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A granular briquet resistance furnace for the electrothermic dry distillation of zinc ores

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A GRANULAR BRIQUET RESISTANCE FURNACE FOR THE ELECTROTHERMIC DRY DISTILLATION OF ZINC ORES.

-By-

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A

THESIS

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C O N T E N T S.

I L L U S T R A T I O N S.

T A B L E S.

P R E F A C E.

This thesis is presented 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 Metallurgical Engineer. It describes experiments carried on at the Mississippi Valley Experiment Station of the United States Bureau of Mines, cooperating with the School of Mines and Metallurgy of the University of Missouri, which had for their purpose the development of a "granular briquet resistance furnace" for the electrothermic dry distillation of zinc ores.

I am greatly indebted to Dr. Charles H. Fulton, Director of the School of Mines and Metallurgy and Mr. B. M. O'Harra, Acting Superintendent of the Bureau of Mines station, under whose direction the work was carried on, for their permission to make use of the data obtained.

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INTRODUCTION.

The work here described had for its purpose the development of a granular briquet resistance furnace for the electrothermic dry distillation of zinc ores.

The history of electric furnaces for the reduction of zinc ores dates back to 1885, at which time E. H. and A. H. Cowles first suggested a design for an electric furnace for that purpose.[1] Their attention was soon directed to other uses for their furnace and nothing further of importance was done in the electrothermic smelting of zinc ores until about 1900. Since that time experiments have been made with numerous types of electric zinc smelting furnaces with little success. There are, however, furnaces operating in Norway and Sweden on a paying basis; their success is partly due to their having very cheap power available. It is doubtful if they would be commercially successful in America, where power is much more expensive, because their power consumption is high. In America

[1] U. S. Patent No. 319,795.

the Fulton[2] process is the only one which gives promise of developing into a commercially successful process. Briefly the Fulton process consists of the following steps: Oxidized zinc ore and crushed coke are briquetted with coal tar pitch as binder. The composition of the briquets varies with the nature of the ore; as a rule the composition is 100 parts ore, about 70 parts coke and 18 to 25 parts of pitch.

The briquets are baked at a temperature of 500° to 600° C. to drive off volatile hydrocarbons; the baked briquet is an electrical conductor, but only to such a degree that it can be used as a resistor by making it a part of an electrical circuit. By using it as an electric resistor in this manner it is heated by an electric current and the zinc is distilled. During this operation the briquet charge is covered by a movable retort and the zinc vapor and carbon monoxide are conducted to a condenser.[3]

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[2]Fulton, Chas. H., Electric resistance furnace of large capacity for zinc ores; Trans. A. I. M. E., Sept. 1919.

[3]For a complete bibliography on the electrothermic metallurgy of zinc and a description of the most important electric zinc furnaces see: O'Harra, B. M., Bibliography on the electrothermic metallurgy of Zinc; Bull. Missouri School of Mines and Metallurgy, Vol. 6, No. 2, March, 1922; See also, O'Harra, B. M., The electrothermic metallurgy of zinc, U. S. Bureau of Mines Bull. 208, 1923.

REASONS FOR UNDERTAKING THE DEVELOPMENT OF A GRANULAR BRIQUET FURNACE.

In the Fulton process large cylindrical or prismatic briquets of uniform size are used, being set up end to end to form a continuous resistor of uniform cross section. These large briquets must be made in a hydraulic press, and their setting up to form a continuous resistor requires considerable hand labor. The experiments described in this paper were undertaken in an effort to develop a furnace that would make use of the same principles as the Fulton furnace but that would enable small egg-shaped or similar small briquets to be used; such briquets can be made in a "roll press", such as is in common use for making coal briquets, more cheaply than the large briquets required in the Fulton furnace, and, with a furnace such as those to be described, can be charged to the furnace and discharged from it with a minimum amount of labor.

EXPERIMENTAL INVESTIGATION.

The Briquet:

In briquetting roasted ore, reducing agent and binder, an intimate mixture is formed. The ore particle is in contact with a particle of reducing agent and both are surrounded with a film of binder, thus forming a coherent mass of all the particles. The briquet thus made will preserve its form and be practically of the same volume after the extraction of metal as it was before. Should the briquet collapse during the distillation process the fines formed will sift to the bottom of the furnace, causing a region of very low resistance. This causes the upper part of the charge to cool and tends to condense the zinc vapor therein and allow only part of the zinc vapor and carbon monoxide to escape into the condenser.

Enough excess reducing agent (coke) must be used to reduce all of the reducible oxides in the ore and leave an amount sufficient to give the briquet the requisite stability and electrical conductivity.

The size of the ore used in the briquets, within a reasonable limit, has little effect on the strength of the briquet or on the extraction of the zinc. Ore that passes through a 10-mesh screen and 86 per cent of which is coarser than 35-mesh shows practically no difference in the results, as compared with ore all of which passes through a 200-mesh screen.

The size of the coke has a strong influence upon the strength of the briquet; fine coke making a much stronger briquet than coarse coke. Coke that passes through a 10-mesh screen is a very satisfactory size for making strong briquets.

The character of the coal tar pitch is very important. The chief points to be noted in selecting a pitch for briquetting purposes are, the character of the coke left after the distillation of the volatile matter, the percentage of coke remaining after distillation, and the melting point of the pitch. In general, a high melting point pitch, 170° to 200° C., leaving 50 to 60 per cent of good firm coke is best.

Size of Briquets:

The size of the briquets used in the charge of the furnaces, for the most part, consisted of approximately one-inch cubes. Due to the lack of apparatus for making briquets of the required size, large briquets were made and then crushed to the size desired. In most of the experiments all material which passed through a one-half inch screen was rejected and constituted the sample for chemical analysis, while that which remained on a one-half inch screen and passed through a one and one-half inch screen formed the charge.

