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ELECTROTHERMIC DRY DISTILLATION OF ZINC.

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-By-

Ira Nathan Goff.

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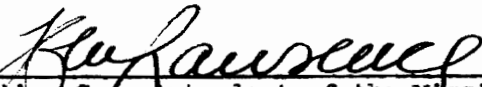
T H E S I S

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
D E G R E E O F
MASTER OF SCIENCE IN METALLURGY.

Rolla, Missouri,

1 9 2 6.

Approved:


Acting Superintendent of the Mississippi
Valley Experiment Station of the U. S.
Bureau of Mines.

Thesis: Electrothermic dry distilla-
tion of zinc. Goff. 1926.

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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 Master of Science in Metallurgy. It describes work carried on at the Mississippi Valley Experiment Station of the United States Bureau of Mines, Department of Commerce, cooperating with the School of Mines and Metallurgy of the University of Missouri, which had for its purpose the study of "Electrothermic Dry Distillation of Zinc" in order to perfect a process that would be suitable to conditions in the United States.

Acknowledgments are due Dr. Charles H. Fulton, Director of the School of Mines and Metallurgy, whose process was used; Mr. H. M. Lawrence, Acting Superintendent of the Mississippi Valley Experiment Station of the United States Bureau of Mines, for his advice and supervision; Mr. William Kahlbaum, Research Metallurgist of the Missouri School of Mines and Metallurgy, with whom the writer was associated in the investigation; and Mr. O. W. Holmes, Chemist of the staff of the State Mining Experiment Station, for the analytical work in connection with the investigation.

ELECTROTHERMIC DRY DISTILLATION OF ZINC.

- By -

Ira Nathan Goff.

INTRODUCTION.

The zinc market of the world is controlled today by the United States and therefore zinc may be called an American metal.¹

¹Notes from Dr. Fink's course in electrochemistry at Columbia University, New York, N. Y.

In considering the production, the three processes for obtaining zinc, in the order of their importance, are: (1) Retort, (2) Electrolytic, and (3) Electrothermic.

Retort Process.

A large percentage of the zinc recovered today is by the retort process, which in principle is simple as it depends upon the reduction of zinc oxide by carbon, carbon monoxide, or both. However, in actual practice this process is complicated. In spite of the fact that progress has been made in late years in the design and thermal efficiency of the zinc smelting furnace, the type of retorts, recovery of zinc, and in treating low grade and complex ores, the chemical and physical properties of zinc are such that this progress seems small compared with the advancement made in the metallurgy of such metals as copper and lead.

Before smelting, zinc ores, if carbonates, are calcined to remove carbon dioxide and water and to disintegrate the ore, while sulphide ores are roasted to remove the sulfur. Sulfur must be eliminated as completely as possible, usually to less than one per cent, and this requires high temperatures for a long period of time, which means additional costs.

In smelting the ore is mixed with approximately 50 per cent of its weight of coke, anthracite, or non-bituminous coal for reduction.¹ This mixture is charged into clay retorts set nearly

¹ O'Harra, B. M., The electrothermic metallurgy of zinc, U. S. Bureau of Mines Bulletin No. 208, Page 1.

horizontal, which are from fifty to sixty inches in length by eight inches inside diameter and hold about one hundred pounds of charge. The limiting factors for the size of the units are the physical properties and the time element for heating the charge.² In regard to

² O'Harra, B. M., Outline of investigation for the development of a granular briquet resistor for the electrothermic distillation furnace.

the physical properties, the zinc retort must possess high tensile strength, resistance to deformation, and resistance to shock at high temperatures. The retort is supported only at the two ends, carries besides its own weight about one hundred pounds of charge distributed uniformly throughout its length, is heated to a maximum temperature of about 1400° C. on the outside which corresponds from 1250° to 1300° C. on the inside, and during charging and discharging is sub-

jected to mechanical shocks and strains. In discharging, a water pipe is inserted at the back of the hot retort and the charge is blown out by the steam which is generated when the water strikes the hot retort. While the retort is still at a yellow heat a cold, wet charge is added.¹ At high temperatures the slags are more or

¹O'Harra, B. M., op. cit. Pages 1 and 2.

less corrosive so the retorts must be able to resist chemical action and penetration of the slags. A non-porous material prevents the escape of zinc vapor and the tendency of the retort to absorb zinc is lessened. Coal, coke or gas is used for heating, depending upon the local conditions. As the zinc volatilizes the gases pass into a condenser where the zinc vapor is condensed to a liquid metal and blue powder. Zinc smelting is an intermittend process, the units are small, the cycle is twenty-four hours, the life of the retorts is from thirty to sixty days, the life of the condensers from eight to fifteen days, while the heating efficiency even in the most modern plants is only about twelve per cent. Zinc losses are heavy, usually at least ten per cent. Careful control of impurities is necessary to prevent excessive corrosion of retort walls. Because of the above facts the retort process cannot handle low grade or complex ores and is also inefficient both in labor and the recovery of zinc.

Electrolytic Process.

The disadvantages of the retort process acted as an incentive in the development of the electrolytic process, which is based upon the usual hydrometallurgical steps up to the time of purifying the zinc sulphate solution.¹ The ore is roasted at about 600° to 650° C.,

¹Prof. Hanley's course in advanced non-ferrous metallurgy at the Missouri School of Mines and Metallurgy, Rolla, Missouri.

manganese dioxide is added, if sufficient is not present in the ore, to aid in the oxidation of the iron. If the temperature in roasting is too high, ferrite of zinc forms, which is much less soluble. After roasting, the zinc in the ore is in the form of the sulphate, oxide, ferrite, and sulphide. The ore is then leached with sulphuric acid solution, iron is precipitated, and gelatinous silica is coagulated and made granular. The impurities in the leached solution in the order of their injurious effects are antimony, cobalt, nickel, and arsenic. The keynote of the purification system is, that in the re-leaching of basic mud by dilute sulphuric acid, the re-solution of impurities does not equal the amount of impurities thrown out at first. Zinc is deposited by electrolysis using pure lead anodes and aluminum cathodes.² One of the more important factors contributing

²Liddell, D. M., Handbook of Chemical Engineering, Vol. 2, Page 801; McGraw-Hill Book Co., New York City, 1922.

to the success of this process in recent years is that flotation pro-

cesses now give a relatively clean high grade ore, while previously work had been done with low grade impure run of mine ore.

The essential conditions for successful electrolytic deposition of zinc are: High degree of purity of the electrolyte, anodes, and cathodes. Costs for the deposition of the zinc in addition to the roasting, leaching, purifying, melting, etc., make the electrolytic process expensive. These costs must be offset by superior advantages in other respects, such as purer product, higher extraction of zinc, and the recovery of other metals in the ore, especially silver. Extraction of zinc by electrolysis even though the smelting method is crude can only compete with the retort process when ore and power are cheap.¹

¹O'Harra, B. M., op. cit., Pages 2-32.

Electrothermic Process.

The first electric furnace for the distillation of zinc ore was patented by Cowles Brothers in 1885. However, other uses for this furnace offered a more promising field so nothing of importance was accomplished along this line until about 1890. Since that time there have been numerous experiments with various types of electric furnaces for the production of zinc. The more important of these are: De Laval's Cyclone Furnace, Casoretti and Bertani Furnace, experiments by Stansfield, Salgues Furnace, Johnson's Process, Imbert-Thompson-Fitzgerald Process, Thierry Furnace, The Taylor Shaft Furnace, Cote and Pierron Process, Gin Induction Furnace, Snyder Furnaces, Pouvrier

Furnaces, The Queneau Furnace, electrothermic zinc smelting by the Canadian Government, experiments at McGill, experiments at Nelson, British Columbia, Peterson's experiments, Fulton's electrothermic dry distillation of zinc, The Nathusius Furnace, the zinc plant of the Elektrometallurgische Werke, Horrem, Germany,¹ Bain's continu-

¹O'Harra, B. M., op. cit., Pages 33-56.

ous electrothermic furnace for complex ores,² and E. G. T. Gustafsson³

²Bain, T. M., Jr., Continuous electrothermic furnace for complex ores, E&MJ-P, Vol. 119, Pages 244-6, 1925.

³British Patent No. 215,004, April 25, 1923.

and H. C. Flodin Process. Other miscellaneous processes and furnaces have received little recognition.

Heat can be produced in the electric furnace without the necessity of blowing air into the charge so the atmosphere can be made reducing and the volume of gases leaving the furnace should be about the same as that leaving the retorts in the usual retort process. Due to the fact that heat is produced, electrically, in the midst of the charge the furnace may be made of any convenient size; this reduces the labor costs, and the furnace walls may be made more permanent as the heat is not transferred through these wall; consequently another source of expense will be eliminated.⁴

⁴Stansfield, A., The electric furnace, page 310; McGraw-Hill Book Co., New York, N. Y., 1914.

Electrosmelting of zinc up to the present time appears to have reached a commercial success only in Scandinavia and this may be due fundamentally to the very cheap power rather than metallurgical superiority. There are large plants in Scandinavia at Trollhattan, Sweden, Sarpsborg, and Glomfjord, Norway besides a few smaller plants.¹

¹Liddell, D. M., op. cit., Page 800.

In comparing the three processes, assuming practically the same existing conditions, the electrothermic process may be said to have the advantage over the retort process in reduction fuel, labor, metal recovery, ability to treat low grade and complex ores, first cost of plant and cost of retorts and condensers, while the advantages over the electrolytic process may be considered to be in power, labor, metal recovery, first cost of plant, and its adaptability to small scale operation. However, each of these three processes has its own particular field, influenced largely by local conditions.

THE FULTON ELECTROTHERMIC DRY DISTILLATION PROCESS.

Among the various processes and types of electric furnaces for extracting zinc from its ores, one that offers excellent possibilities of commercial success, especially in America, is the Fulton electrothermic dry distillation process. This process is an economical user of power, recovers a high percentage of zinc in the form of liquid metal rather than blue powder, can readily treat low or high grade and complex ores, and the resulting residue contains an increased percentage of other metals that may be in the ore, which can be easily treated for their recovery.

The roasted zinc concentrate is thoroughly mixed with about seventy per cent of its weight of 12-hour by-product coke crushed to pass through a 10 mesh screen, and twenty per cent of a melted coal tar pitch of high fixed carbon content, or material of a similar nature to act as a binder. This mixture is made up into briquets in a hydraulic press at a pressure of about 1000 pounds. The briquets are then heated in a baking oven at a temperature of about 500° C. for a few hours to drive off the volatile hydrocarbons from the pitch. The coke base left unites the by-product coke and the ore particles into a coherent mass, which is electrically conductive. The important features of these briquets are that they retain their strength, original form, and volume during and after distillation of the zinc, while the intimate mixture of coke and ore aids in a rapid and complete reduction of the zinc without forming a slag, which will corrode the

refractory linings. The spent briquets are practically high ash coke, since they contain the unconsumed carbon of the charge and the residues from the ore. The amount of ash varies with the character of the ore. The residue may be re-crushed and again mixed with fresh ore for the manufacture of briquets, thus concentrating the amount of lead, copper, and noble metals that may be present and leaving the second residue in an excellent condition for the recovery of these metals in the blast furnace.

In 1914 experimental work on the Fulton process¹ was

¹Fulton, C. H., Trans. AIME (1921), Vol. 64, pp. 188-226.

started at Cleveland, Ohio, and in 1916 a plant was erected at East St. Louis, Illinois, to work out details on a commercial scale. This plant was in operation until January, 1918 when operations were suspended due to conditions created by the war, in spite of its promising commercial possibilities.

Cylindrical briquets 9.25 inches in diameter by 21 inches long, weighing approximately 90 pounds, and containing 50 pounds of ore were made by mixing the ore and coke, then heating and adding melted coal tar residue and mixing in a pug mill. This mixture was made into briquets in a hydraulic press with a pressure from 500 to 1000 pounds with the temperature of the mold from 75° C. to 90° C.

