the proper temperature region, the weight and volume requirements do not appear to be any less than those for an amount of metal sufficient to make the temperature variation very small. It may be possible that they will appear more satisfactory in a large unit but we feel that from an experimental viewpoint, the use of a metal would appear more favorable. Along the metal line, it would appear that graphite is not too inferior relative to aluminum and might be very easily incorporated in the powder.

In starting such an adiabatic unit, we have planned to pass hot gas through the absorbent bed and use the sensible heat to bring it up to operating temperature. As for making up the heat losses, this can be done by using a hot inlet air stream but has the disadvantage of increasing the temperature during the absorption cycle when a low temperature is desired. It should be possible, but may be not practical, to recirculate preheated oxygen during desorption to make up for the heat loss. Such an operation would be desirable due to the fact that it would tend to raise the temperature during the desorption cycle.

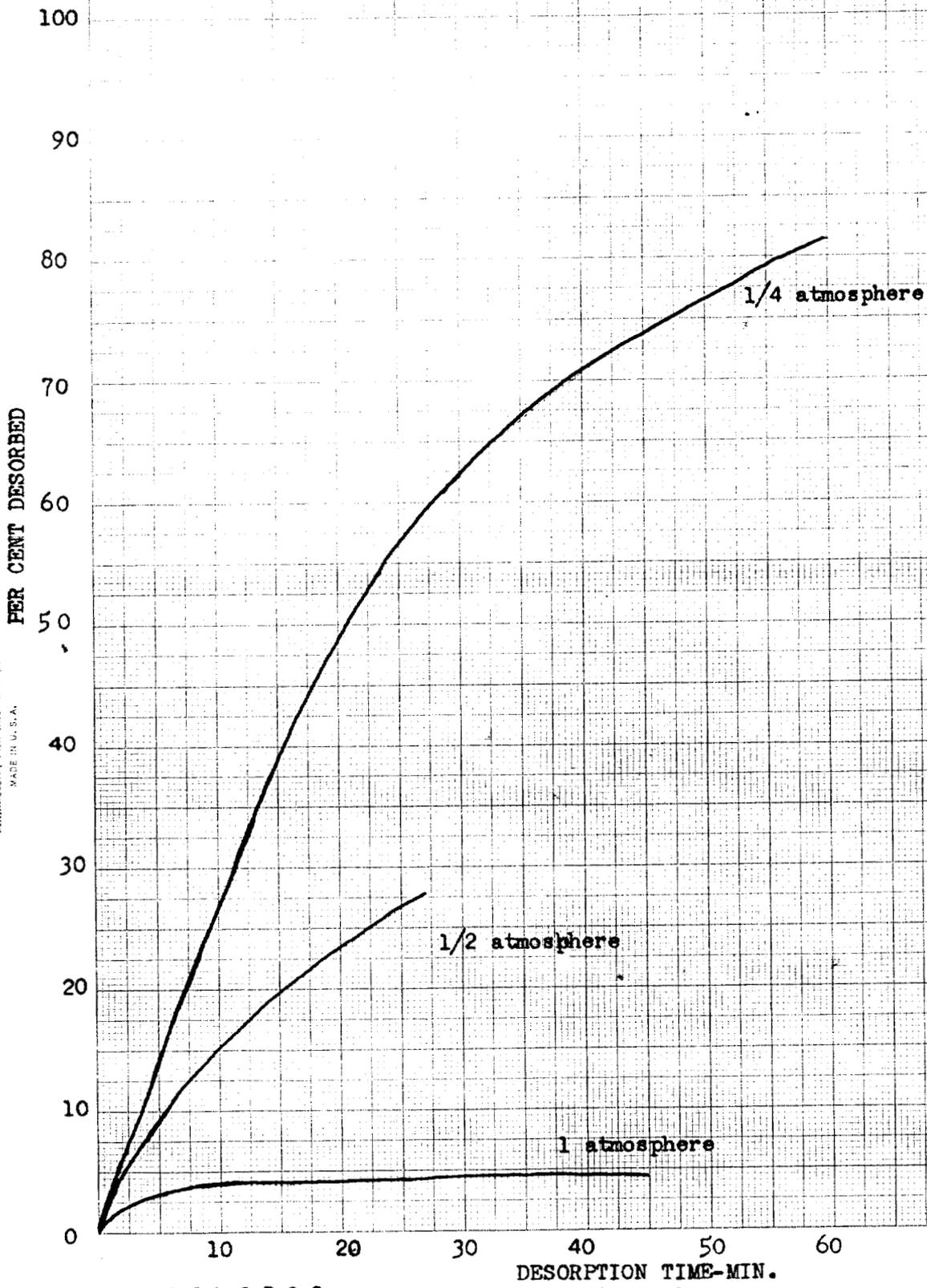
(Signed)
E. R. Gilliland

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Desorption Temperature = 55°C

