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A SERIES OF TESTS ON THE CONDENSATION OF ZINC VAPOR TO METALLIC ZINC.

BY

F. W. CODY

J. W. GREENE.

A

THESIS.

SUBMITTED TO THE FACULTY OF THE SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI IN PARTIAL FULFILLMENT OF THE WORK REQUIRED FOR THE

DEGREE OF

BACHELOR OF SCIENCE IN METALLURGY

AND

BACHELOR OF SCIENCE IN GENERAL SCIENCE

ROLLA MISSOURI

1913.

APPROVED BY D. Copeland

Profeessor of Metallurgy.

15722

SUBJECT.

The condensation of Zinc Vapor to Metallic Zinc and the effect thereon of a number of variables present in the commercial treatment of Zinc Ores.

PURPOSE.

The purpose of this Thesis is to try to show the effect which certain variables, which are always present in practice, have upon the condensation of Zinc Vapors to Metallic Zinc.

That these variables can and do play an important part in the Zinc Smelting Industry can be shown from the fact that all of the zinc obtained at the present time is won from ores by reducing the oxide of zinc to metallic zinc by means of carbon. This operation necessarily takes place at a temperature above the boiling point of zinc. This means that the zinc immediately on being reduced forms a vapor and must be condensed from this gaseous condition in order to be recovered. This condensation would be governed by the same laws which control the condensation of other gases to their liquid form.

The variables which must be considered in the condensation of all vapors to their solid or molten state are:

1. Temperature

2. Time at that temperature

3. The vapor tensions of the particular gas that we we are trying to condense.

4. The percent of the particular gas we are trying to condence is, in the particular mixture with which we are working.

5. The nature of the diluting gases that make up the mixture.

6. Since we know that in practice the zinc must be condensed as molten zinc, and since we know that there is always a large and troublesome production of blue powder we should try to determine the factors which lead to a great or small blue-powder product.

In the following experiments the only variable taken in to consideration was temperature. This includes the variations of temperature in both the furnace and the condenser. We will try to show in this connection the effect which a slight variation in temperature in the furnace has upon the speed of the volatilization of the zinc.

LITERATURE ON THE SUBJECT.

Metallurgy of Zinc and Cadmium ---------------- Ingall

Metallurgical Calculations III ---------------- Richards

Transactions of the American Institute of

Mining Engineers --------------------------------

Current Literature on Mining and Metallurgy. --

METHOD OF ATTACK.

Under this head we will state briefly some of the methods used in attacking this problem and the defect which caused their abandonment.

The first scheme tried was a furnace built of loose bricks using sand crucibles for both retort and condenser and a blast lamp as a source of heat. This scheme proved unsatisfactory for the following reasons:

1. It was impossible to attain a sufficiently high temperature.
2. There was no satisfactory means of controling the condenser temperature.

3. The inconvience incurred in cleaning and changing the condensers.

4. The easy breakage of the crucibles due to quick variations in temperature.

In the next scheme tried the same type of furnace and retort were used but the source of heat consisted of three blast lamps instead of one, as in the proceeding case, and the condencer used was a fire clay tube covered with asbestos and heated by an electrical resistance coil.

This scheme was also discarded on account of the inability to obtain sufficiently high temperature. It is probable that the condenser in this case would have given satisfactory results as the temperature was easily controlled.

In a final endeavor to obtain the temperature required for this experiment, a gasoline furnace was used, and as this type of furnace is capable of reaching 1400^{o}C, the difficulty of insufficient heating was entirely elimenated. The chief difficulty which arose here was the inability of the

Sand crucibles to satisfactorially withstand the temperature of 1200°C or more, particularly if rapid changes in the temperature were required. A fire clay condenser was used but it was finally discarded because the zinc would adhere to the sides and thus cause difficulty in cleaning.

The final plan, and the only one which proved to be successful, was to use a number six graphite crucible as the retort. This crucible gave excellent service and when removed, while showing the effect of the high temperature to whuch it had been subjected, was still apparently good for a few more runs.

The graphite cover to fit this had a hole broken in it to admit the end of the condenser, which was a large rod of graphite - about 3 in. in diameter through which was bored a hole of one inch diameter. The condenser and the lid were held together by a cement of fire clay, and the lid was held to the retort in the same manner.

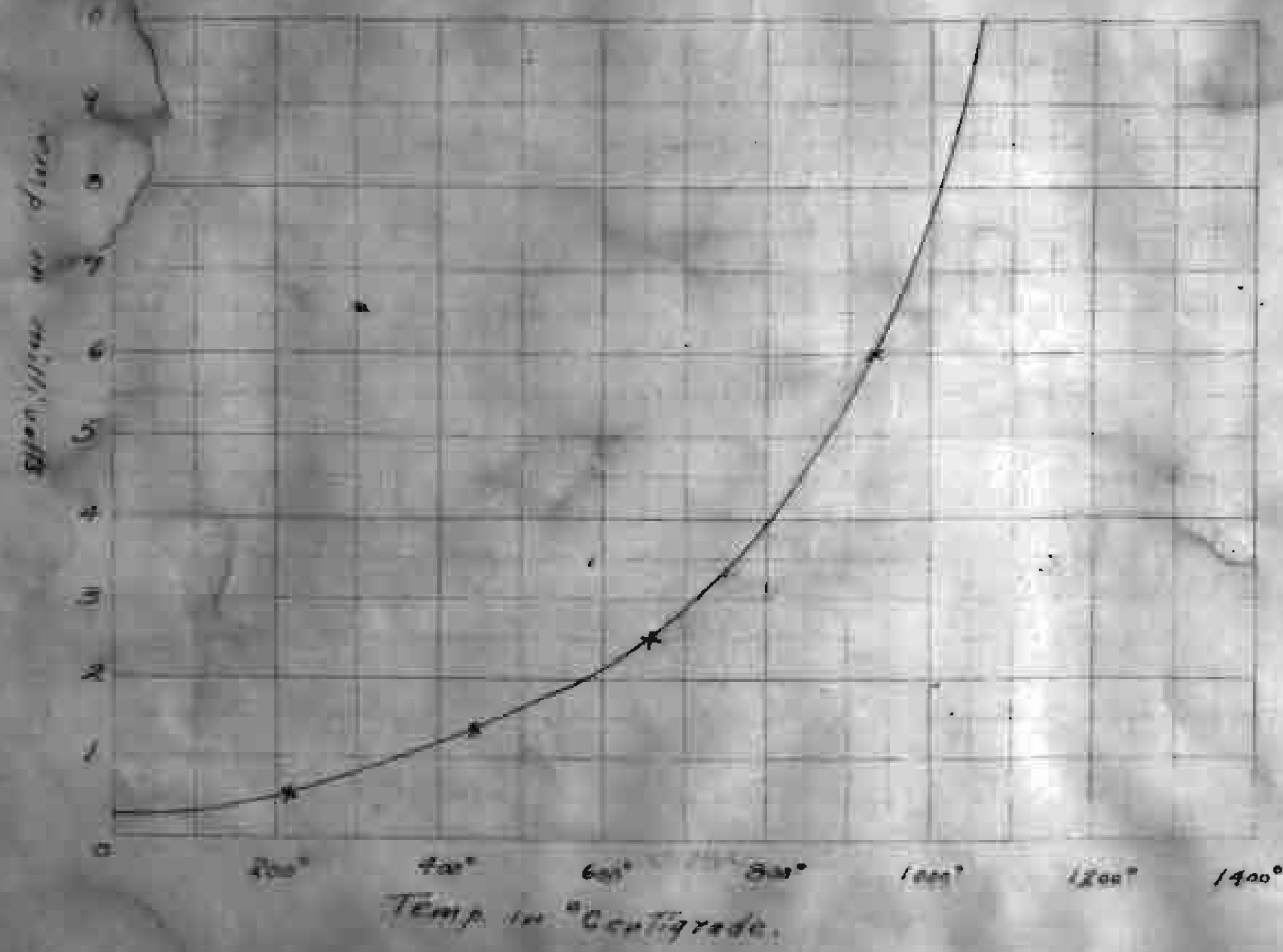
This fire clay cement proved very satisfactory. As soon as the run was well under way, the zinc parmeated the fire clay and seemed to perfectly close the joints and the lid adheared to the crucible very firmly.