It was found that the strength of the briquets after the extraction of the zinc was little effected by the size of the ore particles as practically the same results were obtained with ore that

passed through a 200 mesh screen as that which passed a 10 mesh screen with 86 per cent remaining on a 35 mesh screen. However, the strength of the briquets was affected by the size of the coke particles; a fine coke making a much stronger briquet and requiring more of the tar as a binder, thus increasing the cost. Coke which passed a 10 mesh screen was found to be satisfactory in regard to strength and the amount of pitch required.

All the reducible oxides of the ore must be reduced and an amount of coke left sufficient to give the briquet stability and electrical conductivity; the coke used amounted to from 60 to 85 per cent of the ore. If an infusible residue is left the briquet has more strength after the zinc is extracted than when fused globules are formed. The best results were obtained by adding 10 to 15 per cent of pitch with a melting point of 170° C. to 200° C. and a fixed carbon content of 55 to 60 per cent. Experiments using coking coal to replace a part of the pitch gave less satisfactory results.

In baking, twenty briquets were set on end in a sheet steel cylinder, which was placed in a car, oxidation being prevented by filling the space in the cylinder with crushed coke. The car was then run inside a baking oven where it was left for six or eight hours with a temperature of 450° to 500° C. during the last few hours. Up to 300° C. the briquets passed through a soft stage, increasing in strength as the temperature became higher.

At room temperature the raw briquets had an electrical resistance of 26 to 30 ohms per cubic inch, which gradually decreased until from 450° to 500° C. it fell off sharply to 0.6 to 0.7 ohm, while at 900° to 1100° C. it was only 0.015 to 0.04 ohm per cubic inch.

The charge in the furnace at East St. Louis consisted of 36 briquets, 12 columns of three each, arranged in a circle, and connected to a 3-phase circuit, four columns to a circuit connected in a delta formation. One charge contained about 1,700 pounds of ore and required six hours for distillation. As two hours were necessary for charging and discharging, three charges were run in a 24-hour day, which gave a capacity of 5,100 pounds of ore used.

The power consumption for a number of runs at East St. Louis varied from 1237 to 2270 kw. h. per ton of ore, depending upon the kind of ore treated, and per cent of zinc distilled from the charge. One thousand, three hundred seventy-two kw. h. was calculated as the theoretical power consumption for a sixty per cent ore in the form of briquets, not including radiation losses. Four hundred twenty-one kw. h. was required to preheat the charge to 920° C. Considering the energy in the carbon monoxide produced in extracting the zinc, this would amount to 154 kw. h. and the cooling of the spent charges from 1200° to 25° C. would be 378 kw. h., part of which may be utilized.

In the experimental plant in East St. Louis the only difficulty experienced with the condenser was the disintegration of the fire brick lining in the region where the temperature was about 500° C.¹

¹Fulton, C. H., op. cit. and Trans. AIME (1919), Vol. 60, p. 298.

This appeared to be caused by the deposition of carbon on specks of iron oxide in the lining; the latter were reduced and carbon deposited at these places. This deposition of carbon came from the decomposition of carbon monoxide— $2\text{CO} \longrightarrow \text{CO}_2 + \text{C}$.

The Fulton process is radically different from other electrothermic methods of obtaining zinc from ores, for it is not a smelting but rather a distillation process. Most electrothermic processes mix the ore with a certain amount of reduction fuel and smelt by heating with an electric current, usually a buried arc type. This heating does not give uniform temperatures so carbon dioxide and water vapor are present with zinc vapor and carbon monoxide, which makes condensation to a liquid metal difficult and produces a large amount of blue powder. Slag is also formed as the residue of the ore smelts and this slag may not only attack the refractory linings but contain an appreciable amount of zinc. In Scandinavia, where electrothermic processes are in commercial operation, no attempts are made to condense zinc as liquid metal in the primary smelting; the blue powder formed is re-treated to produce metallic zinc.

The following are the chief advantages presented by the electrothermic dry distillation of zinc from ores:

1. Mechanically operated large units.
2. Low labor costs.
3. Nearly complete extraction of zinc.
4. High recovery of zinc with a large per cent of this as liquid metal.
5. Economical user of power—less than the electrolytic or other electrothermic processes.
6. More complex ores may be treated.
7. Low first cost—less than the retort or electrolytic processes, if electric power is purchased.
8. Simplicity—simpler than the electrolytic or retort processes.
9. No regular consumption of fire clay.
10. Small units may be built wherever power is cheap.
11. Low operating costs—less than the electrolytic or retort processes.
12. It may be used in conjunction with the retort process for re-treating blue powder.

PREVIOUS EXPERIMENTAL WORK AT ROLLA.

The following experimental work along the line of electrothermic metallurgy of zinc for 1923-24 and 1924-25 was carried on at the Mississippi Valley Experiment Station, United States Department of Commerce, Bureau of Mines, Rolla, Missouri:

In order that zinc ores could be smelted more efficiently and cheaply than by the retort process, and that complex ores might be treated economically, a study was made of the electrothermic dry distillation of zinc ores to perfect a process suitable for conditions in the United States.¹ This study included an investigation of the ef-

¹ O'Harra, B. M., and Wheeler, E. S., Experiments on the distillation of zinc from complex zinc-lead-silver ores. Bull. Missouri School of Mines and Metallurgy, 1923, Vol. 6, No. 4, Technical Series.

fect of temperature and time of distillation on the relative rates of volatilizing zinc, lead, and silver from briquets of complex zinc-lead-silver ores. The purpose was to determine results that might occur in the treatment of complex ores by an electrothermic dry distillation process. These results also applied to briquets distilled in a retort furnace, and had some bearing on the results occurring in the ordinary retort process.

In 1923-24 a complete experimental electrothermic dry distillation zinc smelting plant with a capacity of a few hundred pounds of ores was started, using the Fulton process.

In 1924-25 the development of an electrothermic dry distillation zinc furnace which used as a resistor small briquets in bulk, made

from zinc ore, was completed with satisfactory results.

The complete experimental dry distillation zinc plant using the Fulton process, started in 1923, was finished in the fall of 1925. This consists of equipment for crushing ore and coke, a hydraulic press for making the briquets, an oven for baking the raw briquets using the waste heat in the spent charge, and a three retort distillation plant with a common condenser. This plant is of a semi-commercial size with a capacity of a few hundred pounds of zinc per day, and is designed to be a model or demonstration plant.

Detailed Statement of Object of Investigation.

1. To study the electrothermic distillation of zinc ores with the purpose of perfecting a process that will be suited to conditions in the United States, and which will enable zinc ores to be smelted more efficiently and cheaply than by the present retort process; also making possible the economical treatment of complex ores which cannot be commercially worked by present processes.

2. To make a practical demonstration of the Fulton dry distillation process for the production of zinc from ores, using the new experimental electrothermic dry distillation zinc plant as a model unit.

Detailed Description of Process.

Materials Used:

Ore: Roasted Joplin ore containing 69 to 70 per cent zinc and one per cent of sulfur was used in this investigation. The ore was crushed to pass through a 10 mesh screen with a minimum of fines.

Coke: The coke consisted of ordinary 12-hour by-product coke (fixed carbon 81.07 per cent and ash 15.07 per cent), which was crushed to pass through a 10 mesh screen with a small amount of fines.

Pitch: The character of a coal tar pitch used as a binder in the briquets must be carefully studied, especially noting such properties as the melting point, type of coke left after the distillation of the volatile hydrocarbons, and the percentage of coke remaining after distillation. In general a high melting point pitch, between 170 and 200° C., leaving a high percentage of good firm coke between 55 and 60 per cent, gives the best results. The high melting point is essential as the briquet is molded hot and taken from the mold while warm and weak. Thus the lower the melting point of the pitch, the weaker will be the briquet, while the higher the melting point the stronger it will be and the more handling it will stand when taken from the mold. Two functions are performed by the pitch in the briquet: It acts as a binder in its original condition for the raw briquets and, after baking, the residual coke binds together permanently the particles of ore and charge coke. For the latter there must be a rather large amount of fine residual coke. The pitch, which appeared to be most suitable for this purpose, was Barrett Manufacturing Company's "Graigola", which has the following analysis:

Fixed carbon - 52.70 per cent
Free carbon - 37.18 per cent
Ash - 0.38 per cent
Melting point - 298^o F.

Manufacture of Briquets:

One hundred parts of roasted Joplin zinc ore and seventy parts of coke were mixed in a revolving mixer. This material was then transferred to an electrically heated hot plate and twenty-five parts of pitch were added. The entire mass was then thoroughly mixed; the temperature maintained just above the melting point of the pitch. Four and fourteen-hundredths pounds were put into the mold of the press, and the briquets formed at a pressure of 1000 to 1200 pounds per square inch. The size of the briquets was three inches by three inches by six and three-fourths inches. In the pressed briquets a film of pitch surrounds the ore and coke particles, forming a coherent mass that will have the same form and practically the same volume after extraction of the zinc. To obtain the desired stability and electrical conductivity there must be sufficient coke besides reducing all the reducible oxides present.

Baking Briquets:

The object of baking the briquets was to drive off the volatile hydrocarbons and to coke the pitch, which makes them a conductor for the current. Charges of briquets were placed in the baking oven, Figure 3, which was held at a temperature of 500^o to 600^o C. and baked for six to seven hours; a neutral or reducing atmosphere was maintained so the briquets would not oxidize.

Apparatus.

Hot Plates:

Figure 1 shows the electrically heated hot plates upon which the ore, coke, and pitch were mixed for making the briquets. A horizontal layer of granular graphite acted as a resistor for the current which supplied the heat. The surface of the hot plates consisted of half-inch carbon plates placed above the resistor and insulated from it.

Hydraulic Press:

Figure 2 shows the hydraulic press used for making all briquets. The operating lever, plunger, movable mold guided by four vertical columns, and the sliding head-block, against which the mixture is pressed, can be seen in the picture. The pressure used was from 1000 to 1200 pounds per square inch.

Baking Oven:

Figures 3 and 4 show the baking oven from two different angles—Figure 3 with a green charge and Figure 4 with spent charges. The oven is six feet by six feet by twenty-seven inches, and is supported by four posts of two and one-half inch pipe. A course of Sil-o-cel brick on the outside acts as an insulator, while fire brick on the inside, set end-wise, forms four rectangular chambers, eighteen inches by eighteen inches by twenty-three inches in which the raw briquets or spent charges are placed. The four chambers are divided into two units of two chambers each with a large opening near the top connecting the two chambers of each unit which ensures fairly uniform temperatures, while a fire brick wall with a small opening

