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The Roasting and Magnetic
Separation of Wisconsin Ores
and
The Utilization of the By-Products

by

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Approved by: Chas F Clayton

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In the Wisconsin field, the zinc blende is usually found associated with iron sulphide in the form of marcasite. This ore, after being milled and concentrated at the mines, is sent to separating plants for further treatment by roasting and magnetic separation, inasmuch as the mixed blende and marcasite concentrate, owing to the high iron content, is not suitable for the zinc smelter, and the magnetic separation scheme of separation must be resorted to.

The greater portion of the roasting and magnetic separating is done by three companies, the Mineral Point Zinc Company, at Mineral Point, The National Zinc Separating Company, at Cuba City, and the Wisconsin Zinc Company at New Diggings. There are other roasting and separating plants in this district, namely, the Linden Zinc Company, at Linden, which company also has a plant at Cuba City, The Benton, and the Mulcahy Roaster, at Shullsburg, operated by the Oliver Iron Mining Company. Each of the three largest plants has a capacity of 275 tons of ore per 24 hours, and their combined capacity easily takes care of the bulk of the Wisconsin zinc field production. The method of roasting ore at all of these plants is practically the same, inasmuch as each has an auto-roasting 7-hearth furnace. The National plant uses a Wedge Furnace manufactured by the Wedge Mechanical Furnace Company, whereas the plants at Mineral Point and New Diggings have Skinner installations manufactured by the Western Chemical Manufacturing Company, of Denver, Colorado. The treatment of the ores at these plants is so similar that there is here given in detail the present method used at the National Plant.

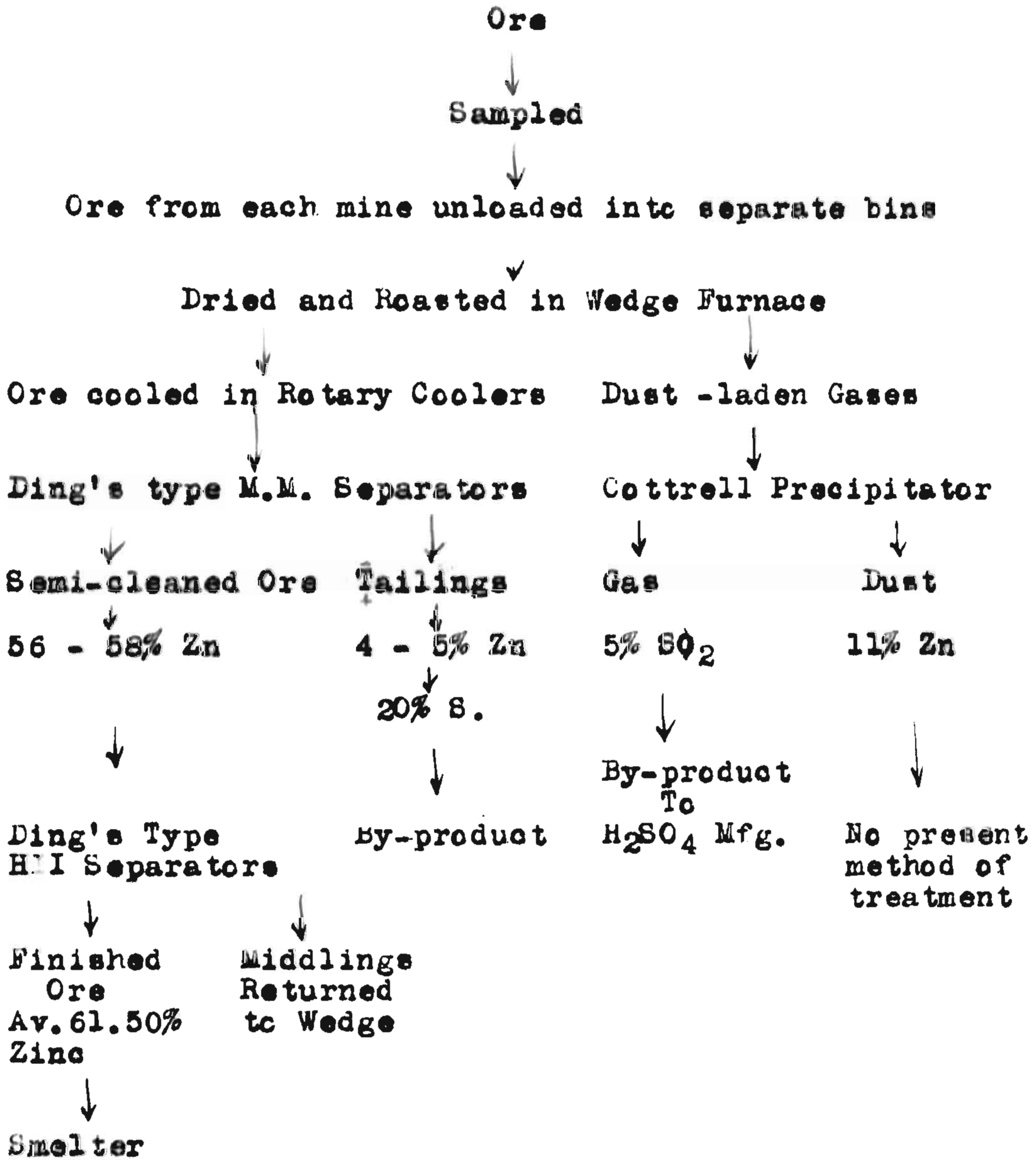
The ore as it is received at the Roasting and Magnetic Separating plants from the mines varies greatly in composition, as shown by the following table:-

Mine	Zinc	Iron	Lead	Lime	Sulphur
Yewdall	21.60%	27.40%	0.82%	2.15%	39.60%
Martin	39.95	17.12	0.20	1.67	35.30
Jefferson	45.90	12.85	0.22	1.50	33.73

Assays were made on the following composite samples for general calculations, as they are representative of the Wisconsin District.

%Zinc	%Fe	%Pb	%CaO	Sulphur
20.1	27.0	.65	2.90	40.6
25.2	24.1	.45	2.25	39.8
29.2	21.5	.45	2.35	38.9
35.5	17.1	1.00	2.95	36.2
40.1	13.8	1.30	2.85	35.0
45.4	11.0	.75	2.30	34.0
50.2	8.0	.25	2.50	34.2

The general flow sheet of the National plant is as follows:-



The ore is shipped to the National plant from the various mines of the district which lie from a few miles to a distance of 25 miles. Shipments are made almost exclusively in box cars, although, in a few instances, where the mines are close to the plant, trucks and teams are used. As the National plant receives a large percentage of its ore in box cars, and also ships out its finished product in box cars, effort has been made to insure maximum unloading and loading tonnage.

The ore as received from the mines contains about 2.0% moisture in the summer time and about 4.0% moisture in the winter time. Therefore, it can be readily seen that adequate precaution must be made for thawing this ore in the winter, as this amount of moisture will cause the ore to be frozen in a solid mass in the car. The method used is to force live steam thru nozzles into the frozen body of ore in the cars and thaw same by means of this live steam. It would naturally be supposed that this introduction of live steam into the ore would raise the moisture content of same to a considerable larger percentage that it originally contained. However, this is not true as can be readily noted by Table No. 1 on the following page.

Table No. 1

1. Mine..............................	Monmouth	Big Dick	Dale	Yewdall
2. Car No............................	2714	515199	2936	53380
3. Gross weight before thawing......	99300	128500	127700	129400
4. Gross weight after thawing.......	98500	128200	126600	128900
5. H_2O before thawing, hand sample..	2.5	3.7	6.0	3.5
6. $\%H_2O$ after thawing, hand sample..	1.9	3.5	2.5	2.5
7. $\%H_2O$ after thawing, gun sample	1.0	3.0	2.5	2.5
8. Empty weight of car...............	33400	46800	36000	37200
9. Net weight before thawing........	65900	81700	91700	92200
10. Net weight after thawing.........	65100	81400	90600	91700
11. Loss due to thawing..............	800	300	1100	500
12. Dry weight figured from 5........	64252	78600	86198	88973
13. Dry weight figured from 6.........	63860	78551	88335	89407
14. Dry weight figured from 7........	64449	78958	88335	89407
15. $\%H_2O$ in top ore after thawing....		2.6	1.8	2.5
16. $\%H_2O$ in middle ore after thawing.		3.5	4.5	2.8
17. $\%H_2O$ in bottom ore after thawing.		5.7	5.7	3.0
Average of 15 - 16 - 17		3.9	3.7	2.8

The box cars are first set out with the side door opposite the green ore storage elevator. The ore is then dragged out of the cars by means of a Clark Single Unloading Shovel into the underground boot of the elevator, which, in turn, carries the ore to the top of the storage bins, where it is distributed into separate bins by means of a belt conveyor and automatic tripper. A large tonnage can be handled by this method. Three men can unload at the rate of one ton per minute, which includes the time of weighing the cars in and out. In loading the finished ore, the ore is spouted from the finished ore bins, which are immediately above the green ore bins, into the side of the car, where it is stacked in the ends of the car by means of a portable belt conveyor.

The sampling of the ores as they are received is done by means of a green ore gun, which consists of a 2 inch pipe about 3 feet long. The roasted ore is sampled with a slotted gun, which consists of a 1¼ inch pipe 4 feet long, with 1¼ inch by ¼ inch slots 1¼ inches apart lengthwise on pipe. The pipe is covered with a sheath when poked into the bed of ore. The sheath is pulled off when the gun is in position, allowing the ore to fall into the slots.

The roasting is done in a Wedge Mechanical Furnace, which has 7 roasting hearths and a top drying hearth, and is 22 feet 6 inches in diameter and 24 feet above the floor. The furnace consists of a cylindrical steel riveted shell in casing walls of red brick. The hearths are constructed of special shaped fire brick and are made horizontal, each hearth, however, forming an arch above the one below.

The central shaft is made of riveted steel plate, being 5 feet in diameter. The outside of the shaft is protected by special insulation and fire brick, which are attached to and revolve with it. The hollow shaft is open at top and bottom, and the natural draft of air thru it keeps it sufficiently cool for workmen to enter at any time, thus giving free access to the rabble arms from outside.

The rabble arms are cast in one piece. They weigh about 1200 pounds each, and are made hollow with a webb horizontally thru the center. The rear end is machined and fitted with pipes for air cooling, one pipe leading from the air manifold at the top of shaft to the upper side of the median webb, and the other leading to a manifold at the bottom of the shaft from the lower side of the median webb. It is from the lower manifold that the heated air can be either increased into the furnace thru the lower hearth, or let out into the furnace space by means of two 3 inch bleeder valves, depending on the condition of the roast. The rabble blades are fitted on the arms with rabble blade holders.

The ore is brought by belt conveyor and elevator to two feeding hoppers at the top of the kiln, where it is fed to the kiln by two revolving arms at the rate of 90 seconds per revolution. These arms can be lengthened or shortened, depending on the desired tonnage to be fed. As the ore drops on the kiln it is stirred by the rabbles and gradually worked to the central shaft and there falls onto the first hearth; here the rabbles gradually work it outward in a spiral path to the periphery, where it falls onto the second hearth. On the first, third, fifth and seventh hearths the ore travels radially outward, and on the second, fourth, and sixth hearths it travels in the opposite direction. The roasted ore is discharged from the periphery of the lowest hearth into two rotary coolers.

With this type of furnace no outside fuel is necessary, except on the initial firing, as the ore is roasted by its own heat of combustion, the ore being heated up gradually on its downward passage in the furnace until it reaches the sixth hearth, where the maximum heat of about 750° to 800° C. is reached. See Table 2, on following page, for complete temperature readings.