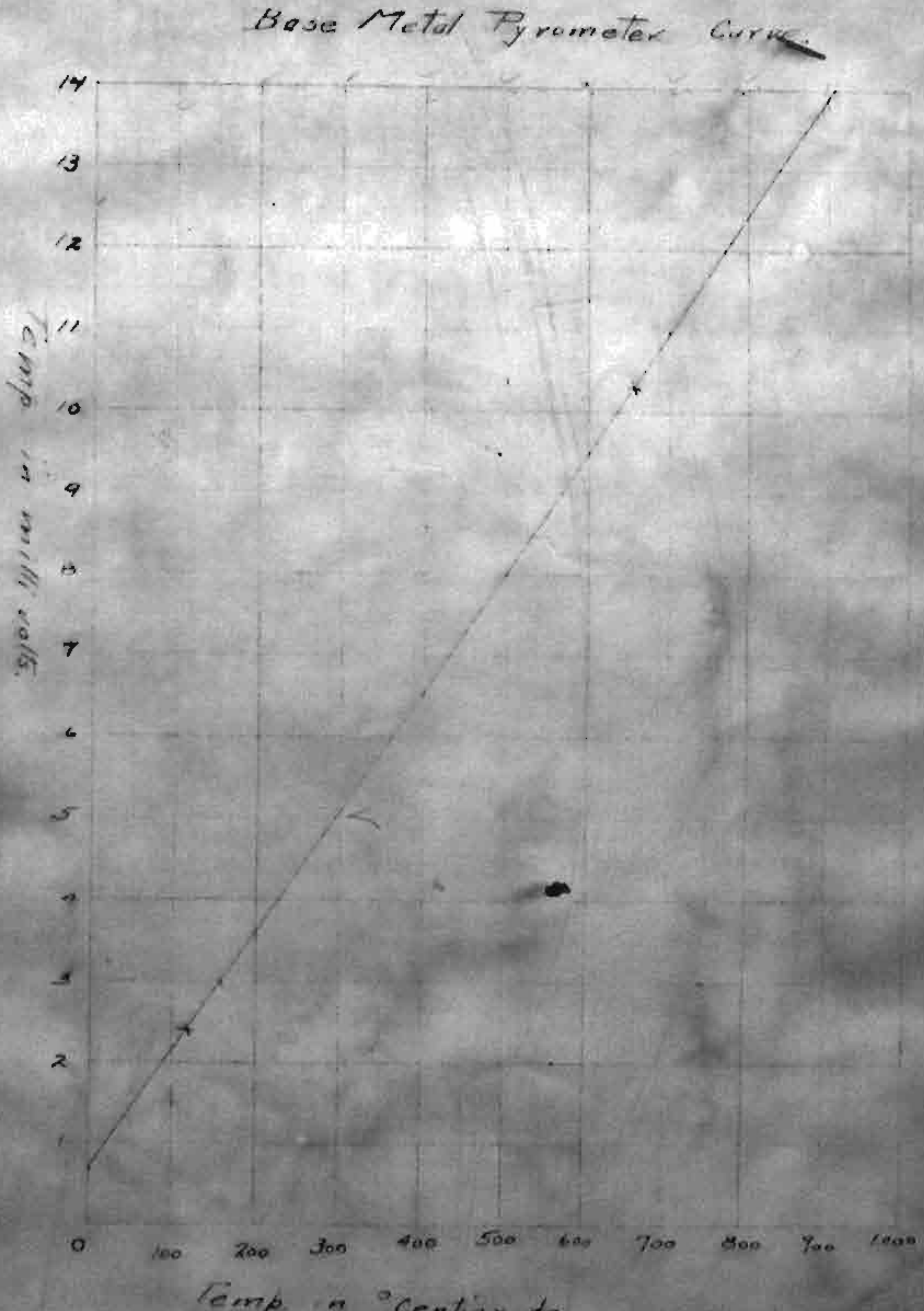
This graphite condenser was about 12" long, at the start, and the lower end had a hole bored into it to receive the pyrometer. In this way it was possible to get a fairly accurate idea of the conditions as to temperature existing in the condenser. As the tube was gradually shortened by sawing off small sections and the pyrometer was moved nearer the retort, the temperature of the gases leaving the condenser became hotter and hotter as the distance of the open end of the graphite tube from the crucible became less and less. The condenser was shortened about three inches at each change in it's length.

The pyrometer used for the condenser was a base metal junction of silver and nickel, while the one used for the furnace readings was platinum, platinum-iridium junction inclosed in a quartz tube.

The following free hand sketches will give some general idea of the appearance and arrangement of the furnace which was used for these experiments.

High Temp. Pyrometer Curve.