Method of Making the Briquets:

The composition of the briquets used in all of the experiments was 100 parts roasted zinc concentrate, 70 parts of minus 10-mesh coke and 25 parts of coal tar pitch. The method employed in making the briquets was as follows: The ore and coke were first mixed thoroughly, then heated with the pitch on a large hot plate, to between 175° and 200° C., or until yellow fumes appeared from the pitch. The ore and coke mixture was spread on the hot plate and the pitch placed in the center of the charge. After the pitch had melted the entire mass was thoroughly mixed. It was then moulded into briquets 3" x 4" x 7", the material being compressed by means of a large screw jack hand press shown in Figure 1.

This press was later replaced by a hydraulic press shown in Figure 2. The pressure varied from 500 to 1500 pounds per square inch; a 1500-pound pressure per square inch producing the best briquet.

Baking the Briquets:

The purpose of baking the briquets is to coke the pitch. The raw briquets were placed in the carbon resistance furnace shown in Figure 3, and gradually heated for a period of eight hours. At the end of this time the briquets had attained a temperature of about 500° to 600° C. The briquets must be kept from oxidizing during the baking; that is, they must be under neutral or reducing conditions. The baked briquets were then crushed to the desired size by hand or by putting them through a Blake crusher set to crush to a very coarse size.

Description of Furnaces and Results Obtained:

Furnace A: The first furnace was hurriedly constructed for the purpose of ascertaining, in a general way, whether sufficient current could be passed through a granular charge of briquets to furnish enough heat to distill the zinc. The furnace had two 12" x 12" x 1" carbon plates placed twelve inches apart. Into the center of the face of the carbon plates two-inch carbon rods, of the

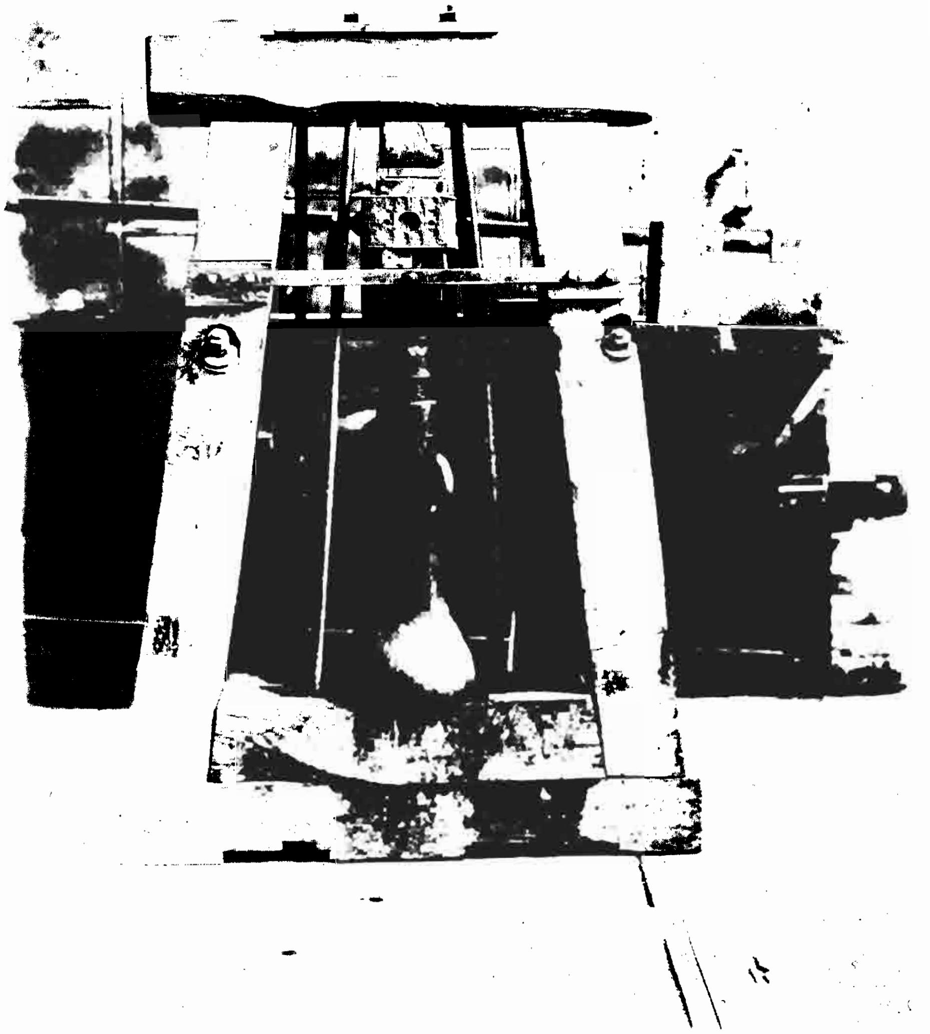

Figure 1. Hand Briquet Press.