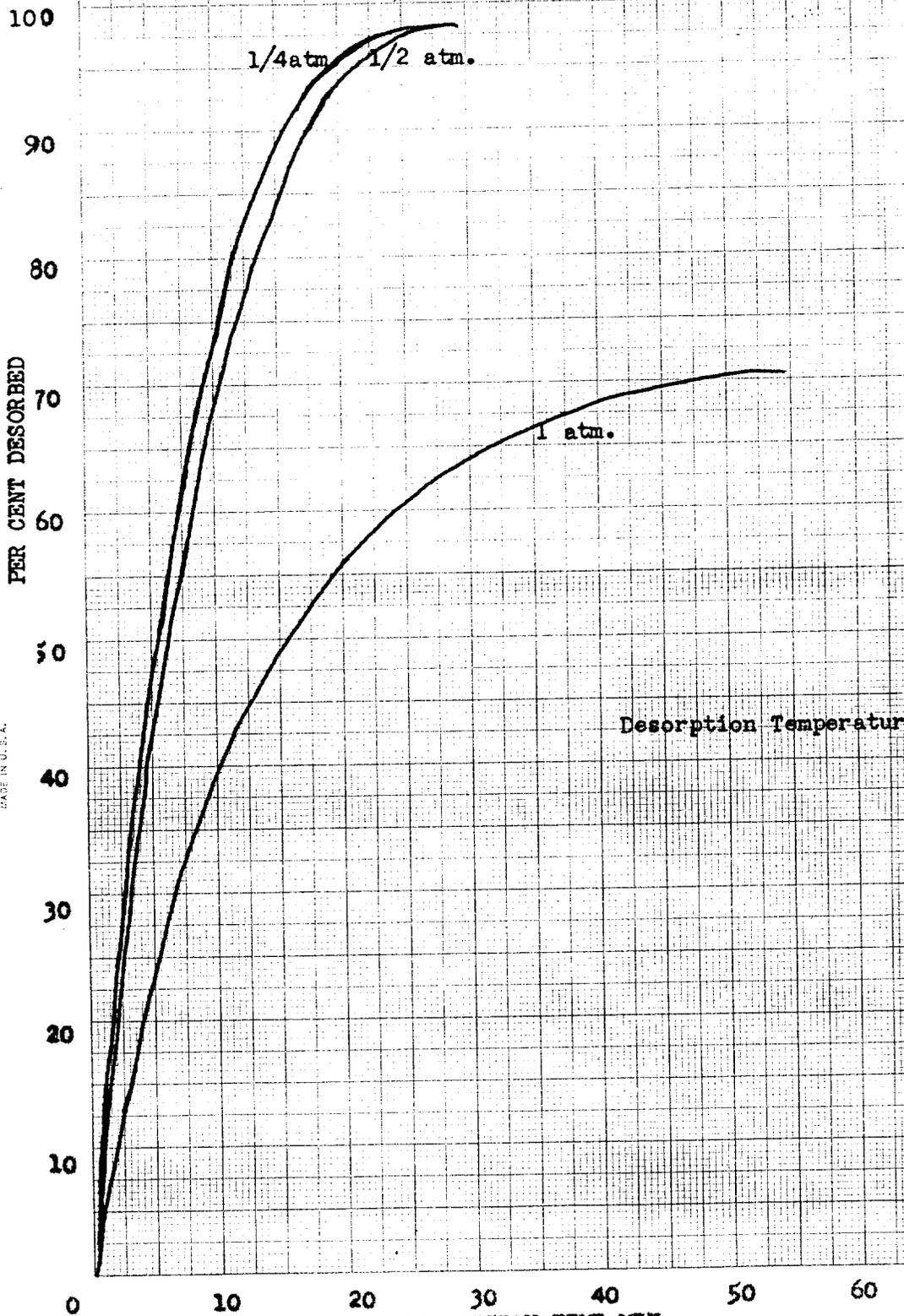
KEUFFEL & ESSER CO., N. Y., NO. 350-14
Millimeters, 1/16th lines heavy.
MADE IN U.S.A.



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DESORPTION TIME-MIN.
Figure 1.

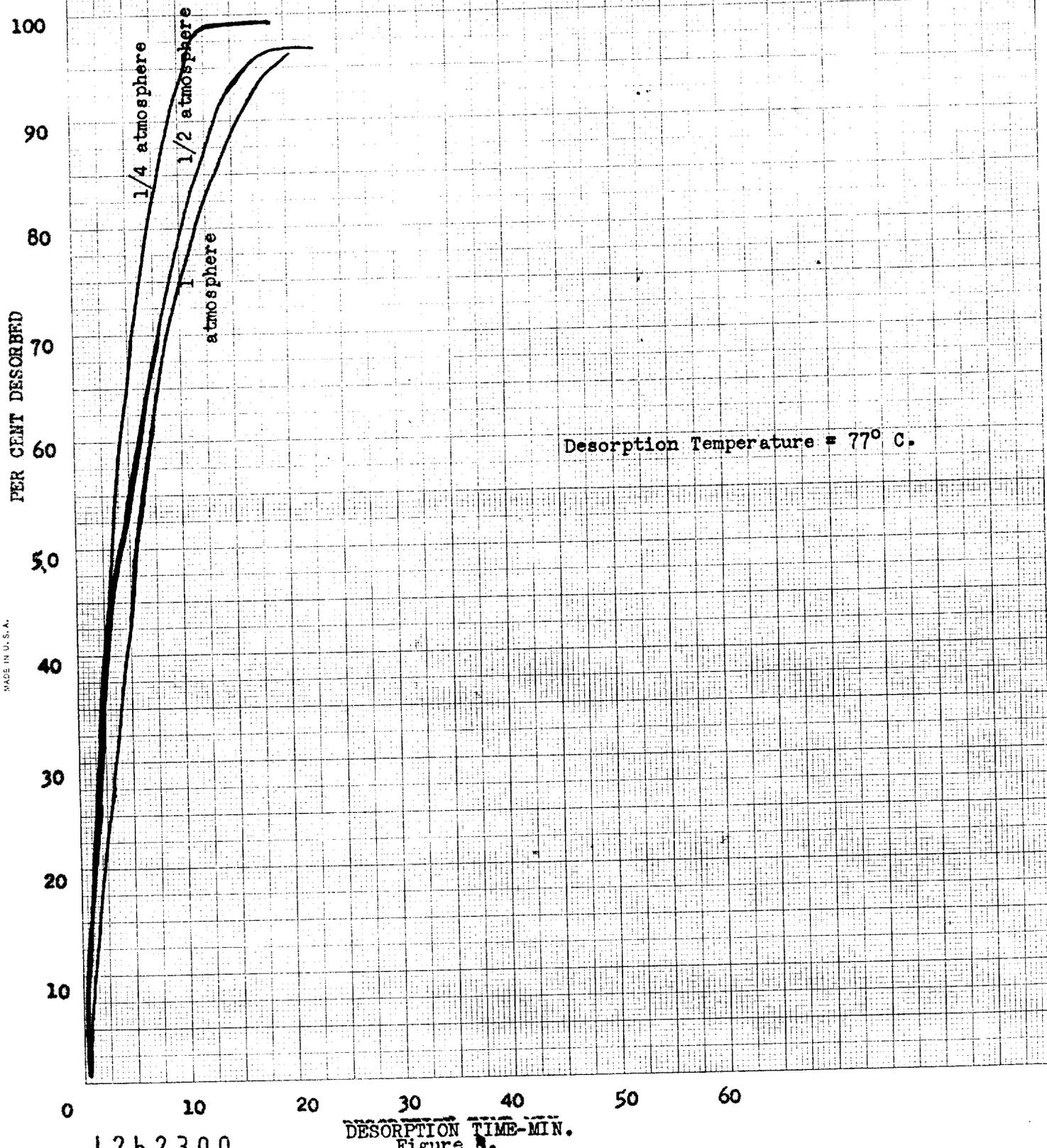
KEUFFEL & ESSER CO., N. Y. NO. 370-14
Millimeters, 10th lines heavy
MADE IN U.S.A.