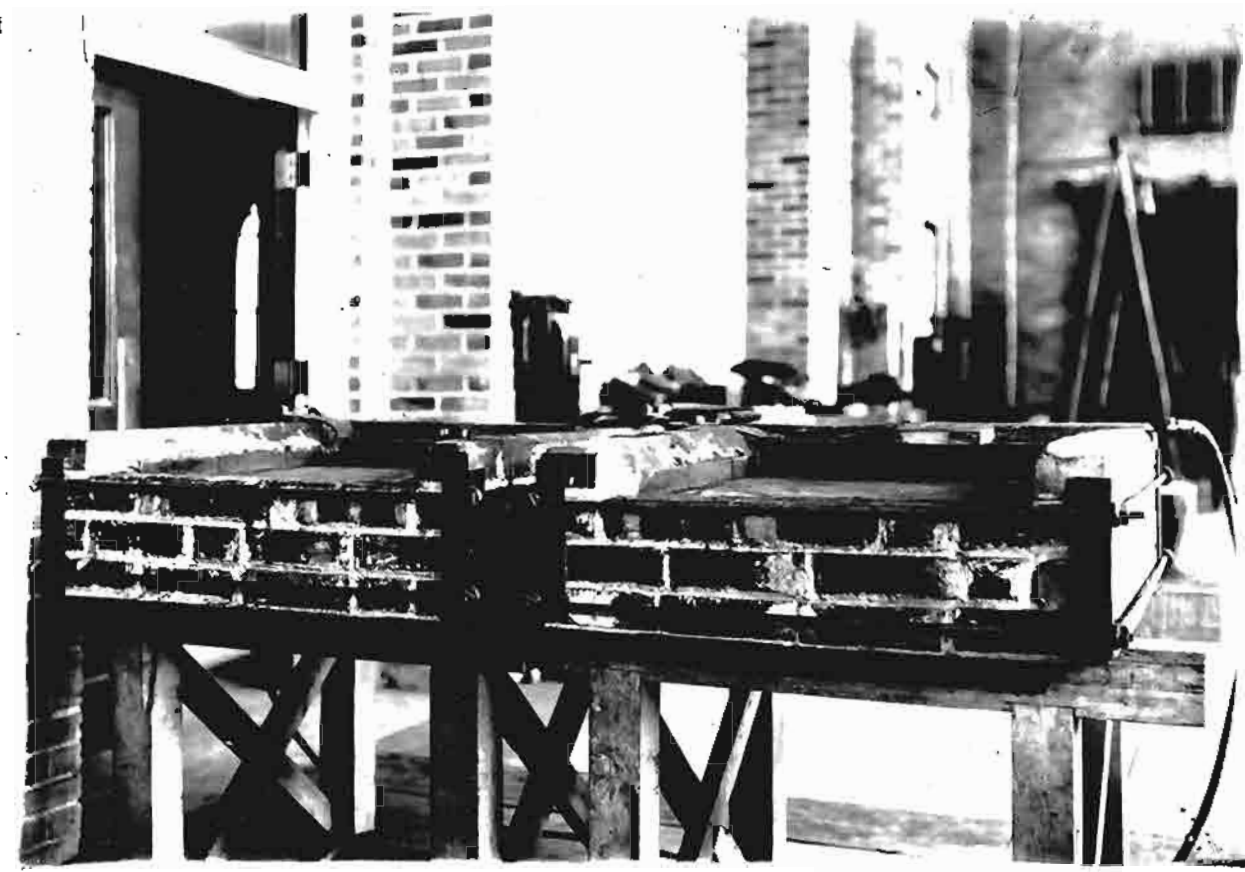


Figure 1.

Electrically Heated Hot Plates.

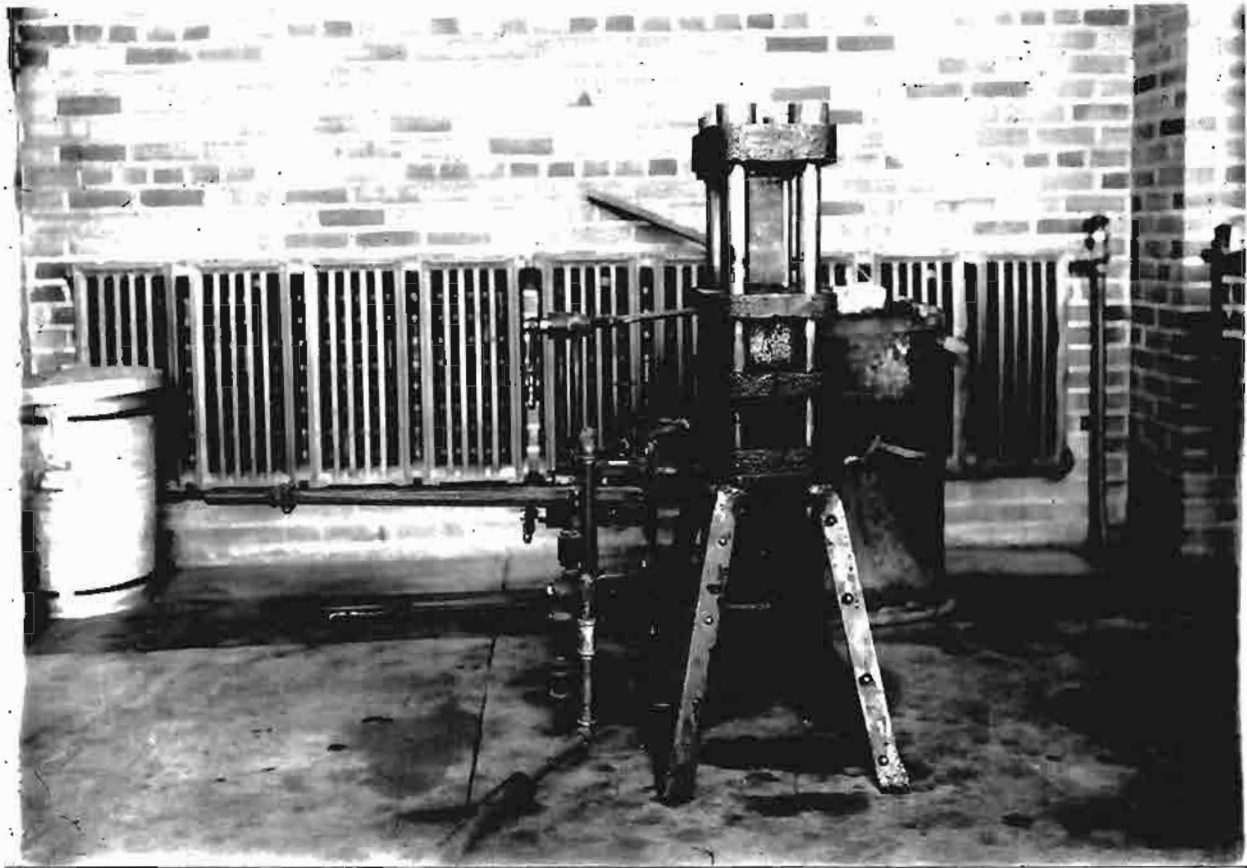


Figure 2.
Hydraulic Briquet Press.



Figure 3.

Baking Oven With A Raw Charge.

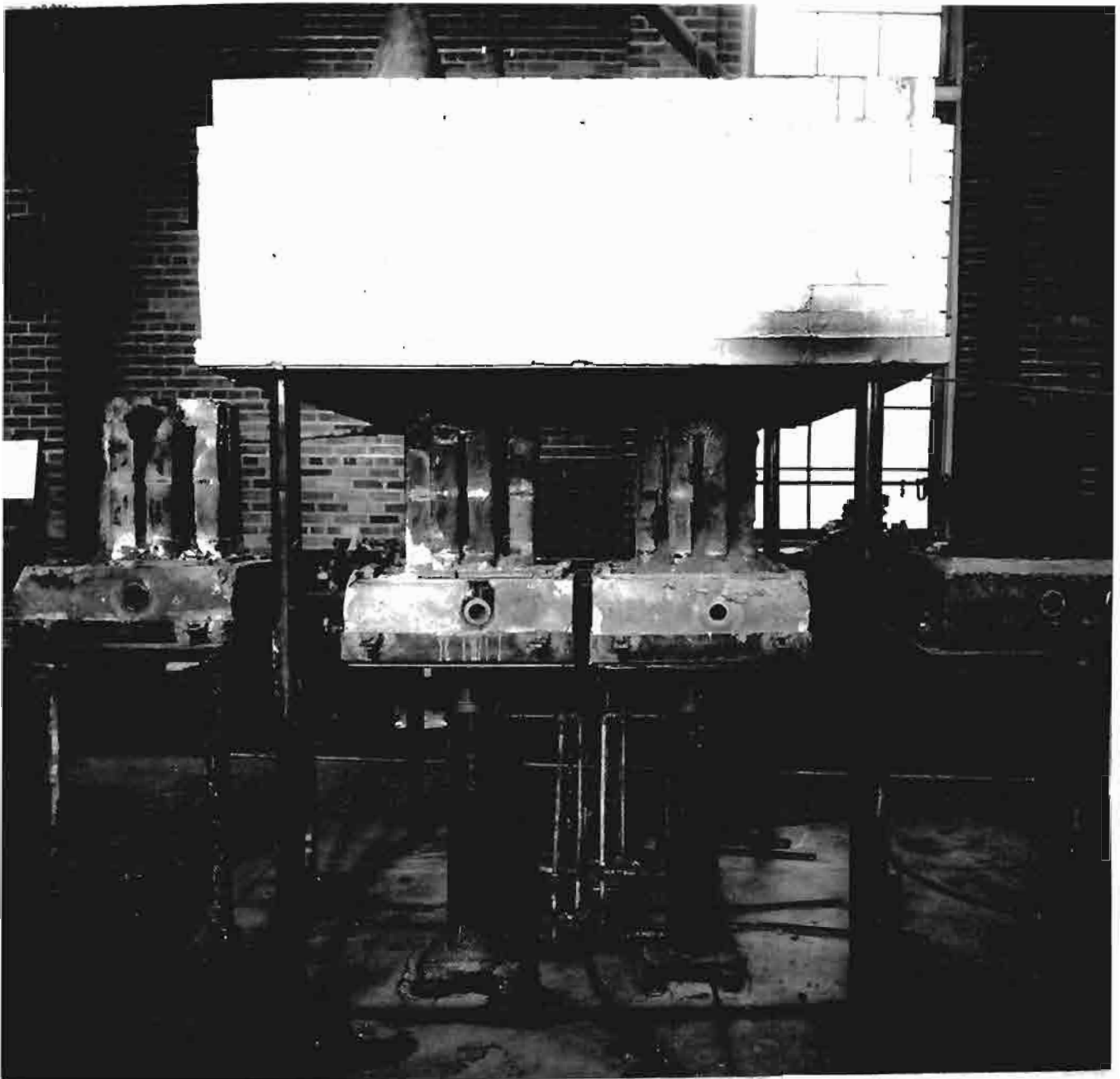


Figure 4.

Baking Oven with Spent Charges.

separates the units. A two and one-half inch pipe connects to a hole in the top of the baking oven and carries the fumes out of the building. Clearance between the briquets and the lining of the chamber is about two inches, while the distance between the columns of briquets is about three-fourths of an inch. Four hydraulic lifts, which raise twenty-one inches, with base plates twenty-four inches by twenty-four inches and set thirty-nine and one-half inches from the floor, lower or raise the charges into the chambers. Guides of one inch angle iron control the truck location in charging or discharging. Temperatures are indicated by two base metal (chromel-alumel) thermocouples, one set in the top of each unit and connected to a Leeds and Northrup recording potentiometer.

Base Block:

Figure 5 shows the base block. This consists of a solid block of refractory material, 24 by 24 by 8 inches, with places on the top for six briquets and holes connecting with a tap on one side of the block to carry off any liquid zinc that might form. The graphite electrode holders are two and three-fourths inches in diameter, arranged to form a delta connection in a 3-phase circuit. Each phase is connected to the graphite electrode by a two-inch graphite rod seven inches long tapered for two and one-half inches from a small diameter of one and one-half inches to a larger diameter of two inches. The above plug is held in a water cooled electrode holder as designed for this service by R. O. Jackson.¹

¹U. S. Patent No. 1,242,554.



Figure 5.

Base Block on Charging Truck.

Charging Truck:

Figure 5 shows a charging truck, which is made of 2 by 2 by 1/4 inch angle iron, three-fourths inch and one and one-quarter inch pipe. The top is twenty-seven inches by twenty-nine inches and the frame stands forty-one inches above the floor. This truck is quite suitable for transferring charges between the baking oven and retorts.

