

The Effect of Time and Temperature of Burning
Upon the Properties of Hydromagnesite

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INTRODUCTION

The Engineering Experiment Station of the Virginia Polytechnic Institute has been investigating the properties of lime with regards to its keeping qualities, its rate of decarbonation, its carbonation, and the hydration of burnt lime for several years.

In this study, many things had been discovered concerning lime which had not been known heretofore, but in the search for new facts, it was decided to diverge from lime to a study of magnesia. Since lime and magnesia are so closely related to each other in every way, a study of the properties of magnesia was undertaken. Magnesium has a number of properties in common with calcium. It is a little more active than calcium, and it forms more compounds than does calcium. This property of magnesium makes it a difficult substance with which to work. Magnesium also displaces part of the calcium in calcium carbonate to form a calcium and magnesium carbonate, which is called dolomite.

In this work on magnesia, the rate of decarbonation of hydromagnesite has been selected as a method of studying the properties of magnesia. This rate of decarbonation at different temperatures gives one a general idea on how tightly the carbon dioxide is held in the carbonate, and is a very good way to compare magnesia with lime, since the research on the rate of decarbonation of lime has already been done. By the rate of hydration of this magnesia we have another means of comparison with the properties of lime.

The main purpose of this work is to illustrate by means of graphs and tables the decarbonation rate of hydromagnesite.

Review of Literature

The word magnesia was first used to designate a magnet or lodestone, but in alchemy it came to mean "a stone shining like silver" and supposed by some to be an ingredient of the philosopher's stone. Paracelsus used the word to mean an amalgam. Its employment in its present sense seems to have originated not in connection with the alchemical use of the word, but by the "magnus carneus" (flesh magnet) applied about 1550 by Cardan to a white earth because it adhered to the lips very strongly. The term "magnesia alba" (in contradistinction to "magnesia nigra" used for the oxide of manganese) was first used by Hoffmann in 1722. In Germany, the terms Bittersalzerde, Bittererde, and Talkerde were employed for magnesia. In 1808, H. Davy proved that magnesia is the oxide of a metal which he named magnium.

Magnesia was not clearly distinguished from lime until the eighteenth century when Black showed that the former gave a soluble sulphate. Magnesia had long been confounded with lime until Hoffmann showed that it differed essentially with lime. It therefore has no individual early history. Magnesia alba appears to have come into commerce from Rome about 1700 A.D. The calcination of magnesium carbonate, like that of calcium carbonate, was explained by Black and Lavoisier (1) whose experiments finally overthrew the phlogiston theory.

Magnesium metal does not occur free in nature. This element is considered to be distributed on the earth's crust. It occurs extensively as the oxide, hydroxide, aluminate, alumin-

ferrite, silicate, and carbonate.

The oxide has been called magnesia, calcined magnesia, burnt magnesite, Talkerde, Bittererde, magnesia usta, magnesia calcinata, etc. It was discovered by A. Scacchi at Monte Somma, Vesuvius, as a mineral called periclase. MgO is a product of the oxidation of the metal. The oxide is also produced by the calcination of many of the salts of magnesium, such as the carbonate, sulphate, nitrate, etc. O. Schumann made it by the calcination of MgS in a current of steam. MgO is made by heating the chloride in a stream of moist air and also by heating magnesium chloride with calcium chloride. Magnesium oxide is generally prepared by burning the metal in air or oxygen or by the thermal decomposition of an oxygen containing compound of magnesium. Commercially it is made from the carbonate, basic carbonate, or hydroxide by heating. The MgO of pharmacy may be prepared in two forms -- magnesia levis and magnesia ponderosa, by igniting the light and heavy basic carbonates respectively. Unlike calcite, magnesia is seldom found in the pure state, and the pure oxide is generally prepared from the precipitated hydroxide or basic carbonate.

The relationship between magnesia and lime is so close that it is often necessary to refer to experiments on lime to properly study magnesia. As is known in the case of quick lime, magnesia changes by absorbing moisture from the atmosphere. When the magnesia starts changing, it is easily noticed because it forms a lumpy sort of material; when it absorbs carbon dioxide from the air, the magnesia remains in the same physical state. The material deteriorates but there is no outward appearance of change on

absorption of carbon dioxide as in the case of absorption of moisture.

The magnesia hydrate reacts with carbon dioxide to form the carbonate as well as does the lime hydrate. Experiments have been in progress at the Virginia Polytechnic Institute on the keeping qualities of hydrated lime for some years. A number of bags of hydrate were obtained some years ago and stored under commercial storage conditions. The original weights of the bags were recorded and the bags were weighed from time to time. By the increase in weight, the reaction of the hydrate with atmospheric carbon dioxide was found to continue steadily. This experiment was started years ago, and the reaction has not come to equilibrium as yet. Thus, when lime is wanted for structural purposes for which there is a definite specification for the carbon dioxide content, the carbon dioxide content of some limes may be higher than the specifications allow if the lime is rather old. Only when the lime is new does one get the carbon dioxide content as specified unless the lime is contained in a carbon dioxide proof bag. In the same way, when one calls for a magnesia with a definite carbon dioxide content, he may or may not get it because the carbon dioxide in the magnesia varies with the time.

With reference to the carbon dioxide content, much work has been done with the action of heat on magnesium carbonate. When magnesium carbonate is subjected to heat, carbon dioxide is evolved. The conclusions drawn by different observers are not always in agreement. H. Rose (2) stated that magnesium carbonate begins to lose carbon dioxide between 200° and 300° and that when heated

to a dull red heat all carbon dioxide is lost. On the contrary, Marchant and Scheerer (3) stated that after a prolonged calcination at a bright red heat, the product still retains carbon dioxide. W. C. Amerson (4) found the lowest temperature at which magnesite begins to lose carbon dioxide is 405° ; with the so-called heavy carbonate, the temperature is 368° ; with the light carbonate, the temperature is 352° ; and with the crystal carbonate, the temperature is 271.5° . In the case of the light and crystal carbonates, expulsion of carbon dioxide was stated to be complete at about 750° while the heavy carbonate retains a little of its carbon dioxide at temperatures over 810° . H. Le Chatelier (5) found that under atmospheric pressure carbon dioxide is given off from magnesite at 680° , and Hedvall (6) gave 546° for the dissociation temperature. K. Friedrich and L.G. Smith (7) found that the decomposition of magnesite begins about 570° , and a maximum thermal change in the heating curve occurs at about 600° . A. Vesterberg (8) found that magnesite decomposes more readily than dolomite, that magnesite first gives off carbon dioxide at 448° , and loses virtually all that gas when heated for an hour at 500° , whereas dolomite loses but little if any carbon dioxide at that temperature.

Hempel and Schubert (9) studied some substances which were heated in an electric furnace, the temperature of which was determined by a Le Chatelier pyrometer. They found that magnesium carbonate began to dissociate at 350° and ended at 900° . Gmelin-Kraut's handbook gives some percentage losses of carbon dioxide (10). After drying at 100° , magnesite loses at 200° 0.04 % CO_2 , and the weight remains constant until 300° . At these temperatures it is

water-free. At 175° , magnesite loses 0.0106 % CO_2 , and even at glowing heat in the Sefstromsches furnace there remains 0.055 % CO_2 . Also at 300° dried magnesite holds still a trace of water, which it loses at glowing heat. Natural magnesite loses under twenty hours at 350° 0.4 of its weight in carbon dioxide, but by raising the temperature, it loses CO_2 very fast. Pure carbonate will lose all its carbon dioxide at 750° to 810° . The basic carbonates on heating decompose with the liberation of carbon dioxide at a much lower temperature than magnesite, a temperature of 230° to 300° being sufficient. Nevertheless, Anderson found that temperatures of 750° and 810° respectively were required for the complete expulsion of the carbon dioxide from light and heavy magnesia alba. It is evident that our knowledge of the chemistry of the carbonates of magnesium -- normal, basic, and hydrated -- is limited, and that the whole subject requires comprehensive investigation.

O. Brill (15) studied the dissociation of artificial normal magnesium carbonate, and, starting with 68.70 grams, he measured the loss in weight after heating ten minutes at different temperatures ranging from 135° to 630° ; the following is a selection from a few of his measurements of the weight after heating to different temperature:

	135°	237°	265°	300°	340°	360°	435°	465°	500°	630°
Grams	68.7	67.9	64.6	64.1	63.4	62.9	59.7	41.1	38.1	32.9

There are a series of breaks in the curve. He considers that this proves that the decomposition of magnesium carbonate takes place in a series of stages corresponding with the formation of a series of basic carbonates each of which has a definite dissociation

temperature. The carbonates with their dissociation temperatures are as follows:

10 MgO.9CO ₂ 265°	9MgO.3CO ₂ 295°	8MgO.7CO ₂ 325°	7MgO.6CO ₂ 340°	6MgO.CO ₂ 380°
	5MgO.4CO ₂ 405°	7MgO.1CO ₂ 510°		

As W. A. Davis (16) pointed out, the fact that O. Brill worked with a hygroscopic magnesium carbonate having the composition $\text{MgCO}_3 \cdot \frac{1}{3}\text{H}_2\text{O}$ deprives his results of real significance as examples of magnesium carbonate. The presence of water caused the material to break down at 230°, a much lower temperature than that required for the dissociation of magnesite, but approaching the temperature required for the decomposition of the trihydrate. R. Marc and A. Simek also emphasize the fact that precipitated magnesium carbonate retains water very tenaciously. Accordingly, the breaks are probably due to some cause other than the formation of basic carbonates.

In 1914 Tzentnershver and Bruzs (17) studied the thermal dissociation of magnesite by a dynamic method which permits of a complete separation of the dissociation products. The substance is heated in a nickel boat in an electric furnace, and the temperature and pressure are measured. The dissociation temperature is determined from the form of the heating and cooling curves. Kahlbaum's pure magnesium carbonate which had been dried for over three days at 220° in a stream of carbon dioxide began to dissociate at 270° but on repeating the test, the dissociation temperature rose to 368°. Water vapor came off during the experiment due to the presence of water of constitution in the sample of carbonate. The sample dried at 220° was therefore further dried at 320° in a

stream of carbon dioxide for three days. The carbonate thus prepared dissociated according to the following stages:

- (1) $2 \text{MgCO}_3 = \text{MgO} \cdot \text{MgCO}_3 + \text{CO}_2$; dissociation temperature 373°
- (2) $2 (\text{MgO} \cdot \text{MgCO}_3) = 3 \text{MgO} \cdot \text{MgCO}_3 + \text{CO}_2$; diss. temp. 442°
- (3) $3 \text{MgO} \cdot \text{MgCO}_3 = 4 \text{MgO} + \text{CO}_2$; diss. temp. 469°

With natural rhombic magnesite, the stages were as follows:

- (1) $4 \text{MgCO}_3 = \text{MgO} \cdot 3\text{MgCO}_3 + \text{CO}_2$; diss. temp. 402°
- (2) $\text{MgO} \cdot 3\text{MgCO}_3 = 2 (\text{MgO} \cdot \text{MgCO}_3) + \text{CO}_2$, or
 $2\text{MgCO}_3 = \text{MgO} \cdot \text{MgCO}_3 + \text{CO}_2$; diss. temp. 437°
- (3) $\text{MgO} \cdot \text{MgCO}_3 = 2 \text{MgO} + \text{CO}_2$; diss. temp. 480°

The first dissociation product of magnesite, monoxycarbonate, is metastable. Experiments in which the completely or partially dissociated carbonate was heated in a stream of carbon dioxide between 200° and 230° showed that under these conditions, the reverse formation of carbonate takes place extremely slowly.

By measuring the carbon dioxide evolved, Tzentnershver and Bruss (18) measured the rate of decomposition of the magnesium carbonates, which follows: MgCO_3 at 395° ; $\text{MgO} \cdot \text{MgCO}_3$ at 452 , 460 , 462 ; $3 \text{MgO} \cdot \text{MgCO}_3$ at 487° . The reaction is always of the first order, and the total process can be resolved in a series of consecutive steps. The reaction constant is an exponential function of the temperature.

Marc and Simak (11) studied the dissociation pressure of magnesium carbonate at various temperatures. This was made difficult by the extreme slowness of the reactions with completely dry carbonate at 300° to 400° . The maximum pressure was not attained in weeks, and dry magnesia could be left in contact with carbon dioxide at 100° below the dissociation temperature for days with-

out any being taken up. Both processes went slower the more completely dried the material used; on the other hand, water catalyzed both reactions. Because of the above disturbing factors, attempts to determine the pressure-temperature curve of magnesium carbonate by the static method failed; in some experiments KNO_3 was added as catalyzer, but bad results were obtained.

Pure magnesia prepared from the hydroxide or carbonate at low temperature readily combines with water to form the hydroxide. The oxide, prepared by heating the native carbonate or nitrate to redness, when mixed with a limited amount of water sets hard, but this property of hard setting is not shared by all oxides made at low temperature. Some low temperature oxides, like lime, absorb water without setting. If the oxide is heated to a high temperature, it loses the property of absorbing water, especially if it contains a trace of iron oxide.

Magnesia gradually absorbs moisture and carbon dioxide from the atmosphere. Rhodes, Jones, and Dougan (19) ran experiments on MgO prepared at low temperature. They found that magnesia prepared at low temperature and exposed to ordinary air absorbs both water and carbon dioxide. They showed that a magnesian lime absorbs water and carbon dioxide concurrently, whilst a pure CaO absorbs very little carbon dioxide until it is fully hydrated. This suggests that MgO , unlike CaO , absorbs carbon dioxide directly at ordinary temperatures, but this matter requires further investigation.

E. De M. Campbell (20) has measured the rate of hydration of MgO burned at different temperatures. The hydration of magnesia burned between 600° and 800° is practically complete in three days. A change in the constitution of MgO sets in between 1000° and 1100°

resulting in a marked decrease in the rate of hydration, but even this high temperature product combines slowly with water. This change becomes more and more marked with rise of burning temperature until at 1450° , or nearly the temperature employed in burning portland cement, the MgO combines with only 61.4 % water required for complete hydration. It has been found that the MgO undergoes a change at 800° very slowly, and probably involves a conversion from amorphous to crystalline periclase. It has already been discovered that there are two main types of magnesite -- one type is amorphous or a gel colloid and is represented by the compact, granular, and cleavable forms; the other type occurs in distinct rhombohedral or trigonal crystals.