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Figure 2.

KEUFFEL & ESSER CO., N. Y., NO. 350-14
Millimeters, 10th line heavy.
MADE IN U.S.A.

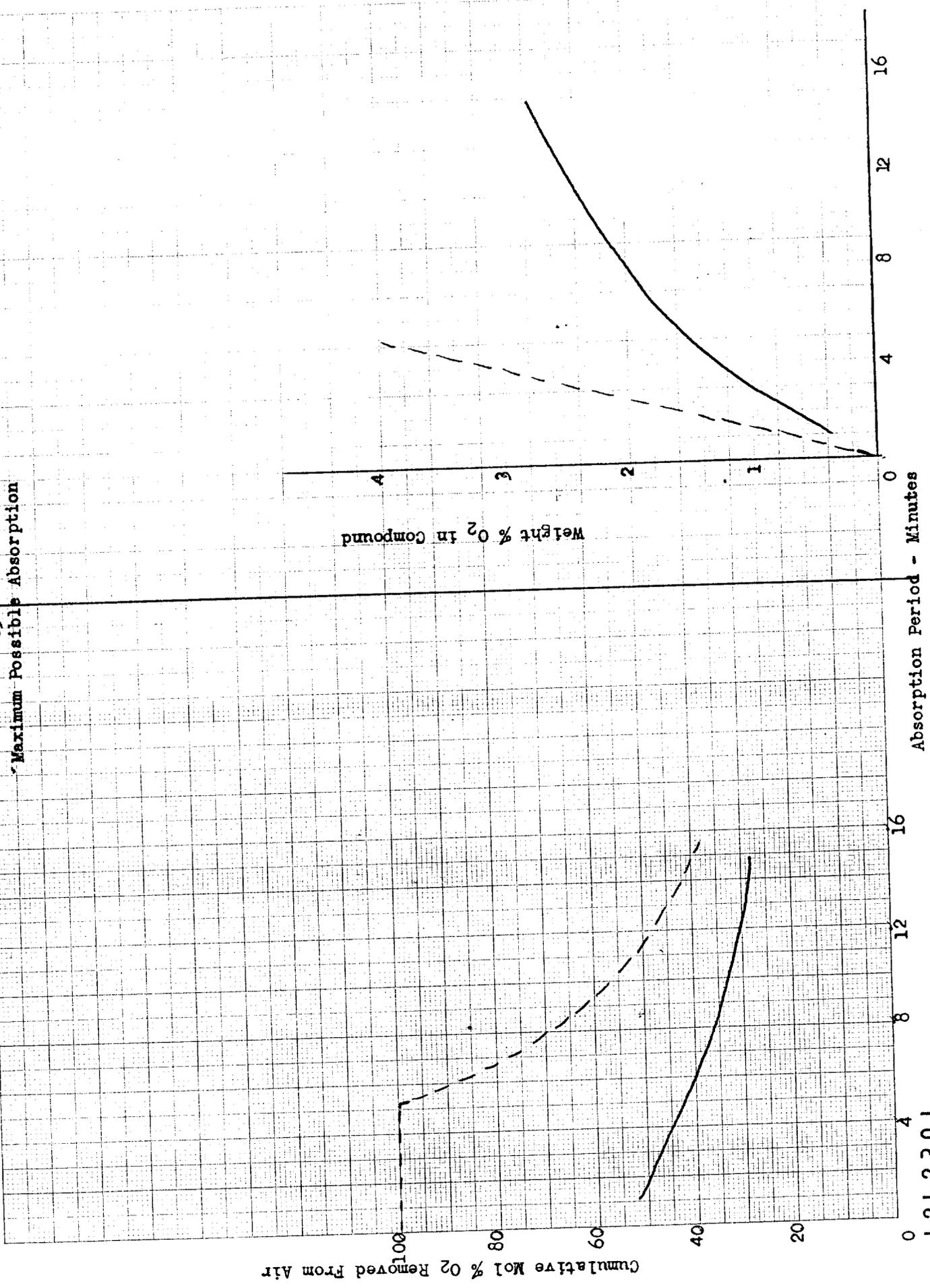


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DESCRIPTION TIME-MIN.
Figure 3.