Retorts:

Figure 6 shows a view of the three retorts. Each retort consists of one-eighth inch cylindrical steel shell, thirty-eight inches in diameter and thirty-two inches high, with a one and one-half inch angle iron at the bottom, bolted to the shell. One course of high grade fire brick, set end-wise against the shell, forms a circular opening, eighteen and one-half inches in diameter by twenty-two inches high, for the charges. The top is insulated with Sil-o-cel brick covered with loose Sil-o-cel, an opening being left for a thermocouple. The clearance between the briquets and the lining of the retort is about two inches. A clay mixture consisting of three parts clay and two parts of powdered coke on the base plate forms a tight joint. About eight inches from the top of each retort is a short welded connection to the common condenser, consisting of a nine inch pipe, fourteen inches in length. The distance between each retort and the common condenser is four inches. These connections are insulated on both the in and outside, leaving a hole of about two and one-half inches for the vapors to pass from the retorts to the condenser; a graphite stop-cock controls the flow of gases. The retorts

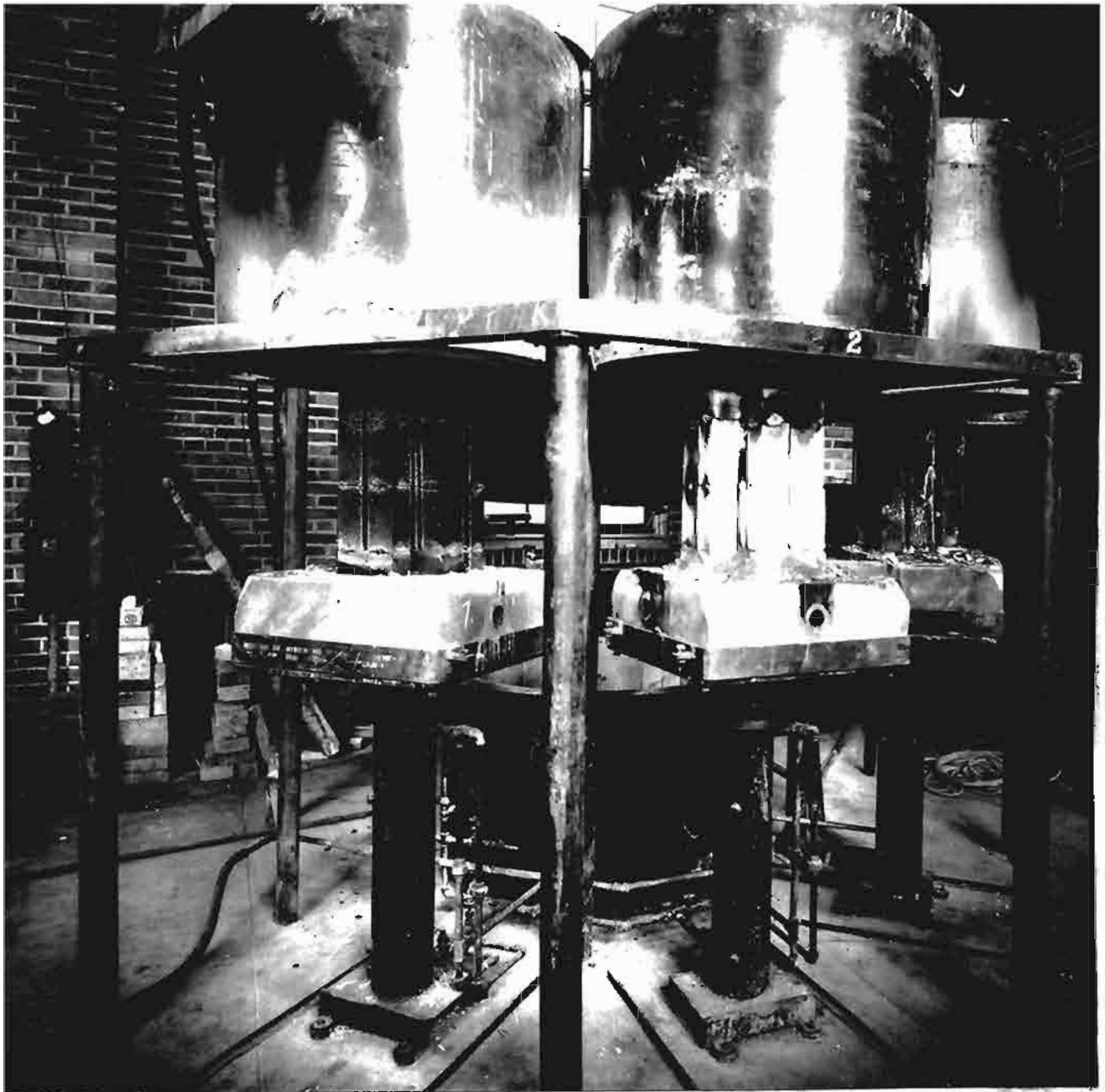


Figure 6.

Retorts Showing One Baked and Two Spent Charges.

rest upon two and one-half inch by three inch angle irons placed seventy inches from the floor and supported by eight posts of three-inch pipe. Three hydraulic lifts of the same type and size as those used in the baking oven, lower or raise the charges in place, while one-inch angle irons for tracks, guide the trucks when placing the charges in position.

The Condenser:

Figure 7 shows a view of the condenser and retorts. The condenser is made of the same sheet steel (one-eighth inch) stock as the retort shell with a diameter of forty-nine inches by forty-four and one-half inches high. The shell consists of two pieces bolted together, one thirty inches high and the other fourteen inches, while the bottom is welded to the edges of the shell. About seven inches from the bottom is a tap hole, five and one-half inches by four and one-half inches. Two courses of two and one-half inch Sil-o-cel brick act as insulation and one inside course of, two and one-half inch, pure silica brick practically free from iron, serves as a lining. One of the essentials of the condenser lining is a refractory material free from iron as the presence of iron oxide particles leads to its destruction by carbon deposition from carbon monoxide gas. This reaction is very active at about 500^o C., a temperature which exists in some regions of the condenser. The inside diameter is thirty-five inches and the depth is twenty-two inches with a number of two and one-half inch partitions acting as baffles as shown in Figure 8.



Figure 7.

Condenser and Retorts.

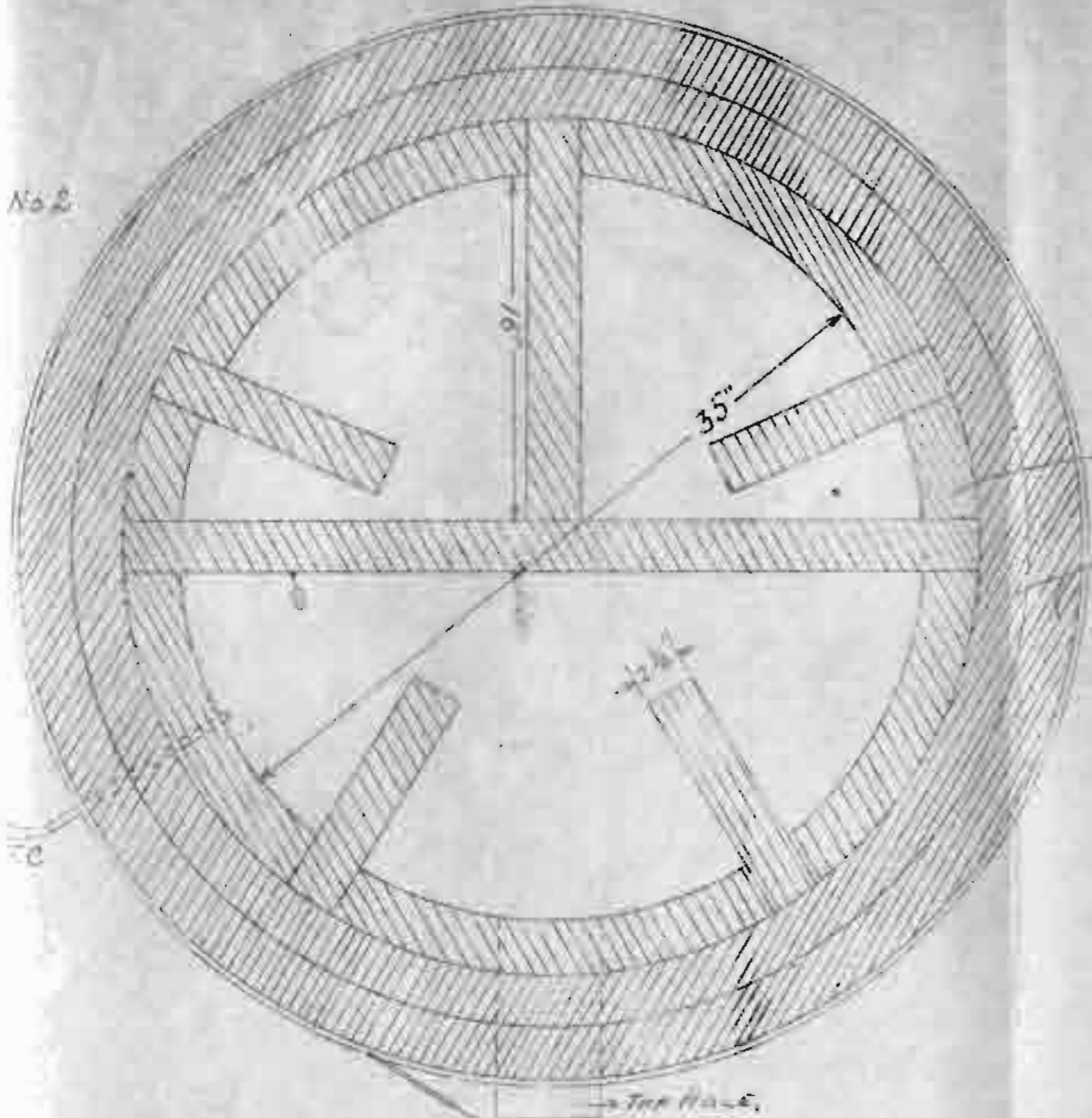
In Figure 7 the condenser is shown in position suspended from a double A-frame constructed from two and one-half inch pipe, supported by two adjustable pipe hangers on a two and one-half inch horizontal pipe across the two A-frames. The bottom of the condenser is fifty-three inches above the floor. The drawing on the right in Figure 8 shows only one, two and one-half inch course of Sil-o-cel brick with an air space and an inner steel shell, while in the drawing on the left is shown the inner steel shell and air space replaced by a two and one-half inch course of Sil-o-cel brick. The arrangement of the three retorts allows a continuous flow of zinc vapor to the condenser and with this combination the volume of the condenser should be not less than twice the volume of a retort.¹

¹Fulton, C. H., A proposed plant for the electrothermic dry distillation of zinc ores, E&MJ, (1922), Vol. 114, pp. 8-14.

Temperatures were shown by two platinum-platinum-rhodium thermocouples, one set in the top and one in the side of the condenser near the bottom. These thermocouples were connected to a Leeds and Northrup recording potentiometer.

The charge is shown in Figure 3, which consists of thirty-six briquets, or six columns arranged in a rectangle and connected to a three-phase circuit, four columns to a circuit in a delta formation. The bottom course consists of six briquets placed horizontally as shown in Figure 9, the next two courses of twelve briquets each are placed vertically on the bottom course, while the top layer of six briquets is arranged horizontally in the opposite order to the bottom row. A charge weighs about one hundred forty-nine pounds and requires from six to seven hours for distillation. The average length of current travel is 80.25 inches per phase.



