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SUBJECT

THE PRODUCTION OF POTASSIUM FROM SODIUM AND POTASSIUM CARBONATE ON A PILOT PLANT SCALE

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NAVAL RESEARCH LABORATORY

BELLEVUE, D. C.

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NAVY DEPARTMENT

Fourteenth Partial Report
on
Oxygen Source Material.

The Production of Potassium from Sodium and Potassium Carbonate on a Pilot Plant Scale.

NAVAL RESEARCH LABORATORY
ANACOSTIA STATION
WASHINGTON, D. C.

Number of Pages: Text - 26 Plates - 21

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TABLE OF CONTENTS

APPENDICES

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ABSTRACT

The production of potassium from the reaction of sodium with solid potassium carbonate at reduced pressure is chemically and mechanically sound. A pilot plant has been erected at the Naval Research Laboratory which has clearly demonstrated this fact. The process employed is a combination of reaction and fractionation carried out in an oxygen-free, closed system at a pressure near 50 mm of mercury. Sodium vapor is boiled through a vertical column (6' high and 7" diameter) of porous lumps of potassium carbonate. The sodium reacts with the potassium carbonate and frees the more volatile potassium which moves upward through the column and is eventually condensed and collected in the receiver. The proper regulation of the process is dependent on the temperature distribution along the potassium carbonate column. The process is safe even though air be admitted to the reaction system during a run. From this pilot plant unit, potassium (ave. 95 wt%K) can be produced at a rate of 31 lbs per 5-1/4 hour cycle per column. Pure potassium can be produced, if desired, at a slightly slower rate. The cost of potassium produced on a commercial scale by this process should not exceed $1.00 per pound.

PERSONNEL

The majority of the design, construction, and operation of the pilot plant was accomplished by the efforts of the following men:

A. S. Lehmann (Chem. Engr. in Chge.)
J. A. Barclay
L. M. Foster
J. G. Coli
W. D. Wessinger
S. L. Belknap
J. B. Mack
S. L. Walters
W. S. Wooster
J. E. Whitney

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AUTHORIZATION

1. Bureau of Ships Project Order 243/42, S77-2(275), dated 30 September 1941.

STATEMENT OF PROBLEM

2. General, "A Study of the Chemical Sources of Oxygen". This investigation concerns the chemical and mechanical soundness of producing pure metallic potassium from sodium and potassium carbonate at reduced pressure on a pilot plant scale.

PREVIOUS WORK ON PROBLEM

3. The most promising method of producing metallic potassium as stated in Report P-2011 entitled "An Investigation of Methods for the Production of Metallic Potassium" was from the reaction of sodium with solid potassium carbonate. This conclusion was based on a thorough laboratory scale research of the available potassium salts and reducing agents, and with due consideration of the economic and mechanical factors involved.

4. A small scale pilot plant was constructed at the Naval Research Laboratory, reported in P-2026 entitled "The Production of Potassium from Potassium Carbonate" which verified the chemical and mechanical soundness of the reaction from the small scale viewpoint.

5. The production of the desired end product, potassium tetroxide, from metallic potassium as carried out with potassium made in the plant described in this report may be found in Report P-1999 entitled "The Burning of Potassium Metal to Potassium Tetroxide with a DeVilbiss Spray Gun".

THEORETICAL CONSIDERATIONS

6. This pilot plant unit has been constructed to produce metallic potassium by the reaction

$$K_2CO_3 + 2Na \rightleftharpoons 2K + Na_2CO_3.$$

The application of this reaction is unique in that it combines chemical reaction with fractionation in such a manner that essentially pure potassium results instead of a sodium-potassium alloy of equilibrium composition.

7. The process as employed in this pilot plant is to pass sodium vapor upward through a column of porous lumps of potassium carbonate in such a way that the sodium vapor and condensed sodium react with the solid K_2CO_3 and liberate the more volatile potassium, which proceeds upward and is ultimately condensed and collected in the receiver. By the process of fractionation, the less volatile sodium is left behind until it reacts with the K_2CO_3 and distills over as potassium.

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8. The reaction is carried out in a closed system in the absence of air, and at reduced pressure (normally 50 mm of Hg). Induced reflux is employed to the extent necessary to assure adequate rectification of the effluent vapor at a practical production rate. The porous lumps of K_2CO_3 serve not only to react the sodium in the rising vapor, but also to offer a surface for condensation similar to packing normally used in a fractionating column. The combination of these effects yields essentially pure potassium.

9. Theoretical quantities of reactants, i.e. one atom of sodium per equivalent of K_2CO_3, can be processed with yields (based on sodium) averaging over 90%.

10. The heat of reaction in as far as can be observed is slightly exothermic, but for most purposes may be considered to be zero.

11. Hereinafter, the term salt will refer to potassium carbonate, and alloy to sodium-potassium alloys. A table of the more important physical properties of these substances will be found in Appendix VI.

RAW MATERIALS

12. Potassium Carbonate. A commercial grade of "Granular Calcined Regular Carbonate of Potash" which averages greater than 98% K_2CO_3 is prepared by a method described in Appendix III in porous lumps 3/4 - 1/4 mesh and thoroughly dried. Potassium carbonate in less carload lots costs around 7 cents a pound delivered.

13. Sodium. A "commercial quality" untrimmed brick 2-1/2 ± 1/4 lb. each of sodium packed 280 lbs to a steel barrel is purchased from Electrochemical Division of DuPont Co. at a cost of 14.26 cents per pound. Since it is melted and filtered before using it is quite satisfactory for use in the pilot plant.

14. Nitrogen and Other Gases. Nitrogen, water pumped, is obtained from Southern Oxygen Co. in steel cylinders containing 200 cu.ft. of gas. The oxygen content is normally less than 0.2% and since it is further purified in a deoxidizing furnace, it has proved quite suitable for pilot plant use. Approximately 10 cylinders are used per month at a cost of $3.50 per cylinder. Other gases such as oxygen, acetylene and hydrogen are used in relatively small quantities and are obtained from the NRL shop.

CONSTRUCTION OF THE PLANT

15. The pilot plant is housed in a large room 60' x 40' x 23' fitted with three equally spaced 60' monorails on the ceiling from which hoists may be suspended. The operating line of the pilot plant which consists of the preheater-dryer, two identical reaction columns, and the after-cooler is situated directly under the central monorail so that it is readily accessible to

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the hoists. (See Plate 1 & 2) The operating line units are supported by a steel superstructure fitted with a top deck (see Plate 3) 18' x 5'3" which is 11'3" from the floor level and convenient to the top openings. The location of the control panel with respect to the operating line is shown in Plate 4.