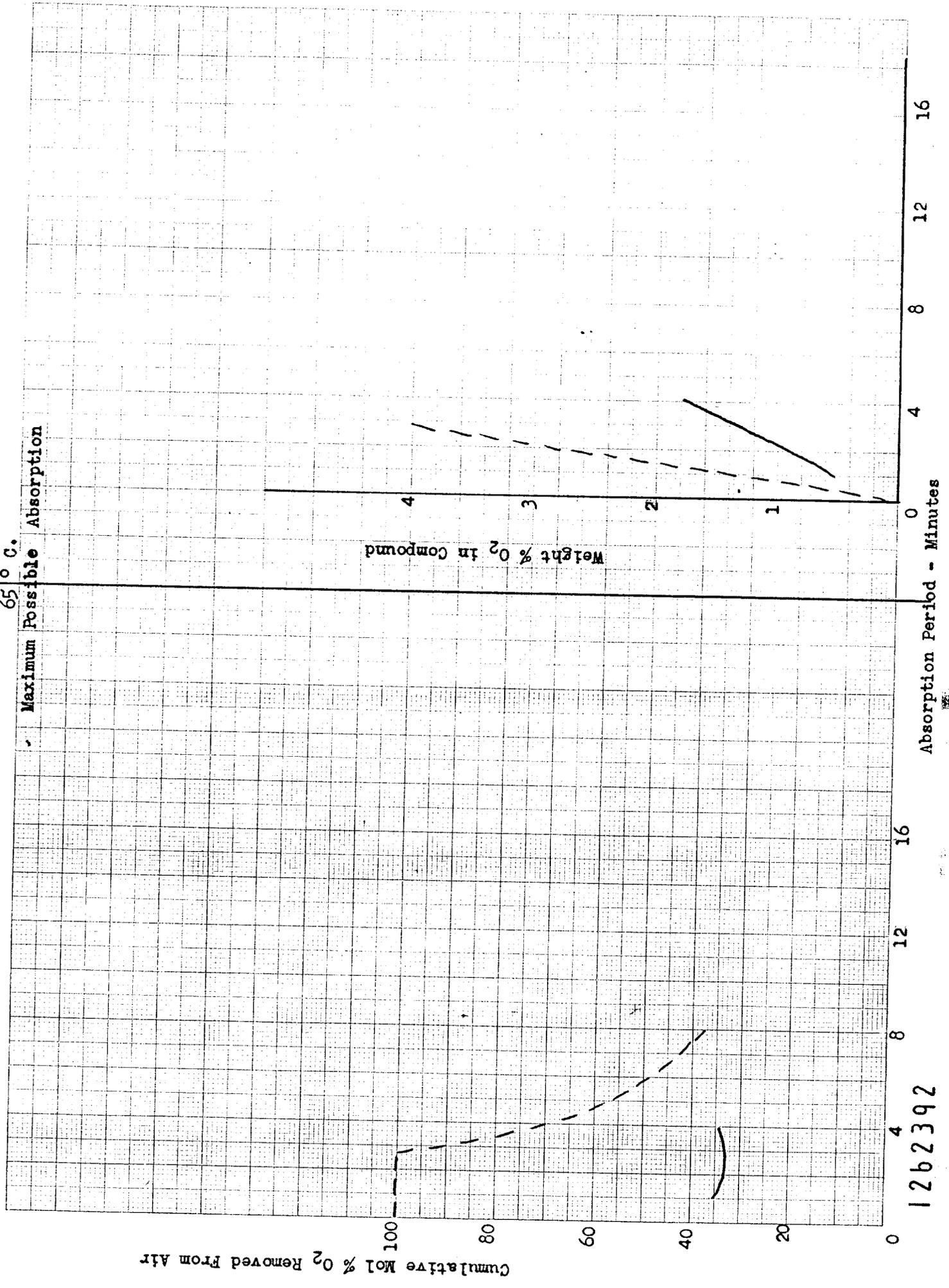
Figure 4
600 Lbs./sq. in.
2 C. F. H.
65° C.

Maximum Possible Absorption



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Figure 5
600 Lbs./sq. in.
4 C.F.H.
65° C.