Parravano and Mazzetti (12) found no discontinuity in the change in the rate of hydration with increase of temperature of calcination. They showed that the velocity of hydration depends on both duration and temperature of heating and on the presence of impurities. They attribute high activity to magnesia in the amorphous condition, which is changed to crystalline periclase on prolonged heating. Moller (13) has also shown that there is no definite temperature of transformation. The change is in all respects similar to that which calcium oxide undergoes, and it is probably to be attributed to an alteration in the exposed surface of the solid. It has been shown that even a highly crystalline oxide is slowly hydrated, and therefore the change is not from the active to the entirely inactive state. Nevertheless, Moller thinks that there are two distinct forms, alpha and beta magnesia, of specific gravities 3.2 and 3.7 respectively.

In his work, Eckel (14) discovered that if magnesite be

heated, the effect as with calcium carbonate is to drive off the carbon dioxide leaving magnesia. A curious and technologically important phenomenon connected with the temperature employed was noted. If the calcination be carried on quickly at a red heat, the magnesia resulting will have a specific gravity of 3.00 to 3.07. If the calcination is long continued or carried on at a higher temperature, the resulting magnesia will be much denser, possessing a specific gravity of 3.61 to 3.80.

The technologic importance of the two forms of magnesia lies in the fact that the lightly-burned magnesia will slake with water and if then exposed to air, it will finally recarbonate and harden slowly, just as lime does. The denser higher-burned magnesia, however, will not take up either water or carbon dioxide from the atmosphere. Another difference of commercial interest lies in the fact that the light form of magnesite possesses a certain amount of plasticity so that it can be molded into shape under heavy pressure, while the dense form of magnesia is entirely devoid of plasticity.

In 1918 Campbell (21) recorded a condition of hydration up to six years. The MgO obtained from the burning of MgCO₃ contained 90.78 % MgO. Completely to hydrate this required an average of 44.59 % of added water. It has already been noted that all samples burned at not over 1100° hydrated completely within three months and that samples burned at 1200° hydrated very slowly. The results of this experiment show that samples burned at 1200° undergo complete hydration in three years while samples burned above 1200° are not completely hydrated even after six years. The temperatures and per-cent of hydration after six years are:

Temperature	1500°	1400°	1450°
Percent	83.5	81	70.3

These results show why the materials containing free MgO, when burned at temperatures approaching those of cement manufacture, will not hydrate completely even when immersed in water for twenty years or more.

Although it has been known for some time that the deleterious expansion of portland cement is due to the presence of MgO, authorities have differed as to the allowable percentage by amounts not easily reconcilable. Campbell (22) has already shown that this expansion is due to the slow hydration of free MgO. From the action of certain natural cements high in magnesia, one could infer that the temperature of burning has an influence on the rate of hydration which would account for the differing opinions as to allowable amount. Campbell has carried out a systematic study of the rate of hydration of MgO obtained from burning a sample of magnesite (containing some calcium carbonate) for one hour at temperatures rising by 100° intervals from 500° to 1450°. The heating was carried out in an electric furnace which could be held constant within a few degrees during the time of heating. The percentage gain in weight by hydration was then determined for various time intervals from a few days to eighteen months. The results showed that the decomposition of MgCO₃ is not complete at 500° in one hour, but is at 600°, while CaCO₃ requires a still higher temperature. The hydration of MgO burned at 600° to 800° is practically complete in three days. Between 800° and 900°, the dissociation of CaCO₃ completes itself and combination between basic and acid oxides produces

aluminates and silicates which require more water for hydration than the theoretical for all the CaO and MgO present. Between 1000° and 1100° , a change sets in which results in a slower rate of hydration of MgO so that at 1450° (practically the temperature of cement burning) MgO is only 61.4 % hydrated after eighteen months.

The study of the carbon dioxide content in magnesite, and, the hydration of magnesia is very important in the cement industry. If the magnesia has been prepared from magnesite, it always contains carbon dioxide, and although a cement made from such impure magnesia sets very rapidly and yields a very hard concrete, the gas passes off during the setting and gives rise to the formation of cracks. Supposing it to be sufficiently pure to contain about 90 % MgO, the cement prepared therefrom sets and hardens less rapidly than that mentioned above, but it yields a concrete quite free from cracks.

It has been known that magnesia prepared by heating $MgCl_2$ or the nitrate to redness has hydraulic properties in that when mixed with a limited quantity of water, it sets rigid like portland cement, and the product is hard enough to scratch marble. On the other hand, magnesia prepared by calcination for twelve hours at a white heat no longer sets with water, or, if it does, the setting is very slow. Magnesia prepared by calcining the nitrate at 350° , when fashioned into rods and spheres with water, remains soft when under water eight months; that calcined at 440° hardens under water and in two months is like polished marble; that calcined at 560° also is very hard after standing two months under water; while that calcined at 1200° does not harden under water. According to W. C. Anderson, MgO prepared by gently heating the native carbonate reacts

quickly with water, and when in presence of a limited amount of water sets to a firm mass. The product obtained by heating similarly the artificial carbonates does not set, although it also appears to slake rapidly with water; but if these carbonates be treated with nitric acid and the resulting nitrate ignited gently, the oxide left will set similarly to that obtained from the native carbonate by heat. The chemical properties of MgO depend even more than those of CaO on the temperature to which it has been heated and the duration of the heating.

Magnesia, though possessing very marked cementing properties, is at present too expensive to be used as a cementing material for ordinary structural purposes. It merits discussion however because (a) it is the basis of an extensive magnesia brick industry, (b) under certain conditions it possesses hydraulic properties, and (c) the factor brought out in a description of the manufacture of magnesia and magnesia brick may serve to throw some light on the vexed question of the part played by magnesia when present in hydraulic cements.

The Investigation

General

The object of this investigation was to decarbonate hydromagnesite by a thermal method, and after decarbonation, to hydrate the samples until complete hydration resulted. This thermal method consisted of heating the samples to the desired temperature in a furnace, the temperature of which could be controlled within a few degrees. The samples were then taken out at different time intervals at this definite temperature and the loss of carbon dioxide recorded. When the point of complete decarbonation was reached, the sample was put in the hydrating room, and readings were taken until a condition of complete hydration was reached.

It was necessary to use chemically pure magnesium carbonate, which is actually hydromagnesite instead of pure magnesium carbonate, because the latter was not available. The formula for hydromagnesite is $3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$, and this substance has a molecular weight of 365.34. The hydromagnesite that was used in the investigation was crushed to break up the lumpy material and then thoroughly mixed to make it as uniform as possible.

Decarbonation of hydromagnesite

The thermal method was decided upon as the best method to use for the decarbonation of hydromagnesite. Some preliminary work had been done on the decarbonation of hydromagnesite at the Virginia Polytechnic Institute by Dr. Holden and Mr. Jefferson, but their investigation had only extended to one or two different temperatures and the work had never been fully completed. It was first necessary

to carry out at different temperatures ranging from 550° to 1000° at 50° intervals a few preliminary runs to determine approximately what length of time it would take for the carbon dioxide to be given off at each temperature. While running these preliminary tests, it was found that at the high temperatures, some of the magnesium carbonate was being lost. This was explained by the fact that when the crucible was placed in the furnace at a high temperature, the carbon dioxide bubbled through the magnesia so rapidly in its effort to escape that the uprush of carbon dioxide caused some of the magnesia to be blown out of the crucible. To prove this, when the crucible was removed, a white ring of magnesia could be seen surrounding the spot where the crucible had stood. Much time was spent in trying to devise a means of avoiding this loss of material. A crucible cover was the first thing that came to mind, but there was no pair of tongs that could be used to put a crucible in the furnace with the cover on. Finally a method was devised by which a dessicator bottom was used. The crucibles with the covers on were placed in the holes in the dessicator bottom, and the whole bottom was placed in the furnace by means of the tongs. At first it was thought that the dessicator bottom would crack when used at high temperature but nothing like this happened. This method proved very efficient, because it not only permitted three crucibles with their covers to be placed in the furnace at the same time but also it kept the crucibles above the floor of the furnace.

Another difficulty arose at this time. It was suggested that the loss of carbon dioxide when using the crucible covers on the crucibles would not correspond with the loss of carbon dioxide using

the crucibles minus the covers. A test was run at 600° with two samples having the same weight of hydromagnesite. One crucible had a cover on it and the other crucible did not. The test was run for thirty minutes, and upon cooling and weighing the two crucibles, the loss of carbon dioxide checked within 0.4%. The sample in the crucible with the cover on lost 97.7% of its total CO₂, and the sample in the crucible without the cover lost 98.1% of its total CO₂.

The crucibles and crucible covers used in the tests were first heated to a red heat, cooled, and then weighed until a constant weight was reached. A 0.2 gram sample was then weighed out into each of the crucibles. The samples were placed in the furnace, which was at some definite temperature, and at different time intervals, a crucible was taken out of the furnace, cooled, and weighed. The difference between the weight recorded and the original weight of the 0.2 gram sample is the amount of carbon dioxide that was lost. At other intervals crucibles were taken out at this same temperature, until finally a sample showed complete decarbonation.

When it was first decided to place more than one crucible in the furnace at one time, it was thought that the pressure of CO₂ that was first evolved from the samples in these crucibles would be large enough to stop any more evolution of carbon dioxide. To prove that this would not have any appreciable effect, the calculation of the amount of the carbon dioxide in the furnace, when six crucibles containing hydromagnesite (which amount was usually the maximum number put in the furnace at one time) were used at one time, was made. The furnace was a Hoskins Electric Furnace sold by the Fisher Scientific Company of Pittsburgh. It used 220 volts stepped down to 44 volts and required 114 ampere current. The pyrometer was a Brown

Electric Pyrometer sold by the Brown Instrument Company of Philadelphia. The size number of the furnace was 204 and the muffle was measured to be $7 \frac{3}{10} \times 5 \times 12 \frac{1}{2}$ inches. It was calculated that there was 36% CO_2 in the hydromagnesite, and there were therefore 0.432 grams of CO_2 in six crucibles using 0.2 gram sample. From the furnace measurements, the furnace contained 0.264 cubic feet air or 0.0077088 lb. air. The carbon dioxide in the furnace was found to be 0.00008 lb. This showed that the CO_2 pressure in the furnace was negligible.

Among the other preliminary work which was done, the rate of decarbonation of samples burned at 500° was investigated. The samples lost weight quite steadily for two hours until a definite loss was reached. After this point attained, regardless how long the sample was heated, it would not lose any more weight. The carbon dioxide loss at this point averaged 90% of the total carbon dioxide in the sample. Evidently another compound having a basic carbonate structure was formed at this point.

Hydration of magnesia

When the sample was completely decarbonated, it was immediately taken to the hydration chamber, and placed in a dessicator containing water. The sample was placed in the dessicator without any crucible top. At frequent intervals the sample was taken out, allowed to come to room temperature, and weighed. This was done until the calculated point of complete hydration was reached. This point was calculated to be a gain of 0.0404 grams over the weight of the decarbonated product. While weighing, the crucible cover was used to prevent addition of CO_2 as much as possible.

Since the amount of vapor which can exist in the air depends chiefly on the temperature of the air, then, if any moist air is made colder, the relative humidity will be increased; and if it is cooled far enough, the relative humidity will become 100%, and saturation will occur. The dew-point is that temperature of the air at which its invisible moisture begins to condense into visible water drops.

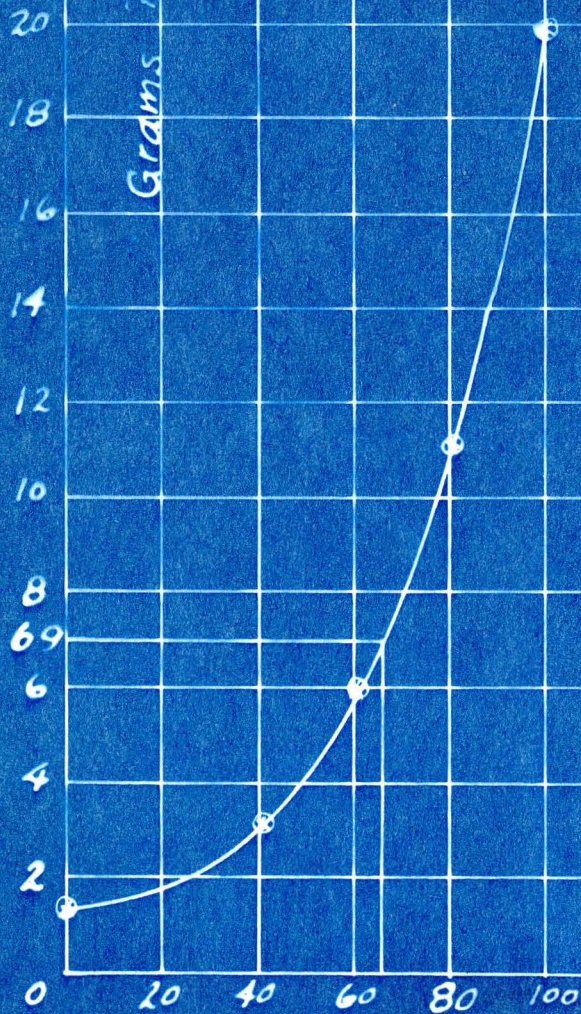
Dew-point °F	0°	40°	60°	80°	100°
Grains Troy	1.3	2.9	5.8	11.0	19.8

The above figures were compiled by Waldo (23), and the curve derived from these figures are shown in Plate 1

The hydration room was a constant-temperature room having a definite temperature of 18° C. This meant that the amount of water vapor present in the dessicator was always constant. From Platel, the grains troy present in the dessicator at this temperature can be found. The temperature 18° C. corresponds to 64.4° F, and from the curve it can be seen that 6.9 grains troy is present at this temperature.