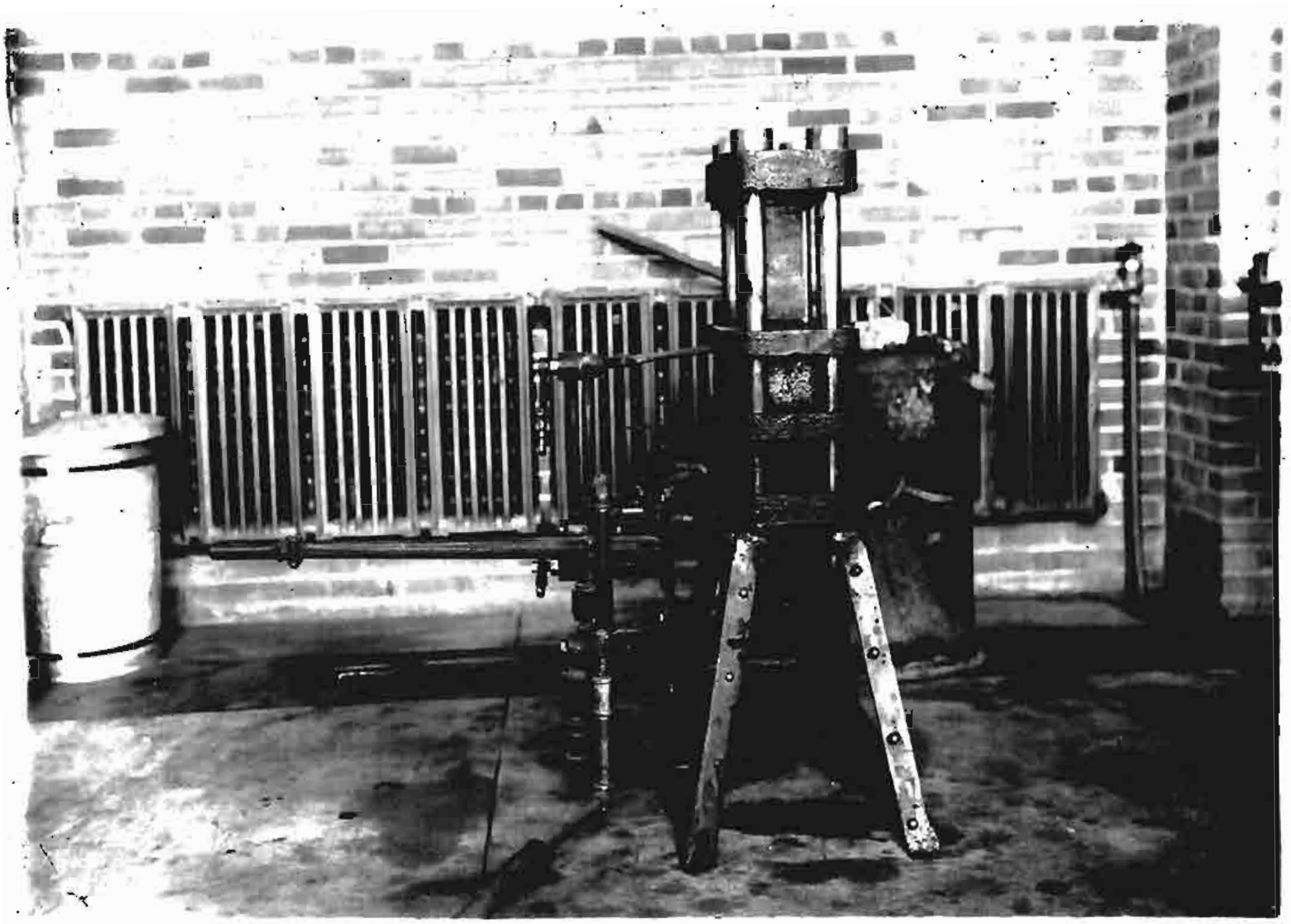

Figure 2. Hydraulic Briquet Press.

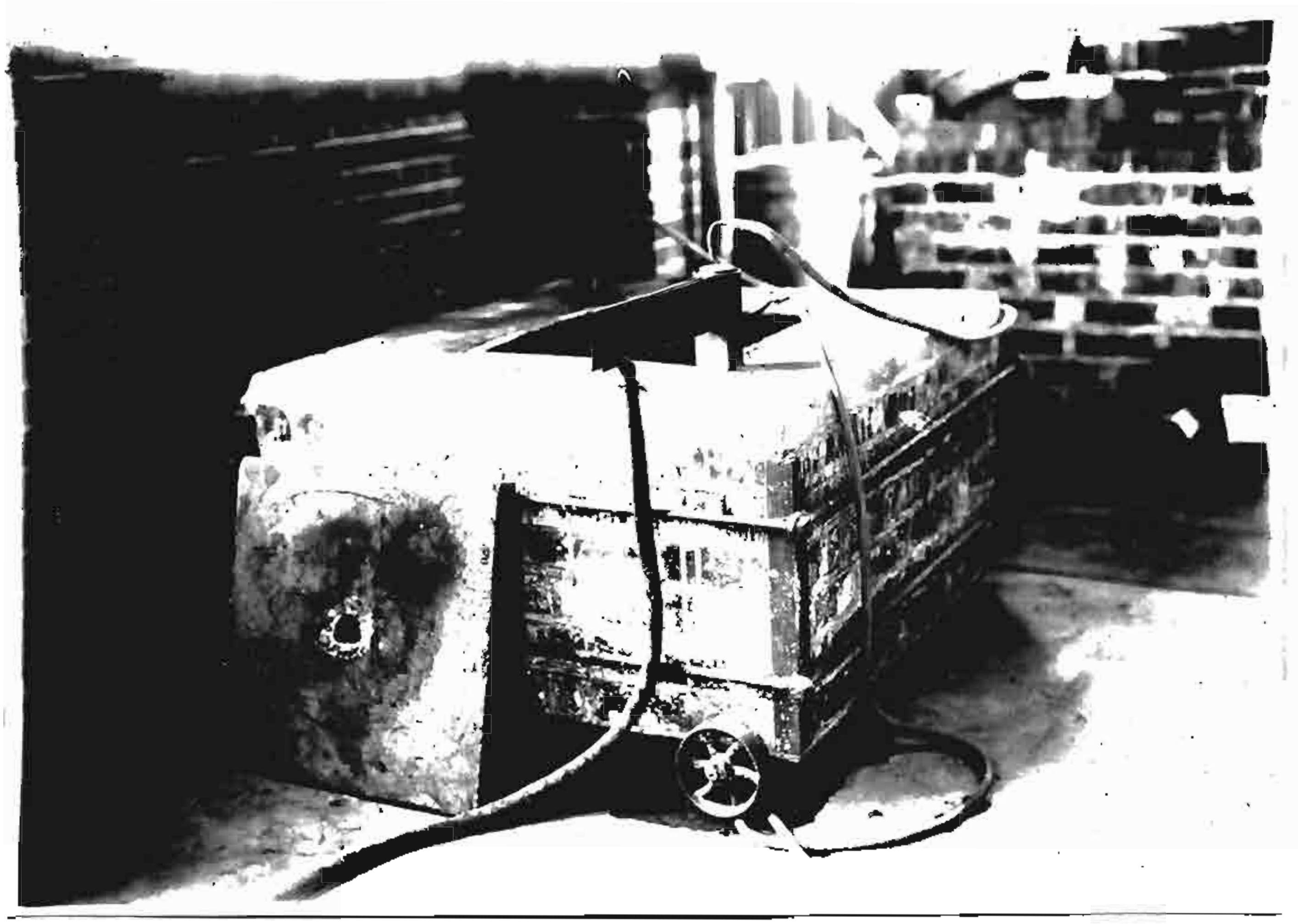

Figure 3. Carbon Resistance Baking Furnace.