Base Metal Pyrometer Curve
14
13
12
11
10
9
8
7
6
5
4
3
2
1
Temp in milli volts
0
100
200
300
400
500
600
700
800
900
1000
Temp in °Centigrade

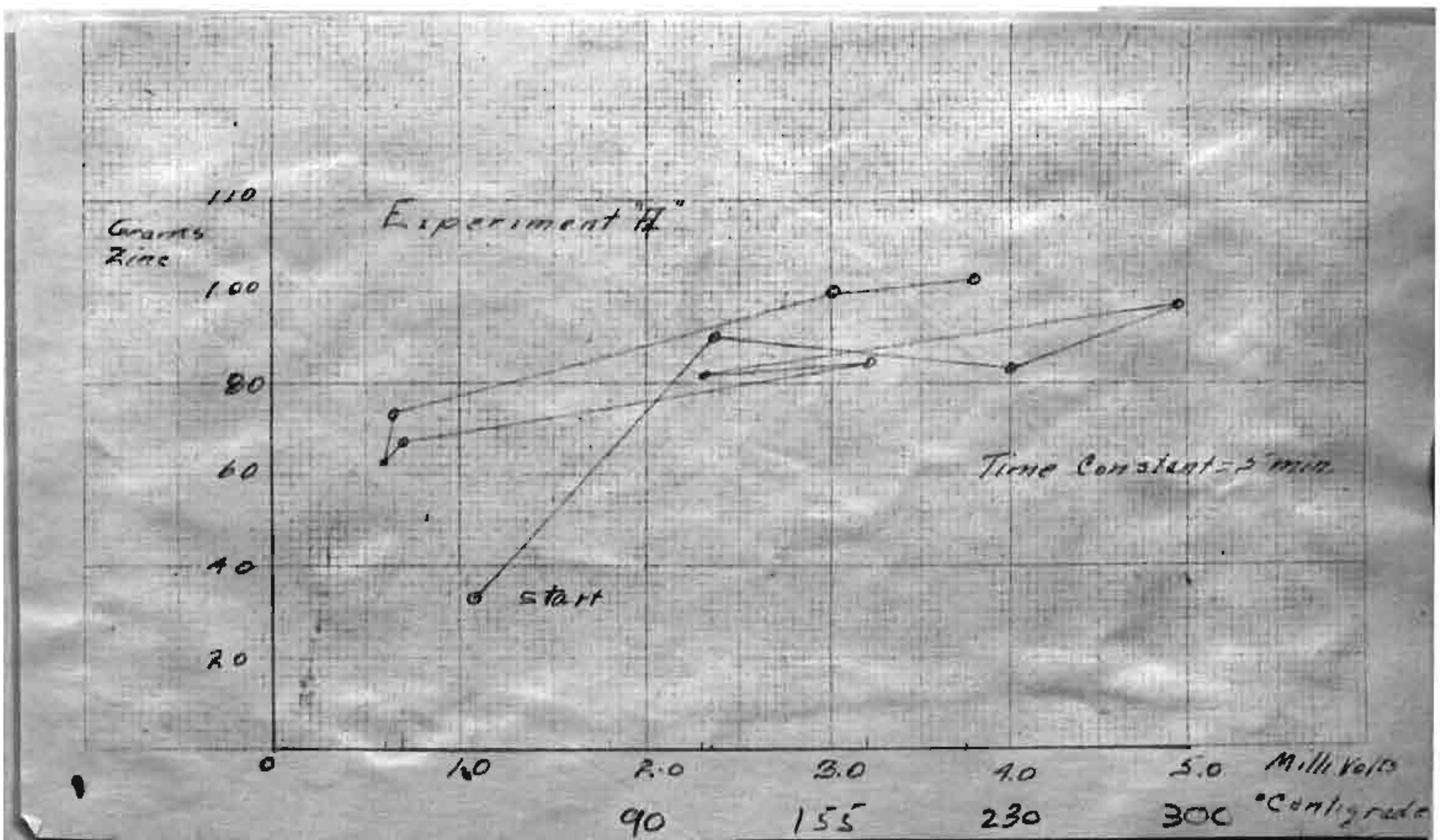
EXPERIMENT A.

This and the following experiment are simple preliminaries that were made in order to get some notion of the speed with which the zinc is boiled over under the conditions of our experiment. It may be seen that the amount of zinc obtained for a given interval varies widely. The temperature of the condenser was such that little zinc escaped uncondensed. Seemingly also there was no great variation in the temperature within the furnace and hence within the crucible retort. However, if the fact that the vapor tention of the zinc must rapidly rise as the temperature rises, the fact that to voletilize a unit of zinc requires absolutely a certain definite amount of heat, and the fact that the amount of heat transfered through the crucible walls is a function of the difference in temperature, i.e., that in the furnace minus that in the retort, such variations need cause no wonder. If the temperature in the furnace be 1000° C and in the retort 930° C then, if with necessarily the same retort, the temperature of the furnace be run up to 1080° C, we have in the first case a head of 70° C, while in the second case we have a head of 150° C, and the speed of distillation might be expected to be twice as rapid in the second case as in the first.

Curve plotted from results obtained in

EXPERIMENT A.

90 gm/hr



temp of retort or condenser?

EXPERIMENT A.

No	Condenser Temp Milli Volts	Condenser Temp °C	Grms Zinc Condensed in 5 Minutes	Remarks
1	1.18	40°	32.2 Grm's	
2	2.35	115°	90.5 "	
3	4.0	225°	82.55 "	
4	4.9	290°	39.05 "	Short time 2 Min $\frac{5}{2}$ x 39.05 = 97.60 estimate
5	2.3	110°	81.7 "	
6	3.2	170°	84.8 "	
7	0.7	40°-	67.00 "	
8	0.6	40°-	62.5 "	
9	0.65	40°-	72.7 "	
10	3.0	160°	99.5 "	
11	3.8	210°	103.4 "	
12	1.5	55°	53.95 "	Zinc in Retort beginning to get low
13	1.8	75°	54.4 "	" " "
14	0.8	40°-	82.8 "	Scrappings No Good. " " "

EXPERIMENT B.