Plate I

Grams Tray



Dew-point °F

Results

Decarbonation of hydromagnesite

Temperature - 550°

<u>Loss in grams</u>	<u>Time</u>
0.0547	5 min.
0.1090	10 min.
0.1098	30 min.
0.1104	1 hour
0.1111	3 hour
0.1132	6 hour

Temperature - 600°

<u>Loss in grams</u>	<u>Time</u>
0.0892	5 min.
0.1113	10 min.
0.1117	30 min.
0.1123	1 hour
0.1130	2 hour

Temperature - 650°

<u>Loss in grams</u>	<u>Time</u>
0.0492	2 min.
0.1007	4 min.
0.1096	8 min.
0.1113	10 min.
0.1122	20 min.
0.1131	30 min.

Temperature - 700°

<u>Loss in grams</u>	<u>Time</u>
0.0558	2 min.
0.1096	4 min.
0.1114	6 min.
0.1120	10 min.
0.1132	15 min.

Temperature - 750°

<u>Loss in grams</u>	<u>Time</u>
0.0939	2 min.
0.1092	4 min.
0.1119	6 min.
0.1125	8 min.
0.1129	10 min.

Temperature - 800°

<u>Loss in grams</u>	<u>Time</u>
0.0380	1 min.
0.0813	2 min.
0.1107	4 min.
0.1124	6 min.
0.1133	8 min.

Temperature - 850°

<u>Loss in grams</u>	<u>Time</u>
0.0417	1 min.
0.1086	2 min.
0.1119	4 min.
0.1133	6 min.

Temperature - 900°

<u>Loss in grams</u>	<u>Time</u>
0.0423	1 min.
0.1090	2 min.
0.1116	3 min.
0.1132	4 min.

Temperature - 950°

<u>Loss in grams</u>	<u>Time</u>
0.0441	1 min.
0.1112	2 min.
0.1133	3 min.

Temperature - 1000°

<u>Loss in grams</u>	<u>Time</u>
0.0882	1 min.
0.1129	2 min.

Hydration of magnesia

Temperature - 550°

<u>Gain in grams</u>	<u>Time</u>
0.0075	30 min.
0.0135	2 hours
0.0165	3 hours
0.0180	5 hours
0.0398	12 hr. 15 min.
0.0404	12 hr. 40 min.

Temperature - 600°

<u>Gain in grams</u>	<u>Time</u>
0.0047	20 min.
0.0073	40 min.
0.0101	1 hr. 30 min.
0.0121	2 hr. 15 min.
0.0138	4 hr. 35 min.
0.0202	5 hr. 15 min.
0.0403	17 hours

Temperature - 650°

<u>Gain in grams</u>	<u>Time</u>
0.0139	3 hours
0.0163	4 hours
0.0173	5 hours
0.0203	6 hours
0.0359	15 hours
0.0402	19 hr. 10 min.

Temperature - 700°

<u>Gain in grams</u>	<u>Time</u>
0.0072	1 hour
0.0096	2 hours
0.232	10 hr. 30 min.
0.0326	14 hr. 40 min.
0.0335	16 hr. 35 min.
0.0354	18 hr. 55 min.
0.0378	20 hr. 55 min.
0.0404	25 hr. 55 min.

Temperature - 750°

<u>Gain in grams</u>	<u>Time</u>
0.0079	2 hours
0.0147	4 hours
0.0165	6 hours
0.0215	10 hours
0.0274	15 hr. 30 min.
0.0304	18 hr. 20 min.
0.0403	29 hr. 30 min.

Temperature - 800°

<u>Gain in grams</u>	<u>Time</u>
0.0074	2 hours
0.0135	4 hours
0.0158	6 hours
0.0264	15 hr. 30 min.
0.0281	18 hr. 20 min.
0.0309	21 hr. 20 min.
0.0381	28 hr. 25 min.
0.0405	32 hr. 30 min.

Temperature - 350°

<u>Gain in grams</u>	<u>Time</u>
0.0071	2 hours
0.0093	4 hours
0.0223	10 hr. 50 min.
0.0247	16 hr. 20 min.
0.0277	18 hr. 30 min.
0.0355	26 hr. 25 min.
0.0403	35 hr. 25 min.

Temperature - 900°

<u>Gain in grams</u>	<u>Time</u>
0.0071	2 hours
0.0087	4 hours
0.0215	13 hr. 30 min.
0.0237	16 hr. 20 min.
0.0267	19 hr. 20 min.
0.0337	26 hr. 25 min.
0.0403	35 hr. 10 min.

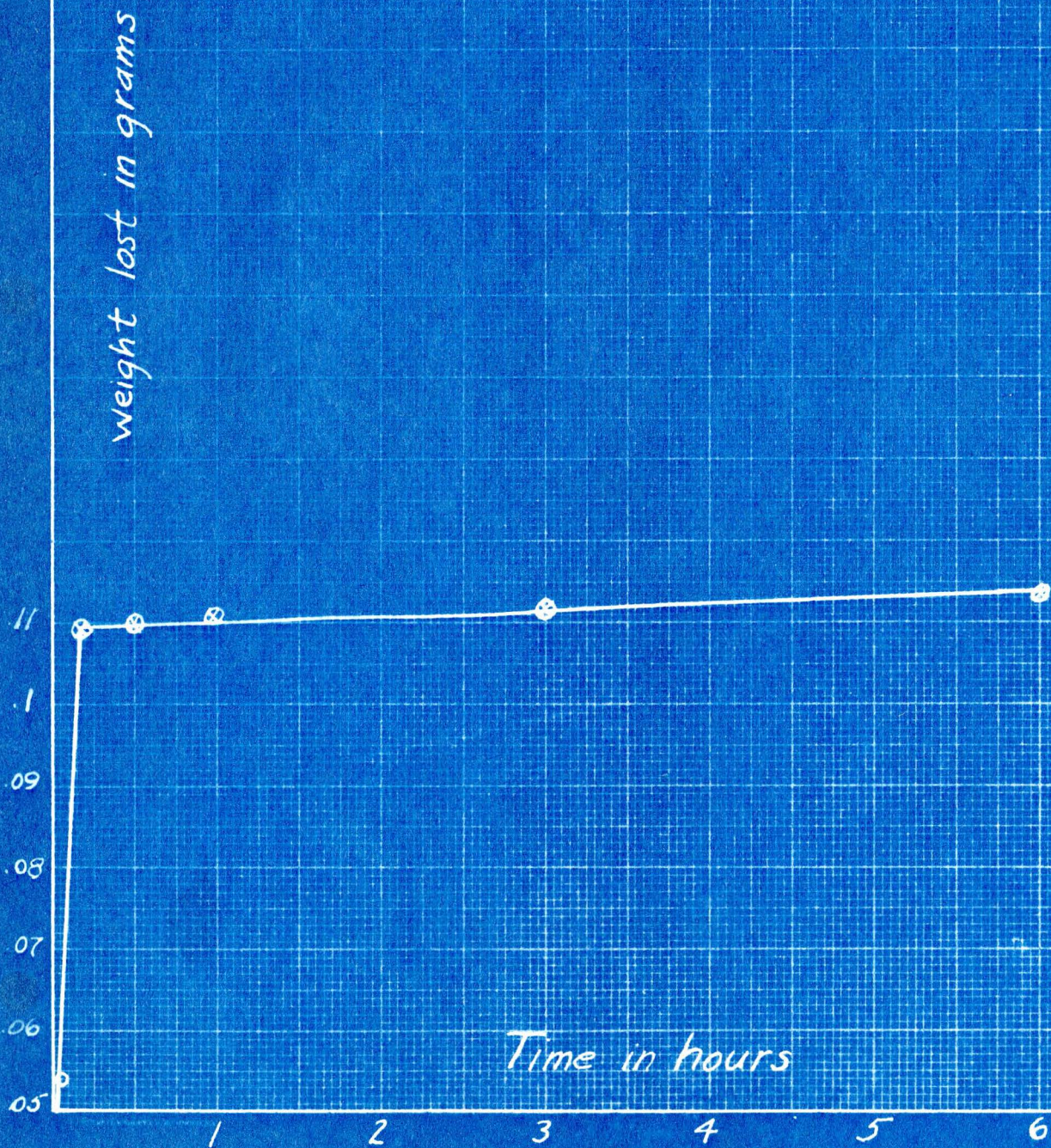
Temperature - 950°

<u>Gain in grams</u>	<u>Time</u>
0.0030	3 hours
0.0166	10 hr. 25 min.
0.0320	23 hr. 10 min.
0.0338	25 hr. 10 min.
0.0403	35 hr. 40 min.

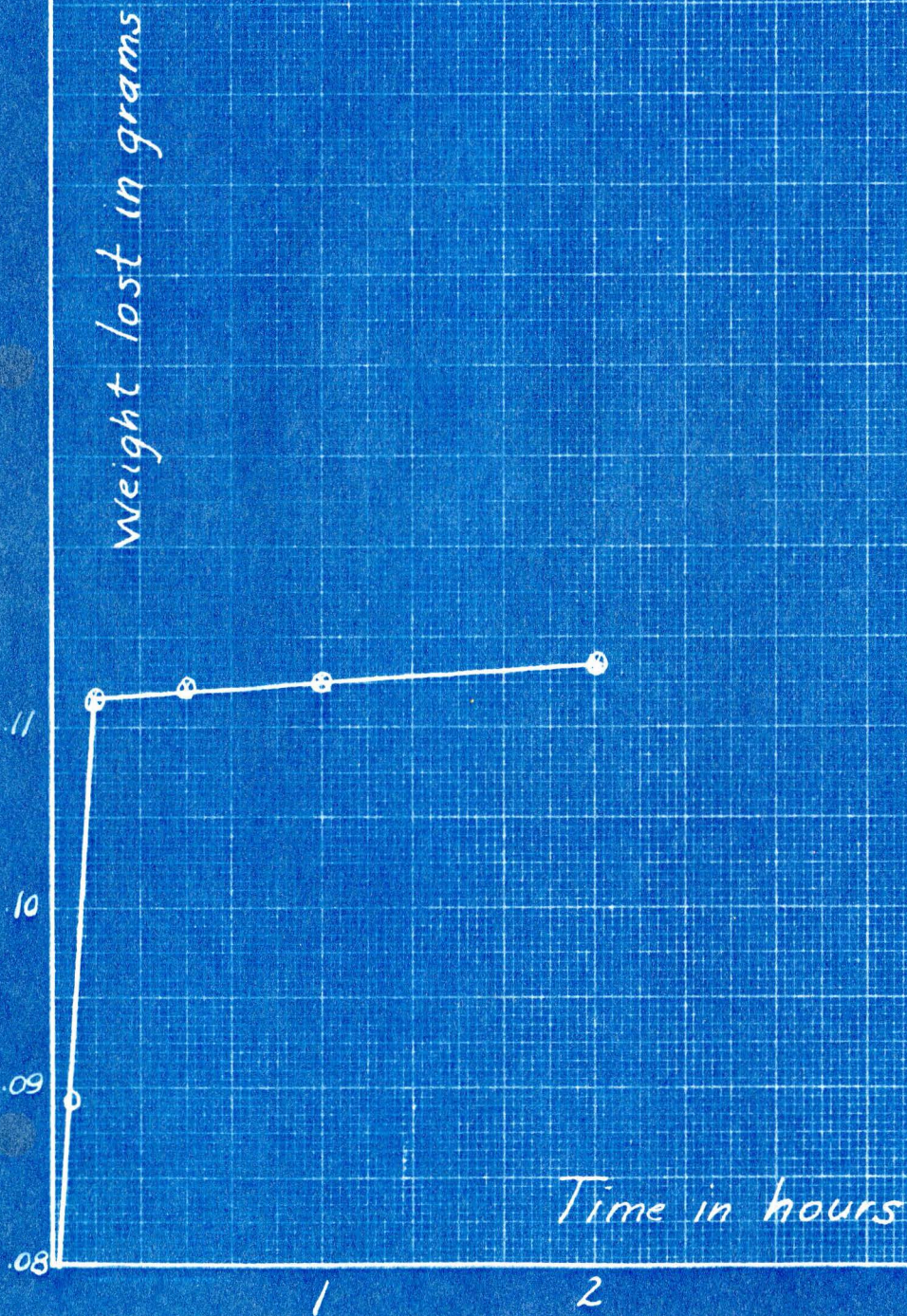
Temperature - 1000°

<u>Gain in grams</u>	<u>Time</u>
0.0140	5 hr. 45 min.
0.0238	13 hr. 30 min.
0.0302	21 hr. 30 min.
0.0335	23 hr. 30 min.
0.0404	36 hr. 15 min.