Table No. 2

Summary: Temperatures Wedge #1. (Feed by-passing first hearth)

Date	Feed	Hearth 7			Hearth 6			Hearth 5			Hearth 4			Hearth 3		
	Tons	Av.	High	Low	Av.	High	Low	Av.	High	Low	Av.	High	Low	Av.	High	Low
12-20	88.25	613	645	585	807	820	800	784	790	780	611	620	600	430	450	415
12-21	88.9	543	590	460	775	825	740	760	785	740	630	660	590	462	485	440
12-22	70.0	502	530	470	735	745	720	724	745	700	619	655	580	452	490	400
12-23	105.0	530	575	450	745	775	720	712	740	690	610	650	590	450	495	405
12-24	75.6	535	615	470	760	780	740	730	750	690	664	700	640	490	550	410
12-25	75.6	489	520	460	726	750	715	715	740	700	700	715	620	530	580	510
12-26	65.0	480	505	450	722	730	710	687	720	670	670	720	640	477	525	460
12-27	72.0	535	600	540	740	780	665	677	710	610	663	700	620	486	520	420
12-28	87.9	557	580	530	752	790	665	690	715	685	702	740	680	506	550	470
12-29	95.8	580	600	550	784	800	750	682	710	650	684	720	650	483	525	445
12-30	93.7	577	600	530	750	790	740	670	710	650	640	660	620	447	490	425
12-31	145	581	600	565	758	775	740	712	730	620	644	690	555	470	530	430
1- 1	1o9	633	690	570	770	800	750	726	780	655	620	670	570	490	540	445
1- 2	95.8	630	660	610	732	780	720	752	740	620	570	600	520	450	510	420
1- 3	104.5	572	630	545	720	760	700	700	740	650	568	610	520	456	490	410
Av.	15 days	557	590	519	756	780	730	715	740	674	640	674	600	472	518	433

By maintaining these temperatures with varying feed, 4.5 to 9% sulphur dioxide gas, suitable for sulphuric acid manufacture is evolved, and tailings averaging 20% sulphur, suitable for further roasting for manufacturing sulphuric acid, are recovered from the magnetic separators. The utilization of the by-product gases and tailings are taken up in detail later in this report. If the temperature becomes too high, the valves on the lower manifold are opened, and the air is allowed to escape into the furnace space, and if more heat is desired these valves are closed.

With this type of roasting furnace, the marcasite (FeS_2) in the ore receives a strong oxidizing roast though the heat maintained is not sufficient to oxidize any of the blende (ZnS). The marcasite particles are converted on the outside to the magnetic Fe_3O_4, the cores of the particles remaining Fe_nS_{n+1}, and, to some extent, unaltered FeS_2. During the roasting and separating the Pb and CaO content will also vary from the green to the finished ore.

The Pb increases about .o15% for every .1%

contained in the ore. The CaO, on the other hand, decreases about .012% for every .1% contained therein. This decrease is due to the fact that in the process of roasting partial decrepitation of the ore takes place, and the CaO, being lighter in gravity, has greater tendency to be carried in the flue dust than does the ore itself.

Screen analysis before and after Roasting.

Size	Before	After
On 1/4 inch mesh	.20%	.10%
On 1/8 inch mesh	21.70	9.70
On 10 inch mesh	17.90	7.80
On 20 inch mesh	28.10	23.30
On 40 inch mesh	22.90	29.10
Thru40 inch mesh	9.20	30.00

From the Wedge furnace the ore passes thru four rotary cooling drums, revolving at 6 R.P.M. They are each 2 feet I.D. and 26 feet long, and are made of 5/16 inch riveted plate steel. The drums are water cooled by means of a spray directly above them. No attempt is made to cool the ore so that it is absolutely cold, as it has been found that roasted ore is not as magnetic when cold as when slightly warm to the hand. Each cooler requires about 30 gallons of water per minute to cool the ore to the proper temperature.

The ore is now ready for magnetic separation. It passes first over a rougher separator of the Ding's Type M.M. Machine. This machine occupies a floor space of about 4x10 feet, and has an extreme height of 83 inches. The ore is received in a hopper at the upper end of the machine, which feeds it in an evenly regulated flow on to a vibrating conveyor or shaking table, actuated by two eccentrics on the driving shaft rotating at 450 R.P.M. causing the ore to travel thru the zones of separation. The table is 21 inches wide by 7 feet 10 inches long, is constructed of maple frame-work with ¼ inch pressed asbestos (Transite Board) top, and is inclinee at 4 inches per foot. The lower end of the table is 21 inches above the floor. Above the tables are two sets of magnets placed in the same inclined position as the table. The first set, or rougher pair, is 5½ inches O.D. x29½ inches long, and has a maximum strength of about 2 amperes at 250 volts when heated to 100° F. The second pair, or semi-finisher coils, are 8 7/8 inches O.D. x 19 inches long, having a maximum strength of about 3 amperes when heated to 100° F. Under each pair of magnets are revolving bronze spiders 26 inches in diameter with 32 laminated electric steel secondar y magnets set in the rims of the spiders about 1½ inches apart and held in place by brass keepers. The upper part of these secondary magnets are U shaped and are inductively energized by the lip of the pole pieces of the stationary magnets immediately above, which they embrace while over the conveyor. This close contact presents a very large area of surface between the primary and secondary magnets, and reduces the magnetic resistance at this point to a minimum. The lower part of these secondary magnets are set about 3/8 inches to 1/2 inch above the top of the asbestos table. The magnetic iron sulphide, as it is picked out of the ore traveling down the table, is carried in circular motion out of the magnetic circuit to the neutral positions where the spider rims hang over the conveyor, and is dropped into hoppers made of non-magnetic sheet metal (Manganese steel). It is then delivered by belt conveyor and elevator to a storage bin. The tailings, or magnetic product, thus made average about 20% sulphur and 4% to 5% Zn. Details regarding the further roasting of this material is taken up later in the report. The semi-finished ore passes over the end of the table and down into a Ding's separator of higher magnetic intensity, which is used to make the final or finished product. The ore, as it is delivered to the finishing machine, will average 56 to 58% Zn abd 3 to 4% Fe. The capacity of this machine is about 60 tons per 24 hours.

The "Finisher" magnetic separator is made by the Ding's Electro Magnetic Separator Company of Milwaukee, and is known as the company's H.I. Machine. It is 5 feet 8 inches x 8 feet 5 inches overall at the base, and 7 feet 4 inches in height. The feed is received at each end of the machine thru a feed hopper, which discharges onto two 12 inch endless belts 6 feet 8 inches long, moving at 70 feet per minute toward the center of the machine between a magnetic and revolving armature which picks out any magnetic material (middlings) and carries it in circular motion to the neutral positions where it is dropped into hoppers at each side of the machine. The non-magnetic material (high grade zinc blende) drops down into a small hopper under the revolving armature onto a 7 inch belt, which delivers it to the end of the separators. There are four magnetic coils, in series, under the revolving armature, 11½ inches O.D. x 20 inches long with 6 inch cores, and having the following current regulation at 225 volts:

Maximum current Hot (100° F)	15 amps
Maximum current Cold	19 amps
Minimum current Hot (100° F)	7½ amps
Minimum current Cold	9 amps

The armature which completes the magnetic field is made of solid steel, being 24 inches in diameter x 6 inches wide on a vertical shaft setting about ¾ inches to 7/8 inches above the pole pieces of the magnets. The weight of this armature and the downward pull are taken care of by a ball thrust bearing.

The capacity of this machine varies from 40 to 45 tons per 24 hours.

The dust-laden gases pass out thru the side of the furnace from the first hearth, being first led into a dust chamber, and thence thru a Cottrell Precipitator.

The Cottrell Precipitator consists of two units which are so arranged that either unit may be operated alone, or in parallel with the other. At present each unit is being operated separately and alternately for periods of about two weeks. The precipitator is handling about 5000 cubic feet of gas per minute at 290° F. and at a velocity of about 3 feet per second. The combined capacity is 18,000 cubic feet per minute for the two units when operated in parallel.

Each unit is 9 feet x 13 feet 6 inches from the center line to center line of column supports, and has an overall height from ground level to base of stack of 34 feet 8 inches. The dust-laden gases enter near the top of the bottom header from opposite sides. The gases flow around the pipes that extend 5 feet into the bottom header and tend to heat these pipes equally. This causes the pipes to draw equally, and as a result an equal amount of gas tends to pass up thru the pipes which are discharged at the top of the precipitator. The fines are collected on the walls of the pipes, or collecting electrodes, whence it drops or is shaken down into the receiving hopper at the bottom. The gases pass on upward into the gas collecting headers at the top and then down thru a steel flue into the purification system of the acid plant.

The collecting electrodes, of which there are 36 in each unit, are pipes 12 inches I.D. x 15 feet long, made of No. 14 sheet steel. The high tension current used in connection with the Cottrell is made fom 250 A.C. current. The electrical apparatus consists of a transformer, switch board, synchronous motor, and a mechanical rectifier. The rectified current used in connection with the precipitator is about 65,000 volts and the amperage varying from 1 to 60. The high tension current is transmitted to the precipitator by a line passing thru the top of the electrical building to the caged housing at the top of the precipitator.

The dust recovered is about 97 to 99% of the total amount lost in the gases, and will approximate about 1.6 % of the amount of ore roasted in the Wedge furnace.

The gases as they have passed thru the Cottrell Precipitator have had the greater portion of the dust removed from them, but require a further cleaning when they reach the acid plant.

The acid plant is of the contact type and consists of three general steps.

1. Purification of the gases
    - Apparatus used
        1. Scrubbing tower using hot water
        2. Scrubbing tower using 60° Be' H_2SO_4
            - 2 sets of lead gas coolers
            - 3 quartz filters
            - 10 mineral wool filters
2. Conversion of SO_2 to SO_3
    - Apparatus used
        - 2 Connerville Blowers
        - 2 Preheaters (2 units per preheater)
        - 4 Heat interchangers
        - 4 converters
3. Absorption of SO_3 by H_2SO_4
    - Apparatus used
        - 2 Fuming (Oleum) Towers
        - 2 Final absorption towers

In the contact process the gases must be freed from certain gaseous impurities which have an inhibiting effect upon the oxidizing power of the catalyzer. In Wisconsin ores these impurities consist almost entirely of lead sulphate, chlorine and arsenic.

The lead sulphate should be removed by the Cottrell Precipitator with the dust, but at times enough creeps over into the acid plant to give trouble unless precautions are taken to insure against it. For this reason the first step taken in our process is to wash the gases with hot water (a waste product from another part of the plant) to cleanse from the gases most of the lead sulphate and such chlorine and arsenic as may be present. The water is hot enough (60°C.) to prevent any material loss of SO_2 by absorption.

Details of the Tower construction are given later in this report.

The gases then pass thru two sets of lead coolers in series. Each set of coolers consists of about 800 lineal feet of the lead cooling pipe of about 12 inch inside diameter. The pipe is arranged in 5 parallel rows of 4 pipes each. These pipes are water sprayed for cooling. These installations collect a large quantity of such dust and other impurities such as may escape any preliminary cleaning. Any moisture or weak acid collected is removed by boots at the bottom of the pipes, but any dust or other impurities collected therein must be washed out at intervals of about every 45 to 60 days.

The gases then pass thru two sets of quartz filters, each containing about a 36 inch bed of finely divided quartz, having a down flow of the gases to precipitate any impurities or moisture possible. As soon as they become foul they are washed with water sprays at intervals of about 60 days. The construction of these filters is given later in the report.