No. 2



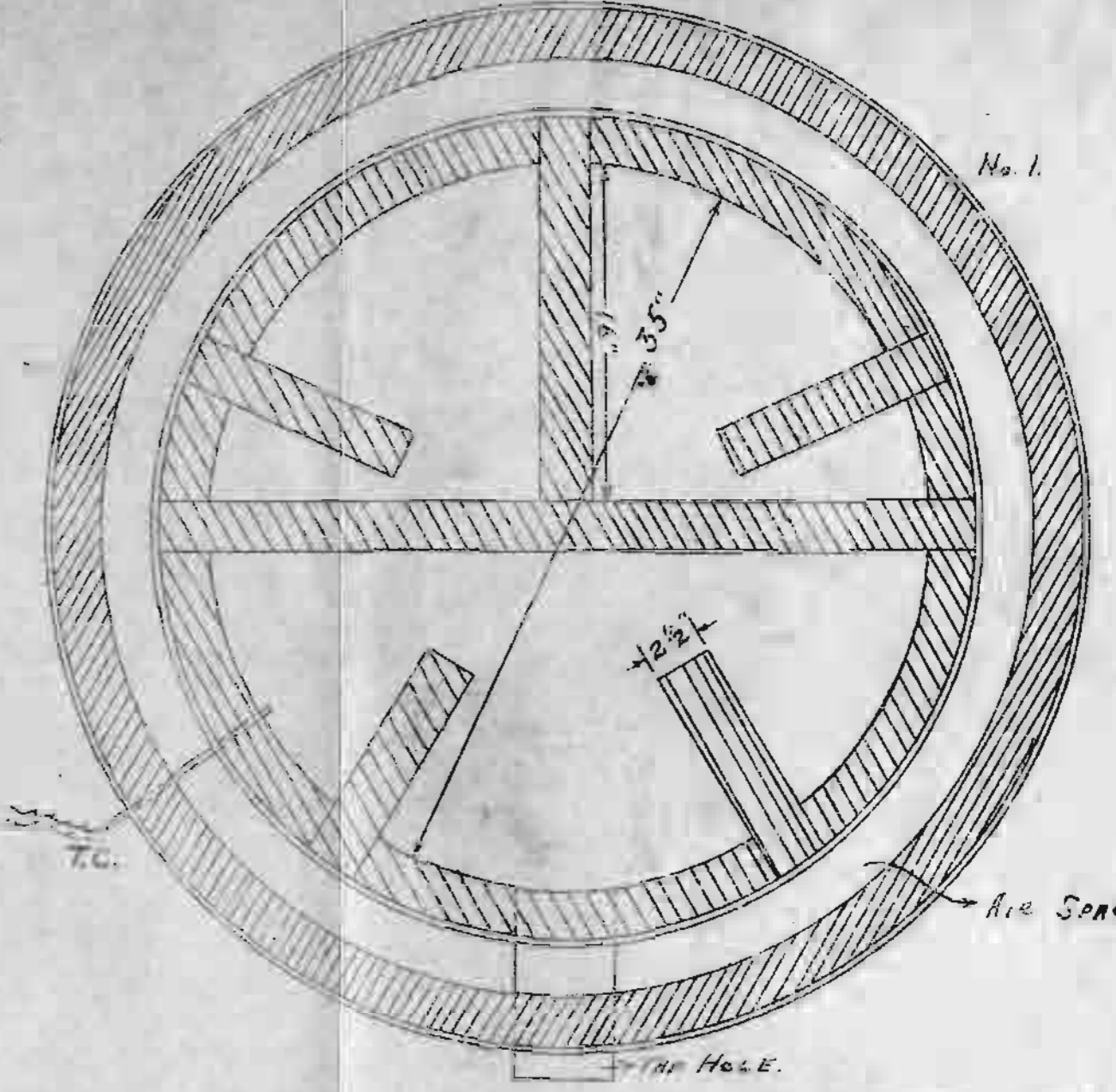
TOP VIEW WITH COVER REMOVED

2 1/2" SILICA BRICK
 3 COURSES OF 2 1/2" INSULATING BRICK

SCALE
 1/8" = 1".

 SILICA BRICK
 INSULATING BRICK

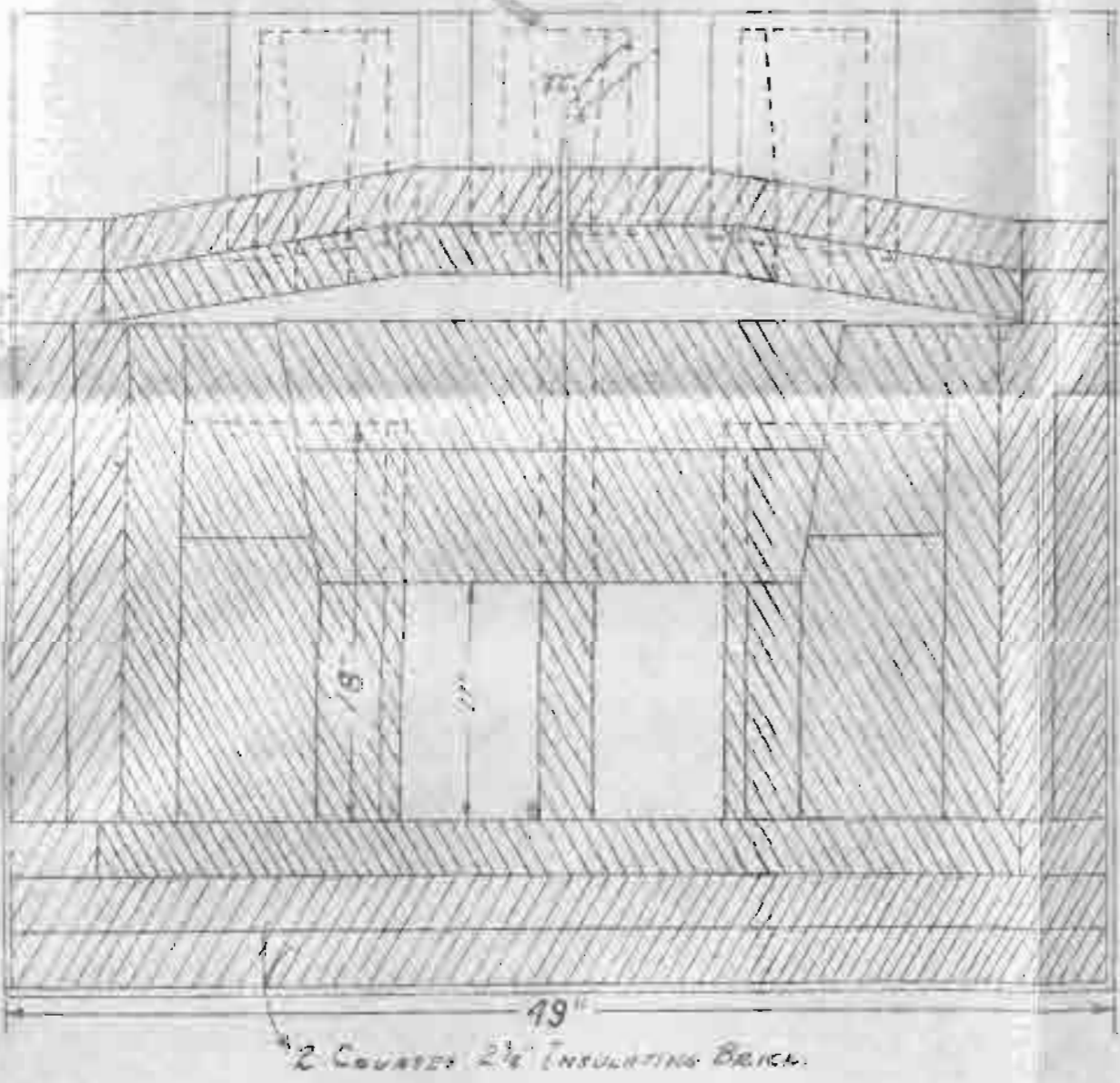
No. 1



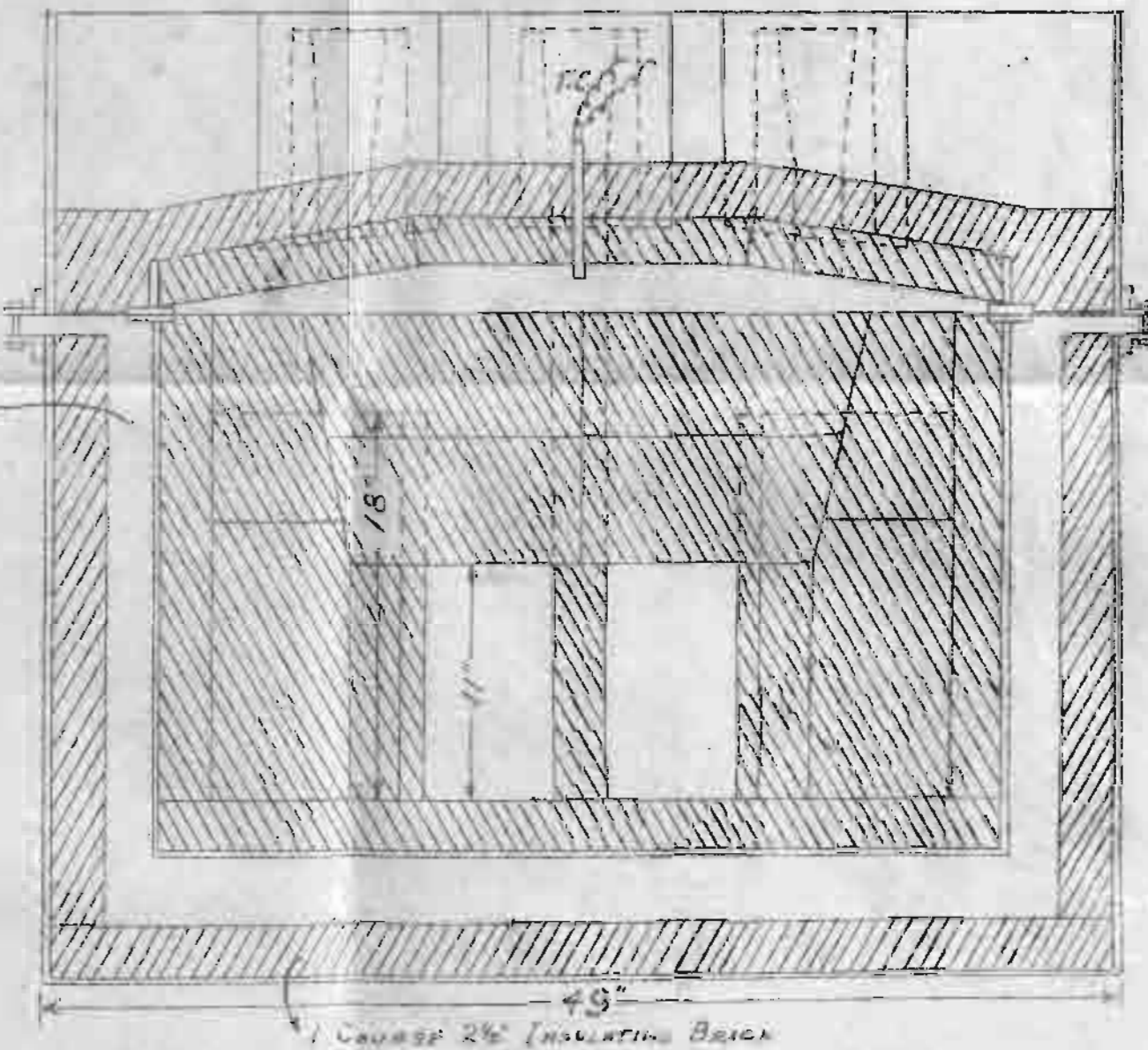
T.C.

Air SPACE.

TOP HOLE.



49"
 2 COURSES 2 1/2" INSULATING BRICK.