Decarbonation Curve
Temperature - 550°

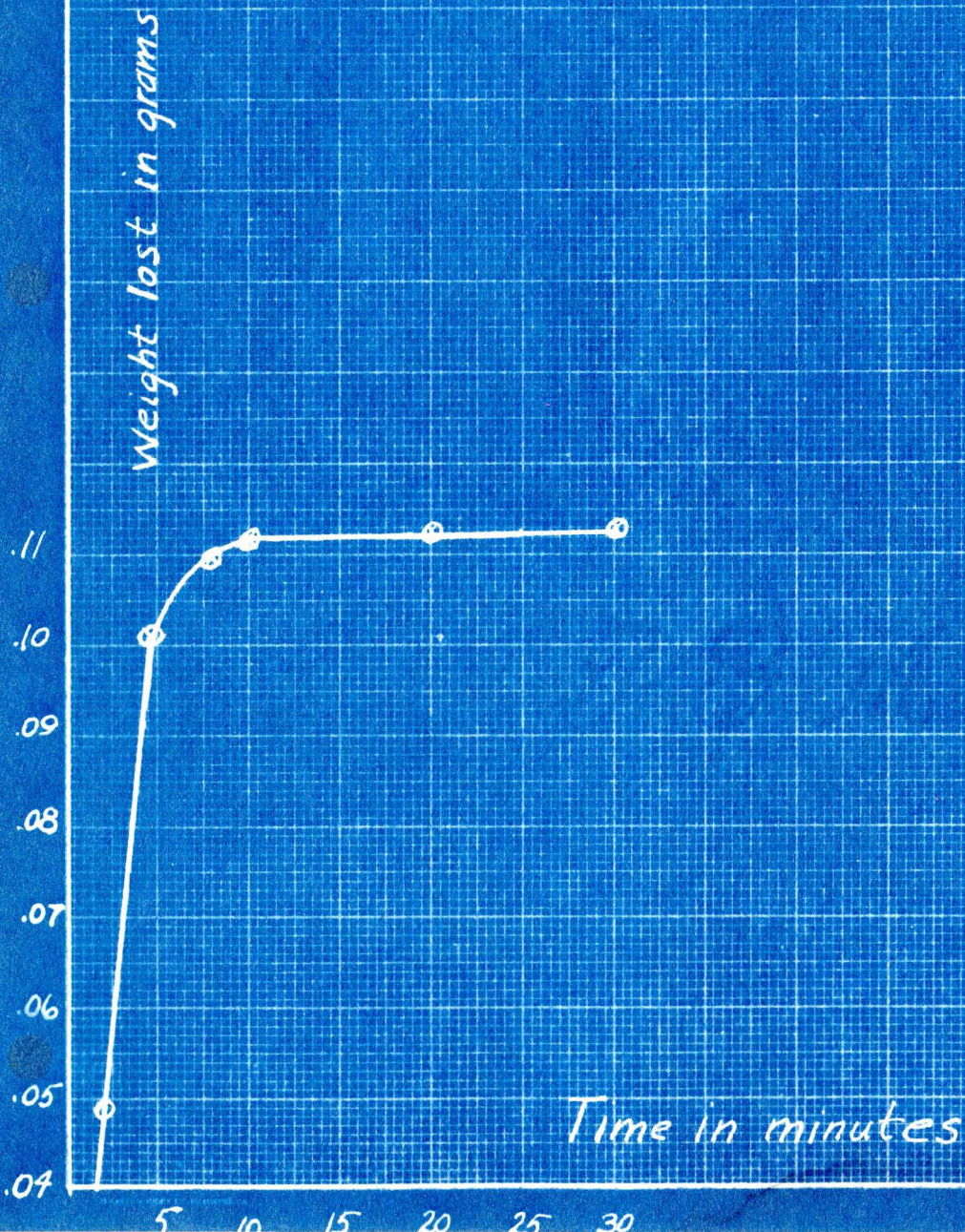


Decarbonation Curve
Temperature - 600°



Decarbonation Curve

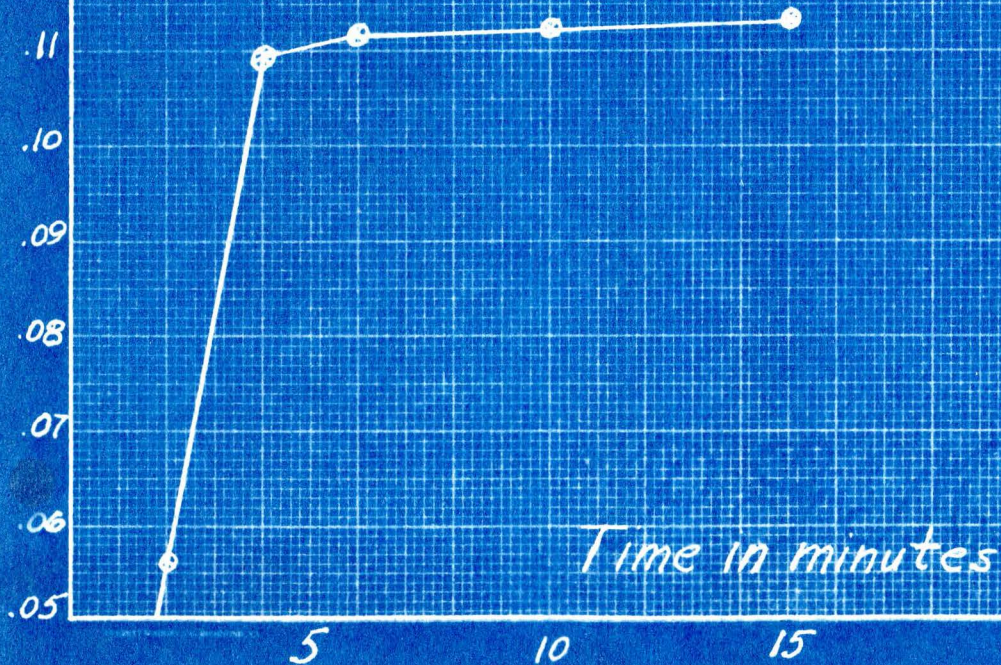
Temperature - 650°



Decarbonation Curve

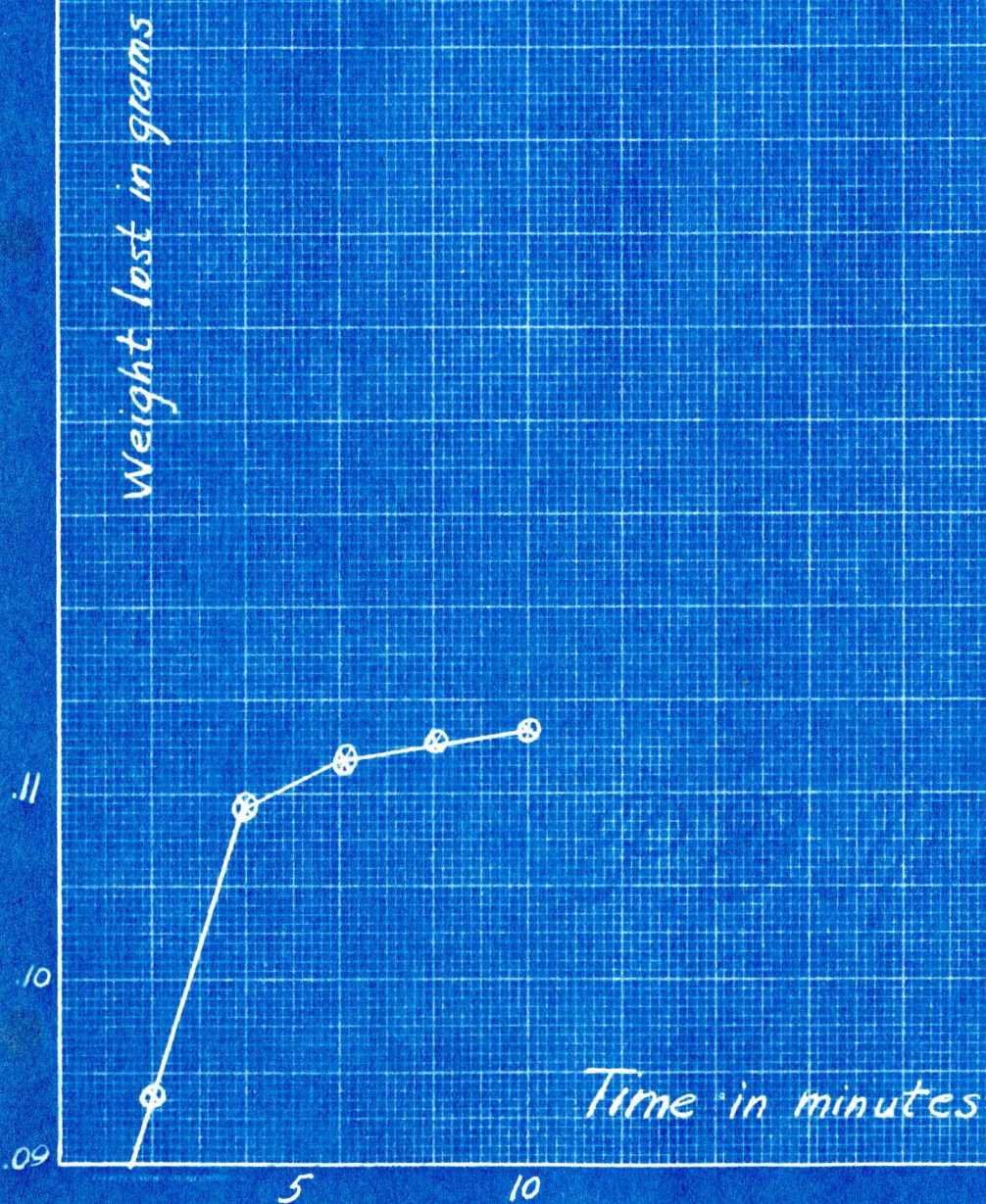
Temperature - 700°

Weight lost in grams

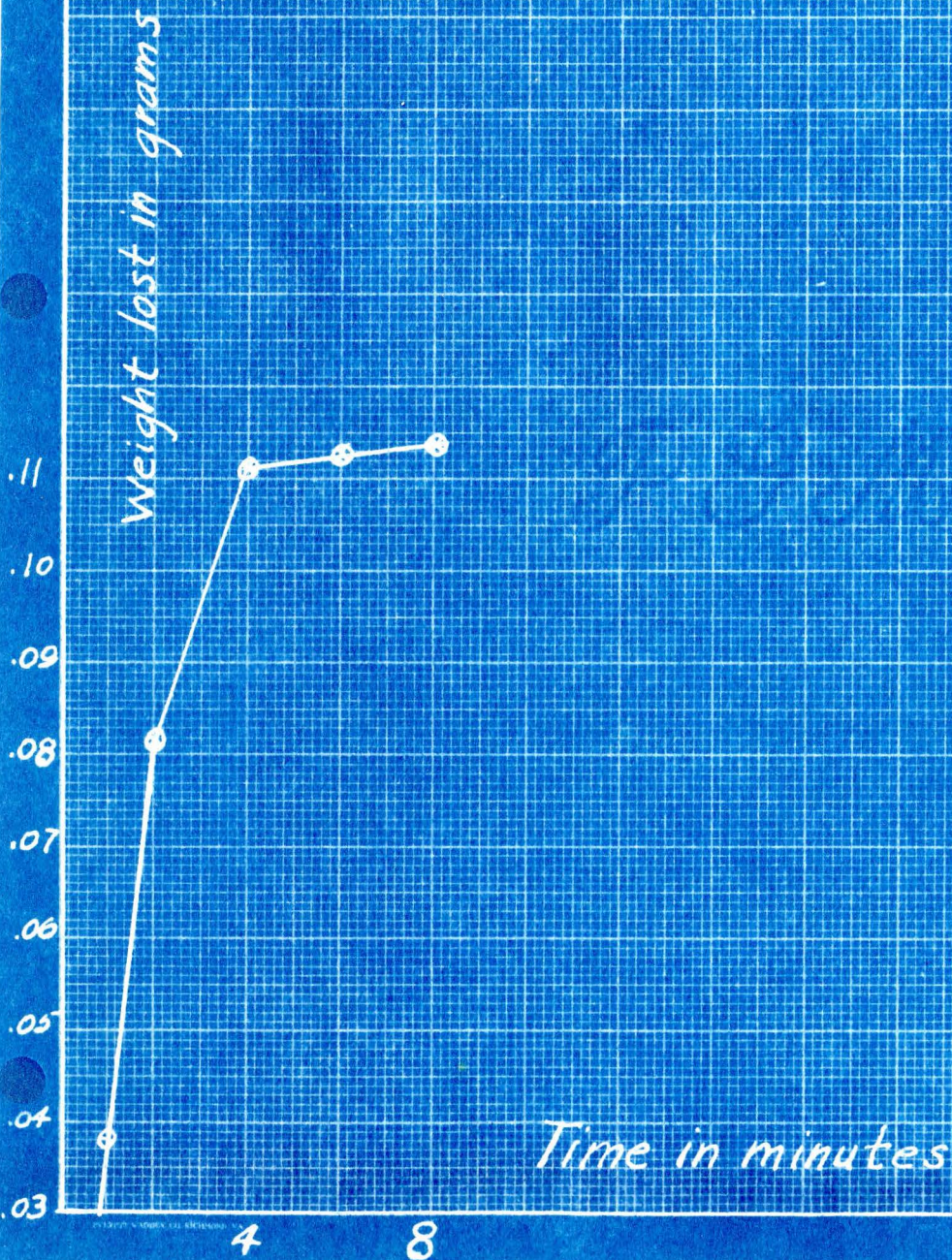


Decarbonation Curve

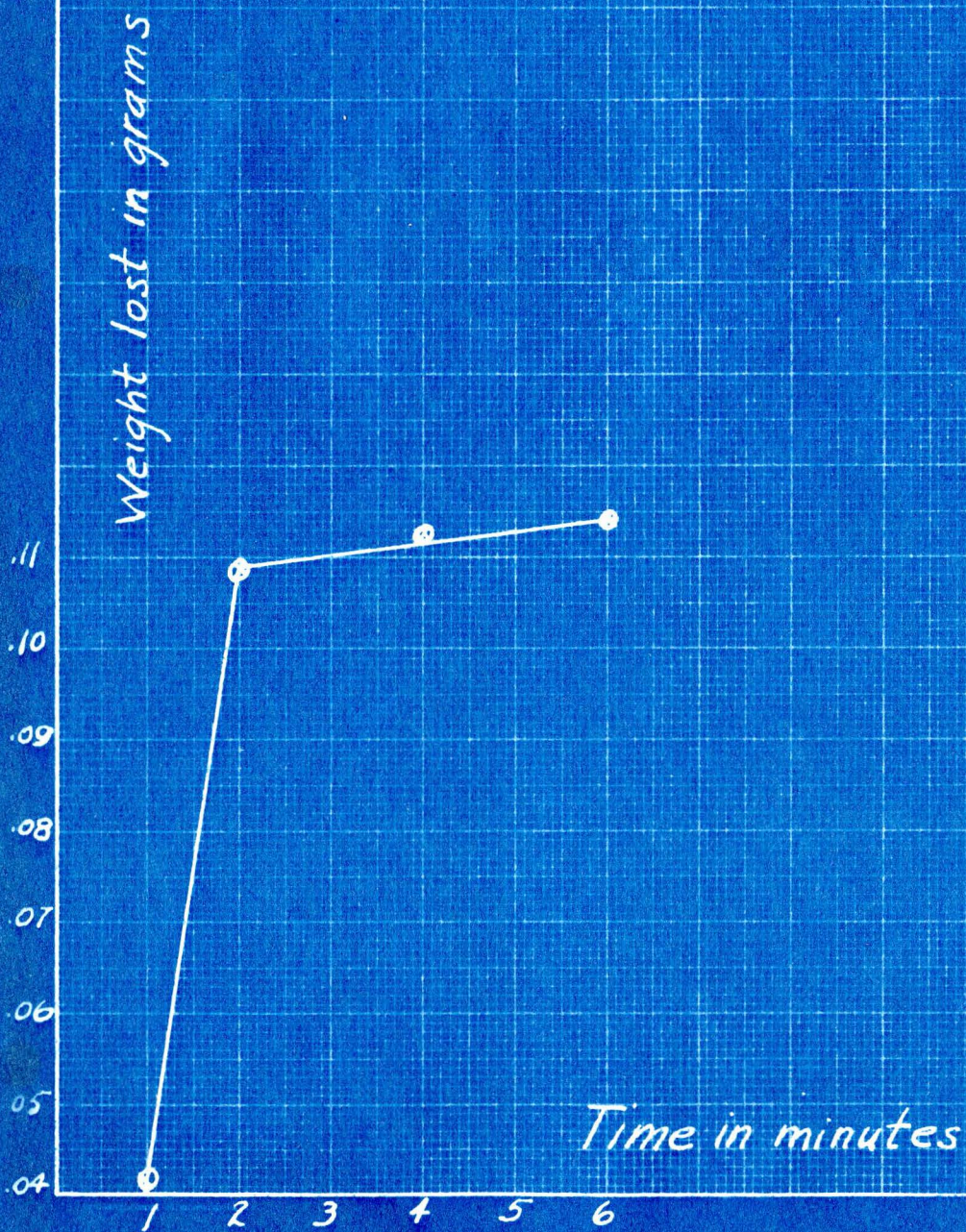
Temperature - 750°



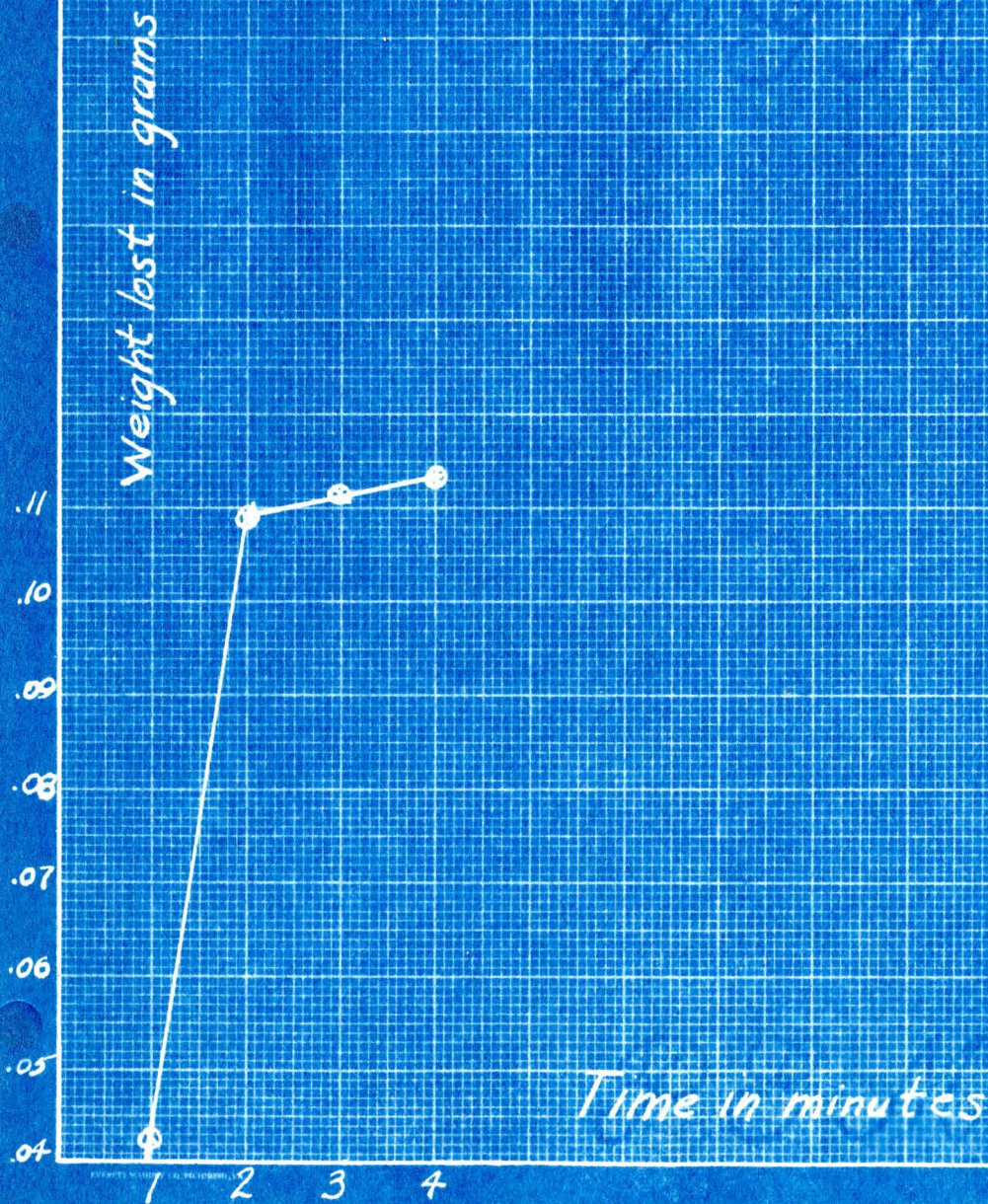
Decarbonation Curve
Temperature - 800°



Decarbonation Curve
Temperature - 850°



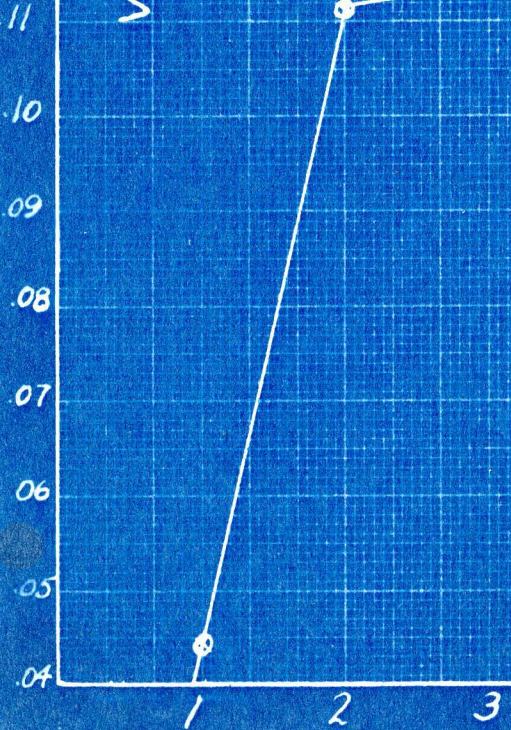
Decarbonation Curve
Temperature - 900°



Decarbonation Curve

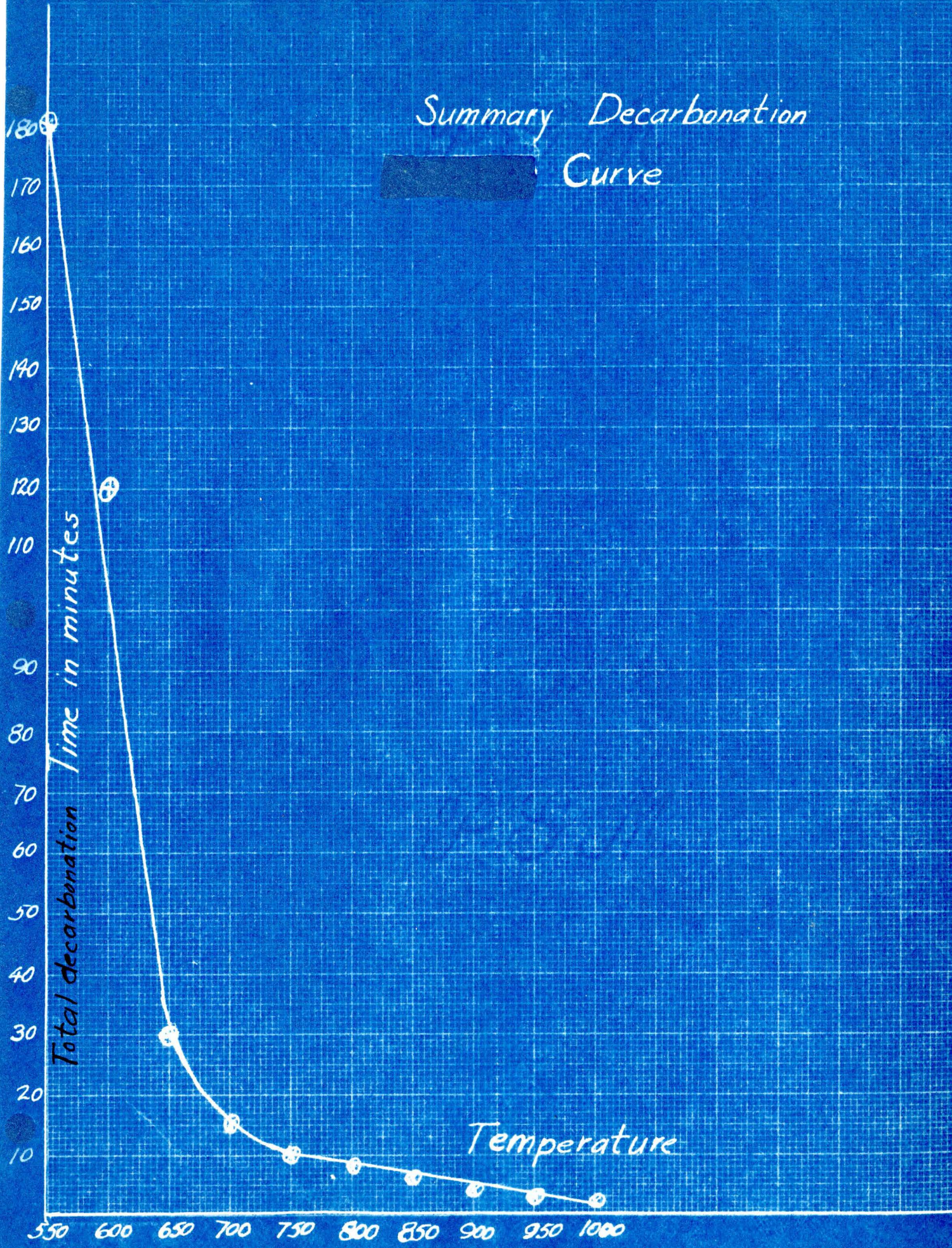
Temperature - 950°

Weight lost in grams



Time in minutes

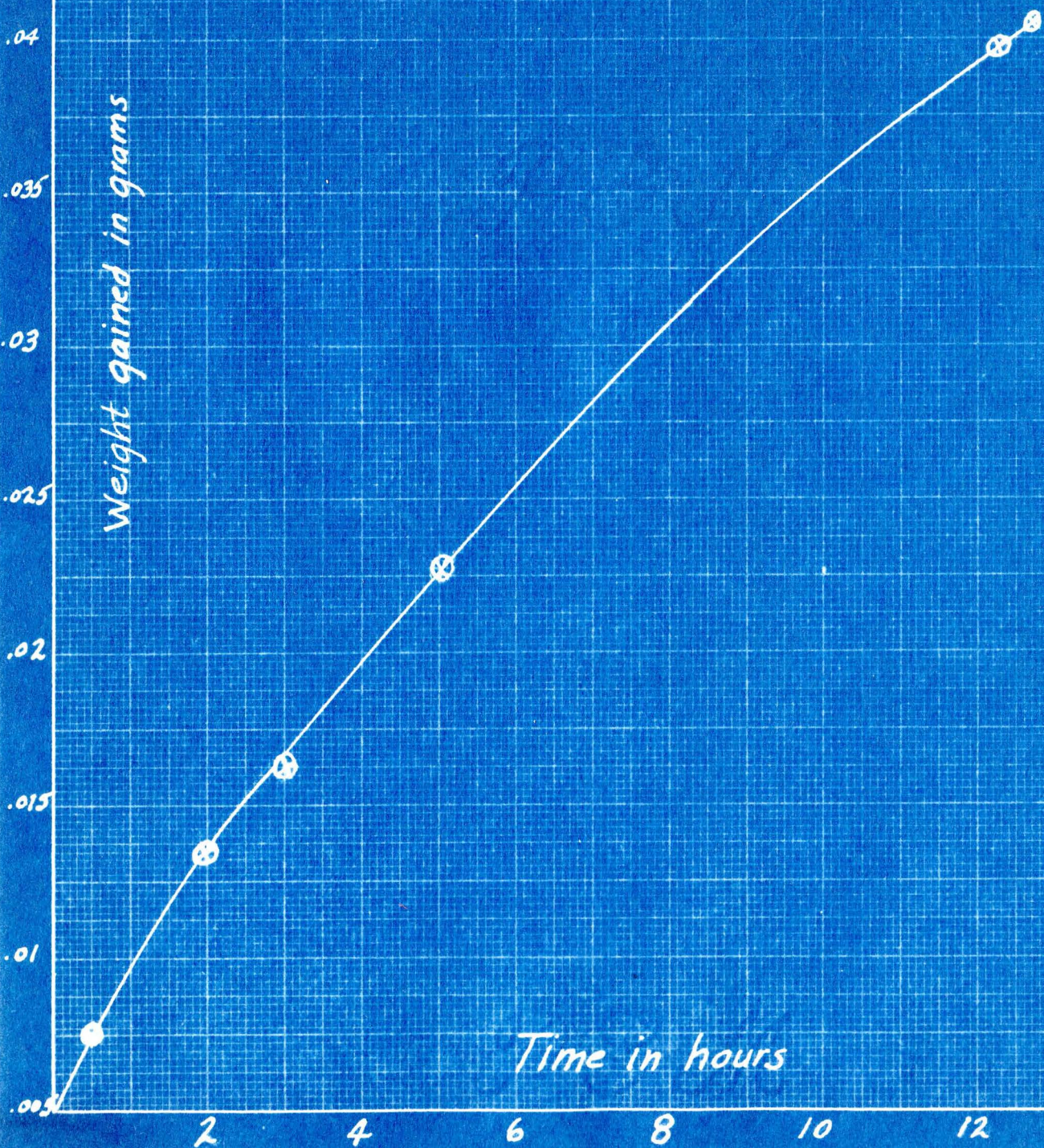
Summary Decarbonation Curve



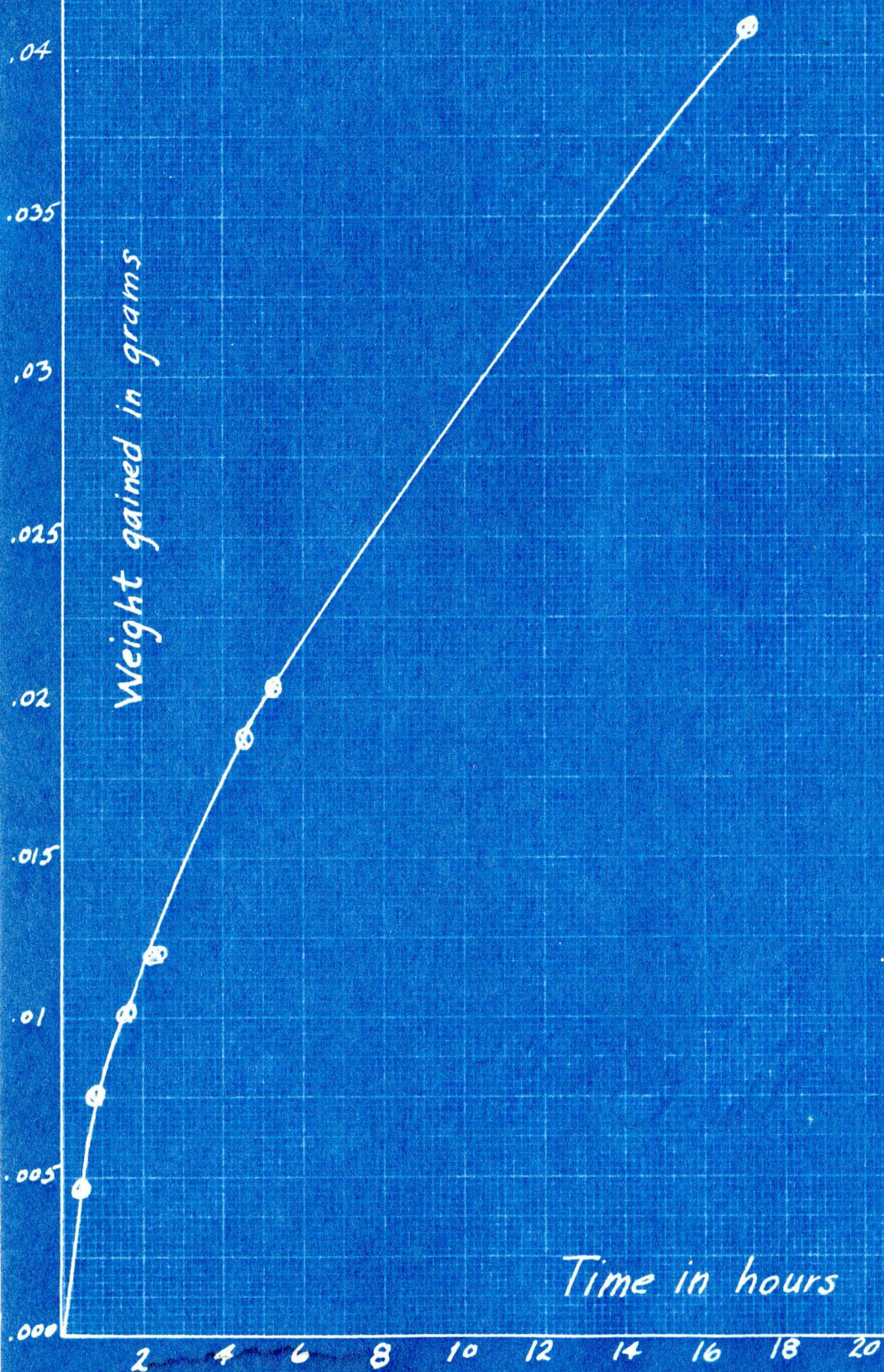
Hydration Curve
Temperature - 550°

Weight gained in grams

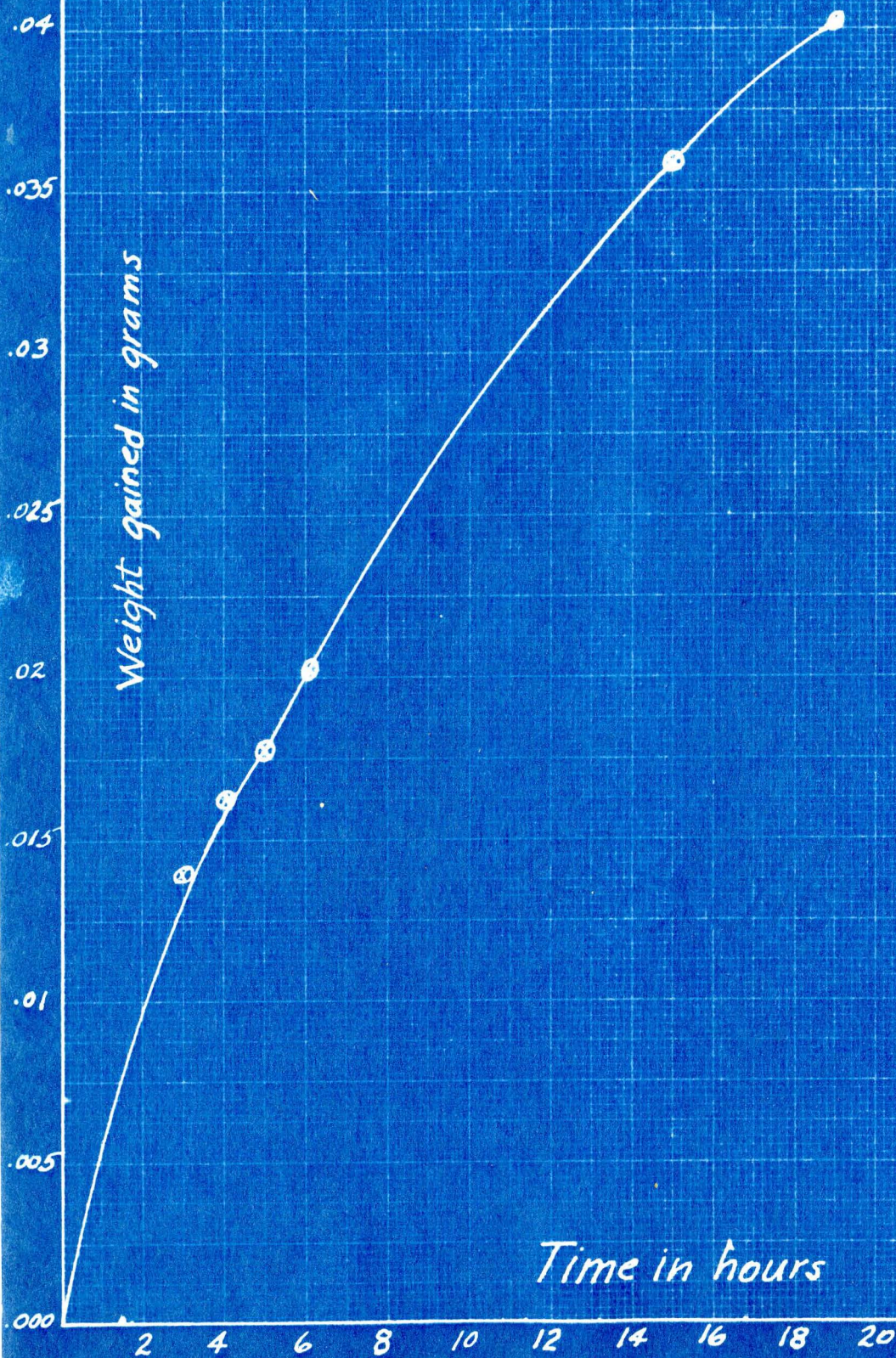
Time in hours



Hydration Curve
Temperature - 600°



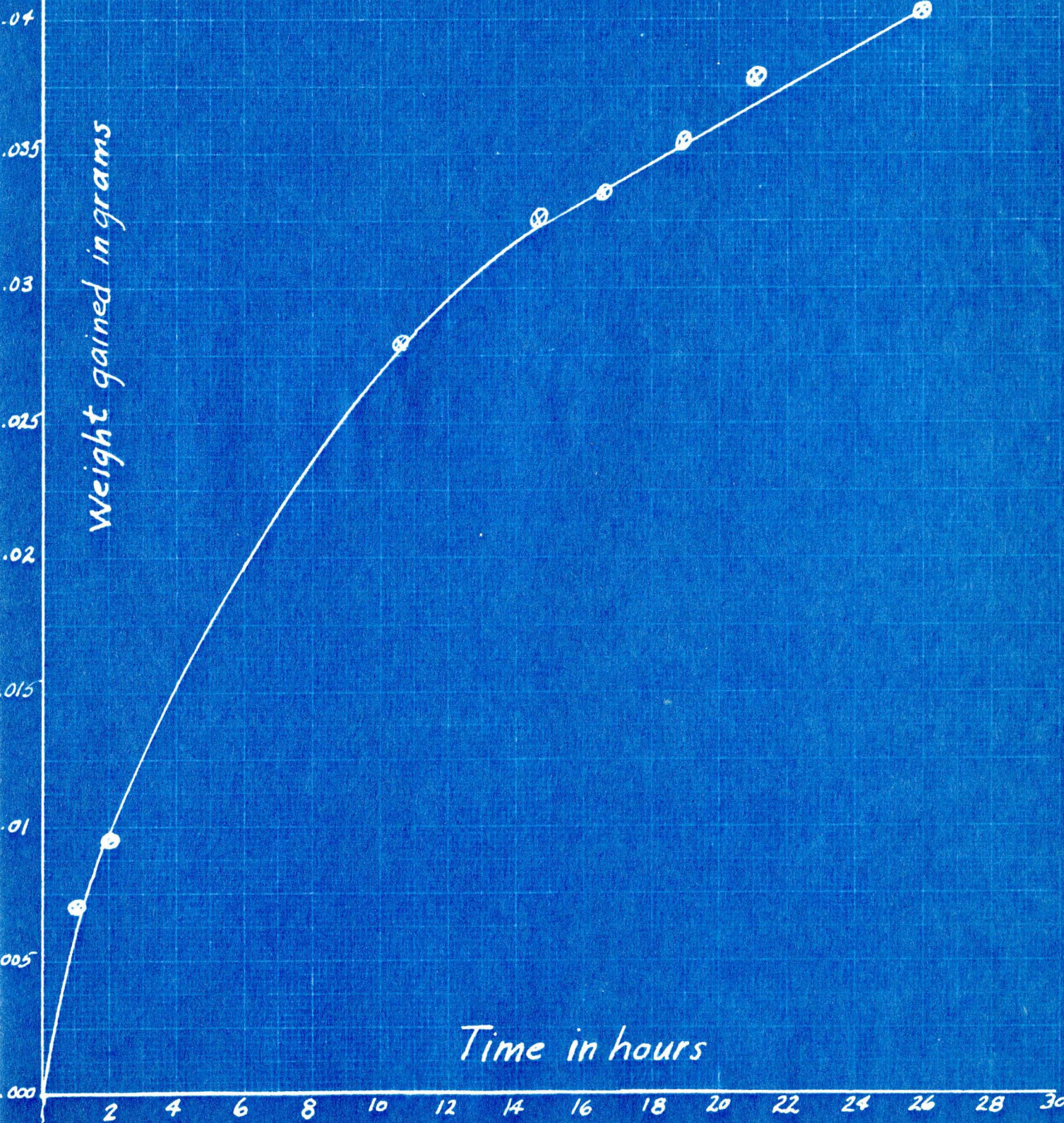
Hydration Curve
Temperature - 650°



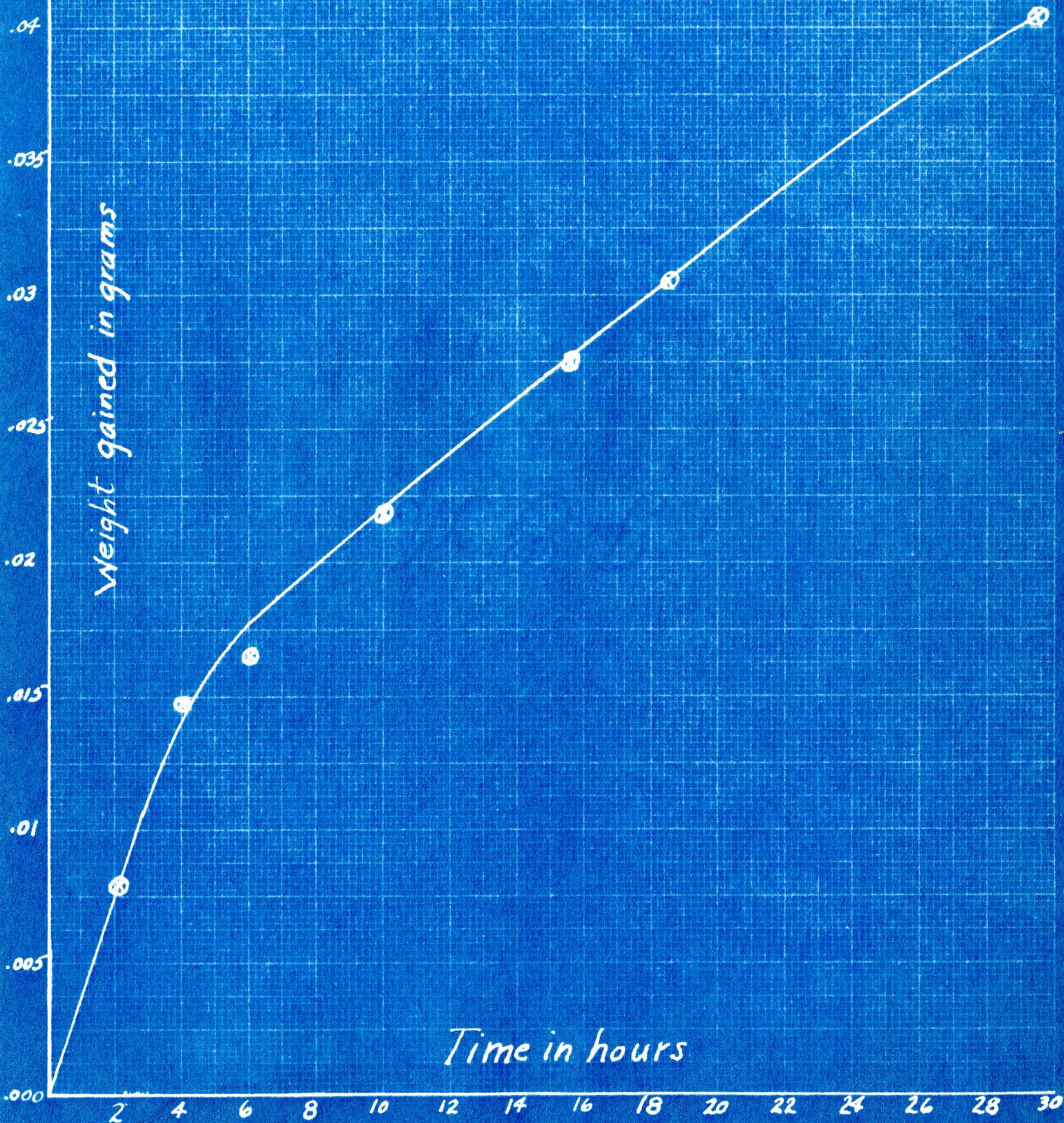