GENERAL DESCRIPTION OF EQUIPMENT

16. A more detailed description of the equipment described below may be found in Appendix I.

17. Preheater-Dryer. This unit consists of cylindrical electric furnace situated on top of an oil fired furnace box so that the top opening of the electric furnace is flush with the top deck. The electric furnace is equipped with an inner sleeve large enough to accommodate a potassium carbonate container. The bottom of this sleeve is hemispherical and is fitted with a pipe which leads directly into the oil fired furnace. Compressed air is passed through a helical coil in the oil fired furnace box and into the bottom of the electric furnace sleeve where it is forced upwards through the potassium carbonate thereby heating and drying it. (See Plate 5)

18. Reaction Columns. Two identical reaction columns are set up in the operating line. Each originally consisted of a stainless steel tubular column with an increased diameter at the top to accommodate the top closing device, a cylindrical stainless steel potassium carbonate container which fits concentrically into the column, a condenser arm, and a receiver to collect the distillate. (See Plate 6)

19. The original columns were fabricated from Type 302 stainless steel rolled and welded. The lower portion was 8' high and 8" in diameter x 1/8" wall. Baffle rings were welded at 1 foot intervals along the exterior of the column to reinforce it and reduce convection between the column and the furnace windings. The upper portion of the column was of enlarged diameter to accommodate the original top closing device and provided with a secondary seal as shown in Plate 6. The primary seal was formed by a tongue and groove lead seated flange secured by six swing bolts.

20. The original top closing device was found to be more elaborate than necessary and was discarded in favor of the "New Top Closing Device" as seen in Plate 7. The new closure eliminates the secondary seal and the flexible bellows formerly employed, and has a superior type reflux condenser.

21. Approximately 18 inches above the bottom of the reaction column an annular ring was welded to the inside walls. The top side of this ring was machined to a spherical radius and serves to seal and support the potassium carbonate container in the column. The portion below the ring is known as the sodium boiler. Liquid sodium is introduced into the column through a pipe welded on the side of the sodium boiler and protruding

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through the wall of the column furnace.

22. The potassium carbonate containers are 6' high and 7" in diameter holding about 60 lbs of K_2CO_3. They are fitted with a lifting bar on top and a screen and a spherical seat on the bottom which allows the sodium vapor to pass upward through the K_2CO_3 but not to leak around the edge of the container.

23. A condenser arm was welded to the column slightly above the top of the salt container and runs out and down into a receiver where the distillate was collected. The receiver was fitted with a filter and a vacuum valve on the lead to the vacuum pumps and has suitable connections to the nitrogen system and to the closed manometer. The last four feet of the condenser arm were electrically heated to keep the potassium produced in the liquid state.

24. The steel columns (see Plate 14) which replaced the stainless ones differ only slightly from the original design. The body of the column is fabricated from 8" seamless steel pipe and is the same diameter throughout. The salt containers are supported in the same fashion, and the same "new top closure" is used. The condenser arm and receiver have been salvaged from the old stainless columns and used on the new steel ones.

25. Reaction Column Furnace. The weight of the column is supported by a firebrick lined and insulated furnace box which also holds the four Globar heating elements horizontally below the sodium boiler. Each Globar is rated at 2.0 kw, the four being connected in series.

26. The furnace box supports the cylindrical, longitudinally split electric furnace that supplies heat to the sides of the reaction column. (See Plate 6) The electric furnace has nine individually controllable heating units, each approximately 1' high and of 2.0 kw capacity. Thermocouples are provided along the column furnace, in the sodium boiler, and in the effluent vapor to give a complete description of the temperature behavior during a run.

27. After-Cooler. (See Plate 8) This unit serves to cool the hot salt containers when they are removed from the reaction column. It has two openings flush with the top deck that can accommodate two hot salt containers and is equipped with a blower and a vent to carry off any smoke that may be present. This unit serves as a convenient quick way of disposing of the hot shell thereby reducing the hazard to the operator. The salt containers are readily removed and dumped when cool.

28. Control Panel. (See Plate 9 & 4) The control panel is divided into three parts. The preheater-dryer is controlled by the left portion, the Number Two reaction column by the middle and the Number One reaction column by the right. The panel is arranged with a time clock so that the above units may be turned on automatically at any desired time.

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29. In the left panel a Brown Recording Potentiometer maintains the proper temperature of the electric preheating-drying furnace and controls the temperature of the drying air by interrupting the circuit to the oil burner.

30. The reaction column control panels are identical. Each has a Brown Recording Potentiometer (5 point recording, one controlling) which records the desired column temperatures and records and controls the boiler temperature continuously.

31. Voltmeters, ammeters, and pilot lights are provided to indicate the proper performance of the electrical equipment. The dials visible in Plate 9 indicate the settings of the variable auto-transformers delivering power to the various sections of the column furnace and auxiliary equipment.

32. Sodium Injection System. (See Plate 8 & 11) The sodium injection system consists of a melting pot, metering tube, lead pipe, and valve into the reaction column. The melting pot is a cylindrical steel tube with a conical bottom fitted with a pressure tight cover, a 2 kw immersion heater, a screen filter in the bottom and a thermocouple well. The exterior is lagged and a hole in the cover can be opened to introduce blocks of sodium when charging (capacity 75 lbs). The bottom of this pot is connected to bottom of the metering tube by means of 1/2" copper tubing and a cock and tee. The metering tube is 6" seamless steel about 36" long and is fitted with a cover, thermocouple well and insulated movable contact wire. The outside of the meter is wound with resistance wire for heating and lagged. The leads to the reaction column run from the tee at the bottom of the meter to the needle valves on the sodium injection pipes of the reaction columns.

33. Nitrogen Purifying System. (Plate 11 & 12) It is desirable to make sure the nitrogen used in contact with the metallic potassium is free of oxygen and water. To accomplish this, water pumped nitrogen from cylinders is passed through hot copper (450°C) and then over lump potassium hydroxide and activated silica gel before it comes in contact with the alkali metal. Since the nitrogen used is quite pure initially, this treatment removes the final traces of water and oxygen effectively.

34. Vacuum System. The vacuum is produced by two rotary vacuum pumps, one for each column. Each has a 2 hp motor, and a rated capacity of 50 cfm. The pumps are connected to the system by means of 2" galvanized pipe with suitable fittings. Glyptal is used to make the joints tight. Each pump will evacuate its system to a pressure of 1 mm of mercury in about 30 seconds.

35. Salt Preparation Equipment. (See Plate 13) The preparation of the potassium carbonate is carried out in a 25' x 14' x 11' room immediately adjacent to the plant location. The equipment consists of two hot air, tray type, drying ovens,

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a grinder, and a vibrating screen. The ovens are heated by a steam coil blower unit and each can dry 350 lbs of potassium carbonate at a temperature of 125°C at the same time.

36. The grinder is a rotating pick type and breaks the dried cakes into lumps at a rate of 600 - 700 lbs/hr. It feeds directly onto a vibrating screen which classifies the broken material to the desired size. The vibrating screen is a conventional single or double deck type actuated by an eccentric and equipped with various sizes of interchangeable screens.

37. Auxiliary Equipment. Three 1/2 ton electric chain hoists mounted on hand geared trolleys, one on the underhung crane and two on the central monorail, serve the operating line equipment and handle the hot salt containers in a very satisfactory manner. The laboratory in which the pilot plant is housed is fitted with the normal services, air (100 psi, 100 cfm capacity), gas, water, steam (100 psi, 30 hp), AC electricity (4 wire 208-120 v system 50 kw capacity), and 110 volt direct current.

OPERATION OF THE PLANT

38. Personnel. Because of the dangerous and unusual properties of the materials being handled, it was considered advisable to employ men with previous chemical training. The staff has averaged six men throughout the construction and operation of the plant, most of them possessing a B.S. in Chemistry.