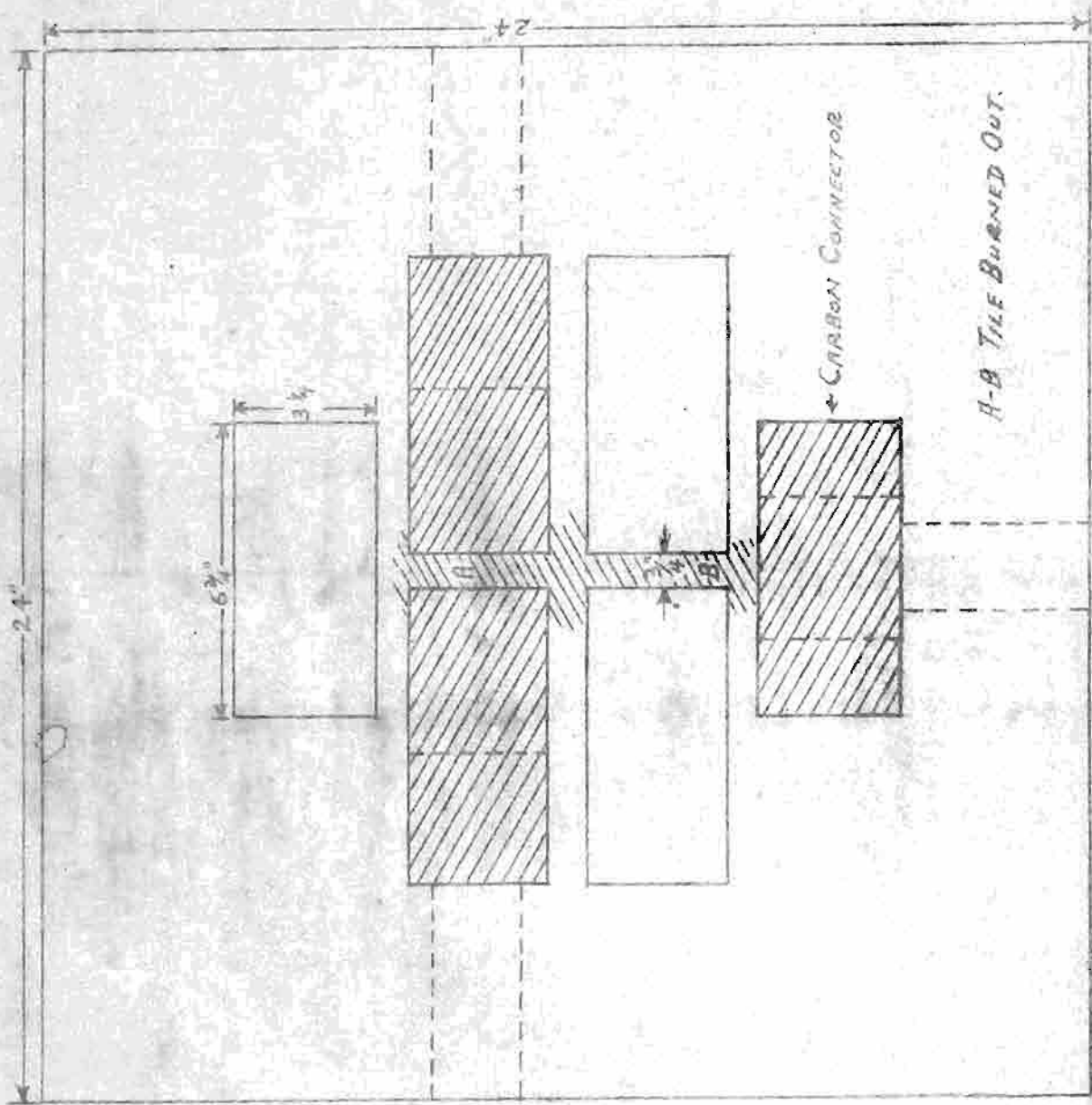


Air SPACE

49"
 1 COURSE 2 1/2" INSULATING BRICK

Figure 8.

Top Views of the Condenser.



TOP VIEW OF TILE BASE FOR ELECTROTHERMIC FURNACE

SCALE 1/4" = 1"

Figure 9.

Top View of Base for Electrothermic Furnace.

Description of First Run.

The first trial run of the new experimental electrothermic dry distillation zinc smelting plant, using the Fulton process, was started on November 28, 1925 with the heating of the retorts, condenser, and baking oven by gas to an operating temperature.

On the afternoon of November 29th, the temperature of the baking oven reached 700° C. so the first charge of raw briquets, consisting of 148.5 pounds, was run into baking oven chamber No. 5 at 5:25 P M and baked until 8:45 A M on the 30th, or fifteen hours and twenty minutes. After baking the charge was placed in No. 1 retort at 9:00 A M. As the temperature of the condenser was still low, pre-heating was continued until about 2:30 P M when it reached 800° C. The current was turned on at 2:40 P M and continued until 9:00 A M on December 1st. Most of the zinc was distilled from this charge, although the lower half of the bottom briquets after distillation contained more zinc, due perhaps to absorption of metal, which had condensed on the cold base.

After distillation the current was shut off and the spent charge was transferred to the baking oven to furnish heat for baking fresh charges. A similar cycle was followed with the other charges treated in this run. Seven charges were distilled; the time of distillation varied from six to twelve hours. As mechanical troubles were interfering with the operation and as the temperature of the condenser was below the melting point of zinc the run was stopped on December 3d, 1925.

A summary of the results for this run are shown in Tables I, II, and III, following:

Table 1.

Analysis of Residue and Condition of Spent Charges.

First Run - December, 1925.

Charge Number	Per cent Zinc				
	No. 1	No. 2	No. 3	No. 4	No. 5
1	0.71	Trace	Trace	9.92	20.50
2	2.14	0.92	2.24	2.45	27.74
3	3.16	2.24	2.75	12.24	37.13
4	0.92	0.51	1.02	1.12	13.46
5	8.63	11.22	23.26	24.48	
6	2.45	1.63	1.73	2.24	22.13
7	2.24	1.02	2.04	1.73	17.54

Condition of Spent Charges.

Charge Number	Condition
1	Badly oxidized; part of charge fell on floor
2	Badly oxidized
3	Slightly oxidized
4	Slightly oxidized
5	Good condition; top briquet crumbled
6	Good condition
7	Good condition

Legend of Sample Numbers.

- No. 1 - Top horizontal briquet
- No. 2 - Upper vertical briquet
- No. 3 - Lower vertical briquet
- No. 4 - Upper half of bottom briquet
- No. 5 - Lower half of bottom briquet

Table II.

Weight in Pounds of Spent Briquets in Charges Nos. 6 and 7.

First Run - December, 1925.

Charge Number	No. 1	No. 2	No. 3	No. 4	No. 5	Loss in Weight
6	9.75	20.0	20.0	4.0	8.50	85.75 pounds or 57.9 per cent
7	10.75	20.5	20.5	4.0	8.00	85.25 pounds or 57.2 per cent

Legend of Sample Numbers

- No. 1 - Top horizontal briquet
- No. 2 - Upper vertical briquet
- No. 3 - Lower vertical briquet
- No. 4 - Upper half of bottom briquet
- No. 5 - Lower half of bottom briquet

Table III.

Summary of Charges Nos. 6 and 7.

First Run - December, 1925.

Charge Number	Original Charge			Residues			Weight lost and zinc distilled			
	Weight in pounds	Percent zinc	Pounds zinc	Weight in pounds	Percent zinc	Pounds zinc	Weight		Zinc	
							Pounds	Percent	Pounds	Percent
6	148	35.9	53.1	62.25	4.43	2.8	85.75	57.9	50.3	94.8
7	149	35.9	53.5	63.75	3.69	2.4	85.25	57.2	51.1	95.5

Residues.

Average assay not including lower section of bottom briquet:

Charge No. 6 - 1.63 per cent zinc

Charge No. 7 - 1.69 per cent zinc.

Average assay including lower section of bottom briquet:

Charge No. 6 - 4.43 per cent zinc

Charge No. 7 - 3.69 per cent zinc.

A Discussion of First Run.

Table I gives the analysis of residues and condition of spent charges showing each charge number, the sample number, legend of sample, and per cent of zinc in each sample. These results are fairly uniform with the exception of charge No. 5. The lower part of the bottom briquet showed a higher percentage of zinc in all cases.

Table II gives the weight in pounds of spent briquets in charges Nos. 6 and 7 for each of the five samples taken per charge, and the loss in weight per charge in pounds and per cent. These results check remarkably well both in pounds and per cent loss.

Table III is a summary of charges Nos. 6 and 7 showing the original weight in pounds, per cent zinc and pounds of zinc. The residues are given in pounds, per cent of zinc, and pounds of zinc, while the weight lost and zinc distilled is shown in pounds and per cent. Charges Nos. 6 and 7 agree closely in original charge, residues, and weight lost and zinc distilled.

A small gas machine, using gasoline, furnished a rich gas which was used in preheating the retorts, condenser, and baking oven. This was a rather lengthy operation and while slow heating of this nature would be less injurious to the refractory linings, it was decided that gas heating was too slow and therefore unsatisfactory for this work.

The baking oven temperature continually decreased, partly due to the loss of heat in transferring the hot spent charges from the retorts to the oven chambers and partly to the radiation losses.

The clay seal at the bottom of the baking oven leaked and allowed air to come in and oxidize the briquets that were being baked. However, the charges of raw briquets were sufficiently baked to stand being transported to the retorts; the baking was completed in the hot retorts.

When placed in the retorts, the charges took the current fairly well. Zinc distilled satisfactorily from the entire charge but some condensed in a metallic state on the cold base of the retorts and was partly absorbed by the bottom briquets, thus causing a large increase in the zinc content of the residues, and occasionally short circuits.

As the electrical transformer equipment permitted only one retort to be operated at a time, the volume of the hot gases generated was not sufficient to maintain the condenser at the required temperature. The condenser, which was preheated to 800° C., steadily decreased to about 350° C. at the top and 240° C. at the bottom, which temperature was maintained nearly constant for the balance of the run. Practically all the zinc vapor condensed in the form of blue powder as the temperatures were largely below the range at which metallic zinc could form. Therefore the connections between the retorts and the condenser and the machined graphite stop-cocks clogged up frequently with blue powder and oxide. Stoppages of this nature caused back pressure in the retorts, which in turn leaked around the bottom, expelling a small amount of metal that had condensed on the base blocks. It was necessary to ream out frequently these graphite cocks and connections between the retorts and condenser.

This was the first time in the electrothermic metallurgy of zinc that carbon top and bottom connectors were not used. The charge was built up of briquets and in this case the briquets used for connectors were the same as those of the rest of the charge. All briquets retained their size and shape during the run, conducted the current well, and the zinc distilled off in a satisfactory manner.

On opening the condenser after the run, and removing the mixture of blue powder and metal, seventy-seven pounds of metal of various sizes and shapes was recovered, and one hundred eight pounds of blue powder, which contained small particles of metallic zinc, was obtained by screening. The metallic zinc was melted and cast into ingots. The lining of the condenser was in excellent condition, some of the bricks were removed and examined and apparently the gases or vapors had no detrimental effects. Between the top sheet and the brick lining around the inlets from the retorts, a small amount of blue powder formed.

The retort linings were in good condition, a light flaky scale that crumbled easily and which was probably zinc oxide formed on the side walls, while a small amount of crusting was noticed around the bottom.

Improvements Made in Equipment Between Runs.

As a number of mechanical defects in the apparatus were evident during the first run, several changes and improvements were made in the equipment before considering another run. The inner steel shell and air space in the condenser was replaced by a course of Sil-o-cel brick as shown in the left hand drawing of Figure 8. An oil burning apparatus was installed in place of the gas burners for preheating the retorts, condenser and baking oven. Holes were cut in the top of the base blocks connecting with a tap on one side, which allowed any liquid zinc that might be formed to be drawn off, thus eliminating a chance of short circuiting. Sheet steel covers seventeen inches in diameter by twenty inches high were made of one-thirty-second inch stock to fit over charges in the baking oven in order to prevent oxidation of the briquets. A half-inch hole in the top of the covers permitted the fumes to escape from whence they were conducted out of the building by a three inch pipe connected to the top of the baking oven. Two lugs on opposite sides near the top of the covers permitted them to be removed by a chain connected to an overhead crane. The clearance between the briquets and the covers was about one inch at the top and one inch on the sides.

Description of Second Run.

The preheating of the retorts, condenser, and baking oven was started at 1:00 P M on April 5, 1925, using the oil burning apparatus which proved to be far more efficient than the gas burning equipment used in the previous run. After heating for about twelve hours the first two charges of raw briquets were placed in baking oven chambers Nos. 6 and 7 at 1:12 and 1:50 A M, respectively, on April 6th. Raw briquet charges were placed in baking oven chambers Nos. 4 and 5 at 11:00 A M on this same day. At this time the oil burners were shut off and from then on the baking oven was heated by the spent charges taken from the retorts.

The first retort was charged on April 6th at 4:00 P M, as the retort temperature had reached 1312° C.; this charge was allowed to bake until 5:30 P M when the current was turned on. Retort No. 3 was charged at 5:30 P M and permitted to bake until the charge in No. 1 retort had been distilled. As soon as a charge was finished it was placed in one of the chambers of the baking oven and a baked charge placed in the empty retort.

A total of nine charges were run, eight of which were completely distilled. The time of distillation varied from five and three-quarters hours to eight and three-quarters hours. Retorts Nos. 1 and 3 were used alternately, only one retort was in operation at a time due to the lack of sufficient transformer equipment.