Hydration Curve
Temperature - 700°

Weight gained in grams

Time in hours



Hydration Curve
Temperature - 750°

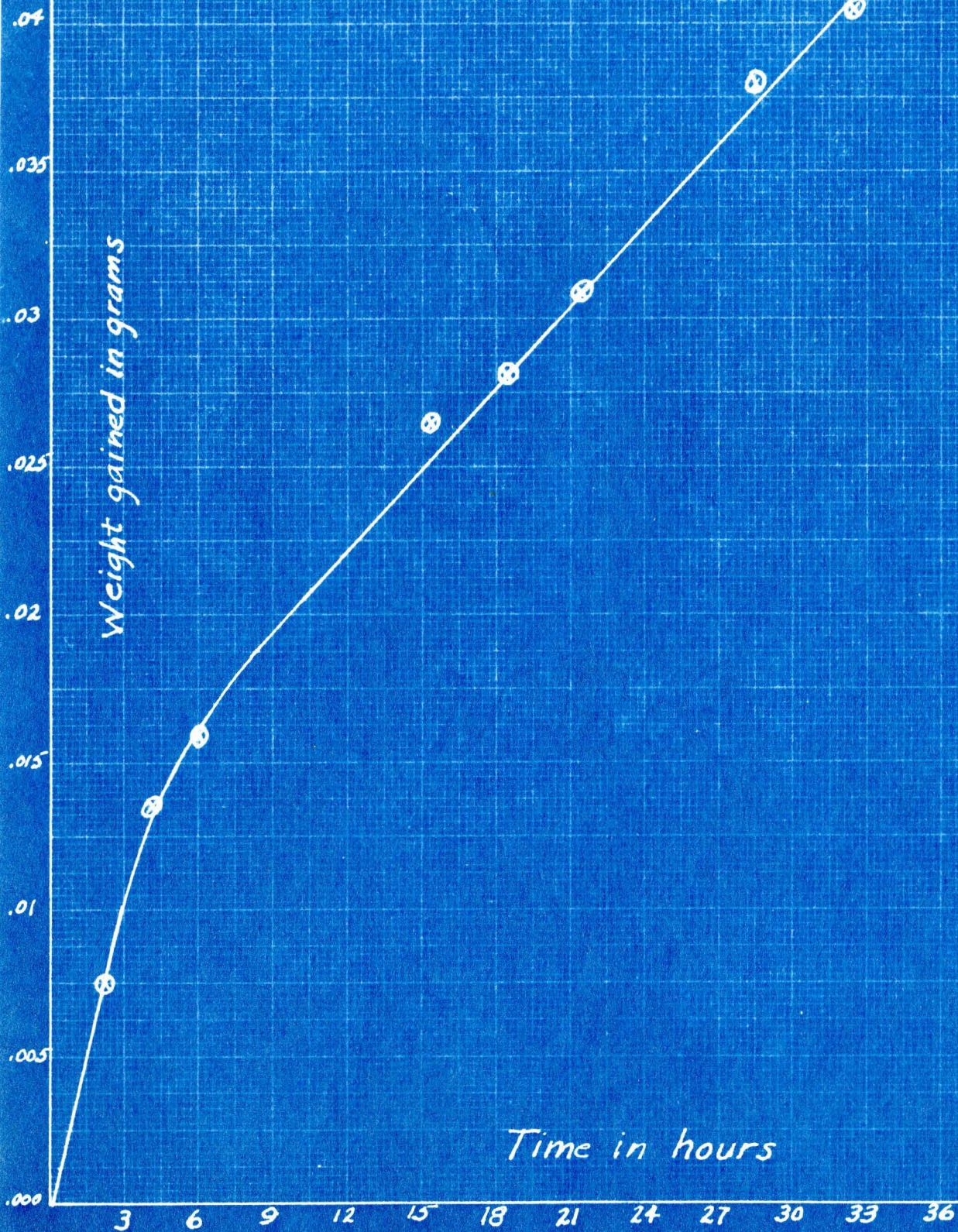


Hydration Curve

Temperature - 800°

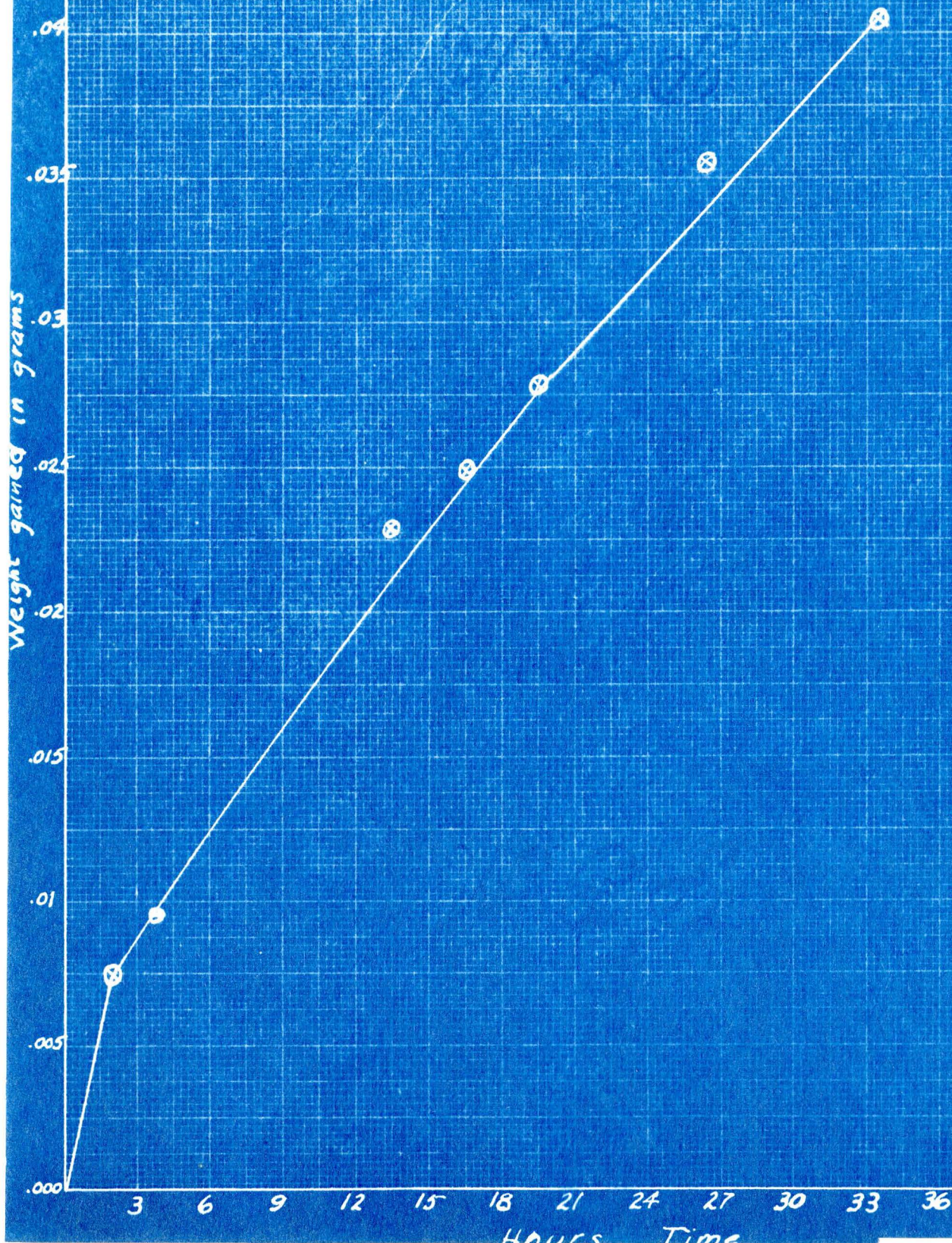
Weight gained in grams

Time in hours

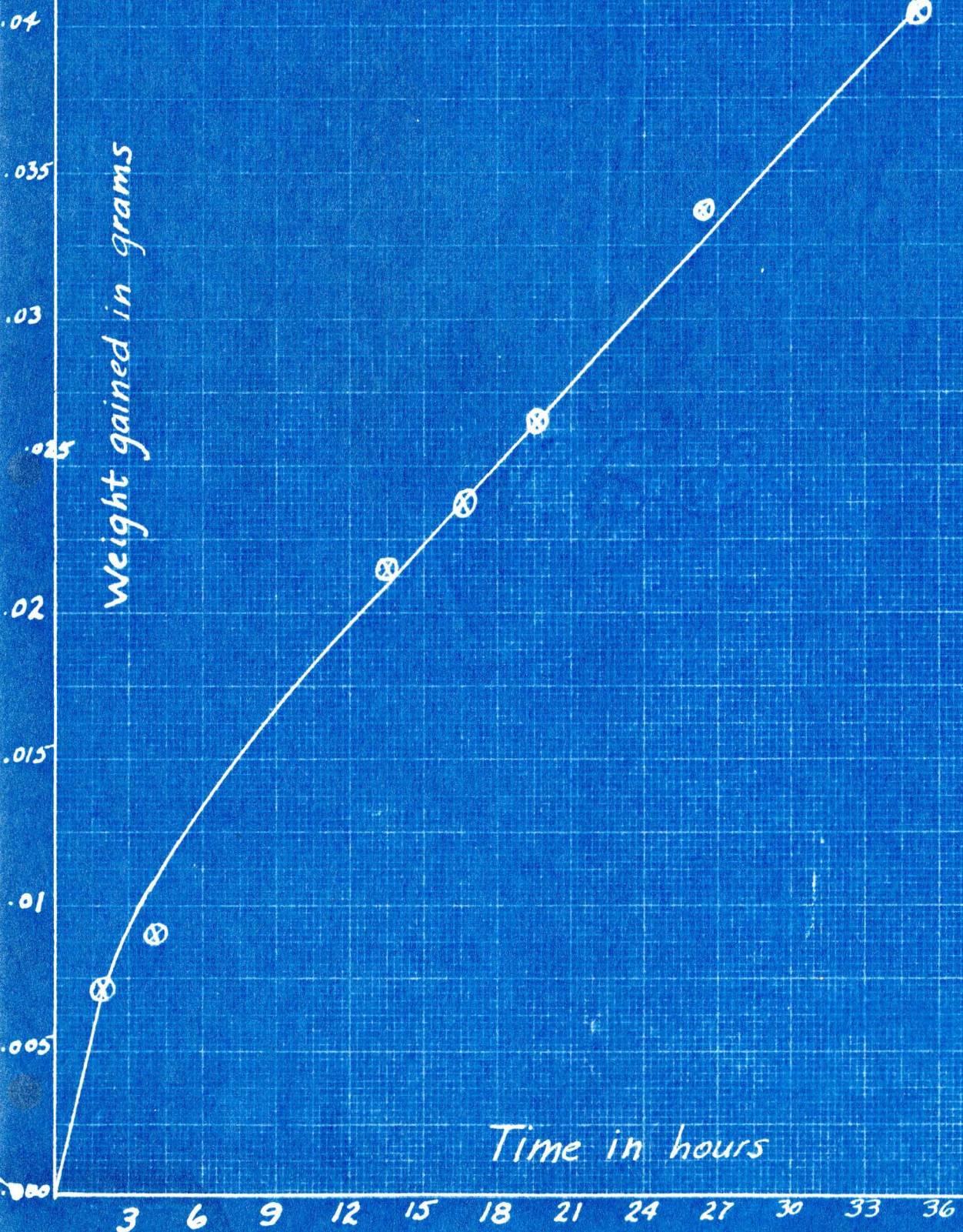


Hydration Curve

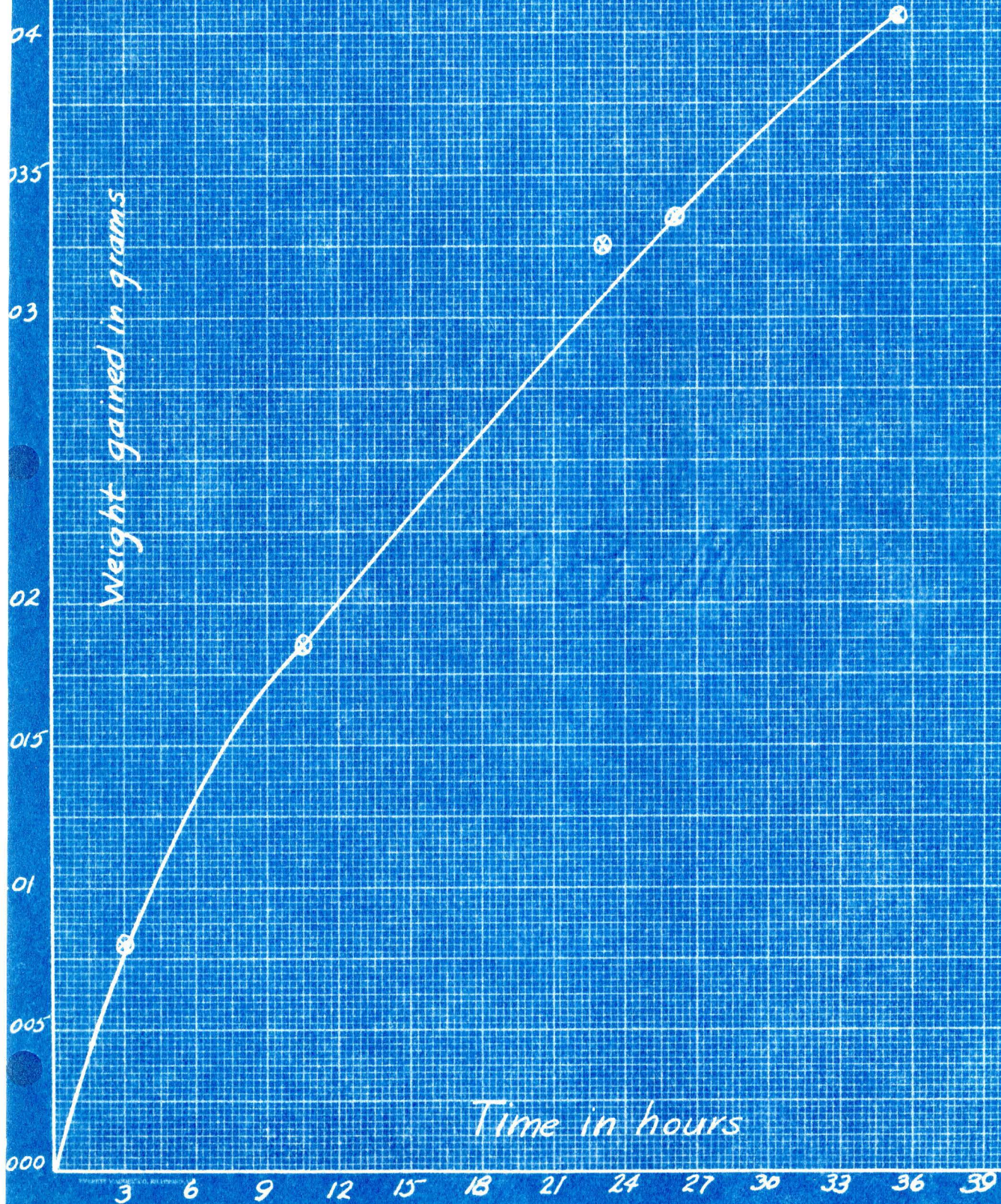
Temperature - 850°



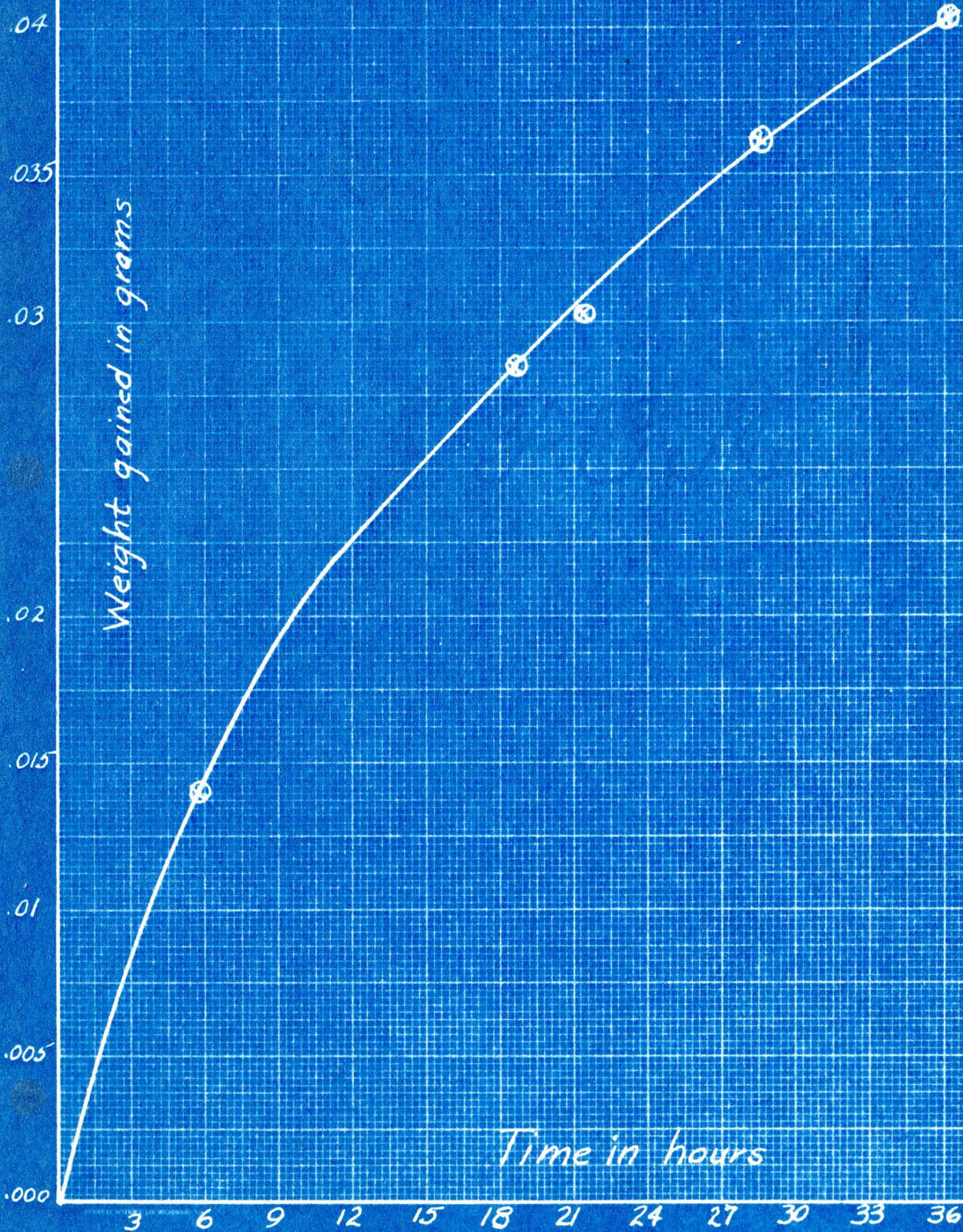
Hydration Curve
Temperature - 900°



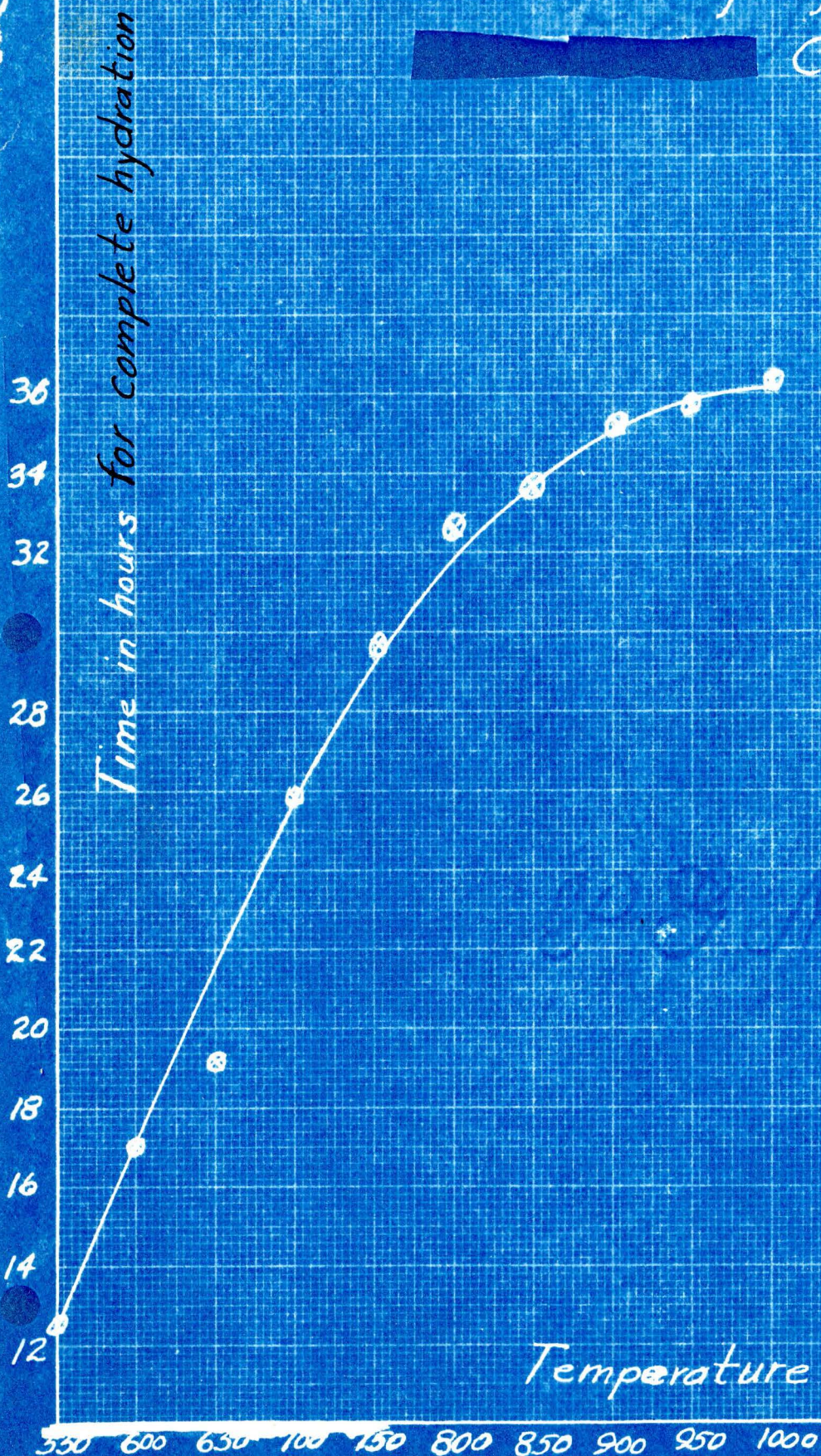
Hydration Curve
Temperature - 950°



Hydration Curve
Temperature - 1000°



Summary Hydration Curve



Discussion

In the decarbonation process of the hydromagnesite samples, most of the carbon dioxide was given off within the first ten minutes at all the temperatures ranging from 550° to 1000° . The samples that were heated at 550° and 600° lost over 90% of their carbon dioxide in the first ten minutes, and then the carbon dioxide came off very slowly until complete decarbonation resulted. These curves seem to have a very definite point at which the carbon dioxide evolution changes suddenly from its very rapid