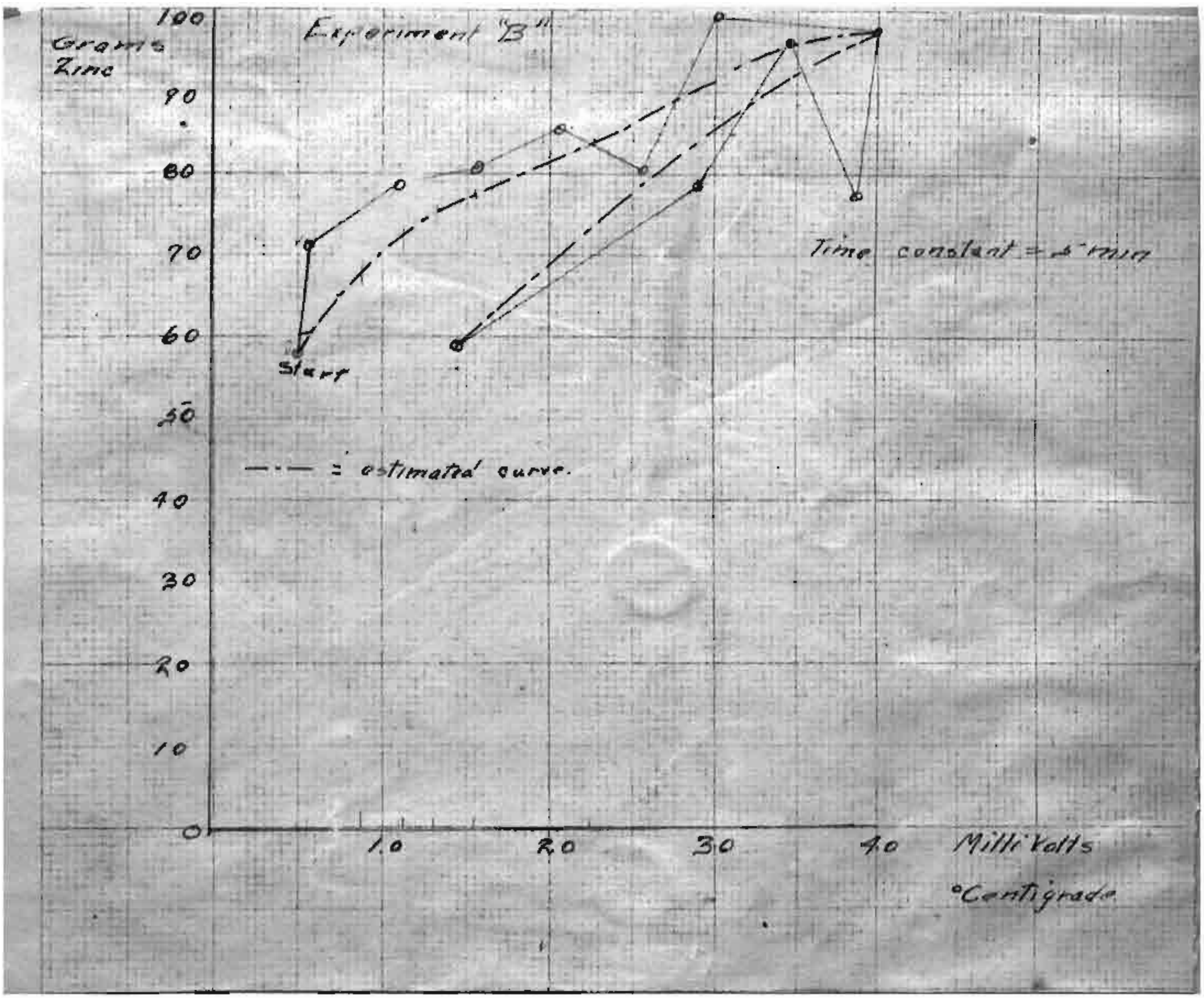
This is the second one of the preliminary experiments and was performed under practically the same conditions as in A. The variations in the results obtained from this experiment were large, but no more than were to be expected in an experiment of this kind.

EXPERIMENT B.

No.	Condenser Lamp Milli Volts	Condenser Temp °C.	Time in Minutes	Grms Condensed	Remarks.
1	0.5	40° –	5 Min	58 Grms	
2	0.7	40° –	5 "	71.6 "	
3	1.18	40°	5 "	78.9 "	
4	1.6	70°	5 "	80.8 "	
5	2.1	100°	5 "	85.85 "	
6	2.6	130°	5 "	80.6 "	
7	3.0	160°	5 "	99.6 "	
8	4.0	225°	5 "	97.85 "	
9	3.75	210°	5 "	77.7 "	
10	3.4	180°	5 "	76.4 "	
11	2.8	140°	5 "	78.6 "	
12	1.4	50°	5 "	58.8 "	

Curve plotted from results obtained in

Experiment B.



CURVE - EXPERIMENT B.

This curve needs very little explanation. All the substances caught in the condenser being weighed as zinc and plotted as such.

The dotted lines show the estimated curve for both the increasing and decreasing amounts of zinc.

EXPERIMENT C.

In the two preceeding experiments the main thing sought after was to get some notion of the amount of zinc which with our apparatus could be distilled and condensed, and to point out the great changes due to small changes in conditions. The object was to thy to show that as the temperature of the condenser increases the amount of blue powder will decrease.

In this experiment the time interval or period allowed the zinc to distill and condense was doubled and besides the condenser temperature another variable was taken into account - namely - the temperature of the furnace.

The pyrometer was placed in the furnace so that it almost touched the retort.

As this is the first experiment in which blue powder has been taken into consideration it may be well to mention the conditions which tend to make the blue powder form.

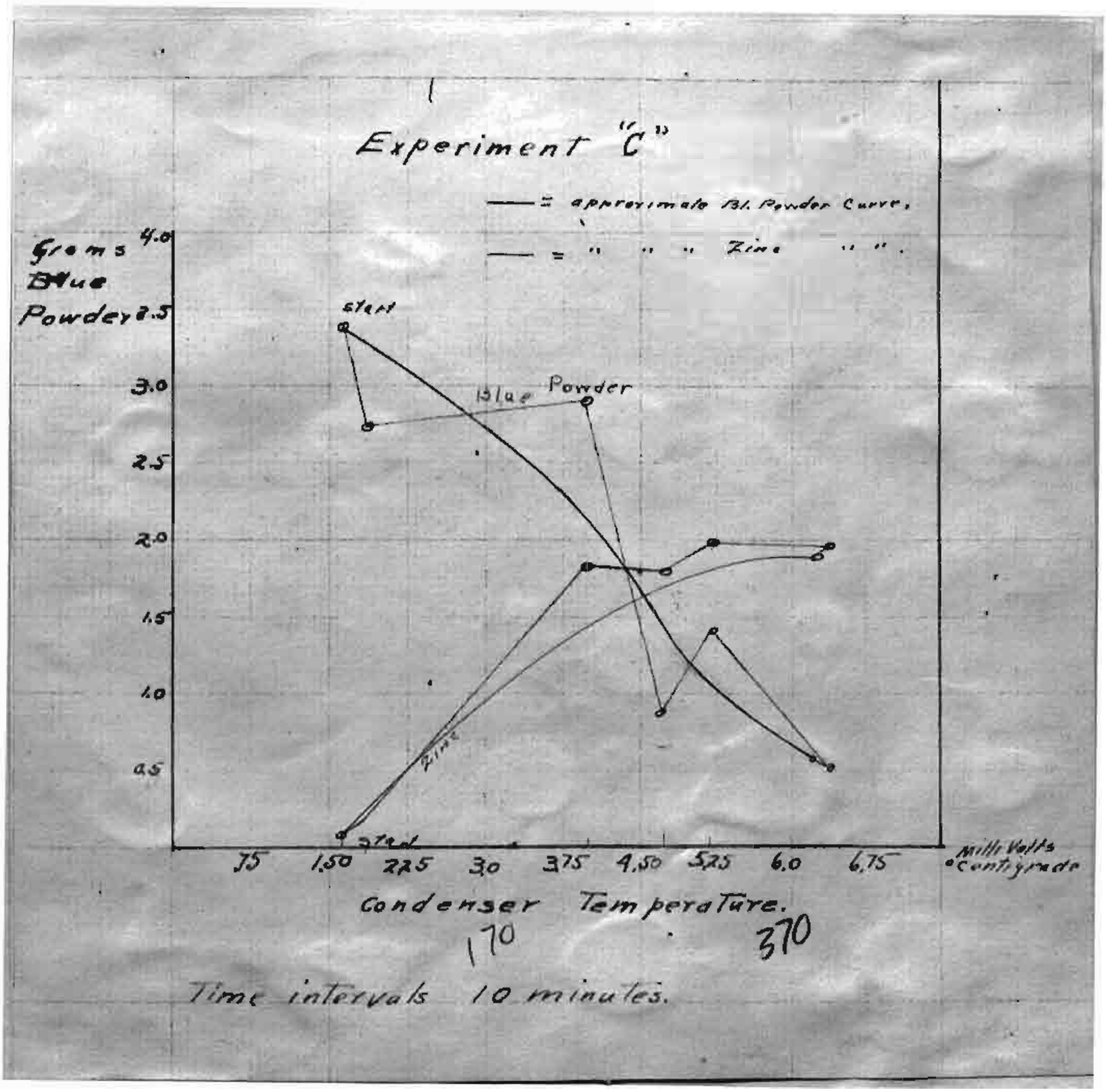
Blue powder seems to be minute particles of metallic zinc which are coated with a thin film of oxide. It's formation can be accounted for in the following manner. At the temperature of boiling zinc each molecule will exist by itself, as this temperature is lowered they will condense to a liquid mass. But these molecules have a certain definite surface tension and will need time to unite so as to form a molten mass. If they are chilled too rapidly the time element is then lacking and a hoar-frost of zinc is formed, this zinc hoar-frost is called blue powder. So at the start of the distillation, while the temperature in the retorts is high enough to volitilize the zinc, the condenser walls are so cold that the zinc vapor will form as a hoar-frost upon them, thus giving a large amount of blue powder at the start. Blue powder will also be formed if too large a quanity of any diluting gass gets into the condenser, the formation being along practically the same lines as those mentioned above. The blue powder which was formed in this experiment was weighed separately from the zinc and a different curve plotted for it. The end of the condenser tube, being open, more zinc oxide will tend to form at the start, do to the diluting gases, than would be in the case of the closed condensers.