39. Operating Procedure. The general procedure used in making potassium metal in this plant is briefly as follows. (For details of temperature, power, etc. see Appendix VI) A container is filled with potassium carbonate and placed in the preheater-dryer for a final drying. This operation takes place while another run is in progress. At the termination of the run in progress the new hot salt container is placed in the hot reaction column and the top secured. The reaction column is then pumped to a pressure of about 1 mm of mercury and checked for leaks. Nitrogen is introduced to a pressure somewhat in excess of the proposed operating pressure and the measured charge of sodium is injected from the sodium melting and metering device into the boiler of the reaction column. The pressure is adjusted to the desired value (normally 50 mm). The charge of sodium is heated to boiling and allowed to boil until the boiler is dry, as is readily indicated on the Brown Instrument recording the temperatures. Any induced reflux is obtained by forcing air through the reflux condenser in the top closure and is normally turned on when potassium vapors reach the top section of the salt column. After the sodium boiler has boiled dry, the reaction column is maintained at the operating pressure and temperature for about 30 min. In this period a fair amount of potassium distills over which increases the yield on the first cut. Nitrogen is introduced into the reaction column to a pressure of about 4 psi and the first cut sample forced from the

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receiver into an auxiliary container where it is weighed and sampled. The run is terminated by pumping the reaction column slowly (about 3mm/min) to the ultimate vacuum obtainable in the system and allowing it to remain there until the condenser arm begins to cool, indicating there is no more metal distilling over (about 30 minutes). The system is again filled with N_2 and the second cut is taken as described above. The top closure is opened, the spent salt container removed and placed in the after-cooler, while a new hot salt container is being placed in the reaction column in preparation for the next run. The complete cycle requires about 5-1/4 hours under normal operating conditions.

EXPERIMENTAL DIFFICULTIES

40. Vacuum Leaks. In any vacuum process a certain amount of care must be exercised to keep the system free from leaks. Maintaining a good vacuum in the reaction system has not been nearly as serious a problem as the structural failures resulting from poor technique in the fabrication of the original reaction columns. The use of tongue and groove flanges with lead gaskets has proven quite satisfactory. Ordinary pipe fittings, unions (carefully ground) and solder fittings have all performed very well under vacuum conditions.

41. Structural Defects. The first two reaction columns were of Type 302 stainless steel and of rolled and welded construction. By far the largest amount of time lost was in repairing failures along the welded seams of these columns. Apparently either flux was trapped in the original weld which gave rise to pin hole leaks, or the parent metal was not sufficiently penetrated by the weld, causing thin weak spaces which tended to crack. The bulk of the difficulties encountered with the stainless steel columns could have been avoided had a seamless tube been available. On examination of the columns after removing them from service (#1-500 hrs, #2-700 hrs), the internal corrosion appeared to be considerable. In the lower portion of the column as much as 1/32" of the wall had been removed by corrosion. It is suggested that if future equipment of this nature is to be made of stainless steel, a different alloy be employed perhaps Type 321 or Type 347.

42. The mild steel columns currently in use have exhibited no faults other than the anticipated amount of external scaling and seem to be quite practical. A smaller degree of internal corrosion is expected although the columns have not been in operation long enough to draw any definite conclusions regarding their life.

FACTORS INFLUENCING THE REACTION

43. Temperature. The successful production of potassium metal by this process is dependent on the proper temperature distribution along the reaction column. In order to obtain pure potassium at a high production rate, it is important that the

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heat input be carefully controlled. This is readily accomplished in the present set-up by means of "variacs" (variable auto-transformers) furnishing power to the column furnace.

44. The lower two feet of the potassium carbonate column (6 feet total) are preheated in the reaction column to a temperature close (+ 0, -15°C) to the boiling point of sodium at the operating pressure. The middle two feet are preheated to a temperature near (+ 10°C) the boiling point of potassium. The top two feet of salt are heated to approximately 40° below the potassium boiling point. As the sodium vapor rises and reaction takes place, the column assumes the temperature of the boiling point of the alloy of the particular composition present in that zone. The purpose of this temperature distribution is to afford the sodium-potassium vapor as rapid as possible transit through the lower portion of the salt column, yet maintain the upper portion equal to, but not greater than the boiling temperature of pure potassium so that only pure potassium vapor can distil through. Initial reflux is made certain because all of the heat necessary to raise the temperature of the top zone must come from the heat of condensation of the potassium; and once the potassium has condensed in a liquid film on the surface of the salt, fractionation is assured.

45. Were the lower sections of the salt column preheated to a value much higher than that suggested, the highly superheated potassium vapor would rise at such a rapid rate that the upper section would become overheated, proper fraction destroyed, and poor quality alloy would result. Preheating the lower sections to a lower temperature would result in time lost raising them to their equilibrium value and hence a slower production rate. Heating the upper sections of the salt column above the potassium boiling temperature would destroy the liquid potassium phase, and thereby upset normal fractionation which would yield lower purity potassium alloys. Insufficient heating in the upper sections slows the production rate and shows no particular advantages.

46. Typical temperature distribution and power requirements can be found in Appendix VI.

47. Pressure. The actual operating pressure at which the reaction is carried out determines the boiling point of the sodium and potassium and hence the preheating temperature for the salt column. The absolute value of the pressure is not as important to the success of the reaction as is the maintaining of that pressure at a constant value throughout the run. Lower pressures (10 - 30 mm) favor the fractionation part of the process since in this region the ratio of the vapor pressure of potassium to sodium is increased. The accompanying lower operating temperature is attractive from the point of view of life of the equipment and the heat required. However, the chemical equilibrium of the reaction seems to be most favorable around 550°C which would set the operating pressure in the neighborhood of 50 mm. The greater vapor density at the higher pressures and

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hence lower linear velocity of the vapor through the salt column favors operation in that region. Further, it is somewhat easier under normal operating conditions to maintain the pressure constant in this higher pressure region. The upper limit, however, is set by the minimum melting mixture of Na_2CO_3-K_2CO_3 system at 705°C. This fixes 50 mm of mercury as the highest safe operating pressure. The majority of the runs reported here have been made at 50 mm pressure.

48. Reflux Ratio. This factor is defined as the ratio of the potassium returned to the column to the total potassium rising and is calculated indirectly since there is no accurate means of measuring the return in this unit. The independent effect of the reflux ratio is difficult to isolate because it is effected by so many other factors, however, it is certain that enough reflux must be employed to assure the presence of a liquid film of potassium in the top of the salt column. The reflux may be of the normal type, that is, vapor condensed to compensate for the normal heat losses of the column; or it may be the induced type, that is, reflux controlled by forcing air through a reflux condenser fitted inside the column. In the case of the stainless steel columns, normal reflux was not adequate to assure good fractionation so induced reflux was required to bring the average reflux ratio for the first cut to approximately 0.25, a value that gave high purity potassium at a reasonable production rate. The mild steel columns currently in operation have considerably higher normal reflux than the stainless because of their higher thermal conductivity with the resulting loss of more heat conducted out the top of the column. For this reason, sufficient reflux is obtained by normal means without the use of induced reflux. Greater reflux than that suggested causes excessive hold-up in the column with the resulting lower yield in the first cut and shows little improvement if any in the purity of the potassium produced for the extra time consumed.