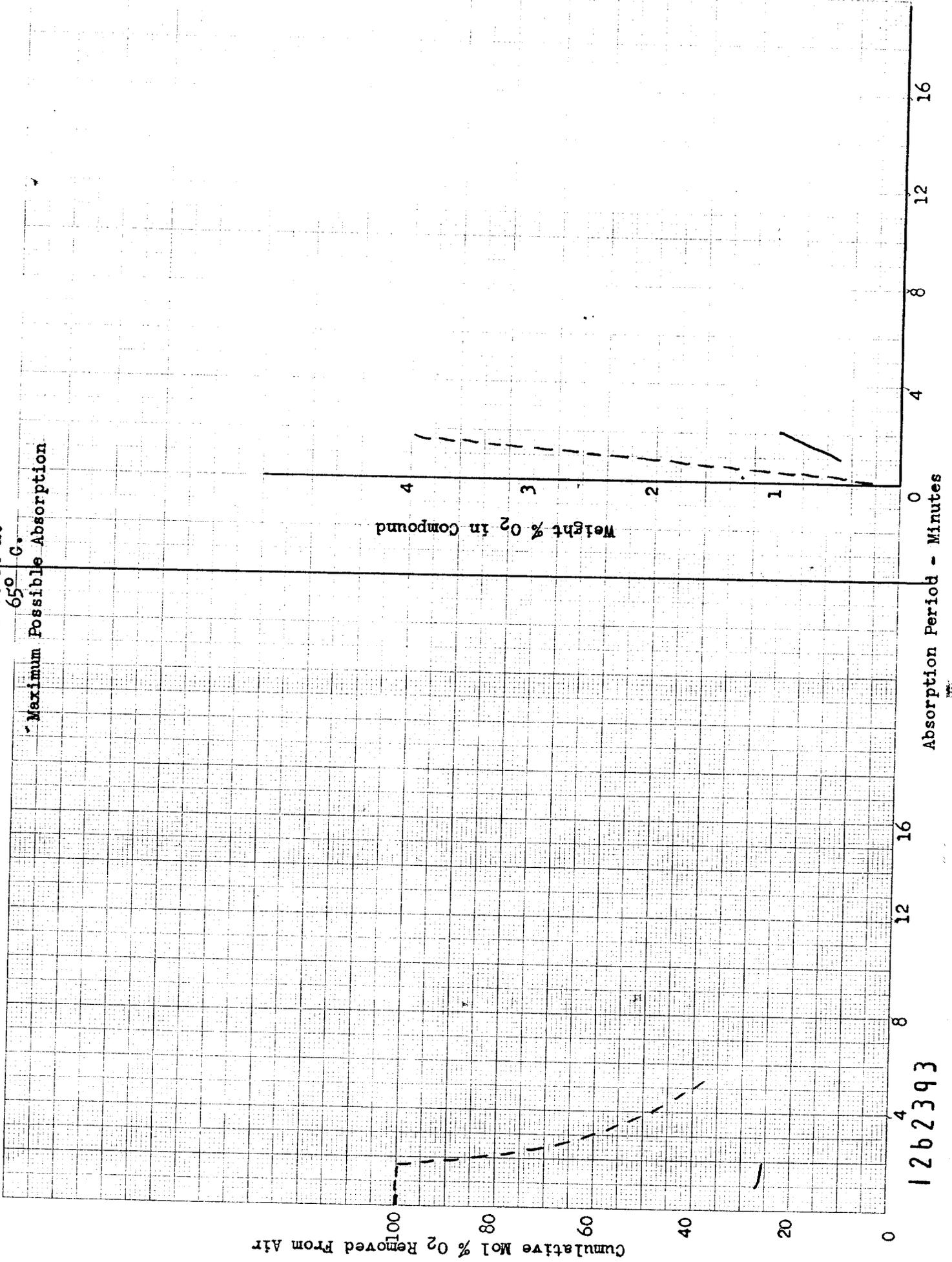


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Figure 6

600 Lbs./sq. in.
6 C.F.H.
650 G.

Maximum Possible Absorption



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Figure 7

600 Lbs./sq. in.
0.87 C. F. d.
65° C.

Maximum Possible Absorption