The gases, after leaving the filter, go to two drying towers thru a 16" lead pipe.

The drying tower is of lead, as described later. The gas inlet enters the side of the tower close to the bottom at a downward angle, so that none of the drying acid can run back into the gas-inlet pipe. The gas outlet is in the side, close to the top.

The acid for scrubbing and drying this is about 59 to 60° Be' H_2SO_4. Acid is delivered to the towers by Norman Hubbard vertical centrifugal pumps, and the discharge goes to coolers which again serve the tower. As this acid is weakened by the absorption of moisture it is strengthened by means of 98% H_2SO_4 in warm weather, and 66° Be' acid in cold weather. In this particular instance we estimated that 3.5 to 4 tons of water are extracted from the gas every 24 hours.

After leaving the scrubbing towers the gas passes into a third quartz box to remove any mist acid that may have been carried mechanically from the scrubbing towers and hence to the ten mineral wool filters.

The mineral wool filter consists of three cast iron trays each about 6 feet in diameter and 1 foot high, having lead covered iron rods in the bottom, on top of which lays a perforated lead screen. The basic mineral wool is laid upon these screens to a depth of about 10 inches. The three sections are then nested together and the joints sealed with a black putty, called chlorine putty. To this point all the construction consisted of lead, whereas from this point on everything consists of steel or cast iron.

From the mineral wool filters the gas passes to the blowers, which have controlled or have been pulling the gas thru all the forementioned apparatus. In other words, the entire plant from the Wedge Furnace to the mineral wool filters have been under suction, whereas from this point on the entire plant is under pressure.

The blowers used are of the Boston gas type, manufactured by the Connersville Blower Company, and are driven by a variable speed motor. Under present operating conditions 5000 cubic feet of gas per minute are handled.

The gases from the blower enter the preheating furnace. A detailed description of a Preheater is given later in this report.

In ordinary practice the gas is usually heated to about 420° C. However, in this plant the heat generated in the exothermic reaction from the oxidation of SO_2 to SO_3 is utilized. This procedure has several distinct advantages, as follows:

1. The fuel consumption is cut to 50% or less.
2. Repairs on the preheating furnace are reduced to a minimum.
3. The hot SO_3 gases, leaving the converter impart some of their heat to the incoming SO_2 gases, thus reducing the cost of the SO_3 cooler installation.

The temperature to which the SO_2 gases are heated in the preheating furnace depends largely on the percentage of SO_2 contained in the gases. In this particular case the percentage of SO_2 varies from 4 to 7%, whereas the temperature to which these gases are preheated varies from 220° to 90° C. As the SO_2 in the gas increases, so does the heat from the exothermic reaction; hence, the lower the temperature of the preheater and a greater saving of fuel.

As the gases leave the preheater they pass into and thru the heat exchanger around a number of vertical tubes and hence into the bottom of the converter, rising to the top of same around its outer shell and then comes in contact with the platinum mass on its downward path thru the center of the converter where the oxidation of SO_2 to SO_3 takes place, resulting in the liberation of heat due to this exothermic reaction. The gas now passes again into the heat exchanger from the bottom, passes up thru the tubes where it liberates some of its heat to the SO_2 gases on the outside of same, and hence to a set of steel SO_3 coolers. Details of the construction of the heat exchanger and converter are given later in this report.

The platinum mass is made of calcined magnesium sulphate. A good commercial grade of magnesium sulphate is used and heated until all the water of crystallization is driven off. In the calcining the salt fuses together and after baking and cooling it is broken up to the desired size, ranging from ½ to 1½ inches in size. The resultant fines are treated with water, refired and again sized.

The platinum is sprayed on this sized material in the form of dilute platinic chloride and then packed in the shelves or trays of the converter. When the hot gases pass over this material, the cloride of platinum is broken up, leaving the metallic platinum in a very fine disseminated state thruout the magnesium sulphate.

The platinum mass is packed in the converters as follows:

Converter No.	Amount of Mass	Actual % Platinum	Troy oz Platinum	Troy oz Platinum Tatal each Converter
No. 1	830.3	.23647	28.63307	
No. 1	3739.2	.157847	86.07385	
Total	4569.5			114.70692
No. 2	804.0	.23647	27.72610	
No. 2	3606.7	.157847	83.01557	
Total	4410.7			110.74169
No. 3	767.5	.23647	26.47138	
No. 3	3396.0	.157847	78.178026	
Total	4163.5			104.64164
No. 4	838.05	.23647	28.87667	
No. 4	3666.05	.157847	84.38995	
Total	4505.10			113.26562

Total Troy oz. Platinum in Converters; 443.35585

For general calculating purposes 14 to 16 oz. of platinum are required per ton of monohydrate (100% H_2SO_4) produced per day, although many plants use less than this amount. At this writing the Silica-Gel Corporation claim to be able to obtain equally as good results with one-sixth this amount of platinum when silica-gel is used for the mass instead of magnesium sulphate.

The SO_3 gases after leaving the converter are passed thru a cooling system before they enter either the oleum or the final absorbing towers. This cooling system consists of a parallel row of pipes comprising 5 rows about 70 feet long, made of ¼ inch steel 12 inches inside diameter. The elbows are constructed of heavy cast iron. These pipes must be protected with a roof in the summer, and roof and sides during the winter time, because moisture falling on these pipes will cause the formation of weak acid and result in a misting from the exit pipes of the final absorbers. Excessive cold weather will cause contraction of the pipes resulting in the breaking of the cast iron elbows. Care must also be taken so that the drip acid formed does not freeze and result in a blockage in the lines at this point.

From the cooling pipes the gas is passed into the base of the oleum tower passing upward thru same and onward to a final absorbing tower and into the base of same, and upward and out to the exit stacks.

Although the gases always pass thru the oleum tower, operation of same is only maintained when the production of acid stronger than 100% H_2SO_4 is desired. In order to have successful operation of this tower the gases must be cooled in the preceding coolers to a temperature of at least 90° C. and lower, if possible. The operation of the manufacture of oleum consists of passing a spray of strong acid down in counter current to the SO_3 gases that are rising from the base of this tower. Usually a quantity of about 30 to 40 tons is used for this operation, and is sent over this tower as often as necessary to be built up to the proper strength. We have found that if we start with an acid assaying 100% the first circuit of this acid will add about .7 of 1%; the second circuit about .6 of 1%; the third .5 of 1%, etc., decreasing in each succeeding one. The foregoing is not a definite table because the percentage of strength added depends on the temperature and strength of the SO_3 gas, the temperature of the acid, and the room temperature. With the equipment we have the highest strength acid we are able to obtain is 105.5%, and requires about 16 to 20 complete circuits of the acid to increase same from 99 to 105% H_2SO_4.

In the final absorbing tower it is desirous to absorb as much of the SO_3 in the gases as possible, and in order to maintain 90% absorption or better, we have found that the absorbing acid for this tower must be kept within the limits of 97½% to 99% H_2SO_4. When the acid falls below 97½% a very perceptible white mist may be seen from the exit stack. When the acid is above 99% a brown fume may be seen. In each case the density of the mist or fume increases as the acid diverges from the required limits. Although great care must be exercised to keep the acid within the required limits, there is no quick and accurate method suitable for the average workman that has been developed to test the strength of this acid quickly and accurately. There are three rough methods employed:-

1. The spit test. By spitting on the acid one may get a fair idea of its strength. If it sizzles like the frying of an egg, it denotes weakness; if it pops, it is a sign it is strong.

2. The acid on top of the final absorbers when it is the proper strength seems to have thin hairs, or whiskers, rising from it. When this is not present the acid has a flat appearance which denotes weakness,

and when the whiskers exceed 8 to 10 inches in length it is a sign that it is too strong.

3. The third test is by observation of the exit stack, and requires long experience by the operator. When the acid is below the required strength a whitish mist may be observed, and when too strong a brownish fume can be seen. Unless the acid is far too weak or too strong it is very difficult for an inexperienced operator to be able to discern one from the other.

At the best these three tests are very crude, and can only be used by one thoroly familar with the details of operations, and it is very often the case that the best operator will notice one test denotes weakness of the acid, whereas by the other it will signify that it is too strong.

As the acid is built up in strength by the addition or absorption of SO_3 in the circulation over the final absorber, it must in turn be porportionately weakened. This is done by diluting the strong absorbing acid with approximately 60° Be' H_2SO_4. The reason for using 60° acid is that less heat is generated than would be if a weaker acid or water was used. A stock of this 60° acid is always kept on hand for this purpose. As this strong absorber acid is diluted it passes thru a series of water sprayed cast iron coolers and is again pumped to the top of the final absorber for recirculation. The operation is continuous. As this circulation acid absorbs the SO_3 and is subsequently diluted the volume naturally increases. This is taken care of by an eccentric tee at the entrance to the coolers, and all acid overflowing into this tee constitutes the "make" acid or production.

The construction of the final absorber is given later in this report.

The product acid is kept in steel tanks 6'0" and 26'0" long, and then stored in 2 steel storage tanks 30'0" and 12'0", each having a capacity of 500 tons.

The acid is all shipped to the consumer in strengths varying from 60° Be' to 104.5% H_2SO_4, in 50 ton steel tank cars, as ordinarily used.

Quartz Filters.

The quartz filters are 20"0' long, 12'0" wide, and 8'2" high. They are made of a skeleton frame work of 3'x8" rough timbers set on 15" centers. The floor is laid of 3x12 timbers set close. The lead lining is then installed, which is made of 7 lb. lead. The wall and roof of the boxes are hung by lead slips around the 3x8 timbers. Each filter has a manhole for entering if necessary.

An acid proof brick grill work is laid on the bottom about 12" high, no cement being used. The vein quartz is then dumped in and graded as follows; about 1 foot 3 to 4", 1 foot 2 to 3", 1 foot 1 to 2", and about 6 inches ½ to 1". The smallest size on top grading down.

The filters are set on a slight pitch so that any moisture or acid which is collected may drain by means of traps set on the low corner of the filters.

The gas enters the box at the top high side by means of a lead manifold and exits on the bottom of the opposite side thru a lead manifold.

Gas Washing Towers Construction.

The Towers are 12'0" square outside measurements, and 20'0" high. Four corner posts of 8"x8" timbers are set and a skeleton framework made of 3"x8" timber morticed in the corner posts set on 2'6" centers having an inside size of 10'0"x10'0".

A heavy lead pan is then laid, made of 15 lb. lead extending up on the sides about 1'0" high. The lead curtains are then hung, being strapped to the skeleton framework with lead cleats or clips. The top is hung from 3"x8" timbers in the same manner as the filters.

The acid or water is distributed to the towers with 1" lead pipe and nozzle on the end of same. Each tower has 16 intakes for acid or water, as the case may be.

The towers are lined, sides and bottom, with a 4" course of acid proof brick set up with acid proof cement, made of 40° Be' silicate of soda and pure silica sand of about the consistency of thick molasses.

An acid-proof brick grill work is then laid to a height of 3'2" from the bottom. Vein quartz is then packed in the towers, as follows; sizes of 12 to 20" for a depth of 3 feet above the grill work. The quartz is then graded down to a size of 3 to 5" within 3'0" from the top of the tower. The tower proper is set on an arched foundation made of concrete, open on two sides, having walls 1'0" wide and a cap 2'0" thick. The foundation is 11'0" high and the opening in same is utilized for overflow, storage and pump tanks.

Construction of Preheaters.