rate to a very slow rate. The curves at all the other temperatures seem to gradually change from this rapid rate of evolution to the slow rate as shown by the more rounded curve. But still all the samples at the temperatures ranging from 650° to 1000° lost very rapidly during the first five minutes. The time for complete decarbonation became less as the temperature rose until at 1000° , only two minutes were required to reach the point of complete decarbonation. At the high temperatures, it became very difficult to obtain very accurate results because crucibles had to be taken out at one minute intervals; this was difficult to do very efficiently with the facilities available. The 1000° degree decarbonation was not plotted because only two points were taken, and these were considered insufficient to plot a curve accurately.

The hydration experiments proved to take much longer to carry out than the decarbonation experiments. At the temperature of 550° , 12 hours and 40 minutes were required for complete hydration. The sample hydrated very steadily throughout the entire hydration. The decarbonation product seemed to hydrate slightly faster during

the first part of the hydration than during the last part, and the samples at temperatures ranging from 550° through 700° acted similarly. The time required for complete hydration became longer as the temperature increased, but the hydration was still very steady throughout with the addition of water being slightly faster during the early part of the hydration. At 750° the sample hydrated rather rapidly for four hours, and then a noticeable change in the rate of hydration became evident. This was noticed at all the higher temperatures. As the temperature increased from 750° to 1000°, the length of time of the fairly rapid hydration became less and less; for example, at 1000°, the change from rapid to slow hydration takes place in less than three hours. The remainder of the hydration took place slowly but steadily. At the temperature of 1000°, the hydration required 36 hours and 15 minutes.

In February, 1934, the pyrometer attached to the furnace used in this investigation was checked with a standardized resistance thermometer. In general, the thermometer read about 30 degrees lower than the pyrometer, except in the case of lags. Therefore the temperatures recorded in this experiment are actually 30 degrees lower.

Note: Samples were not run under a temperature of 550° in this investigation. No attempt was made to distinguish between the loss of CO₂ and water because it was assumed that the water came off at a relatively low temperature.

Conclusion

1. Hydromagnesite gives off carbon dioxide very easily and readily within the first ten minutes at all temperatures ranging from 550° to 1000° , but the carbon dioxide is not given off so easily after this time.
2. The higher the temperature at which the hydromagnesite is burned, the more rapidly is the CO_2 given off.
3. Lower temperatures are required for giving off carbon dioxide from hydromagnesite than from calcium carbonate. It requires only three minutes for the evolution of all the CO_2 from hydromagnesite, whereas it takes fifteen minutes for total dissociation of calcium carbonate *at 950°* .
4. The hydromagnesite does not decompose in stages.
5. The chemical activity of the magnesia is lessened when it is burned at high temperatures.

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