EXPERIMENT C.

No.	Time in Min.	Condenser Temp Milli Volts	Condenser Temp °C.	Grms Zinc in Graphite Tube	Grms [illegible] in Graphite Tube	Muffle Temp Milli Volts	Muffle Temp °C.
1	10 Min	1.6	60°	7.5 Grms	3.40 Grm	10.1	1060
2	10 "	1.82	75°	18.2 "	2.73 "	10.3	1070
3	10 "	2.85	145°	97.9 "	2.85 "	10.43	1080
4	10 "	4.00	225°	183.3 "	2.98 "	10.55	1090
5	10 "	4.73	275°	175.3 "	0.9 "	10.67	1100
6	10 "	5.22	310°	193.8 "	1.42 "	10.8	1110
7 o	10 "	6.35	385°	192.25	0.54 "	10.83	1115
8	10 "	6.22	375°	190.7 "	0.6 "	10.85	1120

EXPERIEMNT C.

Curve.



CONCLUSIONS.

EXPERIMENT C.

It will be noted that in this experiment the production of blue powder at the start was extremely heavy, it being 31 % of the amount condensed in the first period, while at the end of the run it constituted only .31 % of the total amount. At the start the temperture of the condenser end was 60 °C, while at the end this temperture had risen to 375 °C, the amount of zinc condensed increased rapidly - the largest jumps occuring during the third period. In this period there also accured the greatest single increase in temperature. It will also be noted that the furnace temperature was increased as regularly as possible - the rate of increase during the first six periods of the run being approximately .12 millivolts, i.e. about 40 °C per minute.

It seems then, that the decrease in the amount of blue powder produced was largly due to the increase in the condenser temperature, or that the quick chilling of the walls forms the zinc dust or hoar - frost or blue powder.

Curve C shows graphically the amount of zinc condensed and the amount of blue powder formed.

The chief cause of the trouble in this experiment came from the fact that the increased period of time gave a much larger amount of zinc than could be easily handled.

EXPERIMENT D.

This experiment was carried on along the same lines as experiment C except that the time interval was reduced from ten minutes to four minutes.

As before the same variables were taken into account - namely - condenser and furnace temperature.

In this experiment the condenser and furnace temperature were kept within narrow limits as to their respective variations.

This was done in an endeavor to show that the same results will be obtained as in the cases where the chief varaition was in the condenser temperature.

CONCLUSIONS.

The actual plotting of the results obtained show a relatively great variations.

It is true again, in this case as in the others before mentioned, that the blue powder, in general, decreased gradually as the temperature of the condenser walls gradually increased. The reasons for the varations in the amount of molten zinc are not clear. The blue powder had at the last of the run entirely disappeared.

This was due to the high temperature at that time.

In plotting the curve for these results they were all figured for a time interval of four minutes. This was necessary as the some of the periods varied a minute or more in duration. All the results are figured to a constant length of time.

EXPERIMENT D.

Sheet 1.

Remarks	Time	Nature of Material	Blue Powder Grms	Metallic Zinc Grms	Condenser Temp Milli Volts	Condenser Temp °C	Furnace Temp Milli Volts	Furnace Temp °C
Zinc condensing much closer to furnace than Temp. was measured	start Heating 3-43	Blue Powder + a little met Zinc	1.7	0.7	2.2	100	8.6	1030
" "	3-47	Blue Powder + more molten Zinc	1.5	10.6	2.5	120	8.6	1030
" "	3-51	Blue Powder more molten Zinc	2.1	11.6	2.75	140	8.9	1040
" "	3-54	Blue Powder very little nearly all molten Zinc	0.6	18.3	3.10	165	8.7	1035
" "	3-58	Blue Powder more less molten Zinc	1.25	13.5	3.15	170	8.6	1030
" "	4-01	Blue Powder little nearly all Zinc [illegible]	0.7	17.1	3.9	185	8.7	1035
" "	4-04	Ditto above	0.6	16.1	3.4	185	8.7	1035
" "	4-09	Blue Powder little little molten Zinc nearly all Zinc condensed higher in Condenser than Pyrometer Tube	0.4	15.6	3.6	200	8.8	1037
" "	4-15	little Blue Powder little molten Zinc	1.2	24.3	3.5	190	8.9	1040
" "	4-20	Ditto above	1.25	34.3	3.6	200	9.2	1045