49. Equivalent Ratio. This is the ratio of the atoms of sodium employed for the reaction to the number of equivalents of potassium carbonate used, 1:1 would be the theoretical maximum. Ratios less than this indicate that not enough sodium was used to completely react the potassium carbonate present with the resulting loss of economy. Ratios greater than 1:1 can be carried out with a somewhat higher yield of slightly poorer quality potassium, but time and power are required to remove the excess sodium from the spent salt. The effectiveness of this process clearly is demonstrated by the satisfactory reaction of theoretical quantities of reactants with yields averaging over 90%. The ultimate operating ratio will be determined by the economics of the process when the desired purity of potassium is established by the consumer since this ratio does not effect the mechanics of the reaction.

50. Rate of Sodium Evaporation. This factor is expressed as the rate (in lbs/hr) of sodium leaving the boiler and is calculated from the time the sodium boils until the boiler is dry. The rate is dependent primarily on the heat available in the

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immediate vicinity of the boiler and to a smaller extent on the reflux ratio and the size of the salt particles. The greatest portion of the total heat consumed for the reaction is used in boiling the sodium (0.57 kw-hr/lb). For the units described here (7" diam. salt column) a boiling rate of 7 to 8 lbs/hr is as fast as is consistent with the production of high purity potassium. Converting this rate to unit area of salt column it becomes 0.2 lbs/hr/sq.in. salt area. This unit evaporation rate may be considered a safe mean value for purposes of designing larger diameter salt columns. Faster rates have been run with a resulting poorer quality potassium and a decreased yield in the first cut. Too rapid rates tend to overheat the upper sections of the salt column and the effectiveness of the fractionation is destroyed as described earlier. Slower rates give a somewhat increased yield in the first cut but not enough to warrant the extra time consumed. High reflux ratios and fine salt (less than 1/4 mesh) both tend to decrease the rate of sodium evaporation.

51. Size and Condition of Potassium Carbonate. The potassium carbonate used in this process is prepared in a manner described in Appendix III. It has been determined that lumps ranging from 3/4" to 6 mesh (density = .55 - .65) are most satisfactory for the reaction. Finer mesh material tends to increase the resistance to flow for the vapor passing through the salt thereby raising the pressure in the boiler and hence the pressure gradient across the seal from the boiler to the salt container and increasing the possibility of vapor leaks in that direction. Coarser salt has a lower apparent density (density = .5 - .6) and results in a smaller charge per run. It is desirable to place about 6" of fairly coarse salt (3/4" - 1/4" mesh) in the bottom of the salt container. This reduces the tendency for the potassium carbonate, which becomes tacky at reaction temperatures, to mat and stick together and thereby increase the resistance to vapor flow.

52. Care should be exercised that the salt be absolutely dry since the presence of water (greater than 0.3%) in the system is highly undesirable. The preheating-drying operation as described earlier has proved very satisfactory for removing the last traces of water from the salt.

53. It is interesting to note the average distribution of the mixed carbonates in the spent salt since this provides the key to the completeness of the reaction.

TABLE ONE

AVERAGE DISTRIBUTION OF MIXED SODIUM-POTASSIUM CARBONATES
IN THE SPENT SALT. RATIO = 1.0

Distance up From Bottom	Ave. Mol % K_2CO_3	Ave. Wt % NaOH
* 4 feet	49.2	2.3
3 "	17.6	1.8
2 "	4.3	0.9
1 "	1.2	0.5
0 "	0.5	2.4

* Salt shrinks approximately 18" during reaction.

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The last column is calculated as NaOH but conceivably might be a mixture of NaOH, KOH, Na_2O_2, K_2O_4, K, and Na before the sample is put in solution for analysis.

54. It is evident from the above table that a six foot salt column is high enough to satisfactorily convert the lower sections with enough potassium carbonate in the top section to act as a guard against sodium getting into the first cut. Note the sharp break between the three and four foot samples.

55. Other Factors. The above factors are the principal ones that govern the success of the process. However, there are some minor considerations that must not be overlooked. The amount of air allowed to enter the reaction column should be kept at a minimum since the oxygen reacts with the potassium forming oxides which melt at moderate temperatures and are highly corrosive to the columns. Moisture too must be avoided since it will react with the potassium yielding KOH and H_2. Molten KOH is likewise very corrosive to the equipment, and hydrogen when mixed with oxygen forms a highly explosive combination.

56. It is important that the annular ring within the reaction column which supports the salt container be clean so that no sodium vapor can leak from the boiler into the space around the outside of the salt container and by-pass the potassium carbonate.

57. The absolute tightness of the column to vacuum leaks is desirable but not entirely essential. The reaction can be carried out in a satisfactory manner with as high as 200 cc/min leak into the reaction system with no obviously undesirable effects other than causing the pressure to be difficult to maintain at a constant value and contributing to the corrosion on the inside of the reaction system. This fact is mentioned to stress the safety of the process. There is no danger of explosion should air come in contact with potassium vapor during the course of the reaction.

PRODUCTION RESULTS

58. A detailed table of monthly production may be found in Appendix II. The following table shows the results of eight months' research and production from the pilot plant described herein.

TABLE TWO

POTASSIUM PRODUCTION - TOTAL

Number of runs	173	Potassium produced total	4321 lbs
Potassium Carbonate	10,372 lbs	Potassium (greater 97wt%)	3031 "
Sodium	2,912 "	Potassium (94-97wt%)	1290 "

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59. The above figures are grand totals and include all runs attempted, some of which were not entirely successful due to the exploratory nature of the problem. As a result of this research, operating conditions and technique have been improved to the extent that the following production may be anticipated per run from the unit described.

TABLE THREE

AVERAGE PRODUCTION PER RUN

Potassium Carbonate	60 lbs.
Sodium	20 lbs.
Potassium Total	31.0 lbs. (Ave. 95 wt%K)
Potassium 1st Cut	23.5 lbs. (Ave. 99 wt%K)
Potassium 2nd Cut	7.5 lbs. (Ave. 80 wt%K)
Time Consumed	5.25 hrs.
Labor	11.0 man hrs.
Power	10.0 kw

60. These figures are based on producing a maximum of pure potassium in the first cut in a minimum of time. Should the ultimate purity of potassium desired by the consumer be somewhat lower, operating procedures could be altered slightly and effect a considerable saving in time per run.

COSTS

61. Production costs as presented are not representative of the plant scale cost of potassium because the exploratory nature of this investigation necessarily made the labor and equipment items very high, however, they show an upper limit. The principal is omitted because there is no equitable means of depreciating it at this time. The cost of the labor of the complete staff is charged against each pound of pure potassium (99 wt%K and over) produced; overhead is omitted. Power includes cost of operating a 30 hp boiler in addition to the electric power.

TABLE FOUR

PRODUCTION OF PURE POTASSIUM - PARTIAL COSTS PER POUND

Raw Materials	$ 0.32 per lb
Power	0.08 " "
Labor ($1.00/hr)	0.58 " "
Total	0.98 per lb

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62. Since each pound of potassium burned as described in Report P-1999 produces 1.8 lbs of potassium tetroxide and requires little equipment, labor and power, it is estimated that high purity potassium tetroxide (33.0 wt% available O_2 and greater) can readily be produced at $.80 per pound were potassium metal to be available at $1.00 per pound.