The Preheaters are of brick construction and are 23'0" long x 11'9" wide x 14'0" high, set on a concrete foundation 18" deep. In order to avoid radiation surface the preheaters are built in pairs, therefore the total width is 23'6" while the other dimensions remain unchanged. The following details pertain to a double preheater.

The outside walls are made of common brick and are 9" wide. Inside of these walls is a 1" air space and a 9" wall of low grade fire brick.

The fireboxes (one for each unit) have a grate area of 25 sq. ft. and 8'4" overall length extending across the entire front of the preheater. From the grate line the high grade fire brick walls are stepped so that they are the full width of the preheater at a distance of 8'0" from the floor line. At this point there is an opening of 2'0" in the back wall of the firebox which allows the hot combustion gases to heat the cast iron U tubes; they are then deflected downward by a baffle wall, then up by another, and then they pass downward for exit from the U tube chamber for stack exit.

The top of each section of the preheaters is covered with a firebrick arch set on a 9'0" radius running the length of the preheater. This is covered with about 1'0" of cinders to prevent excessive heat radiation.

There are 30U tubes per unit, or a total of 60 in 10 parallel rows.

Description of U Tubes.

The U tubes are about 5½" inside diameter and 7' long. The wall thickness of the preheating tubes is about ¾ of an inch. The gases are delivered from a header into five of these tubes in parallel; the tubes are connected in series with six similar U tubes by means of cast iron return bends. Flange connections between the two bends and the return bends are inadvisable, because the steel bolts of the connection would expand, causing gas leakage. The type of connection between the return bend and the U tube is a rust joint. A shoulder is cast on the return bend about 2½" from the top, upon which is set a cast iron ring having an inside diameter about ¾ of an inch larger than the outside diameter of the U tube. The space between this ring and the joint is then tamped full of a mixture of sal-amoniac and fine iron filings.

Construction of Heat Exchangers.

The heat exchangers are made of 3/8" plate steel and are 5'0" in diameter and 10' high, and contain 587 steel tubes 6'11" long extending within 1'0" from the top and 2'1" from the bottom. The top and bottom heads of the exchangers are of heavy cast iron. The center line of the exit flange on the side of the exchanger is 3'1" from the bottom and the center line of the intake flange on the opposite side is 1'10½" from the top. The following specification covers the construction;

Flanged heads 3/8"; Flange steel shell and baffle plates, tank steel and tubes to be 1¼" O.D. and 1.11" I.D. of standard quality, lap welded mild steel of standard manufacture. Shell to be thoroly caulked so as to be tight under gas pressure of lo pounds per sq. in. and at a temperature of 750° F. Provide caulking strips for nozzles. Holes in heads to be chomfered off and tubes expanded and beaded over so as to be tight under above conditions.

These are lagged, when installed, with 6" of magnesia and 6" of mineral wool, making a total of 12" of insulation on the outside.

Construction of Converters.

The inside shell of the converter is 6'4" I.D. and 12'6" high, and made of 3/8" steel plate. The outside shell of the converter is 7'0" O.D. and 12'0" high, extending from 11" above the bottom of the inside shell and 5" above the top of the inside shell, allowing 3½" air chamber in the upward passage of the gases. The head and bottom plates are made of heavy cast iron. Cast iron angles brackets are riveted on the inside of the center shell. The first set being 1'6" from the bottom and each succeeding set 2'6" above the previous set, making a total of five. Baffle plates of 1/8" steel and 5'0" O.D. are set 2'1½" above the tray brackets, and used for deflecting the gases in their downward path from tray to tray to prevent channeling. The tray bars are of heavy cast iron, upon which are set 3/16" steel screens with 3/8" perforations used for holding the mass. The converters must withstand 10 lbs. per sq. inch of gas pressure at 750° F.

The converters are lagged, when installed, with 6" of magnesia and 6" of mineral wool, giving a total of 12 inches of insulation.

A flanged opening is made on the outer shell thru the inner shell to each tray for pyrometer couplets.

Absorber Construction.

Shell for Oleum Absorber:- The Oleum Absorber is 5'0" O.D. and 11'2" high, made of 5/16" plate steel. A 16" cast iron intake gas nozzle is riveted to the shell, the center line of which is 2'0" from the bottom. A 4" exit acid nozzle made of cast iron is on the opposite side 7½" from the bottom.

The cover for this tower is made of 3/8" steel plate and is bolted to the tower proper. The cover has a 4" cast iron acid intake in the center, and a 16" cast iron gas exit nozzle on the edge of the top.

The Final Absorber is 7'0" I.D. and 15'6" high, and made of 3/8" plate steel. A 16" cast iron intake gas nozzle is riveted to the shell, the center line of which is 1'6" from the bottom. A 6" exit acid nozzle made of cast iron is on the opposite side 9" from the bottom. The gas exit nozzle is 16" in diameter and the center line is 1'8" from the top on the same side as the exit acid nozzle.

The acid intake for the final absorber is a heavy cast iron bowl which sets on the top of the final absorber. The bowl is about 6" deep and is perforated with ½" about 6" apart on circles starting from the center. Each circle being 8" diameter larger than the one previous, until the outer edge of the bowl is reached. The flow of acid is regulated by opening or closing some of these holes.

The final and fuming absorber are each packed alike. They are first lined with a double row of acid proof brick, each 2" wide, set in acid proof cement made of 40° Be' silicate of sodium and pure silica sand which has the consistency of thick molasses when used. A double row of brick are also laid on the bottom. A grill work of piers are set up in like manner from the bottom, about 2'6" high, the space between being about 8".

Vein quartz is then packed in these towers.

Quartz ranging in size from 12 to 20" is packed in directly on top of the grill work to a depth of about 3 feet. The size is then graded down until sizes of about 2 to 3" are reached about 6" below the exit gas nozzle in the case of the Final Absorber, and one foot below the top plate in the case of the Oleum Tower

General.

In order to keep records of the plant operation the following records are made:-

1. Hourly records made of speed of blowers which have a positive displacement of 23.1 cu.ft. of gas per revolution. From this record the volume of gas per 24 hours can be calculated.

2. Hourly tests are made of the SO_2 content of the incoming gases to the acid plant. From this record and from No. 1 the tonnage of sulphur handled per 24 hours can be figured.

3. Hourly tests are made of the SO_2 content of the stack gases as they leave the acid plant. From this record and No. 1 the amount of sulphur lost, and with No. 1 and No. 2 we may calculate the percent of sulphur converted from SO_2 to SO_3.

4. The daily volume of acid made (which is ap-

proximately 98% H_2SO_4) is recorded, so the total recovery of sulphur can be figured, and the amount of sulphur absorbed and be calculated using the foregoing records.

5. An accurate weekly and monthly inventory is made so that the accurate recoveries are made for weekly and monthly reports.

6. The tests made in No. 2 and No. 3 are made by means of the Reich test for SO_2.

7. Hydrostatic guages are placed on all the filters and in various places in the system. The water suction or pressure as noted in these guages is quite an aid in general operations, as a blockage can be worked down to within a small area and thus cleared as soon as possible. In winter beet juice is used in these guages on account of its low freezing point and dark color; and same has a gravity of 1.2 very close to water. Many plants use a low gravity acid that has a low freezing point.

8. The fuel consumed is carefully measured. The average amount of fuel consumed varies from 10 to 11% of the total tonnage of SO_3 made.

9. Hourly records of the heats carried at var-

ious points are tabulated. The points noted are as follows;

a - The gas exit the preheater
b - Temperature of top mass tray
c - Temperature of second mass tray
d - Temperature of third mass tray
e - Temperature of fourth mass tray
f - Temperature of fifth mass tray

If the percent of the SO_2 in the gas increases (say from 4 to 5% SO_2) and holds at the higher percent, it will require dropping of the heats in the preheater. The reason for dropping the heats in the preheater is because the amount of heat liberated in the exothermic reaction will increase and thus has a tendency to increase the temperature thruout the whole converter.

The reverse condition takes place when the percent of SO_2 decreases in the gas.

A representative average of temperatures taken from operation following the above list would be as follows on a 5% SO_2:-

a - 150° C.
b - 420° C.
c - 520° C.
d - 515° C.
e - 510° C.
f - 510° C.

The rise in temperature from a to b, 270°, represents the amount of heat added to the SO_2 gas in passing thru the heat exchanger and from the outer shell of the converter, or the amount taken from the SO_3 gases in the places mentioned.

The rise in temperature from b to c represents the heat evolved from the exothermic reaction in the first step of the conversion.

The drop in temperature from d to e and from e to f is due to the fact that some of the heat has been transferred to the incoming SO_2 gases, and that most of the SO_2 has been converted to SO_3 at this point.

All temperatures are measured by recording pyrometers.

In order to give some idea as to the amount of acid or sulphur that is available from any particular grade of ore which might be roasted, we have compiled the table on the following page;

Calculations for Determining

Average Sulphur Value of Various Grades of Ore

(Recovery Considered)

			80 ton feed per 24 hours		125 ton feed per 24 hours	
% Zinc	% Iron	% Sulphur Available in iron	Tons Sulphur Available per ton ore	Tons 66°Be' H_2SO_4 per ton ore	Tons Sulphur Available per ton ore	Tons 66°Be' H_2SO_4 per ton ore
20	28.0	40.5	.163	.5346	.115	.3772
25	24.0	39.7	.147	.482	.110	.3608
30	20.0	39.0	.131	.4296	.100	.328
35	17.0	36.0	.110	.3608	.085	.2788
40	14.0	35.0	.089	.2952	.075	.2460
45	11.5	34.5	.068	.223	.067	.2197
50	8.0	34.2	.045	.1476	.050	.1640

Note:- Sulphur value varies with daily tonnage handled in Wedge Furnace.

Iron Pyrite Tailings.

In the former practice of this district the ore was roasted in the Mathey type of kilns. This type of kiln consisted of a ½" steel shell about 26'0" long and 6'0" in diameter lined with fire brick. The kiln was set at a slight incline, and revolved at the rate of about one revolution per minute. The ore was fed in at one end and gradually progressed to the other, where the ore was directly fired with either an oil or produce gas flame. The ore thus roasted left the kiln by means of port holes in the shell at the end of same. In extremely old practice a fire box was set in front of the kiln and coal firing was resorted to. In this practice the iron sulphide was not so nearly roasted or desulphurized as in present practice, resulting in a refuse iron pyrite assaying 30 to 33% sulphur. This product could easily be disposed of with a fair margin of profit, as same sold for .50 to $2 per long ton. However, since the installation of auto-roasting furnaces of the Wedge and Skinner type, these iron pyrite tailings are a drug on the market, as same only contain about 20 to 22% sulphur which is not enough to make it an economical proposition to ship to distant acid plant for further recovery of their sulphur values.

Several attempts were made to reroast these tailings in the Wedge Furnace, as described in the first part of this peport, to see if same could be dead roasted, and at the same time produce an SO_2 gas suitable for contact sulphuric acid practice. However, not much success was met in this procedure for the following reasons;

1. The gas was too lean or weak in SO_2 content for contact acid plant operation. The gas averaged 2.5% to 3% SO_2.

2. The cinder or resultant product from the reroasting of these iron pyrites contained 8.0% sulphur. The percentage of sulphur remaining in same was too high to make disposition of same to the blast furnaces or the utilization of same for paint pigment.

3. The SO_3 content in the gases was extremely large, resulting from the conversion of SO_2 to SO_3; the iron oxides present in the pyrites serving as the catalyzer. (This difficulty, however, can be overcome to a great extent).