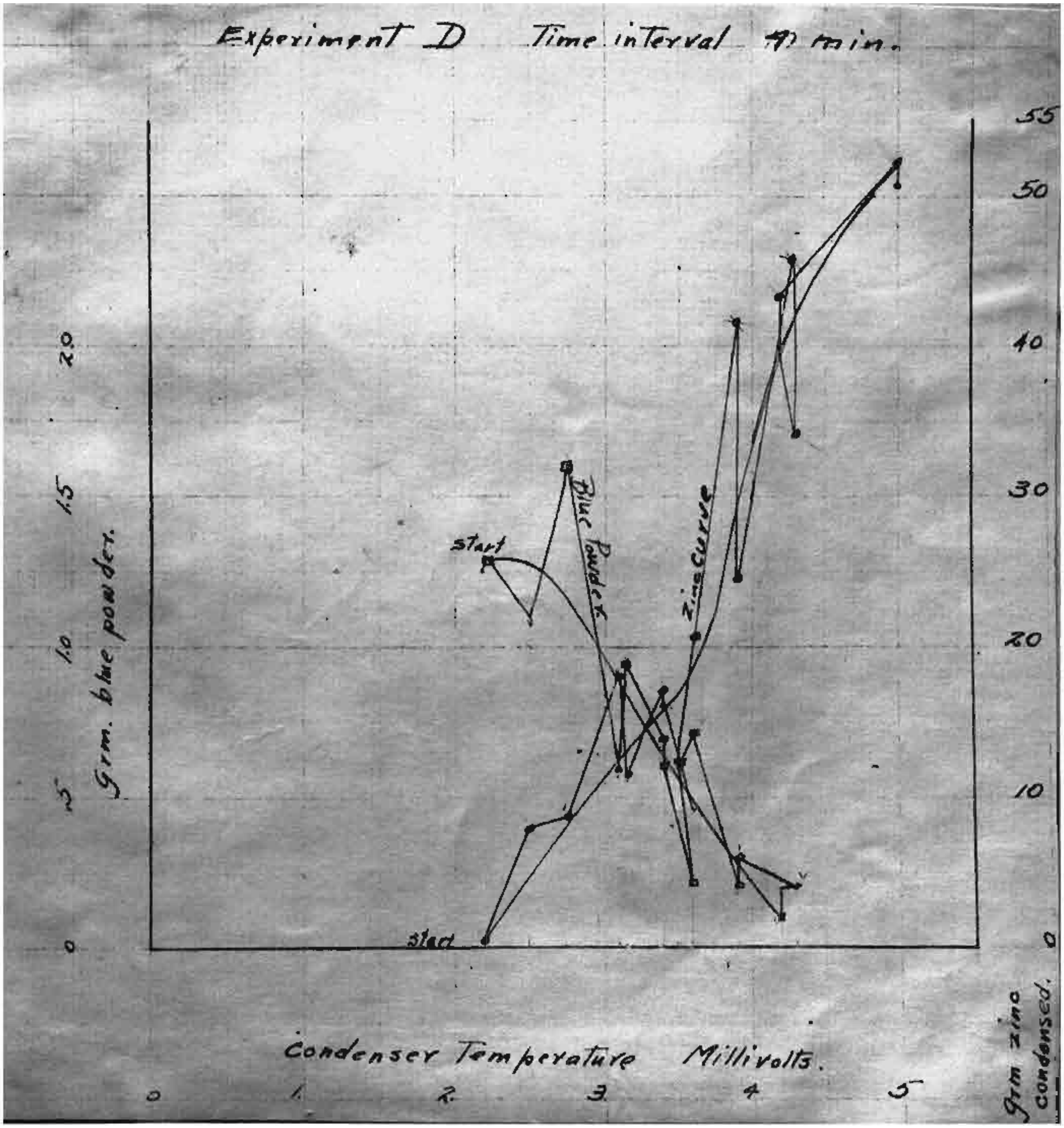
EXPERIMENT D.

Sheet 2.

Remarks	Time Hr. Min	Nature of Material	Blue Powder Grms	Metallic Zinc Grms	Condenser Temp milli Volt	Condenser Temp °C	Furnace Temp Milli Volts	Furnace Temp °C
condenser gets hot slowly	4-26	little Blue Powder much molten Zinc	0.4	83.6	3.9	220	9.3	1050
seems to be a little air up in	4-30	Ditto above	0.4	33.1	3.9	220	9.3	1050
fume since Zinc will catch fire at times	4-34	Blue Powder Little at Zn condensed high up in tube at point where junction is Zn solid [illegible]	0.3	61.4	4.3	250	9.0	1040
↓	4-39	Ditto above	0.3	79.7	4.3	250	9.0	1040
	4-42	Ditto above	0.2	40.0	4.2	240	9.0	1040
Tube caught this	4-44 4-48	Tube on Ditto above	0.2	96.7	4.2	240	9.0	1040
"	4-49 4-54 4-55	Tube on Tube pulled Finished pulling Zinc [illegible]	—	122.5	5.0	295	9.1	1045
"	4-55 4-59 5-00	Tube on Tube pulled Finished pulling Zinc	—	84.7	5.0	295	9.1	1045

EXPERIMENT D.

Curve. ,



EXPERIMENT E AND F.

In these experiments the condenser length was shortened which made the zinc condense much closer to the furnace and therefore at a higher temprature than in the previous experiments. The increase in temperature of the condenser was accompanied ofcourse by an increased escape of zinc vapor. In order to catch this escaping vapor, a glass tube was placed at the end of the condenser. This tube caught and condensed as blue powder nearly all of the zinc vapor which otherwise would have escaped uncondensed. At the end of each period the tube was cleaned out thoroughly.

The object of the tube was to determine, as the temperature of the condensor rose, how much more zinc vapor would have escaped to the air. In this experiment (E) the variations of the condenser and furnace temperatures were kept within as small limits as possible. In experiment (F) instead of trying to confine the condenser temperature within narrow limits, it was varied over a wider range than in any preceeding experiment. Owing to increased temperature the glass tube of experiment (E) was replaced, by one of quartz.