proper length, were screwed, so that they would extend beyond the brick work. On the ends of the rods the electrode holders and coolers[4] were clamped, to which the cables from the current transformers were fastened. The sides and bottom of the furnace were laid up of fire brick. The walls were laid up so as to extend one inch above the carbon plates. The outer faces of the carbon plates were protected by a wall of fire brick to prevent them from being oxidized. Into one of the furnace walls, between the electrodes, an ordinary zinc retort condenser was fitted and the entire furnace surrounded with a wall of insulating brick. A fire-clay slab, on top of which insulating brick was placed, was used for a cover. The furnace was charged and discharged through the top opening.

In the first run made, the charge consisted of briquet material which would all pass through a one-half inch screen. In order to start the current through the cold charge it was found necessary to distribute a small amount of powdered graphite throughout the charge. This charge was allowed to operate for eleven hours and forty-five minutes with an average power input of 14 K. W. at 25 to 35 volts. Some liquid zinc was obtained, the greater part of the recovery was blue powder. Upon opening the furnace, after distillation, it was evident that the charge was too fine, as indicated by hot spots, due to the unequal distribution of the fine material.

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[4]Jackson, R. O., U. S. Patent No. 1,242,554.

No zinc recovery data was taken for this run, but it proved that a granular charge of briquets could be distilled. Two runs followed, the charges consisting of one and one-half inch and one-inch cubes respectively, with all of the minus one-half inch material removed. These charges were allowed to operate for eight hours and thirty minutes each, with an average power input of 15 K. W. at 60 volts. No trouble was experienced in maintaining a constant flow of current. The two runs operated very successfully and showed that small briquets, free from fine material, could be made to work successfully. The results obtained with this furnace are given in Table I.

It was decided at this juncture that all charges would be made up of minus one and one-half inch plus one-half inch mesh briquets and the briquets used in all of the following experiments were of this size unless otherwise specified.

Furnace B: The results obtained from these two runs led to the remodeling and enlarging of the furnace, the carbon plates being placed 24 inches apart in the new furnace. The furnace was charged with one-inch briquets, but the power available was not sufficient to operate the furnace successfully and no results of value were obtained with it.

Table I.

Results for Furnace A.

Run Number	2	3
Weight of Charge, pounds	65	64
Zinc in Charge, ounces	287	286
Weight of Residue, pounds	36	33.5
Zinc in Residue, ounces	62	12
Zinc Recovered as Metal, ounces	104	140
Zinc Recovered as Blue Powder, ounces, ...	81	59
Total Zinc Recovered, ounces	185	199
Total Zinc Accounted for, ounces	247	211
Per cent Zinc Recovered	64.50	65.40
Per cent Zinc in Residue	21.60	4.20
Per cent Zinc Accounted for	86.11	69.60
Duration of Run in Hours	8.25	10.5
Size of Briquets (cubes), inches	1.5	1.0

Furnace C: The next furnace was of a different design. It was constructed so that it could be mechanically discharged. The two 12" x 12" x 1" carbon plates with the two-inch carbon rods secured into the center of the face of the plates were placed 16 inches apart and inside of a one-fourth inch thick 20" x 26" x 10" sheet iron form, resting on two 16" x 36" x 4" fire-clay slabs mounted in an angle iron frame 17" x 38" x 14". A hole 12" x 18" was cut in the center portion of the fire-clay slabs to correspond to the inner dimensions of the furnace, allowing the carbon plates to extend two inches below the upper surface of the slabs. Sufficient space was allowed in making the sheet iron form so that the walls of the furnace could be laid up of four-inch fire brick on the inside of it. The walls were laid up to extend one inch above the carbon plates. In one side wall, between the electrodes, a hole was cut in the sheet iron form to allow for the setting of the retort condenser. Openings were cut in the short walls of the iron form to allow the carbon rods to extend out for electrical connections. The furnace was surrounded with insulating brick. The angle iron frame was provided with extensions at the upper corners with which to pick the furnace up when it was to be discharged. A fire-clay slab was used as a cover. The furnace was charged through the top opening and discharged through the opening in the bottom. The angle iron frame rested upon an iron table, having a top made of fire-clay slabs. Charging and discharging of the furnace were accomplished in the following manner: A ridge of fire-clay luting material was placed in the center portion of the table top, to correspond to the size and shape of the opening in the bottom of the furnace. The furnace was lowered, by crane, onto the ridge of luting material, thus sealing the furnace,and was then ready to receive the charge. To discharge, the furnace was raised by crane just enough to break the bottom seal; it was then moved toward one of the long edges of the table, allowing the spent charge to drop into a wheelbarrow which had been placed there for that purpose. The results obtained with this furnace are given in Table II. The average power input was 13 K. W. at 60 volts.

Table II.

Results for Furnace C.