Although the first two of the foregoing reasons were enough to convince one that the idea of reroasting the iron pyrites was a failure in this type of furnace, it did give us the idea that if fuel in some form or other could be introduced in about the sixth hearth of the Wedge furnace, that a lower calcine could be made with a subsequent higher SO_2 gas for acid plant. Although this idea has since proven to be the solution of this problem, nothing further was done along these lines until an installation was made of a furnace embodying this same idea, as developed by the Ore Roasting Development Company, primarily for the auto-roasting of zinc sulphide ores without the use of extraneous fuel. The plan of the furnace was as follows;

The general plant and size was practically identical to the Wedge Furnace as detailed in the first part of this report for the roasting of the Wisconsin ores, with the following modifications:

1. One extra hearth was added to the top of the furnace, so that there are a total of 8 hearths. This hearth was built so that the interior height was about twice that of any of the other seven.

2. The outside brick walls of the furance were

increased to 24", with the exception of hearth No. 2, to prevent any excessive radiation of heat.

3. Hearth No. 2 (from the top) was built so the outside brick walls were only 12" wide, giving this hearth a diameter 12" greater than the other hearths. This was done so that fifteen drop holes could be constructed equi-distant in the outer circumference of this second hearth to direct the ore as follows:

5 holes, Nos. 1-4-7-10 and 13, directed the ore to the third or next hearth.

5 holes, Nos. 2-5-8-11-and 14, directed the ore directly to the fifth hearth, by passing hearths Nos. 3 and 4.

5 holes, Nos. 3-6-9-12 and 15, directed the ore directly to the 7th hearth, by passing hearths Nos. 3 to 6 inclusive.

Summarizing we have the flow sheet as follows, assuming a 30 ton feed:

10 tons passing thru hearths Nos. 1-2-3-4-5-6-7 and 8.
10 tons passing thru hearths Nos. 1-2-5-6-7 and 8.
10 tons passing thru hearths Nos. 1-2-7 and 8

4. Lighter rabbling arms were used. In this case they only weighed 850 lbs. against 1200 lbs. in the other furnace. (This was only an attempt of

economy on account of an anticipated smaller capacity of the furnace.

5. A rabble blade of an entirely new design was used. It was wedge shaped as it passed thru the ore body, one side of the wedge being smaller than the other side, for the following reasons. As the blades passed thru the ore, past practice has been to advance the ore towards on its downward path thru the furnace. With this type of blade the idea was to advance .6 of the ore and retreat .4. In other words only an actual advance of .2 was made, insuring a more thoro turnover of the product.

There were several other changes also made in the design, but since they were made primarily for the roasting of zinc sulphide and did not have any effect on the roasting of the low sulphur pyrites, no description of same will be made here.

In March 1921, the first tests were made. The following table is a portion of the data as gathered and calculated from the test.

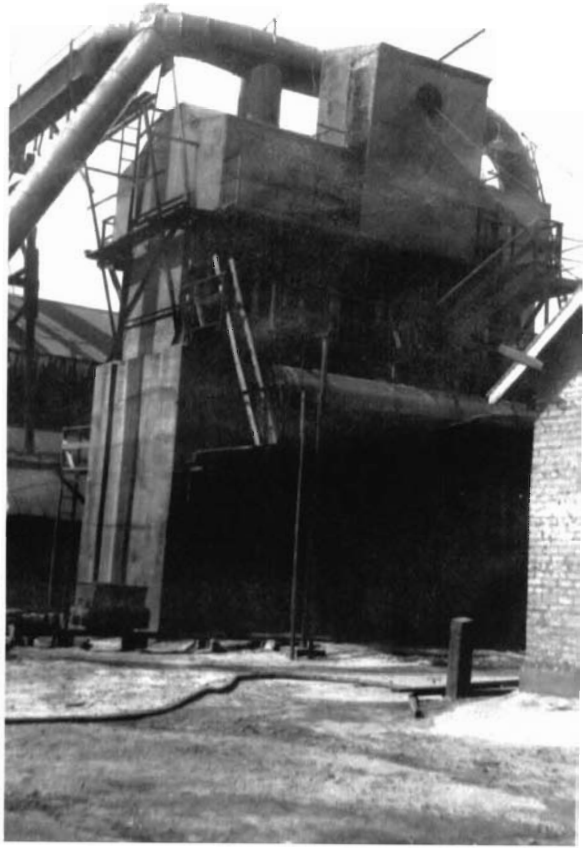
The direct charge of roasting this product is within reasonable limits, therefore it is now only a case of wait for the return of normal business conditions and the entire production of this material will be utilized. The gases, which will average 4 to 5% SO_2, can be used in a sulphuric acid plant. The calcines which we believe we can produce containing about 1.5% sulphur, may be disposed of to the blast furnace, or the paint manufacturers as a paint pigment, or some other industry.

Time	Temp. at beginning of shift °C. Disc. 8th	7th	5th	3rd	2nd	Flue	Feed per min lbs.	Heads Avail. Sulphur	Calcines Avail. Sulphur	Sulphur burnt per min	Cu. ft. gas per min.
7 AM	190	670	672	683	630	455	50	18.2	4.8	6.7	
3 PM	250	674	674	680	640	425	50	19.3	4.4	7.45	1880
11 PM	275	690	690	700	680	442	50	19.5	4.0	7.75	2130
7 AM	260	701	707	710	650	423	58	19.8	4.6	8.82	2060
3 PM	288	694	697	697	760	422	50	20.2	3.7	8.25	2000
11 PM	296	708	712	710	660	435	50	21.9	4.1	8.90	2040
7 AM	308	722	720	720	670	440	50	21.0	6.2	7.40	
3 PM	295	712	717	720	700	448	48.5	21.3	3.4	8.68	2190
11 PM	290	722	725	730	690	470	45.5	22.2	3.3	8.60	1980
7 AM	295	730	732	741	720	480	47	19.4	3.2	7.61	1860
3 PM	270	726	728	743	720	482	46	19.2	2.0	7.91	2240
11 PM	217	710	730	755	720	505	46	22.0	1.5	9.43	2480
7 AM	255	724	740	757	730	500	48	20.3	2.9	8.35	2290
3 PM	300	800	710	732	720	482	48	21.2	2.9	8.78	2520
11 PM	280	770	777	765	750	500	48	20.8	2.9	8.59	2260
7 AM	280	780	820	777	760	520	50	18.2	2.1	8.55	2640
3 PM	210	675	680	733	760	595	50	20.6	1.7	9.45	
11 PM	250	775	790	770	760	567	46	20.2	1.0	8.83	3360

The percent of SO_2 in the gas, in the above test, averaged from 4.1 to 5.4%.

The speed of the kiln or that of the central shaft was one revolution in 111 seconds.

The entire test was made without interruption or shut down.

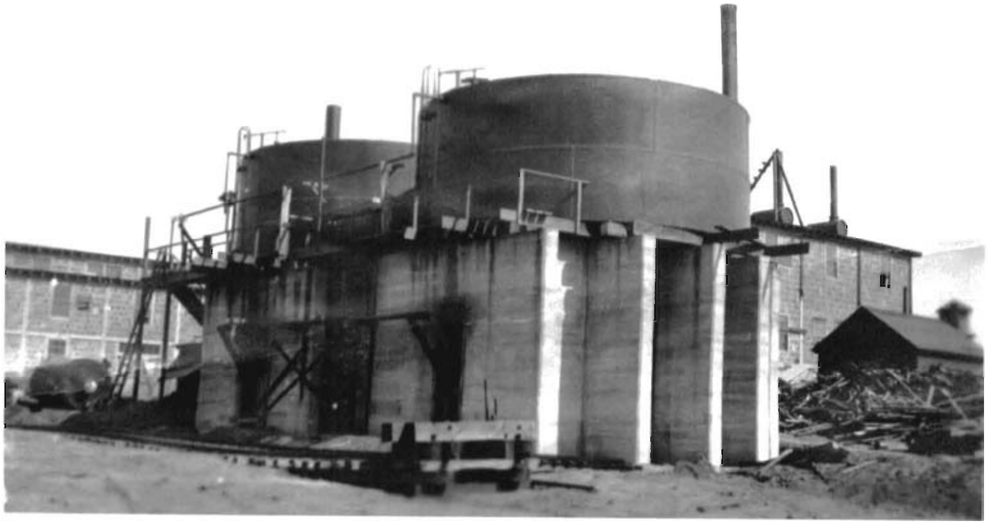


Cottrell Precipitator
as
Described on Page 16

Used for Separating the Dust from the Gases.

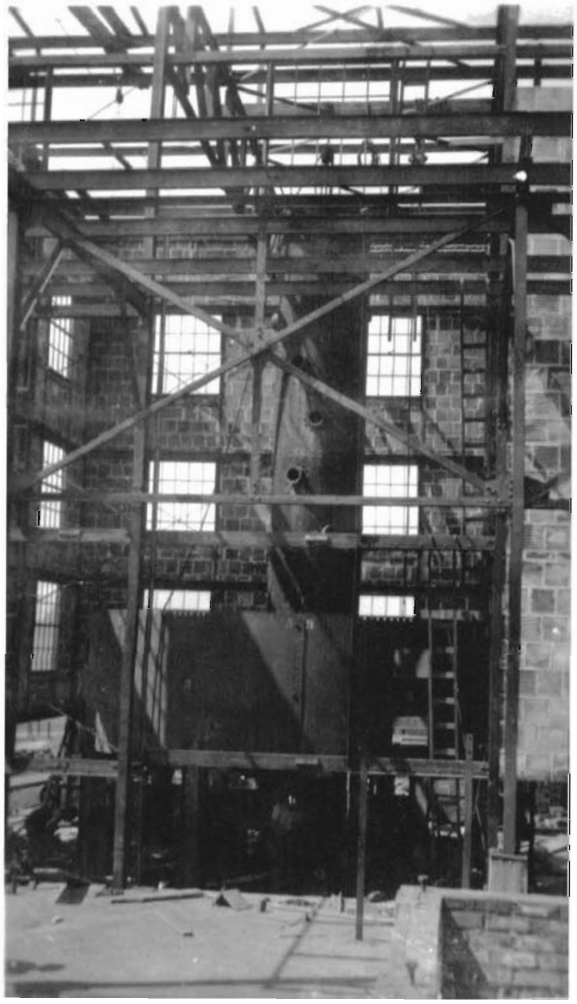
Spray Catcher

Used for collecting traces of acid that might be carried mechanically in the gases from the Drying Towers to the Mineral Wool Filters.