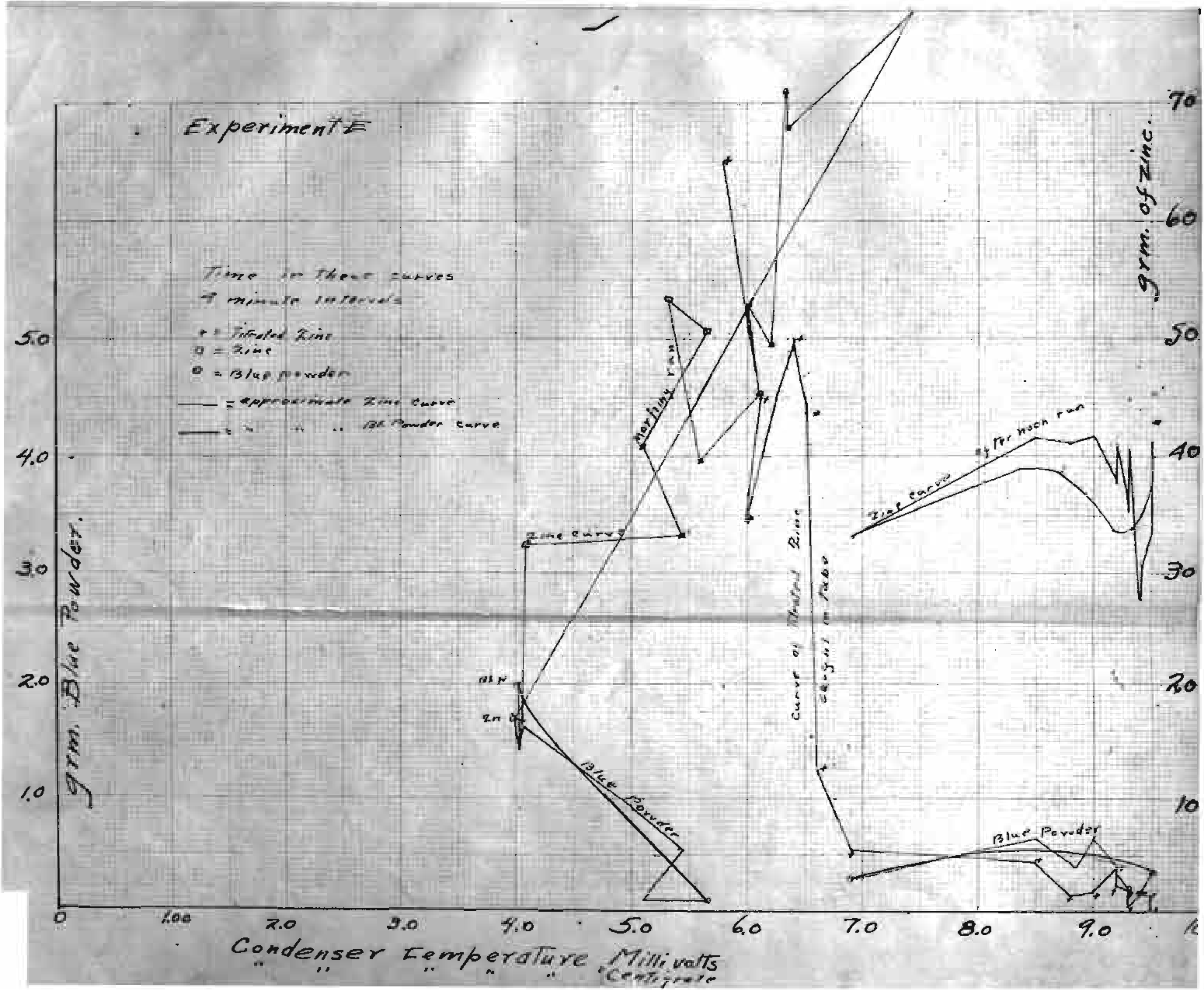
Experiment "E" sheet ①

Remarks	Time Hr-Min	Nature of Material	Blue Powder Grms	Metallic Zinc	Cond. Temp Millivolt	Cond. Temp °C	Furnace Temp Millivolt	Furnace Temp °C	Remarks	
Tube the zinc condenses in much	10-58	much Blue Powder very little Zinc	2.0	15.2	4.2	~~4.2~~ 240	9.2	1047		
shorter temp more accurate	11-02	little Bl. Powder molten Zinc	1.2	14.2	4.5	~~4.7~~ 260	9.3	1050		
	11-05	Ditto above	1.3	13.0	4.7	~~4.7~~ 275	9.3	1050		
	11-08	" "	0.7	24.1	5.0	~~5.0~~ 295	9.2	1047		
	11-12	" "	0.6	33.7	5.4	~~5.4~~ 320	9.2	1047		
	11-16	" "	0.2	41.0	5.2	~~5.2~~ 310	9.3	1050		
	11-20	" "	0.2	50.7	5.3	~~5.3~~ 315	9.3	1050		
no vapor escaped to tube	11-25	" "	0.2	66.7	5.6	~~5.6~~ 335	9.3	1050	Added glass tube here amt. of Zinc in Grms. caught	
Beaker used	11-30	" "	0.2	47.4	5.8	~~5.8~~ 350	9.3	1050	.065 grms	
" "	11-34	" "	–	45.2	6.1	~~6.1~~ 370	9.3	1050	.045 "	
" "	11-39			67.1	6.0	~~6.0~~ 360	9.4	1052	.035 "	
" "	11-44			61.9	6.2	~~6.2~~ 375	9.4	1052	.045 "	
" "	11-47			55.3	6.3	~~6.3~~ 385	9.5	1054	.050 "	
" "	11-51			67.8	6.5	~~6.5~~ 395	9.4	1052	.045 "	
" "	11-54 cont'd			70.5	6.6	~~6.6~~ 400	9.4	1052	.123 "	
	R-19 R-2? R-26	Tube on; Tube cleaned of Zinc	0.3	42.4	6.9	425	9.4	1052	.051 "	
	R-37 R-39 R-40	Ditto above	0.5	41.9	8.5	530	9.3	1050	.045 "	

Experiment F cont'd

Sheet (2)

Time Hrs-Min	Nature of Material	Bye Product Grms	Metallic Zinc	Cond. Temp Milli Volts	Cond. Temp °C	Furnace Temp Milli Volts	Furnace Temp. °C	Remarks.
2.43 2.45½ 2.46½	Tube on	0.3	41.2	8.8	550	9.3	1050	amt. of Zinc caught in tube titrated. .015 grms
2.49 2.49½ 2.52	Tube on	0.15	41.8	9.0	570	9.3	1050	.019 "
2.53 2.55	" "	0.3	37.6	9.2	580	9.2	1047	.037 "
2.55½ 2.57½	Tube on	0.2	41.25	9.2	580	9.2	1047	.023 "
2.58½ 3.00½	Tube on	0.15	36.2	9.3	585	9.2	1045	.019 "
3.01 3.03	Tube on	0.1	35.4	9.3	585	9.2	1045	.018 "
3.03½ 3.05½ 3.06	Tube on	Trace	40.7	9.3	585	9.1	1045	Trace .007 "
3.06 3.08	Tube on	No grm Bl R 0.1	27.8	9.4	595	9.1	1.045	.004 "
3.08½ 3.10½	Tube on	0.1 No Bl. R	30.7	9.4	595	9.1	1045	.016 "
3.11 3.13	Tube on							
3.13½ [illegible]	Tube on	0.2	33.7	9.5	600	9.1	1045	.015 "
3.15½ 3.16		0.3	41.3	9.5	600	9.1	1045	.003 "



Experiment E
Time in these curves
9 minute intervals
+ = Titrated Zinc
□ = Zinc
○ = Blue powder
— = approximate Zinc curve
— = " " " Bl. Powder curve
Morning run
Zinc curve
Blue Powder
Zinc curve after noon run
Blue Powder
Bl. P.
Zn
Grm. Blue Powder.
grm. of Zinc.
Condenser temperature Millivolts
0
1.00
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
1.0
2.0
3.0
4.0
5.0
10
20
30
40
50
60
70

Experiment "F"