CONCLUSIONS

63. The production of potassium from the reaction of sodium with potassium carbonate at reduced pressure is chemically and mechanically sound.

64. The use of low pressure (50 mm) for this process has presented no major problems. Little time has been lost maintaining the system vacuum tight.

65. The reaction is safe and presents little hazard to an operator exercising normal caution.

66. Type 302 stainless steel reaction columns of rolled and welded construction are not satisfactory since they undergo excessive internal corrosion under normal operating conditions.

67. Present indications are that mild steel seamless reaction columns will prove satisfactory for operation at pressures of 50 mm or less.

68. The success of the process is dependent on the proper temperature distribution along the salt column, and the presence of a liquid potassium phase in the top of the salt column.

69. From the unit described, using 60 lbs of potassium carbonate and 20 lbs of sodium, 31 lbs of potassium averaging 95 wt%K should be produced every 5-1/4 hrs.

70. Costs of production of high purity (99 wt%K and over) on a commercial scale by this process should not exceed $1.00 per pound, which should make potassium tetroxide (33% O_2 and higher) available at around $.80 per pound.

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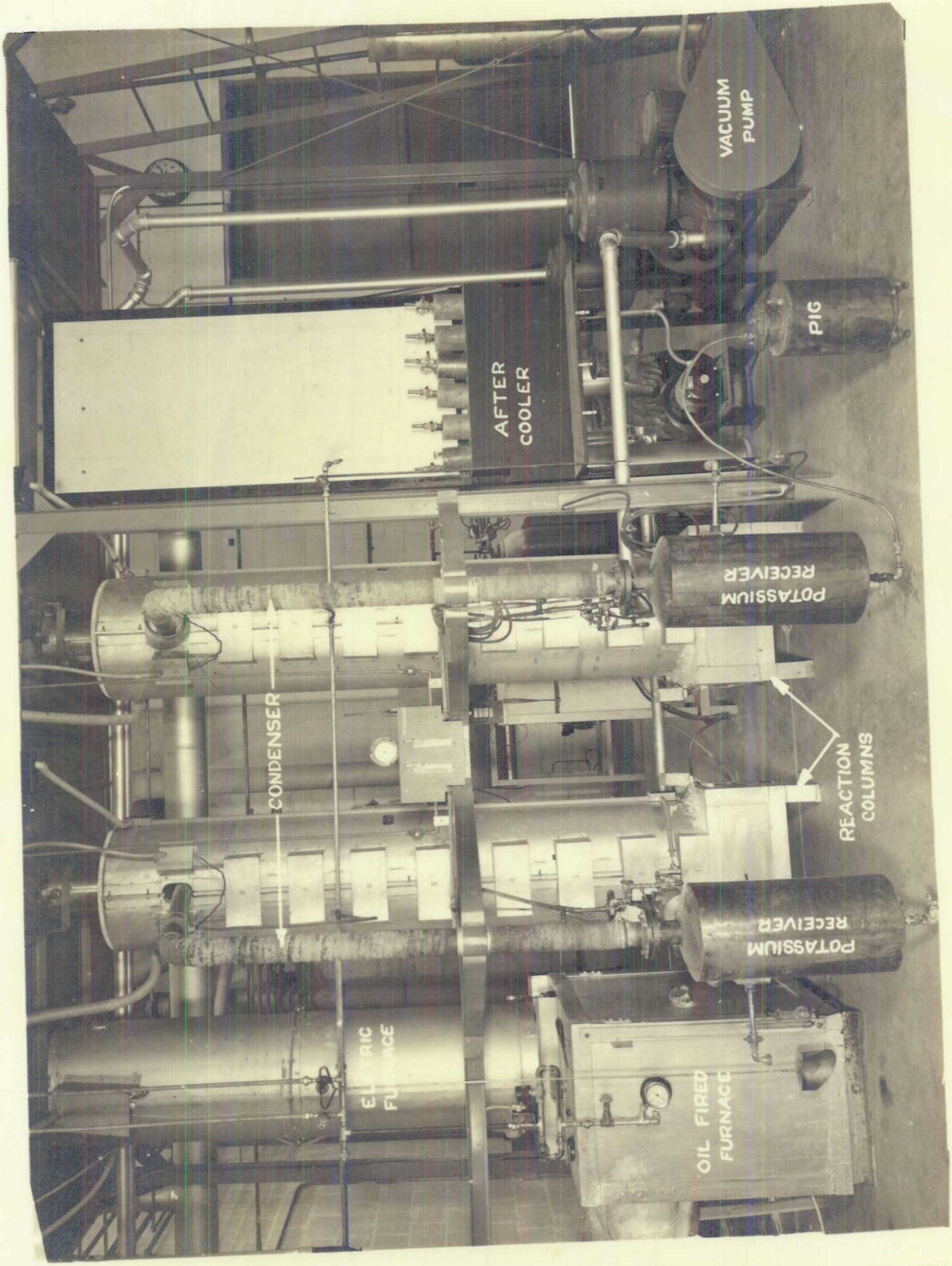