Storage Tanks

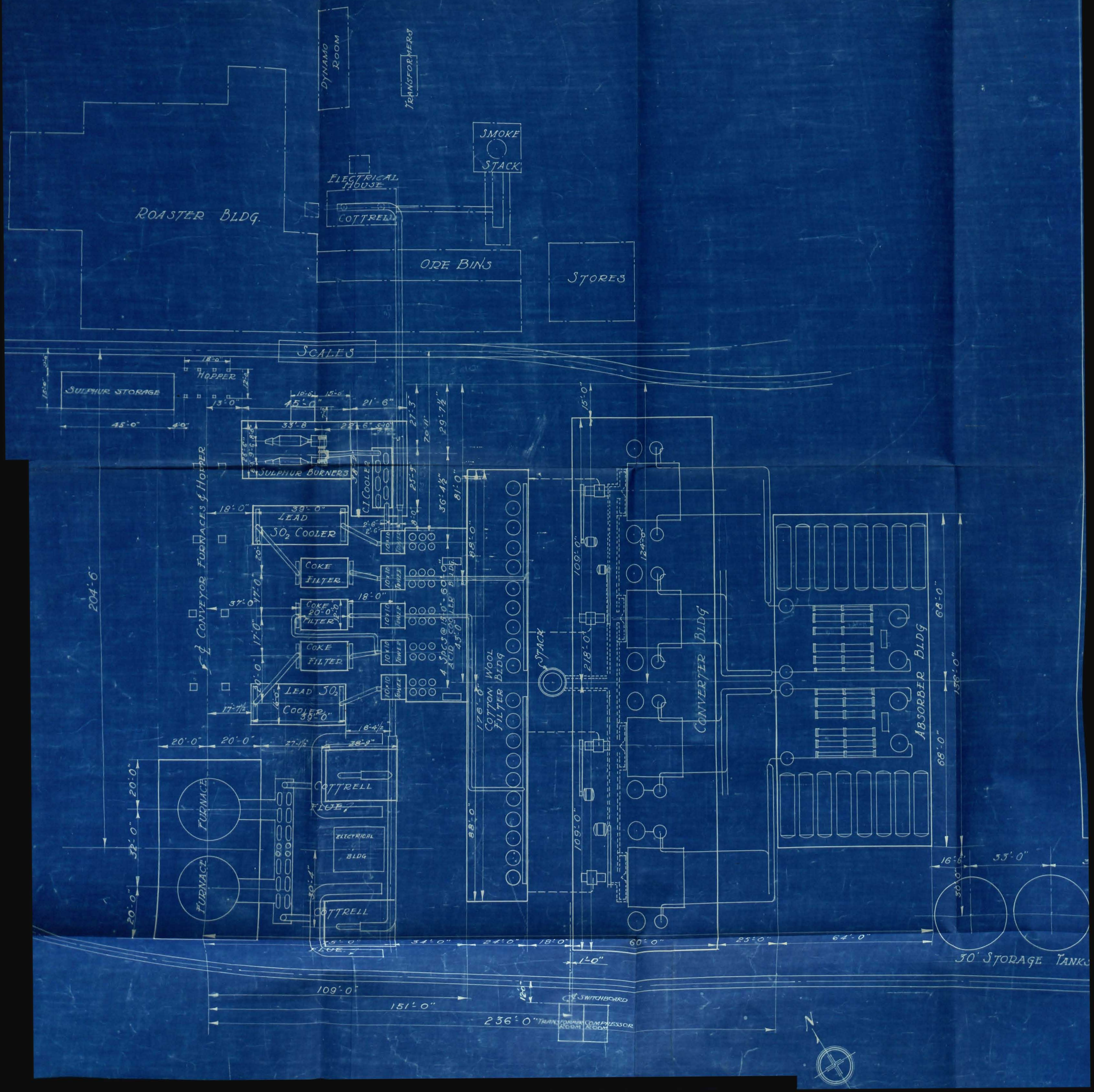
Used for storing acid before loading into Tank Cars for shipment.



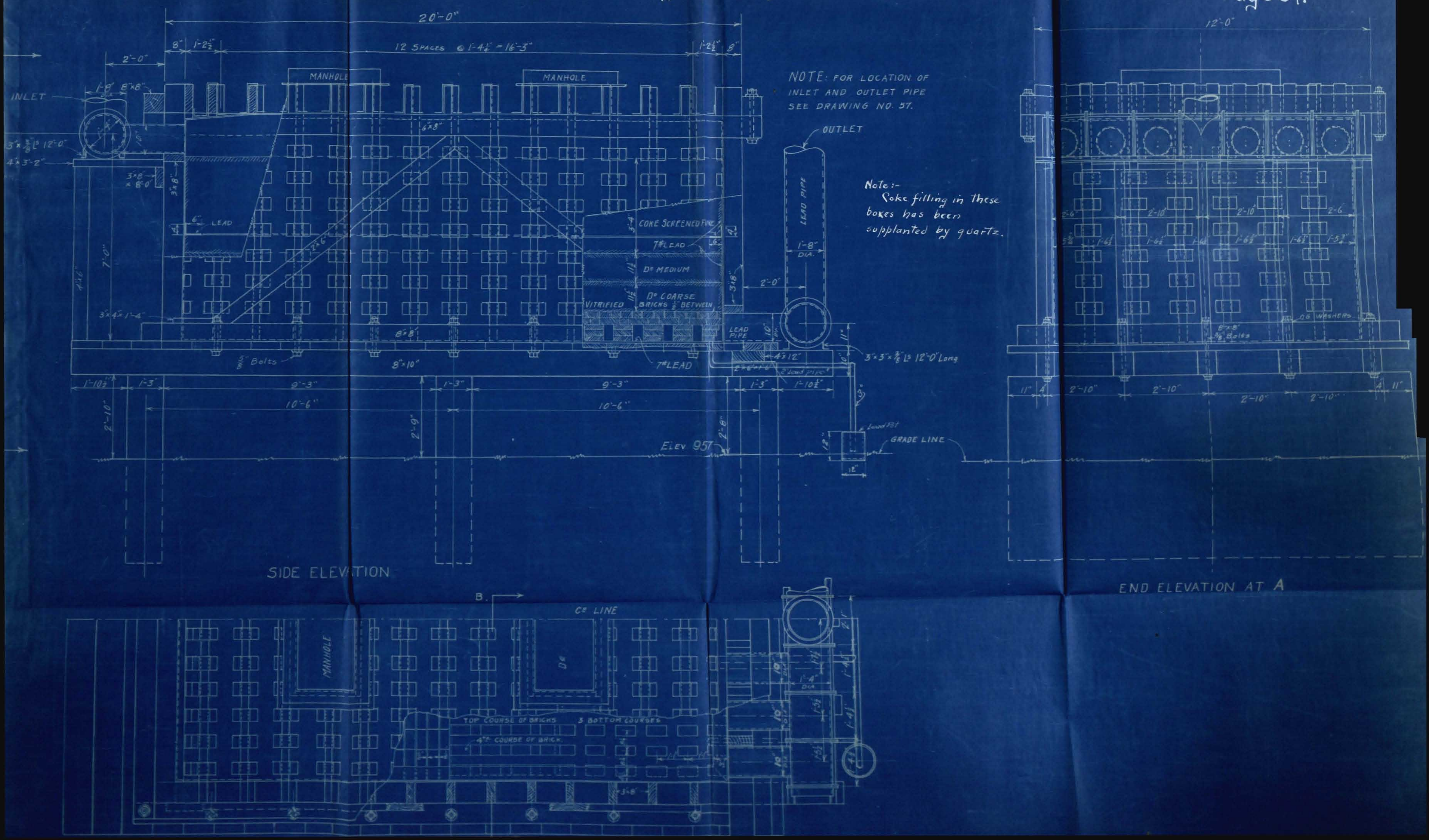
Wedge Furnace in the course of construction.

Note; Central Shaft first erected.
Steel outer casing in first state of erection.
Brick walls and hearths not laid until steel work completed.

General Layout Drawing of Roaster & Acid Plant.



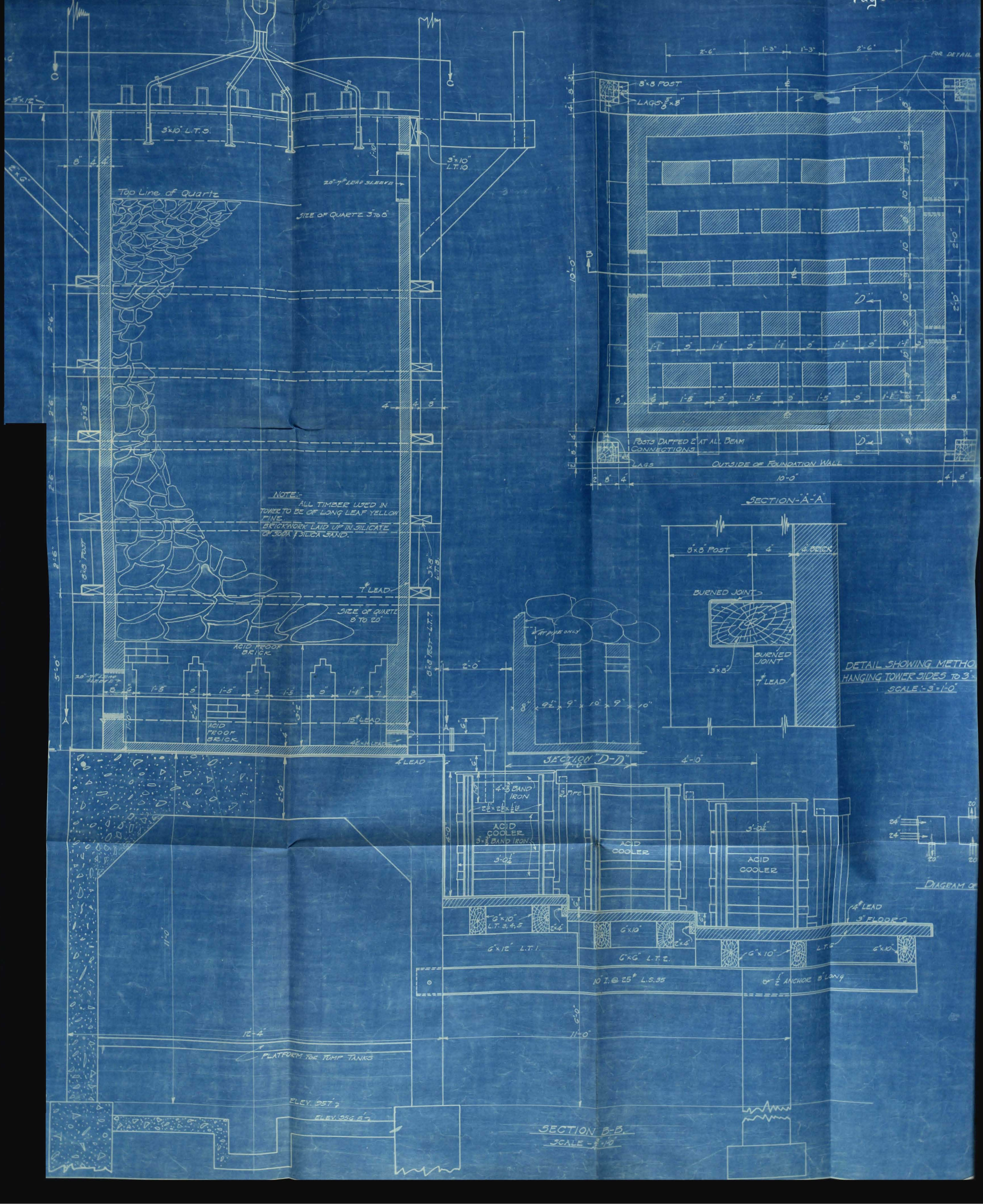
General Drawing of Quartz Filter Boxes.



NOTE: FOR LOCATION OF INLET AND OUTLET PIPE SEE DRAWING NO. 57.

Note:-
Coke filling in these boxes has been supplanted by quartz.

Detail Drawing of Gas Washing Tower.