When the current was turned on the first charge in retort No. 1 the temperature of the condenser was 580° C. at the top and 704° C. in the center near the bottom. These temperatures were not

maintained but gradually dropped from ten to fifteen degrees per hour for the first day, then from five to ten degrees per hour until the top of the condenser reached a temperature of about 290° C. and the bottom about 240° C., at which temperatures the condenser remained throughout the balance of the run.

Three samples were taken from each of the distilled charges, one briquet from the top, two from the middle vertical rows, and one from the bottom course. The remaining briquets on the base block were run into a baking oven chamber without using the sheet steel covers which allowed them to continue burning slowly and gave off sufficient heat to hold the baking oven at a temperature between 550° C. and 650° C.

Discussion of Second Run.

Table IV is a data sheet of the baking oven showing the time, charge, weight, and chamber in which each charge was placed, length of time the charges were baked, condition of baked charges, and the retort to which they were transferred. The time of baking in the oven varied from six hours and twenty-five minutes to nineteen hours and thirty minutes, and all charges were well baked.

Table V is a data sheet of the retorts and shows the time, charge, and retort number into which each baked charge was placed, together with the time when the power was turned on and off, the length of time the charge stood in the retort before the current was turned on, time of discharge, condition of spent charge, and chamber in the baking oven to which the spent charge was transferred. The

Table IV.

Data Sheet of Baking Oven.

Second Run - April, 1926.

Time Charged		Weight of Charge in pounds	Baking Oven Number	Time Discharged		Condition of baked charge	Charged into Retort Number
Date	Hour			Date	Hour		
4-6-26	1:12 A M	149.5	7	4-6-26	5:20 P M	One bot- tom piece crumbled*	3
4-6-26	1:50 A M	149.5	6	4-6-26	3:55 P M	Good	1
4-6-26	4:05 P M	150	6	4-6-26	10:30 P M	Good	1
4-6-26	6:15 P M	150	7	4-7-26	5:35 A M	Good	3
4-6-26	11:10 P M	150	4**	4-7-26	11:30 A M	Good	1
4-6-26	11:35 P M	149.5	5***	4-7-26	7:05 P M	Good	3
4-7-26	12:30 P M	149	4	4-8-26	2:55 A M	Excellent	1
4-7-26	7:50 P M	149	7	4-8-26	10:45 A M	Excellent	3
4-8-26	4:05 A M	149.5	4	4-8-26	7:12 P M	Excellent	1
4-8-26	11:40 P M	149	7	4-9-26	2:25 A M	Excellent	3

* The bottom briquet was replaced, and the charge put into the retort.

** Transferred to No. 6.

*** Transferred to No. 7 at 7:00 A M on 4-7-26.

Table V.

Data Sheet of Retorts.

Second Run—April, 1926.

Time Charged		Retort Number	Power		Time Discharged		Condition of spent charge	Spent Charge in- to Baking Oven Num- ber
Date	Hour		On	Off	Date	Hour		
4-6-26	4:00 P M	1	4:15 P M	10:00 P M	4-6-26	10:40 P M	Excellent	6
4-6-26	5:40 P M	3	10:50 P M	5:30 A M	4-7-26	5:30 A M	One leg crushed	7
4-6-26	10:40 P M	1	5:37 A M	11:20 A M	4-7-26	11:50 A M	Good	4
4-7-26	5:45 A M	3	11:27 A M	6:55 P M	4-7-26	7:12 P M	Good	5
4-7-26	11:45 A M	1	7:00 P M	2:40 A M	4-8-26	3:05 A M	Excellent	6
4-7-26	7:20 P M	3	2:50 A M	10:10 A M	4-8-26	10:50 A M	Good	7
4-8-26	3:10 A M	1	10:20 A M	7:05 P M	4-8-26	7:20 P M	Good	4
4-8-26	11:10 A M	3	7:07 P M	2:28 A M	4-9-26	2:45 A M	Good	7
4-8-26	7:25 P M	1	2:40 A M	7:25 A M	4-9-26	Distillation incomplete; left in retort.		
4-9-26	2:55 A M	3	Not distilled.					

time of distillation varied from five hours and forty-five minutes to eight hours and forty-five minutes, and in all cases the spent charges were in good condition.

Table VI is a typical power sheet during the distillation of charge No. 4 in retort No. 1 and shows the time the charge was started, control meter readings in kilowatts, total kilowatts, volts, amperes, and watt hour meter readings. The total time of distillation, the way the charge took the current, and the total power input may be noted from this table. This charge happens to have been the longest of any during this run, and consequently the kilowatt hours were the largest.

Table VII is a typical temperature sheet during the time charge No. 4 was distilling in No. 1 retort. The temperatures of the retorts, top and bottom of the condenser, and baking oven, were recorded every half hour from Leeds and Northrup recording potentiometers. The gradual increase of the temperature in retort No. 1 from 644^o to 1200^o C. and the slow decrease of the temperatures of the top and bottom of the condenser are important points to be noted in considering the results.

Table VIII gives the time of treatment and power consumption for each charge, showing the retort and charge numbers, time of baking in oven and retorts, time of distillation, and total power consumed during the treatment of each charge.

Table VI.

Typical Power Report; Charge No. 4 - Retort No. 1.

Second Run - April 8, 1926.

Time	Kw.		Total Kw.	Volts			Amperes			Meter Watt Hour
	Power Input			No.1	No.3	No.2	No.1	No.3	No.2	
10:20 AM	11	8	19	77.5	65	90	140	140	150	0490
10:55	8	7	15	43	42	48	210	170	180	0498
11:30	12	6	18	44	41	57	290	170	170	0509
12:00 PM	13	6	19	47	48	52	300	150	250	0518
12:30	12	7	19	40	42	52	340	170	260	0534
1:00	10	6	16	36	41	43	290	170	240	0539
1:30	10	9	19	36	46	51	320	190	230	0549
2:00	12	8	20	35	46	46	380	200	260	0561
2:30	10	7	17	35	44	45	340	200	230	0569
3:00	10	8	18	34	41	42	320	200	260	0581
3:30	9	12	21	33	41	40	320	350	340	0590
4:30	9	12	21	33	41	42	310	330	310	0614
5:00	10	13	23	35	44	45	340	340	326	0626
5:30	10.5	13	23.5	36	46	45	340	300	310	0638
6:00	9.5	16.5	26	35	47	45	330	380	370	0655
6:30	9	16.5	25.5	35	48	45	320	360	350	0668
7:05	8	11	19	33	41	40	280	250	300	0680
Average of Read- ings			19.94	39.2	44.9	48.7	314	238	266	
Total power input										190 kw.h.

Time of distillation period - 8 hours and 45 minutes.

Table VII.

Typical Temperature Report; Charge No. 4 - Retort No. 1.

Second Run - April 8, 1926.

Time	Temperatures degrees Centigrade					
	Retorts		Condenser		Baking Oven	
	No. 1	No. 3	Top	Bottom	4 & 5	6 & 7
10:00 A M	644	1237	295	243	560	688
:30	644	1147	292	241	558	682
11:00	665	1085	291	241	558	680
:30	697	850	290	238	552	649
12:00	718	838	285	238	588	632
:30 P M	776	810	275	233	590	628
1:00	800	800	272	230	590	613
:30	818	795	275	232	593	646
2:00	863	780	275	228	593	645
:30	880	763	273	221	600	645
3:00	900	748	264	221	600	640
:30	935	727	272	217	605	640
4:00	Not taken					
:30	980	710	275	220	600	632
5:00	1000	700	278	215	603	633
:30	1040	680	283	210	603	633
6:00	1100	670	283	210	630	630
:30	1150	650	283	210	630	625
7:00	1200	640	278	210	620	600

NOTES:

Power on No. 1 Retort at 10:20 A M.

Power off No. 1 Retort at 7:05 P M.

Baked charge from No. 7 Oven to No. 3 Retort at 10:45 A M.

Spent charge from No. 3 Retort to No. 7 Oven at 10:50 A M.

Spent charge in No. 5 Oven removed at 11:15 A M.

Spent charge in No. 7 Oven transferred to No. 5 Oven at 11:20 A M.

Green charge into No. 7 Oven at 11:40 A M.

Table VIII.

Time of Treatment and Power Consumption.

Second Run - April, 1926.

Retort Number	Charge Number	Time of Baking				Time of Distillation		Power Consump- tion in Kw. h.
		In Bake Oven		In Retort		Hours	Minutes	
		Hours	Minutes	Hours	Minutes			
1	1	14	5	-	15	5	45	112
3	1	16	8	5	10	6	40	177
1	2	6	25	6	57	5	43	135
3	2	11	20	5	42	7	28	160
1	3	12	20	7	15	7	40	174
3	3	19	30	7	30	7	20	176
1	4	14	25	7	10	8	45	190
3	4	14	55	7	57	7	21	160

Table IX is a summary of temperature readings giving the retort and charge numbers, retort temperatures when charged, when the power was turned on and off, the condenser temperatures for the top and bottom when the current was turned on and off, and the average temperatures when these charges were baked. In each case the retort temperatures fell off after the charges were placed in the retorts, then increased when the current was turned on while the condenser temperatures, both top and bottom, showed a gradual decrease.

Table X is a summary of residues giving the retort and charge numbers, a description of samples from spent charges—a top and two vertical briquets were taken for one sample. Two samples were made of the bottom briquet, one from the upper part and one from the lower part. These samples were weighed, analyzed for zinc content and fixed carbon, and the results tabulated. There is a fair degree of uniformity shown and in all cases the lower half of the bottom briquet has a much larger per cent of zinc than the other samples.

Table XI is a summary of charge No. 4 in retort No. 1 and charge No. 4 in retort No. 3. These show the original charge in pounds, per cent of zinc and pounds of zinc; the residues in pounds, per cent of zinc and pounds of zinc; the weight lost and zinc distilled in pounds and per cent. These results show a marked uniformity in the distillation of the charges.

The changes and improvements in the equipment proved to be very satisfactory. The oil burning apparatus for preheating the retorts, condenser, and baking oven raised the temperatures to the de-

Table IX.

Summary of Temperature Readings.

Second Run - April, 1926.

Retort Number	Charge Number	Temperatures degrees Centigrade							
		Retort			Condenser				Baking Oven
		When charged	Power on	Power off	Top		Bottom		Average
					Power On	Power Off	Power On	Power Off	
1	1	1312	985	1240	612	540	702	649	593
3	1	1030	830	1142	530	458	649	556	591
1	2	925	740	1198	458	427	532	505	601
3	2	898	706	1174	427	380	505	450	671
1	3	870	650	1200	380	350	455	280	573
3	3	880	670	1237	350	295	270	243	637
1	4	845	644	1200	292	278	241	210	583
3	4	850	640	1232	278	265	210	199	635

Table X.

Summary of Residues.

Second Run - April, 1926.

Retort Number	Charge Number	Sample Description			W e i g h t s			Percent Fixed Carbon		
		Percent zinc top and two vertical	Percent zinc Bottom briquet		Top and two vertical	Bottom		Top and two verti- cal	Bottom	
			Lower part	Upper part		Lower part	Upper part		Lower part	Upper part
1	1	3.22*	--	--	--	--	--	78.44*	--	--
3	1	9.32	--	--	--	--	--	69.70	--	--
1	2	3.42	19.68 ²	3.42	5.25*	1.31*	0.81	59.75	74.90 ²	61.30
3	2	3.11	34.08	8.60	5.37	1.81	.87	76.00	76.75	45.75
1	3	3.63	13.36	4.35	5.12	1.12	.87	75.20	66.85	75.75
3	3	3.73	34.71	18.96	5.19	2.56	.75	70.55	45.45	75.55
1	4	3.21		4.56 ³	4.94		1.81	75.00		74.30 ³
3	4	3.73		2.28	5.12		1.87	76.15		78.60

* From top briquet only.

² One-half of bottom briquet (base section).³ Entire briquet.

Table XI.

Summary of Typical Charges.

Second Run - April, 1926.