PLATE I

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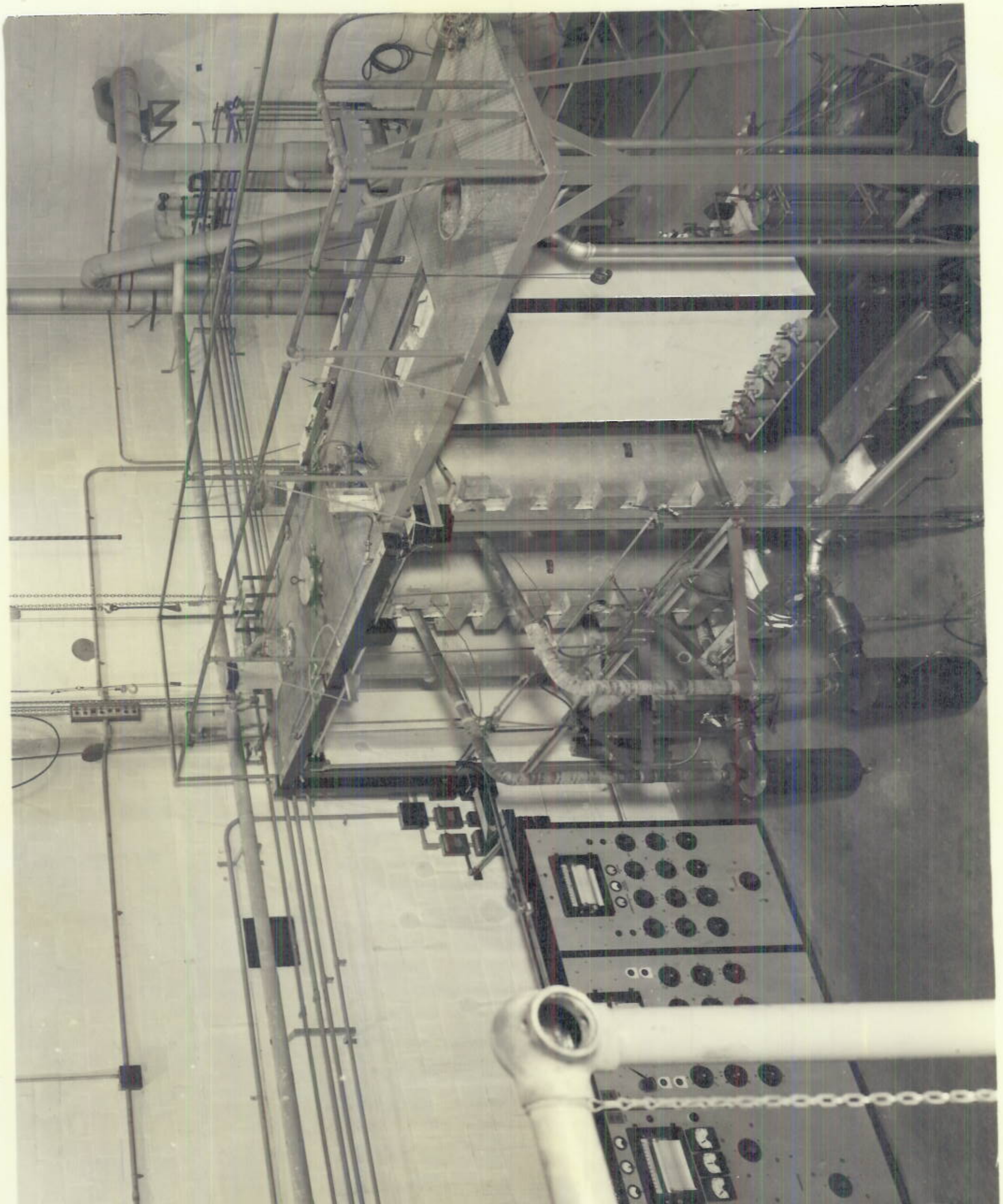

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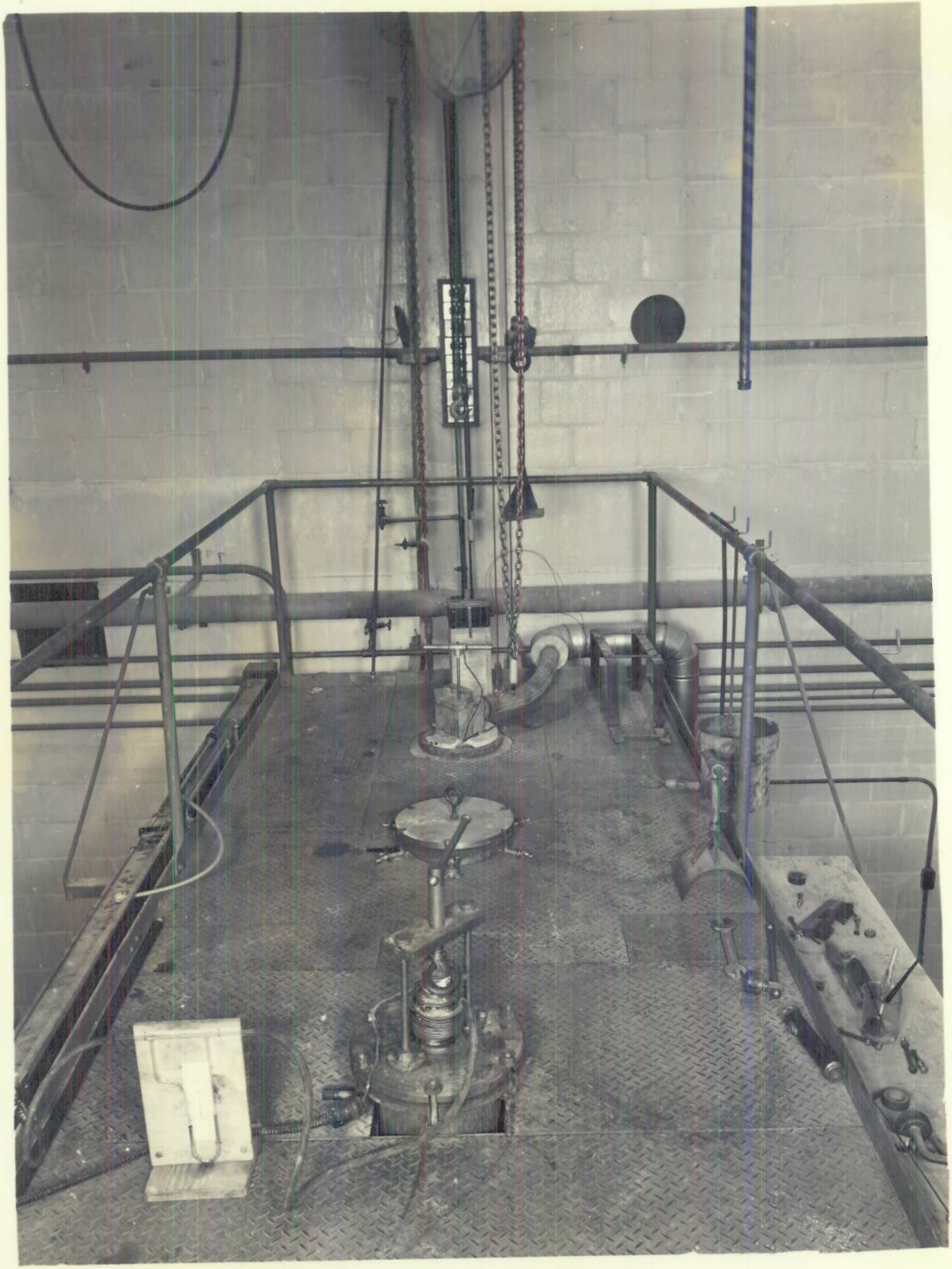
PLATE 2