Run Number	1	3	4	5	6	7	8
Weight of Charge, pounds	60	69.5	69.25	69	69	70	72
Zinc in Charge, ounces	264	416	408	407	420	420	426
Weight of Residue, pounds	33	36.25	38	41	45	29	33
Zinc in Residue, ounces	10	70	85	51	76	18	18
Zinc Recovered as Metal, ounces	140	124	132	201	183	195	177
Zinc Recovered as Blue Powder, ounces	16	106	84	98	102	125	135
Total Zinc Recovered, ounces	156	230	216	299	276	320	312
Total Zinc Accounted for, ounces	166	300	301	350	361	338	330
Percent Zinc Recovered	59.10	56.50	52.90	72.08	67.60	76.31	73.22
Percent Zinc in Residue	3.80	16.80	20.90	12.57	18.13	4.32	4.31
Percent Zinc Accounted for	62.90	73.30	73.80	84.65	85.73	80.63	77.54
Duration of Run in Hours	11	10	10	12	11.25	13	15

Furnace D: This furnace was of the stationary type and was designed to be charged and discharged continuously after the manner of a blast furnace. The briquets were charged into a hopper on top of the furnace and the spent charge drawn out of an opening in the bottom of the furnace. The internal volume of this furnace was the same as that of Furnace B, but the dimensions of the carbon plates were 18" x 8" x 2". The carbon plates were placed sixteen inches apart. Two-inch carbon rods were secured into the center of the faces of the plates for electrical connections. The walls and base of the smelting chamber were laid up of fire brick. Into one wall, and in between the carbon plates, and two inches below the top of the smelting chamber, an ordinary retort condenser was fitted. Below the condenser an opening 6" x 8" was left, extending through the furnace wall. The bottom of the opening was on the same level with the bottom of the smelting chamber. The fire brick walls and carbon plates were surrounded by a course of insulating brick and a course of red brick. The furnace was tied together by means of tie rods and angle irons. On top of the smelting chamber walls a hopper fifteen inches high was built of brick with a top opening 6" x 16". The opening in the hopper just above the smelting chamber was 3" x 16".

One run of twenty-two hours and thirty minutes was made in this furnace with little success. Trouble was experienced in getting the material in the hopper to feed down into the smelting chamber. The region in the lower part of the hopper sintered together, due to the condensation of zinc in that part of the charge, forming an arch which would not allow the material to feed down into the smelting chamber. No trouble was experienced in drawing the spent charge.

It was decided that the chances of developing a successful continuous furnace were not promising, therefore the work on this furnace was abandoned.

Furnace E: To form Furnace E the hopper was removed from Furnace D and another condenser added. A fire-clay slab was used for a cover. The internal dimensions and discharge opening remained the same. The construction of this furnace is shown in detail in Figure 4. The results obtained with this furnace are given in Table III.

Furnace F: The construction of this furnace is shown in detail in Figure 5. The two condensers were built into one of the walls and the discharge opening into the opposite one. It was observed, during the first run, that the condensers were not heating up as they should. After the run the insulating brick were removed from around and below the condensers and laid up about a foot from the furnace wall, in order to allow more heat to radiate to the condensers.

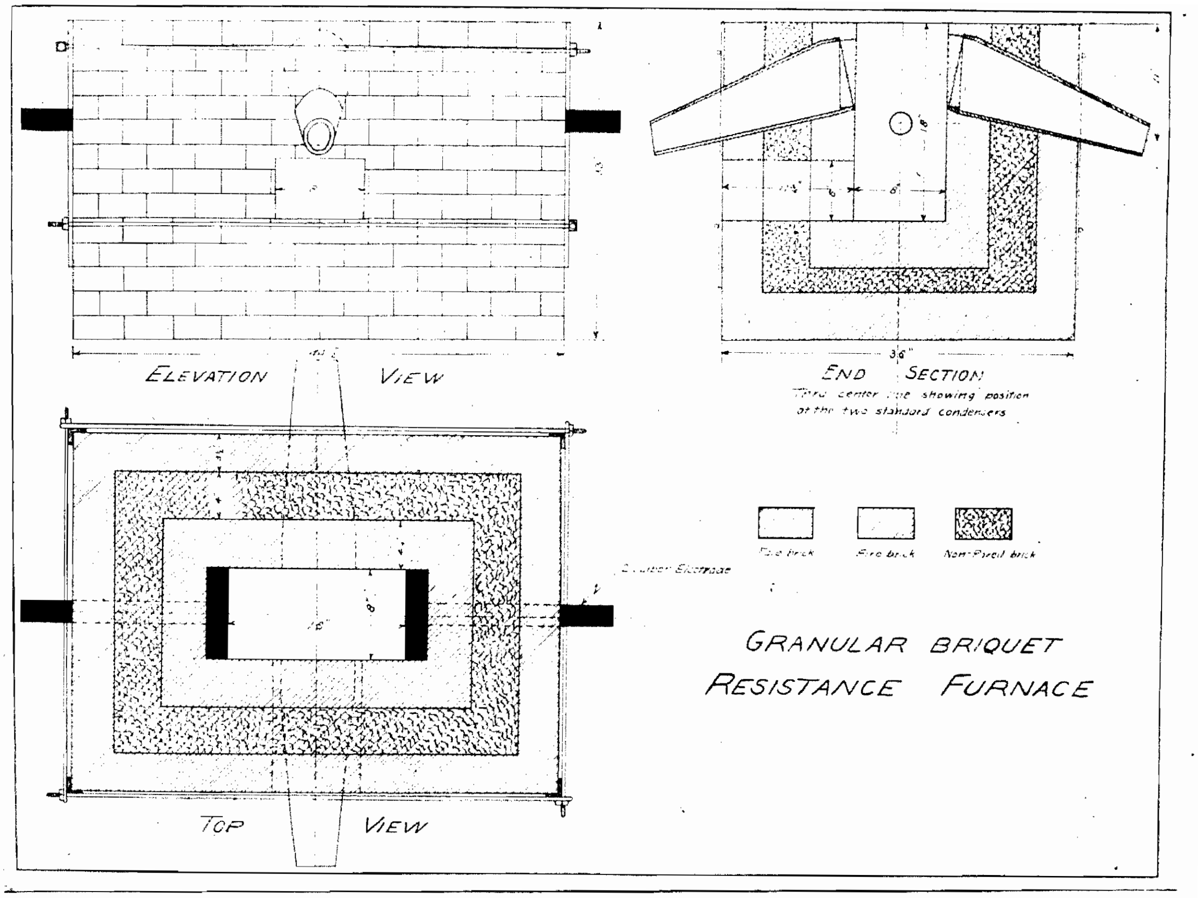

Figure 4. Furnace E.