Cumulative Mol % O_2 Removed from Air

Weight % O_2 in Compound

0

20

40

60

80

100

0

4

8

12

16

0

1

2

3

4

12

16

16

Absorption Period - Minutes

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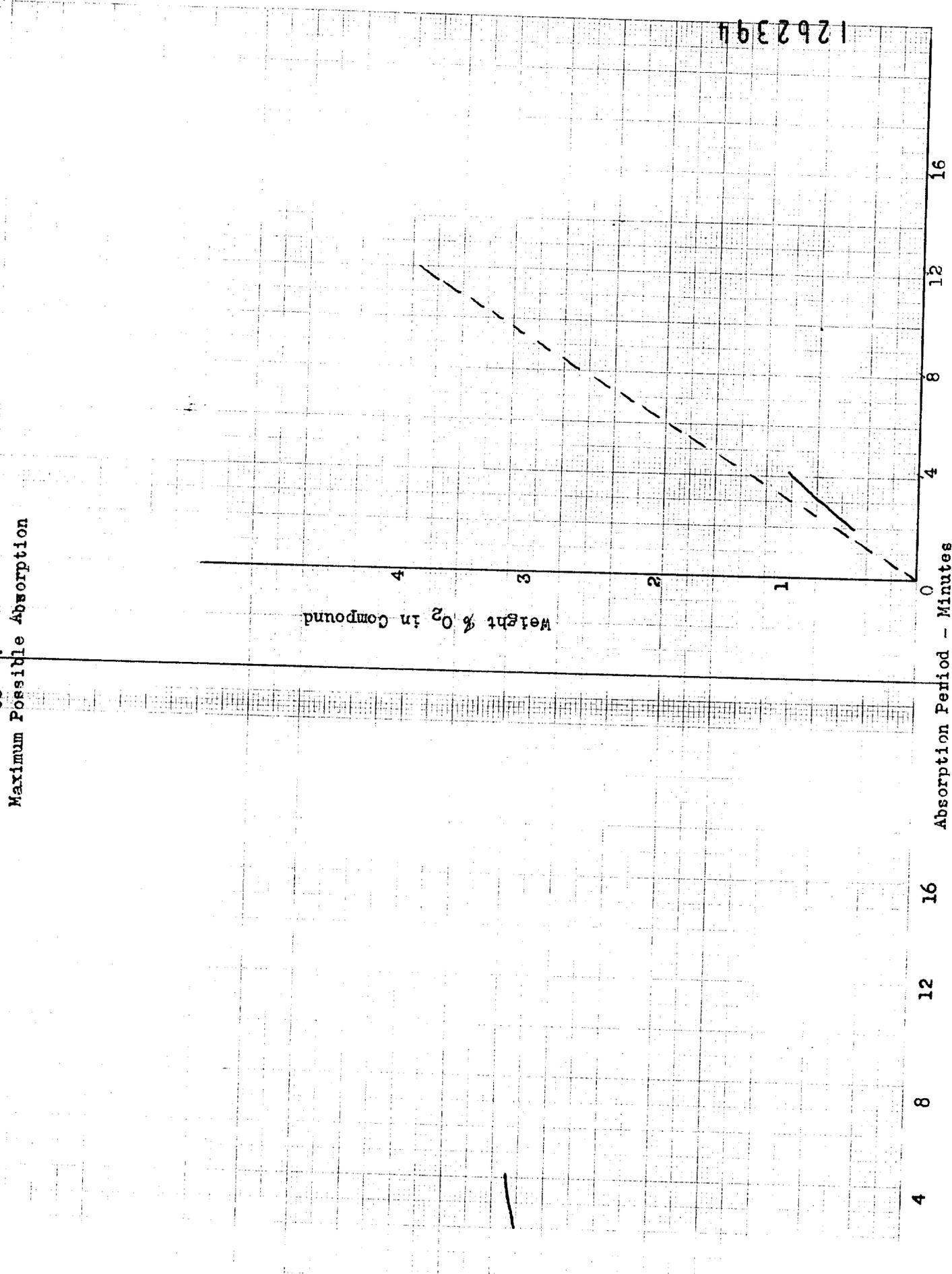
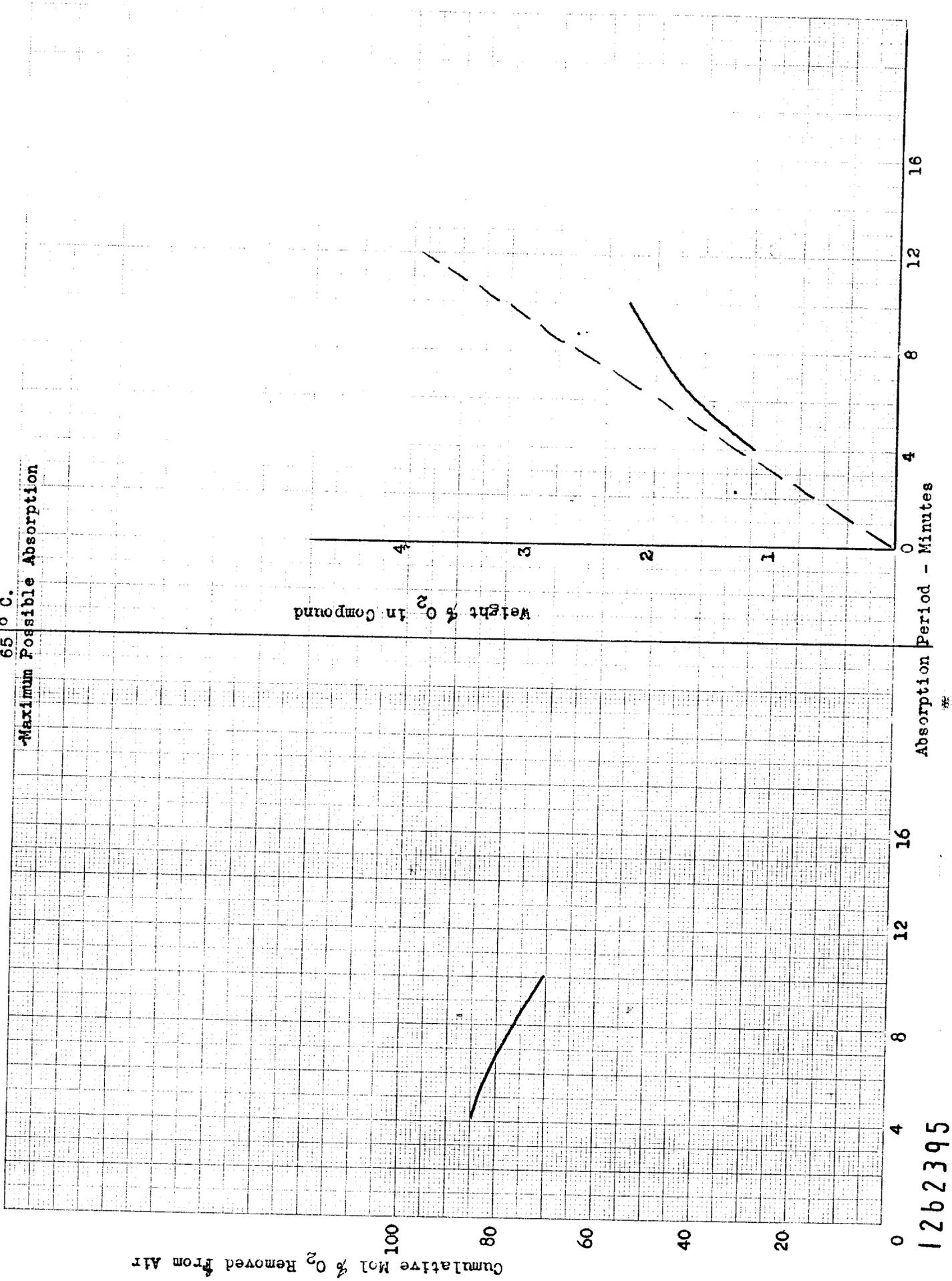