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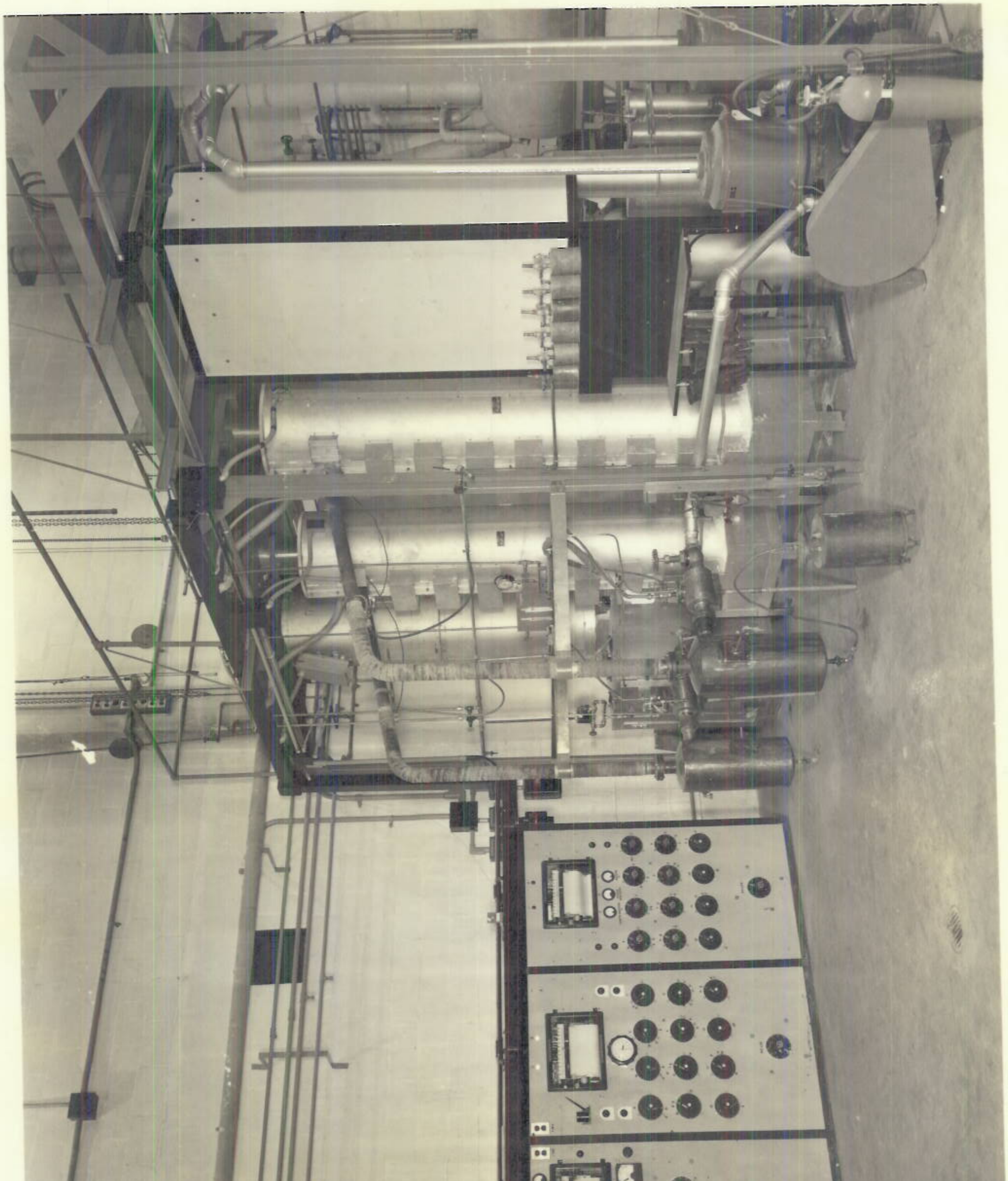

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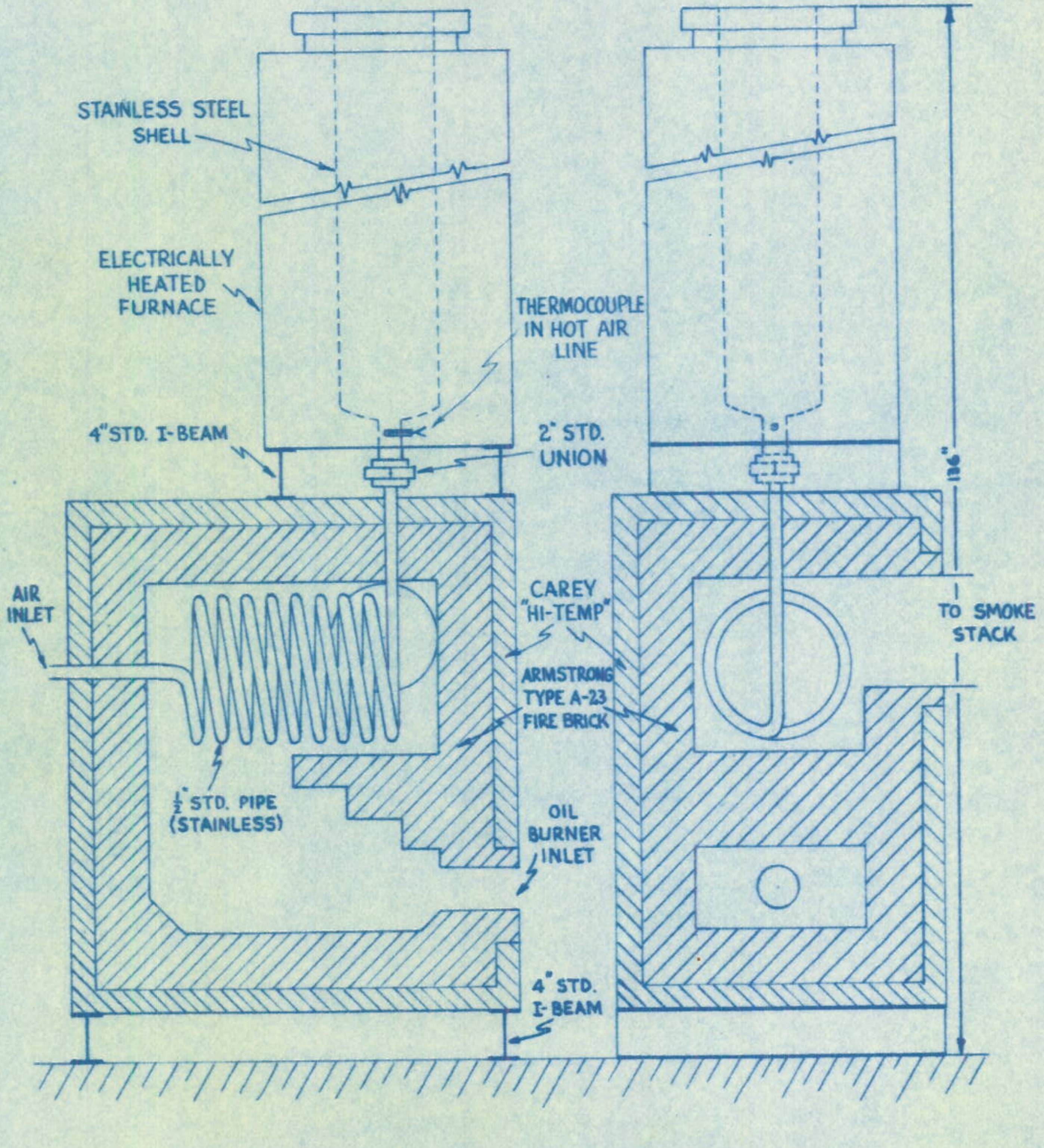
PLATE 4

PREHEATING FURNACE



SCALE 3/32" = 1"