Table III.

Results for Furnace E.

Run Number	2	3
Weight of Charge, pounds	78	78
Zinc in Charge, ounces	464	459
Weight of Residue, pounds	38.5	37
Zinc in Residue, ounces	73	26
Zinc Recovered as Metal, ounces	183	159
Zinc Recovered as Blue Powder, ounces	114	194
Total Zinc Recovered, ounces	297	353
Total Zinc Accounted for, ounces	370	379
Per cent Zinc Recovered	63.99	76.90
Per cent Zinc in Residue	15.73	5.66
Per cent Zinc Accounted for	79.72	82.56
Duration of Run in Hours	12	12

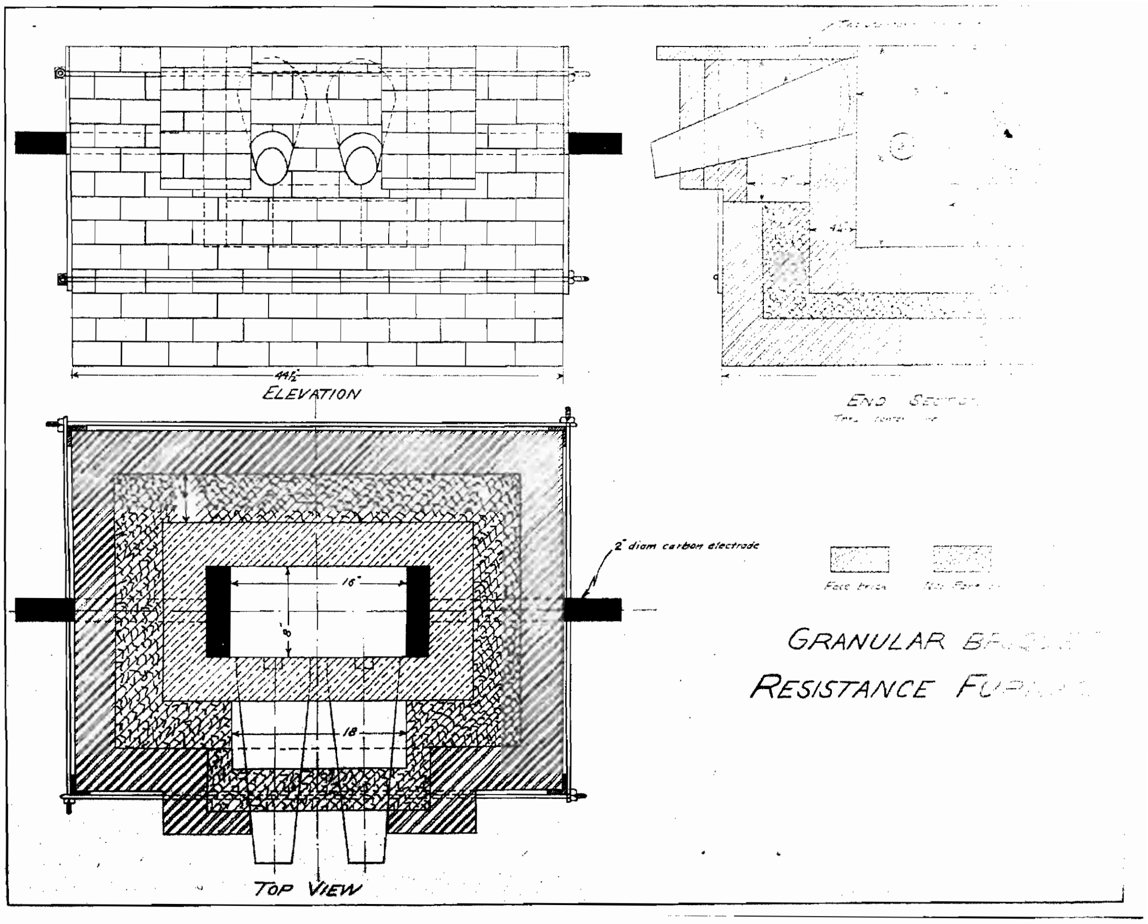

Figure 5. Furnace F.