Charge Number	Original Charge			Residues			Weight lost and zinc distilled			
	Weight in pounds	Per cent zinc	Pounds zinc	Weight in pounds	Per cent zinc	Pounds zinc	Weight		Zinc	
							Pounds	Per cent	Pounds	Per cent
Charge 4 Retort 1	149.0	35.9	53.5	62.5	3.47	2.17	86.5	58.0	51.3	95.9
Charge 4 Retort 3	149.5	35.9	53.7	60.3	3.15	1.90	89.2	59.7	51.8	96.4

Power Consumption.

Charge No. 4, Retort No. 1 - 190 kw. hours or 3.70 kw. hours per pound zinc distilled.

Charge No. 4, Retort No. 3 - 160 kw. hours or 3.09 kw. hours per pound zinc distilled.

sired points much faster than when gas was used and with less burner adjustment. Practically no difficulty was experienced with short circuits in the base blocks and only a small amount of zinc was tapped from the outlets in the side of the blocks. The sheet steel covers placed over the raw charges to prevent oxidation while baking were very successful as none of the briquets showed any sign of oxidation. The baking sufficiently cemented together the briquets so that no trouble occurred in transferring the charges from the oven chambers to the retorts. As a rule the briquets took the current readily. The added row of insulating brick in the walls of the condenser, as shown in Figure 8, probably decreased radiation losses. However, the small volume of hot gases from the retort did not maintain the desired temperature range in the condenser. Zinc as blue powder, condensed and choked the connections between the retorts and condenser just as in the previous run. The connections had to be frequently reamed out, and while this reaming was in progress a slight explosion of gases in the condenser caused the top brick-work to cave in and ended the run.

After the current had been on a fresh charge for a short while the three phases were very uniform and although there were signs of local heating at times, the briquets held up remarkably well.

During the run no zinc was drawn off from the condenser. When the furnace was shut down one hundred twelve pounds of metal and one hundred seventy-three pounds of blue powder were recovered.

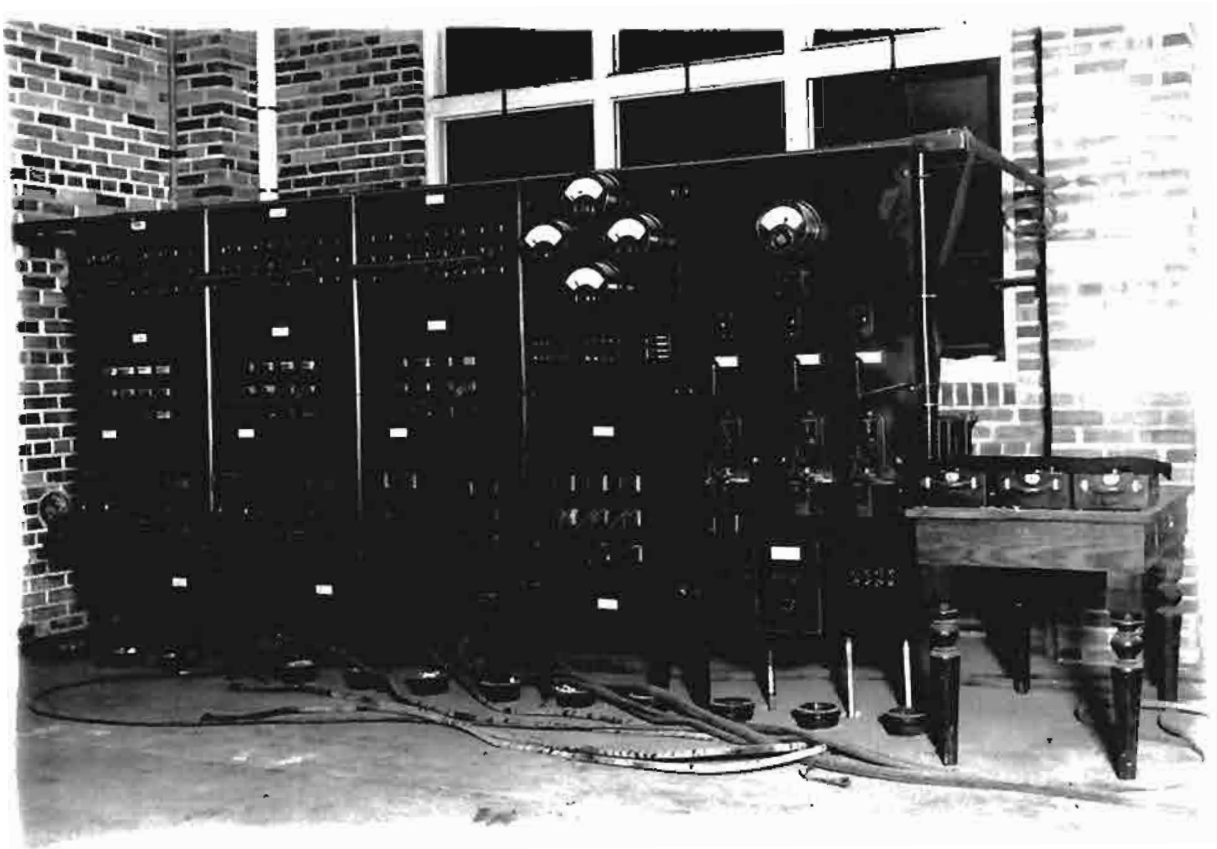


Figure 10.

Switch Board for Controlling Current to Furnaces.

DISCUSSION OF THE CONDENSATION OF ZINC.

The usual gas laws apply to the condensation of zinc from the vapor phase. In an atmosphere, containing zinc vapor only, zinc will condense as dust, when the temperature is below the melting point of zinc— 419° C. In a chamber where the temperature range is from 419° to 930° C., zinc will condense to liquid metal from an atmosphere containing zinc vapor as long as the vapor tension exceeds the maximum tension of zinc at the temperature of the condensing chamber. As the tension of zinc vapor decreases the temperature must also decrease to continue the condensation of liquid zinc. Further, at any temperature within the range at which zinc is molten, some zinc vapor, corresponding to the maximum vapor tension of zinc at that temperature, will remain uncondensed. When other gases are present zinc vapor sustains only a part of the pressure, hence condensation cannot begin until a lower temperature is reached; at this temperature the partial pressure of the zinc vapor in the mixed gases equals the vapor tension of zinc. Also at this temperature condensation to liquid metal may commence and continue as the temperature decreases, until the melting point of zinc is reached. During condensation the gas mixture is always saturated with zinc vapor. This is similar to the precipitation of water from air as the temperature of the air decreases. The temperature at which zinc vapor begins to condense depends upon the vapor tension curve of the metal and the amount of other uncondensable gases, with which it is mixed. These other gases reduce the pressure of the

zinc vapor. An exact analogy may be seen to the phenomena of the dew-point of moist air, the temperature at which water forms.

In the condensation of zinc vapors, certain amounts of blue powder are formed. Two kinds of blue powder—chemical and physical or mechanical—are generally recognized and their causes are different.

The chief cause for the formation of chemical blue powder is thought to be oxidation, either by carbon dioxide, oxygen, or water vapor in the gas mixture. As the oxide is lighter than the metal it probably floats on the surface of the liquid globules and forms a protective coating over them. In order to minimize the formation of carbon dioxide from the decomposition of carbon monoxide ($2 \text{ CO} \rightleftharpoons \text{CO}_2 + \text{C}$) with consequent oxidation of zinc vapor, rapid condensation is desirable. Chemical forces other than oxidation may contribute to the formation of blue powder. The presence of cadmium, arsenic, antimony, tellurium, selenium, and sulfur is detrimental to efficient condensation. Small dust particles may act as nuclei around which zinc vapor may condense and increase the amount of blue powder formed.

Blue powder produced by physical or mechanical causes is largely due to condenser design and improper temperature regulation, but atomic and molecular, and mechanical forces may have some effect. The force expressed in heat units required to overcome atomic and molecular forces which hold zinc in the liquid state is a direct measure of the force that tends to condense it. On condensation, the same amount of heat is given out as is absorbed when zinc metal is

vaporized. It is essential that the separate particles be close together for the action of atomic and molecular forces to take place. From an atmosphere in which the zinc is much diluted by gases, either inert or oxidizing, more blue powder is formed. The condenser walls and baffles, dissipating the heat given out and also their location with reference to the gas flow, are important factors.

In electrothermic zinc smelting in the past certain amounts of blue powder have been produced. The blue powder formed in some of the campaigns in the Fulton electrothermic dry distillation process in East St. Louis was very small. In the experimental work covered by this thesis considerable blue powder was formed, but this could not be avoided, as the temperature of the condenser was below the melting point of zinc—419° C.—for much of the time.

The forces that act on the zinc globules and gases to bring the zinc atoms and molecules within the range of intermolecular forces are gravity and mass action. Mass action is due to the change of volume by the addition or subtraction of heat. Gravity is constant but opposed by several resistances. A rapid evolution of zinc vapor from the retort chamber is not necessarily a disadvantage, but an irregular flow of gases is detrimental. The condenser governs the velocity and direction of issuing gas currents.

The temperature range within which condensation is possible cannot be varied and must be between the melting and boiling points of zinc. The vapor should gradually cool from entrance to exit. A rapid and uniform rate of distillation should decrease the heat loss by radia-

tion. If the loss of heat in the condenser is too rapid or too local and cannot be prevented, external heat must be added; if the reverse occurs, cooling with air or water must be used. Regulation of the condenser temperature consists of three parts: (1) Obtaining heat sufficient for the start, (2) Conserving the heat during the normal working so the condenser may be as large as possible, and (3) Supplying or removing heat as desired.

Condensation is a complicated phenomena. In liquids with low boiling points the liquid globules increase rapidly when their presence in the gas is indicated by a nebular zone. Solid particles or neuclei throughout the cooling gas, around which condensation occurs, aid in the formation of the liquid globules. At a certain size these globules fall by gravity at a measured rate and collect as a liquid. Zinc vapor probably acts in a somewhat similar manner and to thoroughly understand condensation it is essential to be familiar with the laws of diffusion, viscosity, vapor tension of gases, surface tension, capularity, and adhesion and cohesion of liquids, as these forces are involved in the formation of the liquid globules and their union with a liquid mass.

SUMMARY.

Although the work described in this thesis cannot be considered as complete, until certain mechanical changes have been made and the furnace operated continuously over a considerable period of time, this electrothermic dry distillation process for the extraction of zinc from ores offers excellent commercial possibilities.

The literature has been reviewed, the process that was used at East St. Louis described, and results of work at the Mississippi Valley Experiment Station of the United States Bureau of Mines, Department of Commerce, Rolla, Missouri along the line of electrothermic metallurgy of zinc considered.

In the Fulton process the runs in November, 1925 and April, 1926 both showed that the briquets baked in a satisfactory manner in the baking oven; the zinc distilled off readily in the retorts and the briquets maintained their original form and volume after treatment. Considering the fact that the temperatures in the condenser for about fifty per cent of the time were below that at which zinc could possibly condense as a metal, an unusually large amount of metallic zinc was obtained. In the first run seventy-seven pounds of metal with one hundred eight pounds of blue powder were recovered, while in the second run one hundred twelve pounds of metal with one hundred seventy-three pounds of blue powder were obtained from the condenser. This is an average of about forty per cent in the form of metal.

The per cent of zinc distilled in Charges No. 6 and No. 7 of the first run was 94.8 and 95.5 per cent. In the second run Charge No. 4 in No. 1 Retort and Charge No. 4 in No. 3 Retort showed 95.9 and 96.4 per cent, respectively, which are excellent recoveries.

The work described in this thesis was carried out in a small model plant. This furnace is of unusual interest from a commercial view point, as there is a real need for a properly designed electrothermic process in which the furnace would have a daily capacity of from five to ten tons. The power consumption¹ from the

¹Fulton, C. H., Large electric-resistance furnace for zinc ores, Trans. Am. Inst. Min. & Met. Engrs., Vol. LXIV, p. 222, 1921.

tests in the model plant are not indicative of the results of actual operation on a large scale, but will vary with the grade of the ore treated and will be higher for low grade ores. However, the difference may be compensated by the subsequent recovery of other metals, if any are present.

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