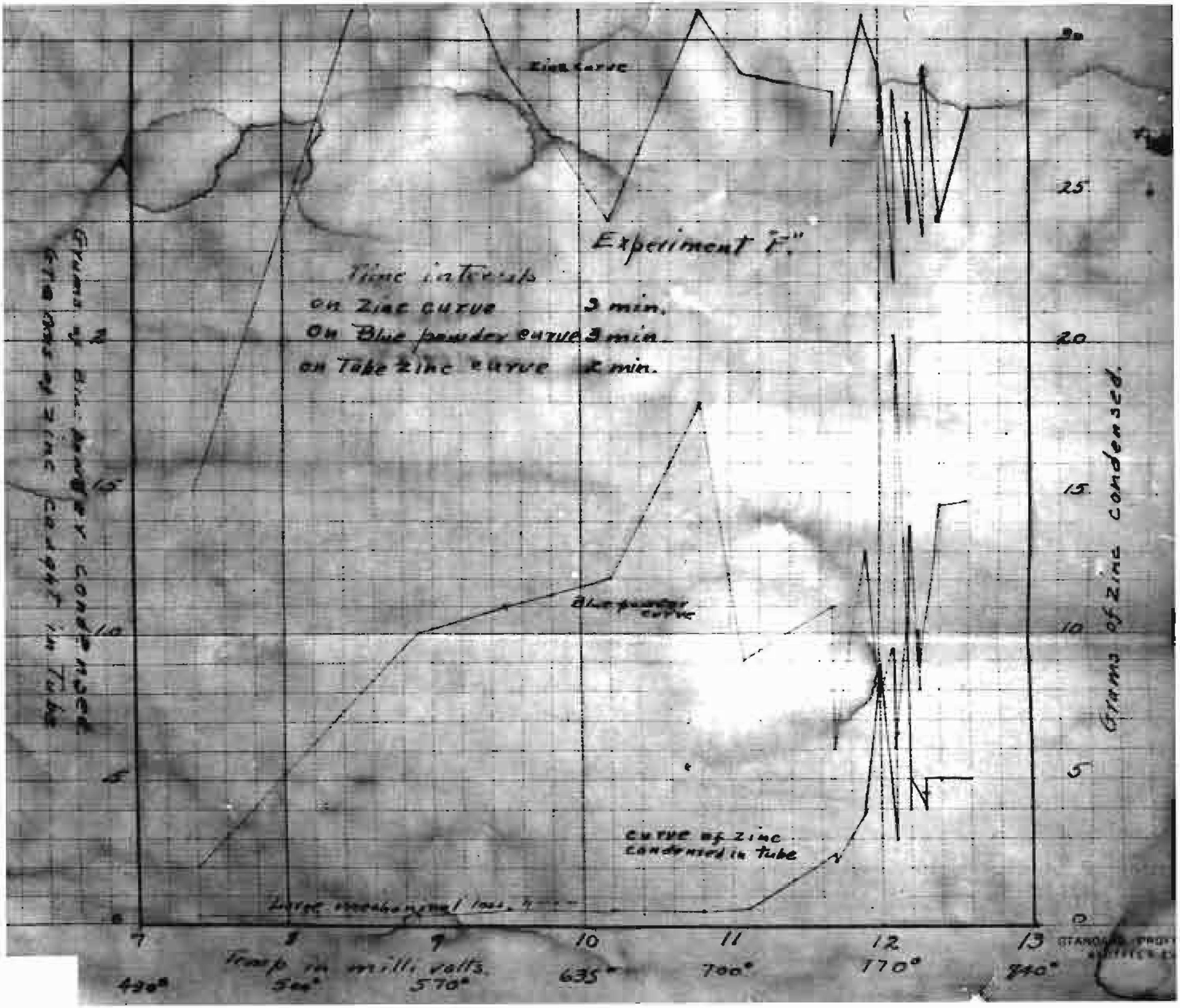
sheet (1).

Qtz Tube Short	Time Hr. Min		Blue Powder Grams	Metallic Zinc Grams	Cond' Temp Milli Volt	Cond' Temp °C	Furnace Temp. Milli Volt	Furnace Temp. °C	Remarks. Zinc caught in Qtz. Tube and aerated amt in Grams.
	8-55 9-00½		0.4	30.53	7.4	440	9.4	1052	.072
	9-01 9-01½ 9-04½		1.5	37.1	8.9	560	9.4	1052	.053
	9-05½ 9-08½		1.5	38.7	9.5	600	9.3	1050	.087
	9-09½ 9-12½ 9-13		1.7	31.4	10.2	650	9.2	1047	.091
	9-15 9-16		1.8	26.6	10.8	690	9.2	1050	.064 .053
	9-18 9-19		0.4	28.9	11.1	710	9.2	1049	~~.243~~ ? .061
	9-21 9-22		1.1	28.3	11.7	750	9.2	1049	.243
	9-24 9-25½		0.7	26.5	11.7	750	9.2	1047	.394 .223
	9-27½ 9-28½		1.3	30.9	11.9	765	9.2	1047	.396
	9-30½ 9-31½		0.8	24.1	12.0	770	9.1	1052	.885

Experiment "F" cont'd

Sheet (2)

Qtz Tube Short	Time Hr Min			Blue Powder Grams	Metallic Zinc Grams	Cond'r Temp Milli Volt	Cond'r Temp °C	Furnace Temp Milli Volt	Furnace Temp °C	Remarks: Zinc caught in Qtz Tube and Filtrated amount in Grams
	9-31½ 9-34 9-35			~~1.1~~ 1.1	25.5	12.2	775	9.1	1045	.365
	9-37 9-38			~~0.4~~ 0.6	28.9	12.1	775	9.1	1045	2.09.5
	9-38 9-40 9-42			~~0.6~~ 1.4	31.9	12.2	785	9.1	1045	.875
	9-44 9-45½			0.6 0.4	32.0	12.2	785	9.1	1045	2.07
	9-47½ 9-49			0.6	31.8	12.2	785	9.1	1045	1.37
	9-51½ 9-53½			0.6	34.3	12.3	790	9.1	1045	1.10
	9-55½ 9-57½			0.6	38.3	12.3	790	9.1	1045	1.05
	9-59½ 10-01½			0.7	38.6	12.3	790	9.1	1045	0.80
	10-03½ 10-05½			0.7	32.2	12.4	800	9.1	1045	1.46
	10-07½ 10-09½			0.6	31.5	12.6	810	9.1	1045	1.47



Zinc curve
Experiment "F."
Time intervals
on Zinc curve 3 min.
On Blue powder curve 3 min.
on Tube zinc curve 2 min.
Blue powder curve
curve of zinc condensed in tube
Grams of Zinc condensed.
Temp in milli volts.
7
8
9
10
11
12
13
570°
635°
700°
770°
840°
30
25
20
15
10
5
0

CONCLUSION..

EXPERIMENTS E AND F.

In these experiments as in the preceeding ones, the general trend of the blue powder was to decrease in amount as the temperature rose, while on the other hand, with the zinc condensed the opposite was true.

The time interval on experiment (F) was decreased to three minutes for the zinc and the blue powder, while for the zinc caught in the tube, the time interval was two minutes. The most noticable fact in experiment (E) was that the amount of zinc condensed in the glass tube decreased as the condenser temperature increased, while in experiment (F) the amount increased with the condenser temperature.

The only satisfactory explaination for the decrease of zinc cought in the tube seems to be that it was due to a mechanical loss.