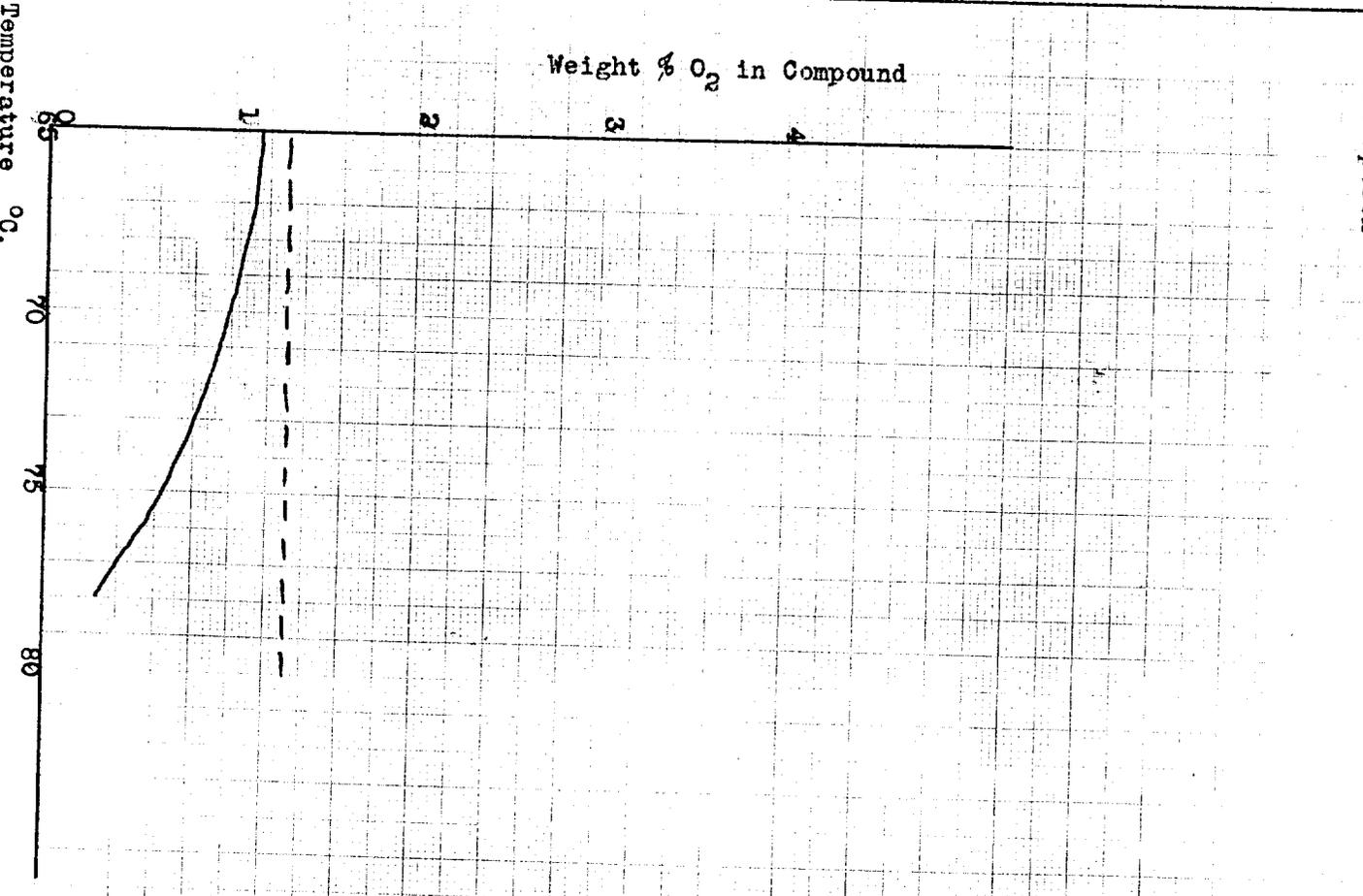
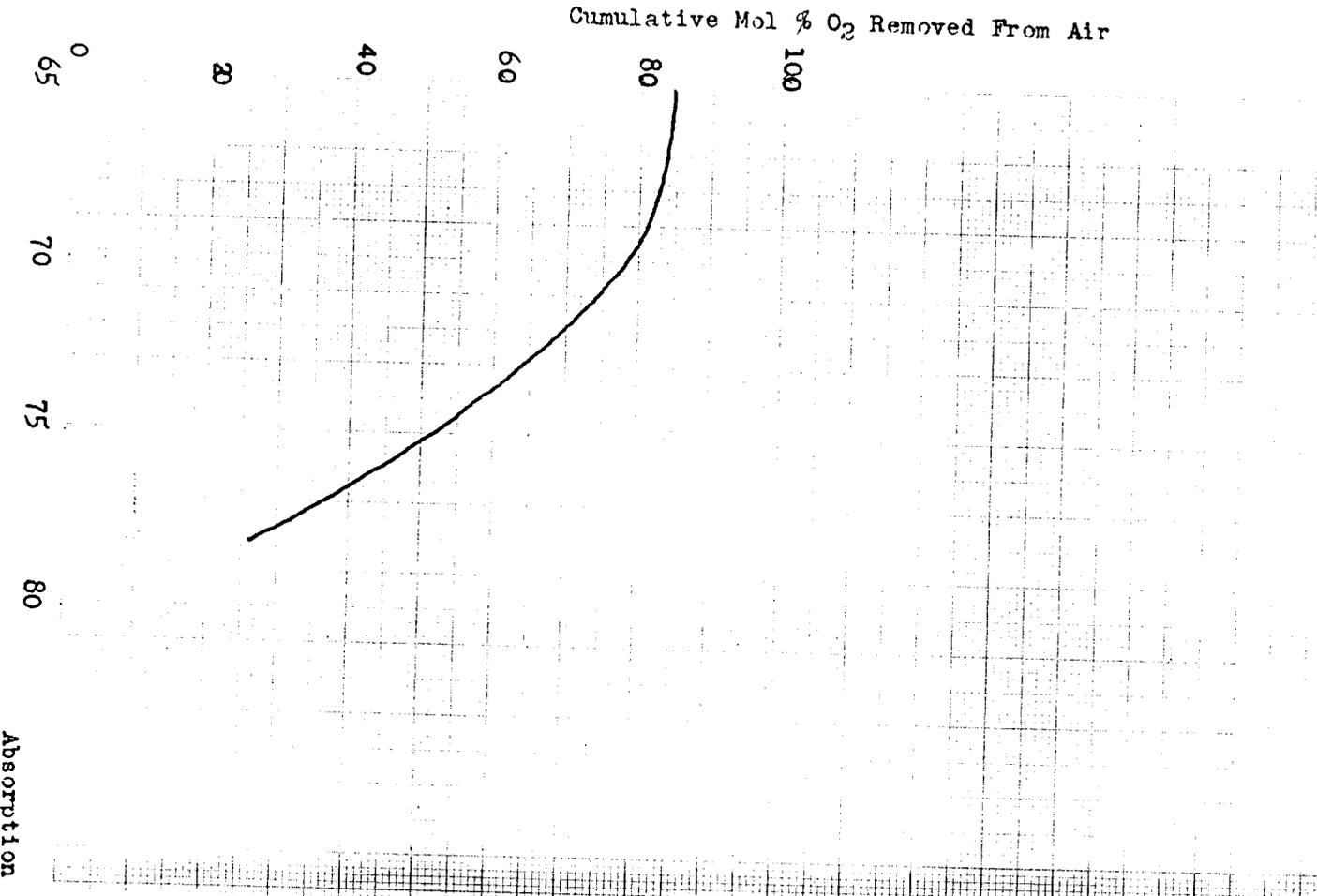
Figure 8
800 lbs./sq. in.
0.87 C. F. H.
65° C.