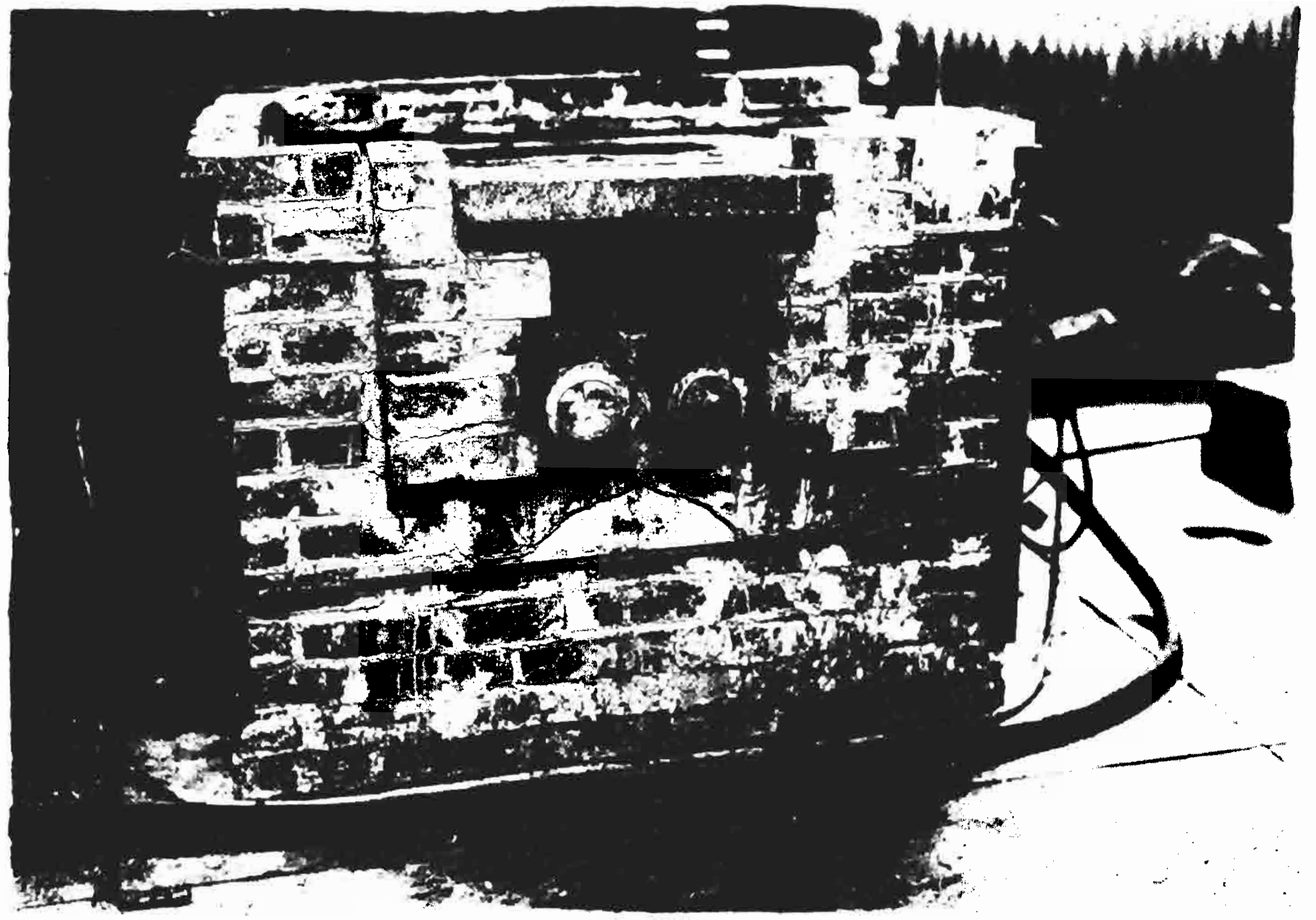

Figure 6. Furnace F.

A two-inch fire brick was fitted, in a vertical position, into the condenser inlets in order that the smelting chamber could be completely filled with the charge, thereby avoiding the condensation of zinc on the surface of the charge, which, up to that time had not completely filled the smelting chamber around the condenser inlets. These two changes proved very beneficial. The results obtained with this furnace are given in Table IV.

Table IV.

Results for Furnace F.

Run Number	5	6	7	8	10	11
Weight of Charge, pounds	78	77.5	77.5	77	76.5	80
Zinc in Charge, ounces	455	457	467	457	363	493
Weight of Residue, pounds	38	39.5	38	35	43	36
Zinc in Residue, ounces	63	88	64	20	91	59
Zinc Recovered as Metal, ounces	154	144	226	288	184	310
Zinc Recovered as Blue Powder, ounces	106	104	107	84	35	60
Total Zinc Recovered, ounces	260	248	333	372	219	370
Total Zinc Accounted for, ounces	323	336	397	392	310	429
Per cent Zinc Recovered	57.13	54.25	71.31	81.56	60.32	75.05
Per cent Zinc in Residue	13.85	19.25	13.70	4.39	25.06	11.96
Per cent Zinc Accounted for	70.98	73.50	85.01	85.95	85.38	87.01
Duration of Run in Hours	12	12	12	16	14	16

Table V.

TYPICAL DATA SHEET TAKEN DURING A RUN.

Run Number 7 November 7, 1924.

Weight - 77.5 pounds

Analysis of Charge - 37.64 per cent Zinc

Zinc in Charge - 29 pounds and 3 ounces

Running Log.

Time	Volts	K. W.	Remarks
5:25 A M	264		
6:00	280		Gas ignited
6:30	228		Very good flame
7:00	208		Very good flame
7:30	164		Zinc in flame
8:00	140	12	Zinc in flame
8:30	120	13	Good flame
9:00	105	13	Good flame
9:30	105	13	Good flame
10:30	90	13.5	Good flame
11:00	93	13.5	Good flame
11:30	89	14	Good flame
12:00 P M	84	14	Good flame
12:30	76	14	Good flame
1:00	78	14	Good flame
1:30	73	14	Good flame
2:15	73	13.5	Good flame
2:20	67	12	Strong zinc flame
2:50	70	12.5	Strong zinc flame
3:15	69	13.5	Strong zinc flame
3:35	70	13	Good flame
4:00	70	12.5	Good flame
4:30	66	12.5	Good flame
5:00	63	12.5	Good flame
5:10	63	12.5	Power off

Weight of Residue - 38 pounds

Analysis of Residue - 10.57 per cent zinc

Zinc in Residue - 4 pounds

Loss in weight of Charge - 50.65 per cent

TYPICAL DATA SHEET TAKEN DURING A RUN (Continued).

Metal Recovered:

<u>North Condenser</u>:

Zinc	9 pounds
Blue Powder in Condenser	1 pound 12 ounces
Blue Powder in Prolong	1 pound 1 ounce

<u>South Condenser</u>:

Zinc	5 pounds 2 ounces
Blue Powder in Condenser	2 pounds 13 ounces
Blue Powder in Prolong	1 pound

T O T A L

Zinc	14 pounds 2 ounces
Blue Powder	6 pounds 10 ounces

SUMMARY

Zinc Recovered	Weight	Per cent	Accumulative per cent
Zinc	14 lb. 2 oz.	48.4	48.40
Blue Powder in Condenser	4 lb. 9 oz.	15.63	64.03
Blue Powder in Prolong ...	2 lb. 2 oz.	7.28	71.31

Zinc Accounted for	Weight	Per cent	Accumulative per cent
Zinc Recovered	20 lb. 13 oz.	71.31	71.31
Zinc in Residue	4 lb.	13.70	85.01

SUMMARY AND CONCLUSIONS.