SECTION "A"-"A"

DETAIL SHOWING METHO
HANGING TOWER SIDES TO 3"
SCALE :- 3" = 1'-0"

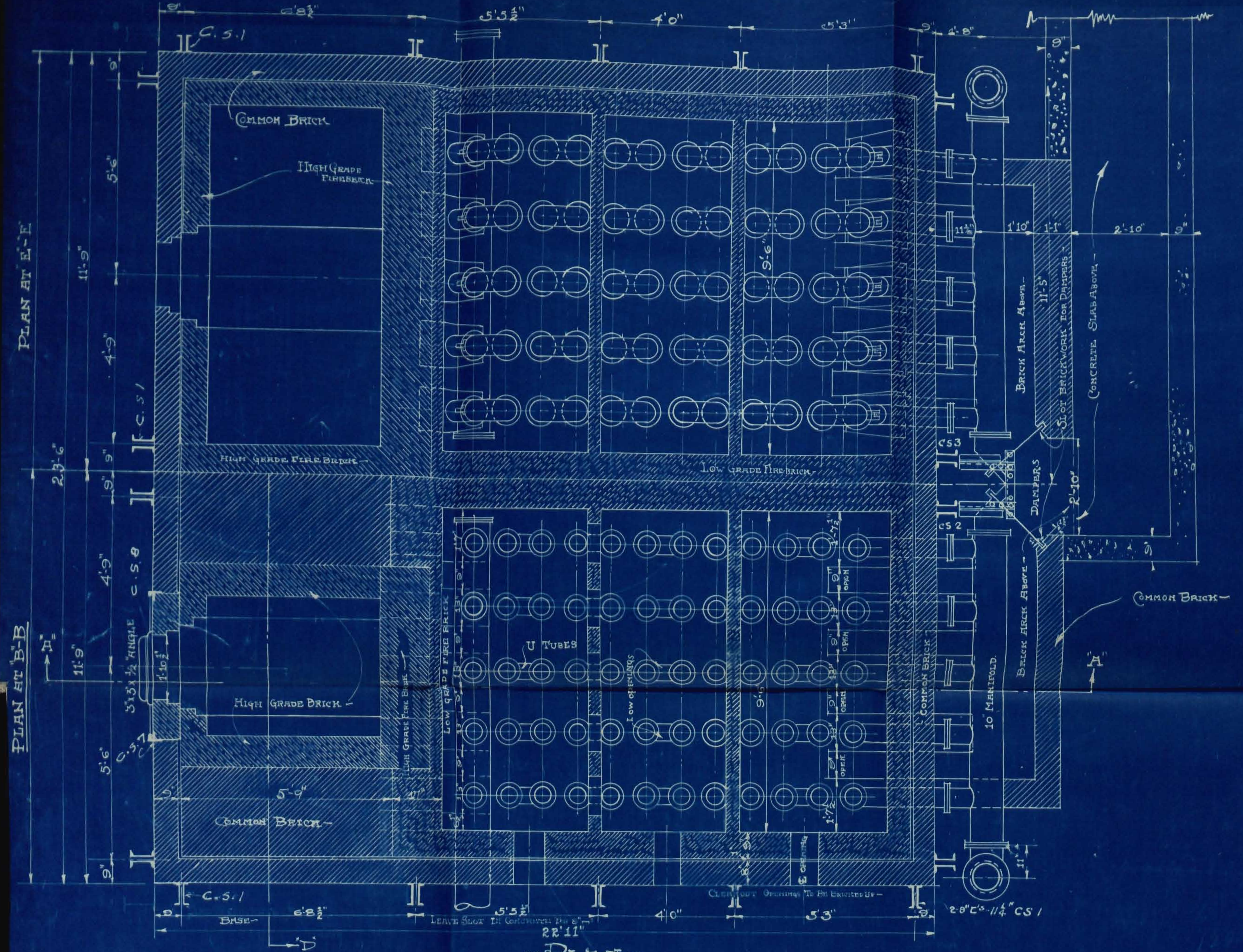
SECTION "D-D"

SECTION B-B.
SCALE - 3/8" = 1'-0"

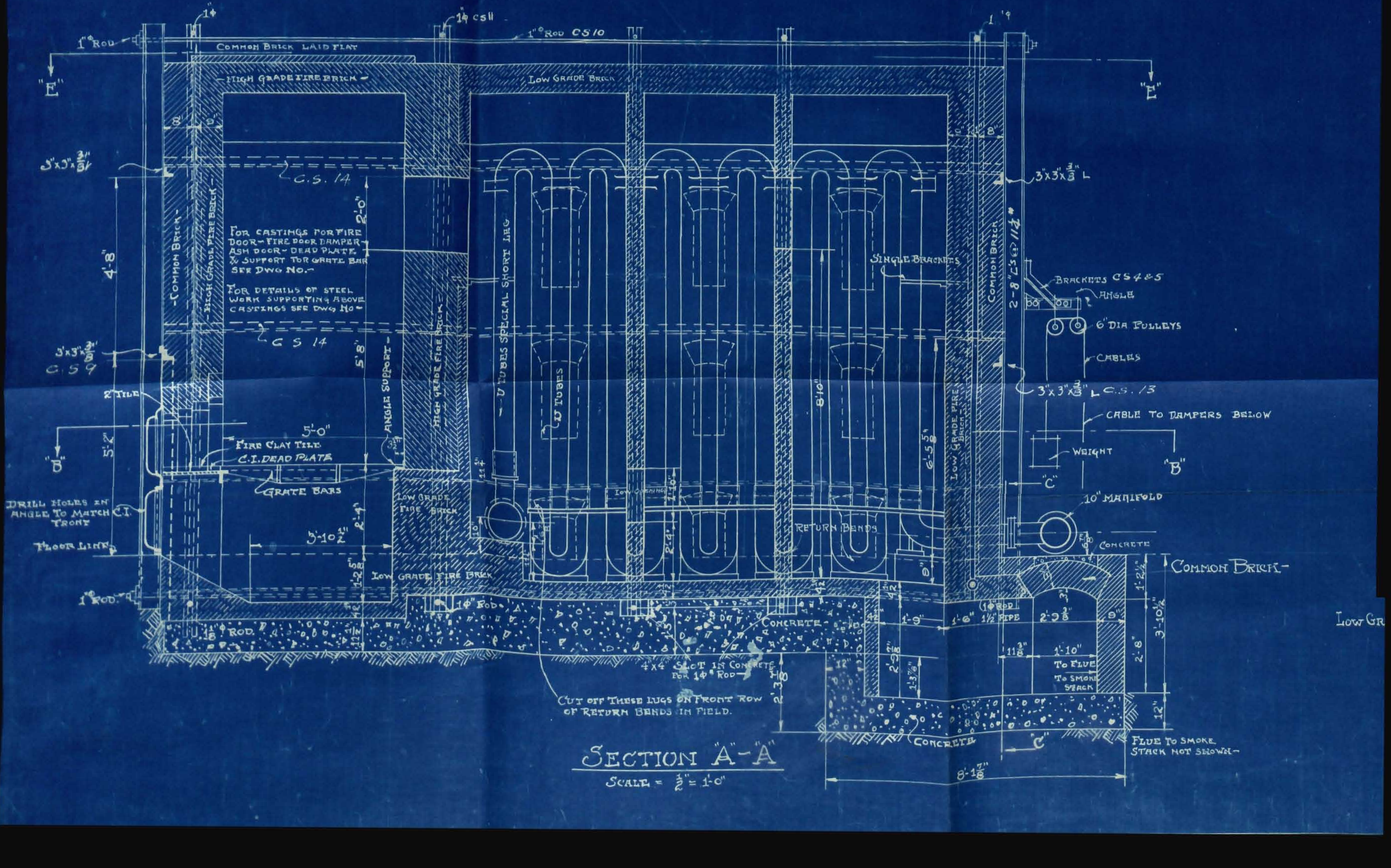
DIAGRAM OF

NOTE:-
ALL TIMBER USED IN TOWER TO BE OF LONG LEAF YELLOW PINE.
BRICKWORK LAID UP IN SILICATE OF SODA & SILICA SAND.

Details of Preheaters.