Maximum Possible Absorption



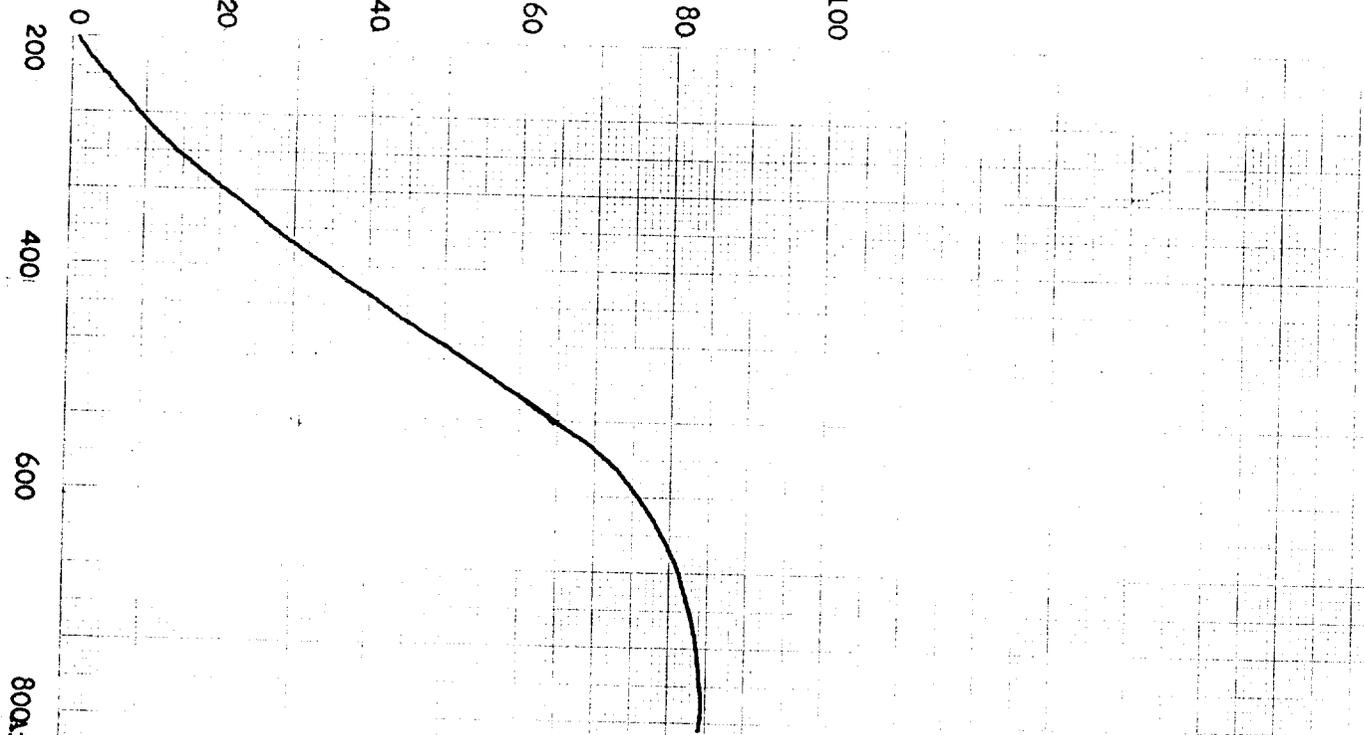
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Figure 9
 0.87 C. F. H.
 800 Lbs./sq. in.
 4 Min. Absorption Period
 Maximum Possible Absorption



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Cumulative Mol % O₂ Removed From Air

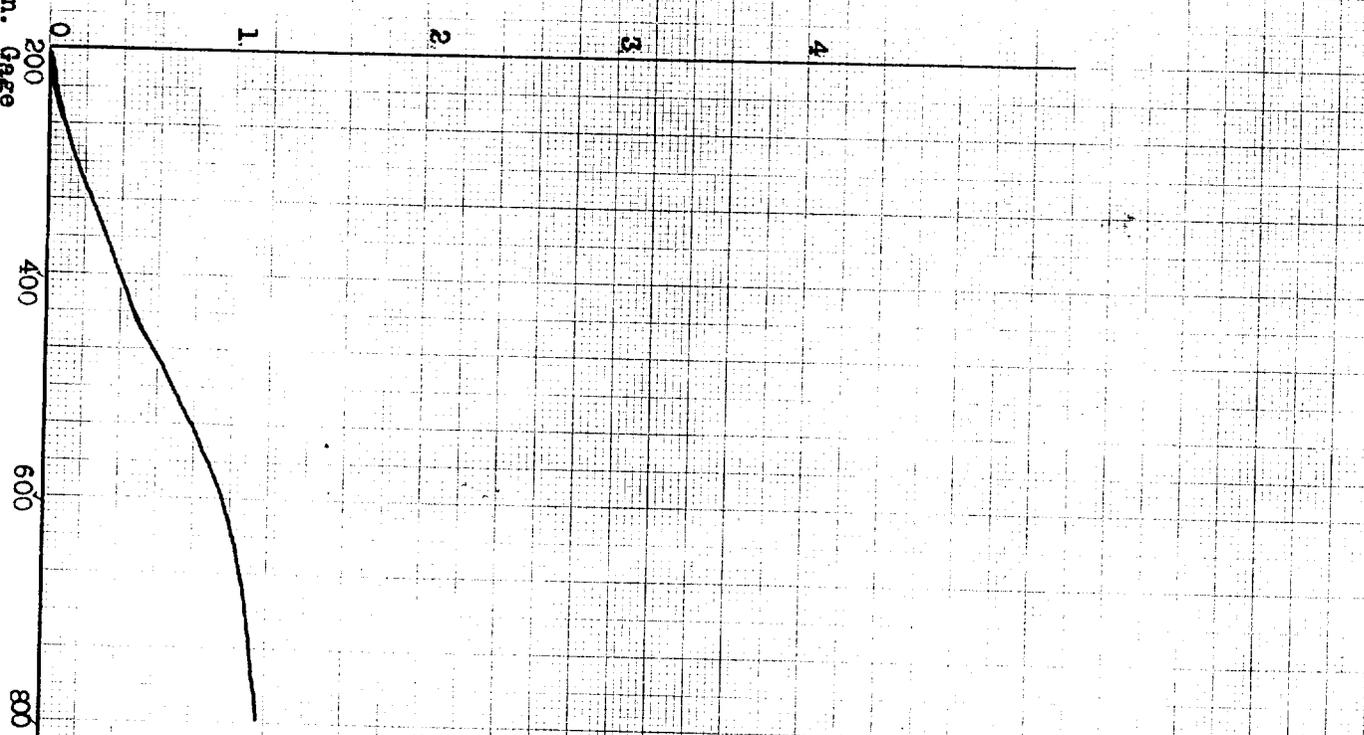


800 Air Pressure - Lbs/Sq. In. Gage

4 Min. Absorption Period
Maximum Possible Absorption
65° C.

Figure 10
0.87 C. F. H.
65° C.

Weight % O₂ in Compound



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