Although the work described in this thesis cannot be considered complete until larger furnaces of the same type have been tried, and operated continuously over considerable periods of time under semi-commercial conditions, the results of the experiments indicate that the granular briquet resistance furnace has considerable promise as a furnace for the distillation of zinc ores.

The recoveries obtained in the experiments that have been described may be criticized. It must be remembered, however, that a number of different furnaces were worked with, under a variety of conditions. In many cases only one or two charges were distilled in a given furnace; if these runs indicated that changes in the furnace design were desirable the changes were made at once and the new furnace tried. Under these conditions there was little opportunity to standardize operating conditions or to ascertain the time necessary to distill the charge completely. Enough charges were distilled to completion to prove that with proper operating conditions all but three to five per cent of the zinc can be distilled. For the same reasons not all the zinc distilled was condensed, in many of the runs; this fact, together with the fact that none of the furnaces were operated for a sufficiently long time for the walls to become saturated with zinc, explains the considerable amounts of zinc unaccounted for in all the runs. The proportion of zinc condensed to metal, rather than blue powder, was very satisfactory, especially in the later types of furnace.

As the power consumption in a small furnace, because of the large radiating surface as compared to volume, is very much larger than in a commercial sized furnace, no attempt was made to obtain any data as to power consumption. Reasoning by analogy, it seems that the power consumption in the granannular briquet furnace should be about the same as in the Fulton furnace.

The laboratory furnace that has been developed would necessarily be changed in many respects in enlarging it to commercial size. Various possible forms for a commercial furnace suggest themselves, but as they have not been worked out in detail they will not be discussed here. It seems that there should be no difficulty in designing a large furnace so that it could be charged from a hopper, or by crane, and discharged with a minimum of labor. The condenser for a furnace of large capacity would, of course, be entirely different; the same type of condenser that is used for the Fulton furnace should be satisfactory.

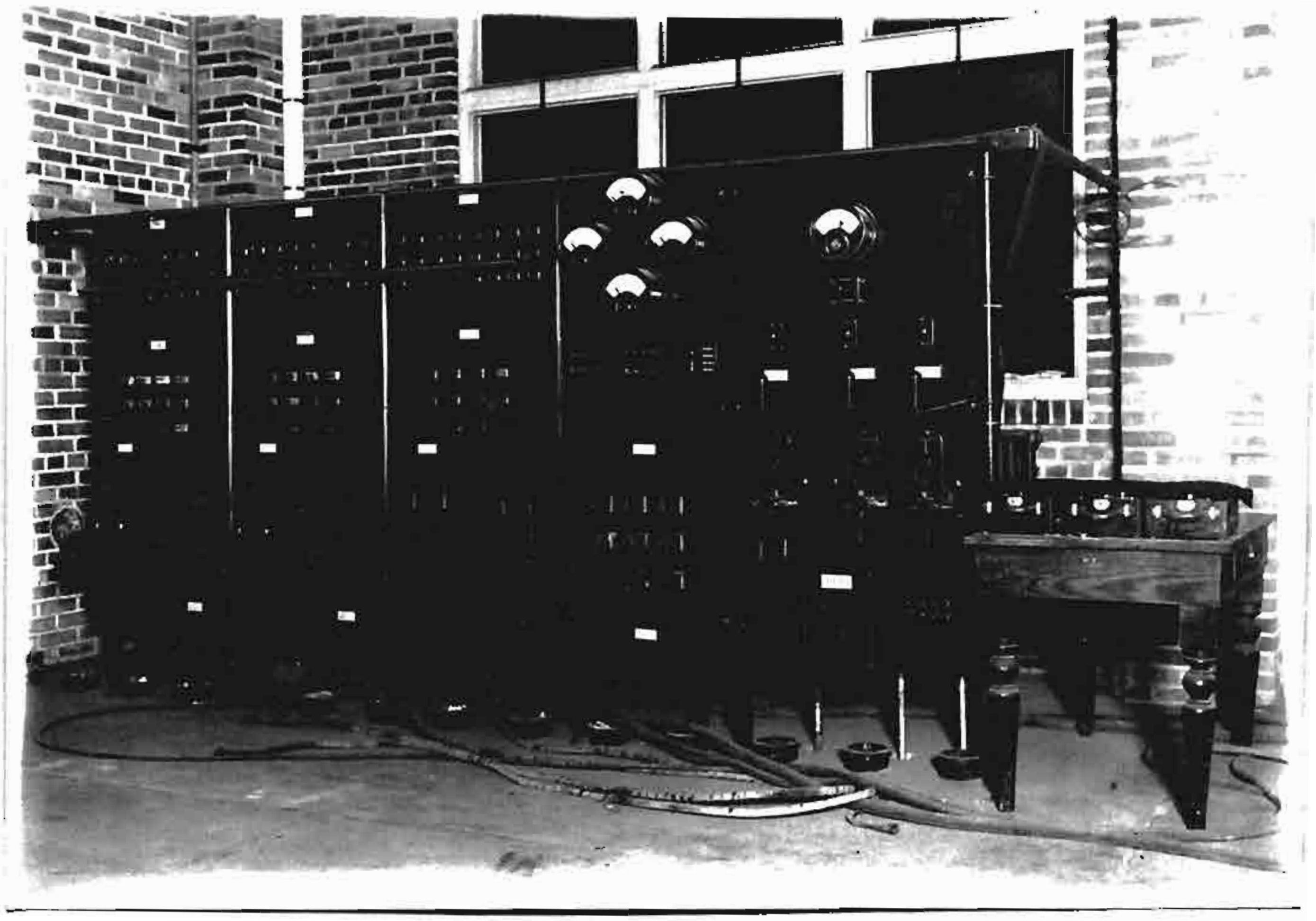

Figure 7. Switch Board for Controlling Current to the Furnaces.