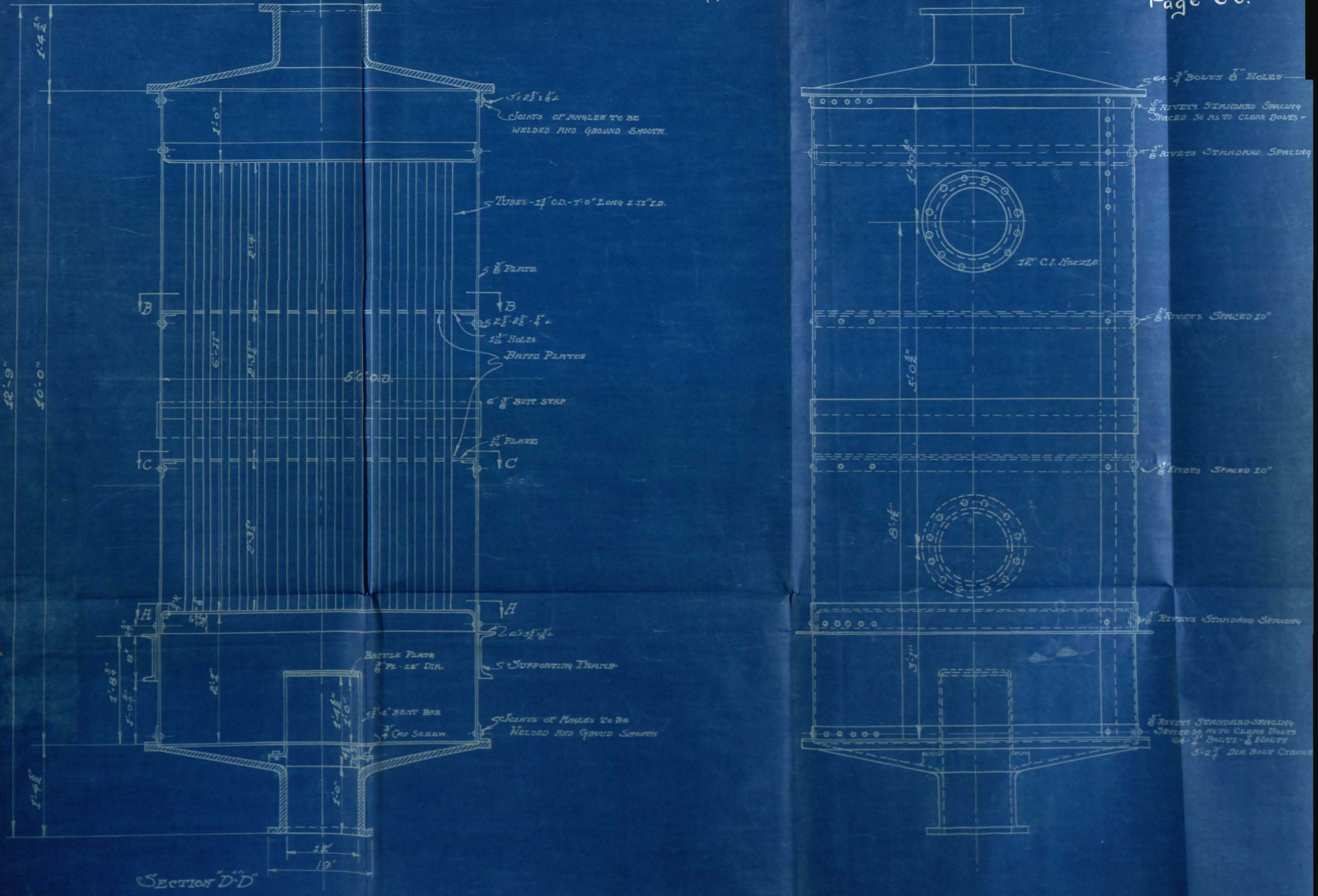
-PLAN-



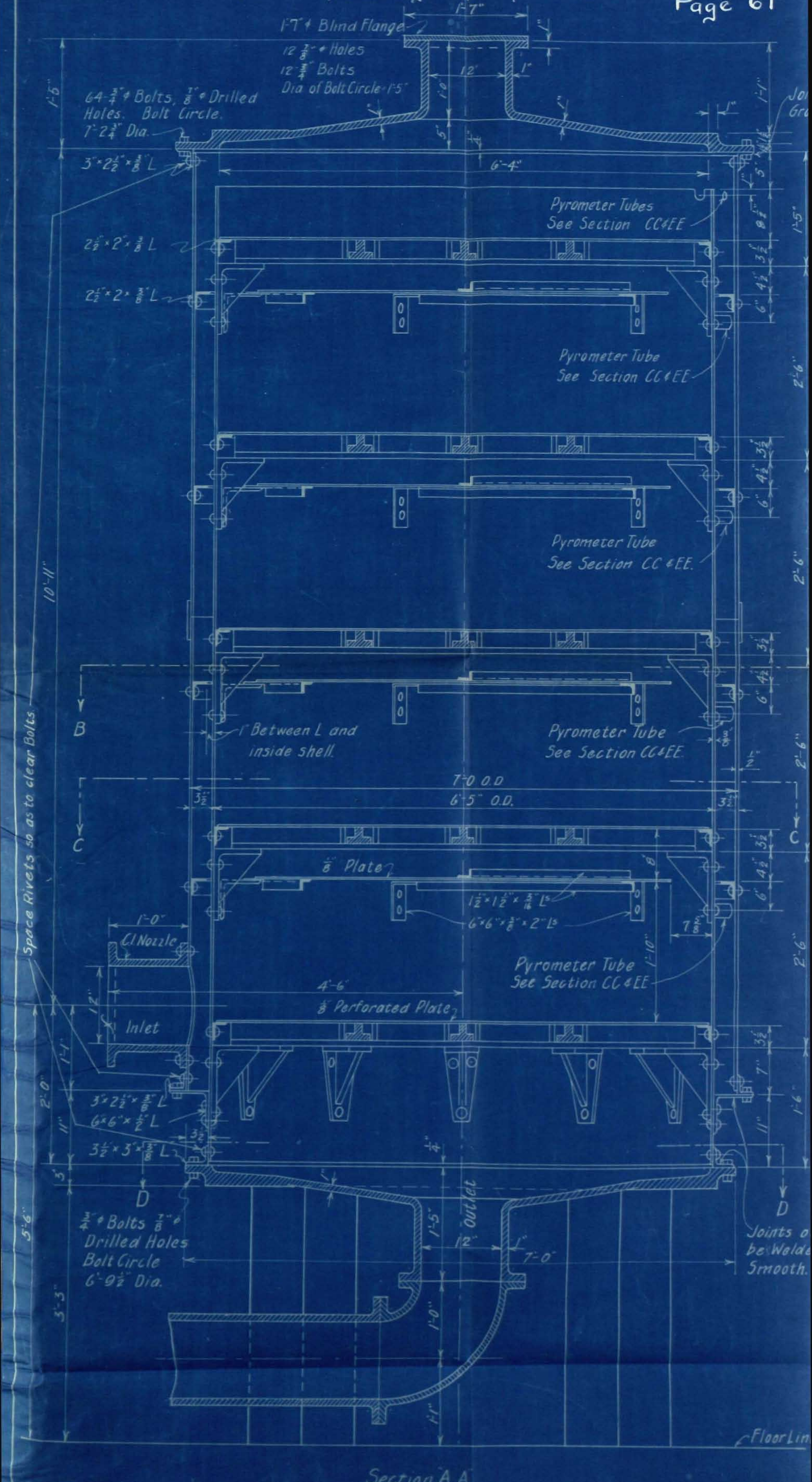
SECTION "A"-"A"

SCALE = 1/2" = 1'-0"

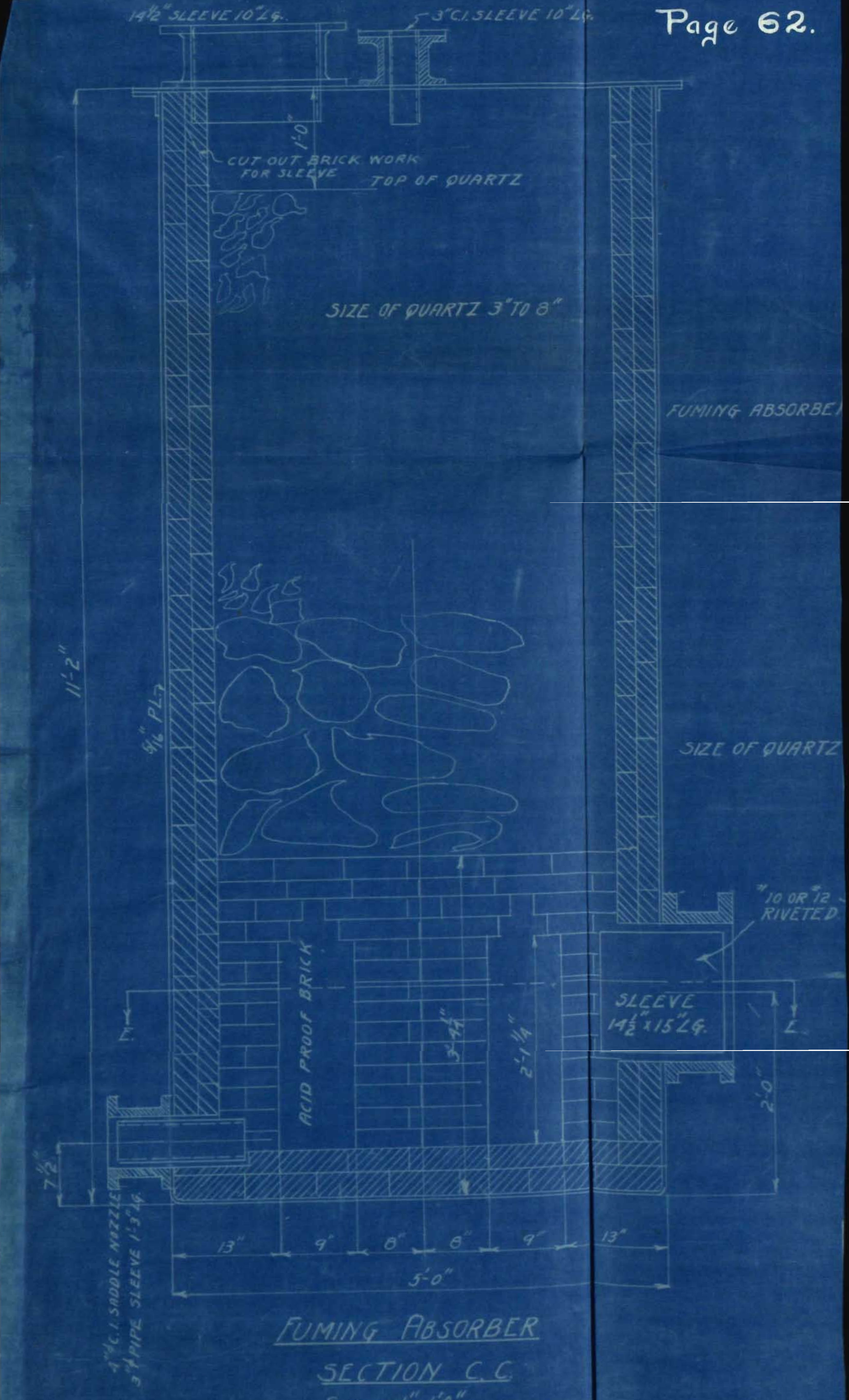
Details of Heat Exchanger



Detail Drawing of Converter



Section A.A



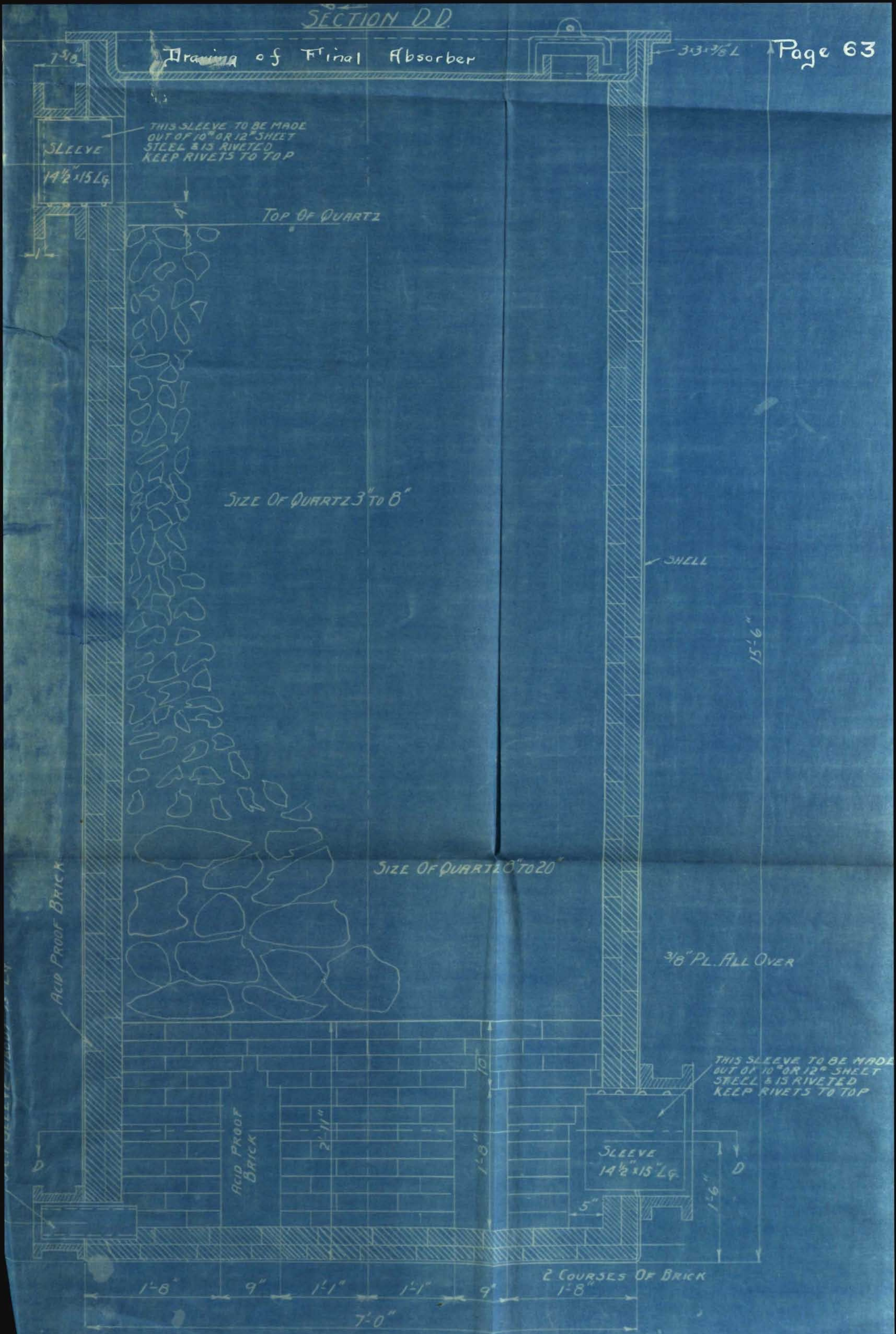
FUMING ABSORBER

SECTION C.C

SCALE 1"=1'0"

S ARE APPROXIMATE & SUBJECT
BE SELECTED BRICK TO BE LAID
SILICA SAND

Drawing of Final Absorber



Index.