PLATE 5

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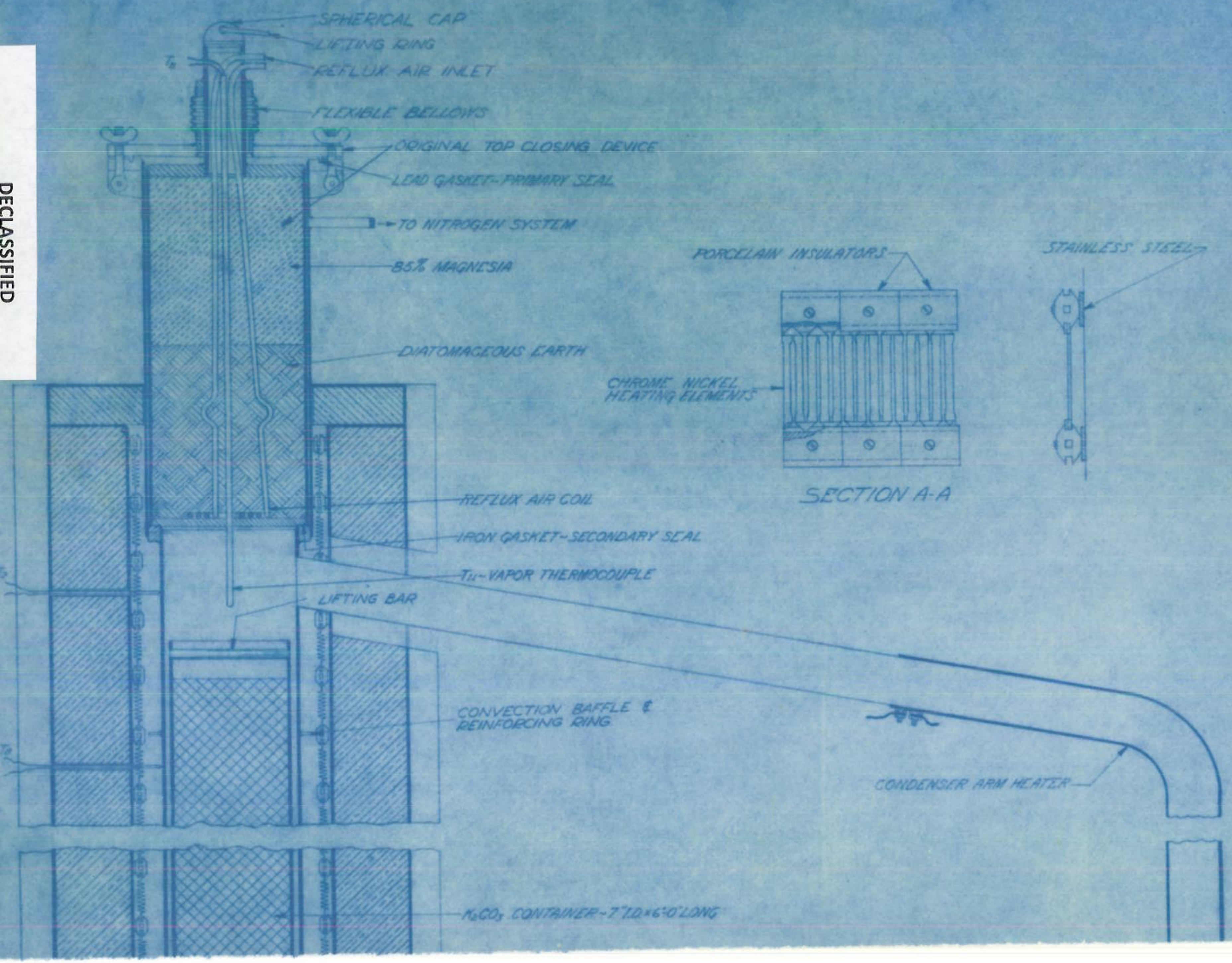


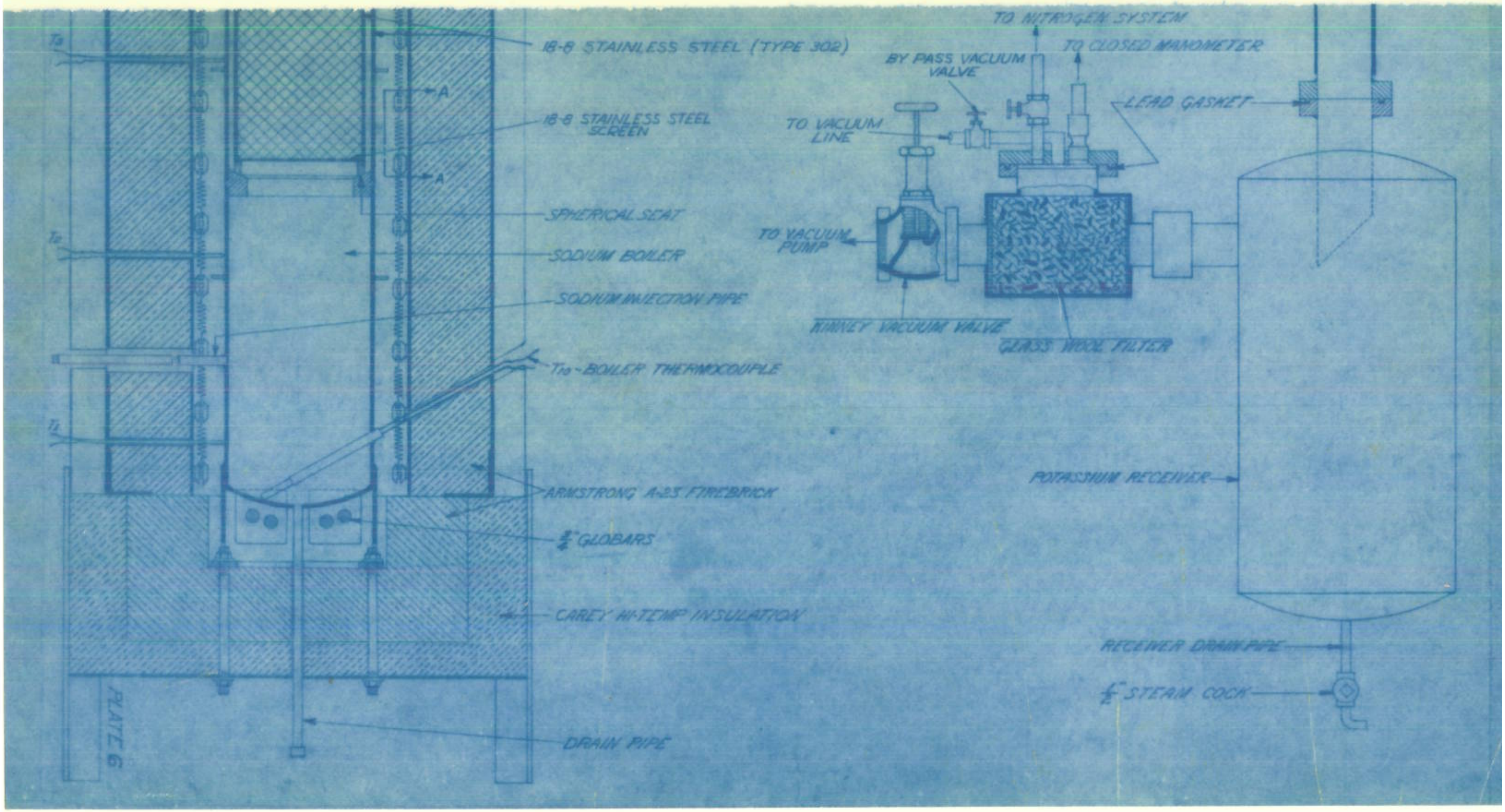
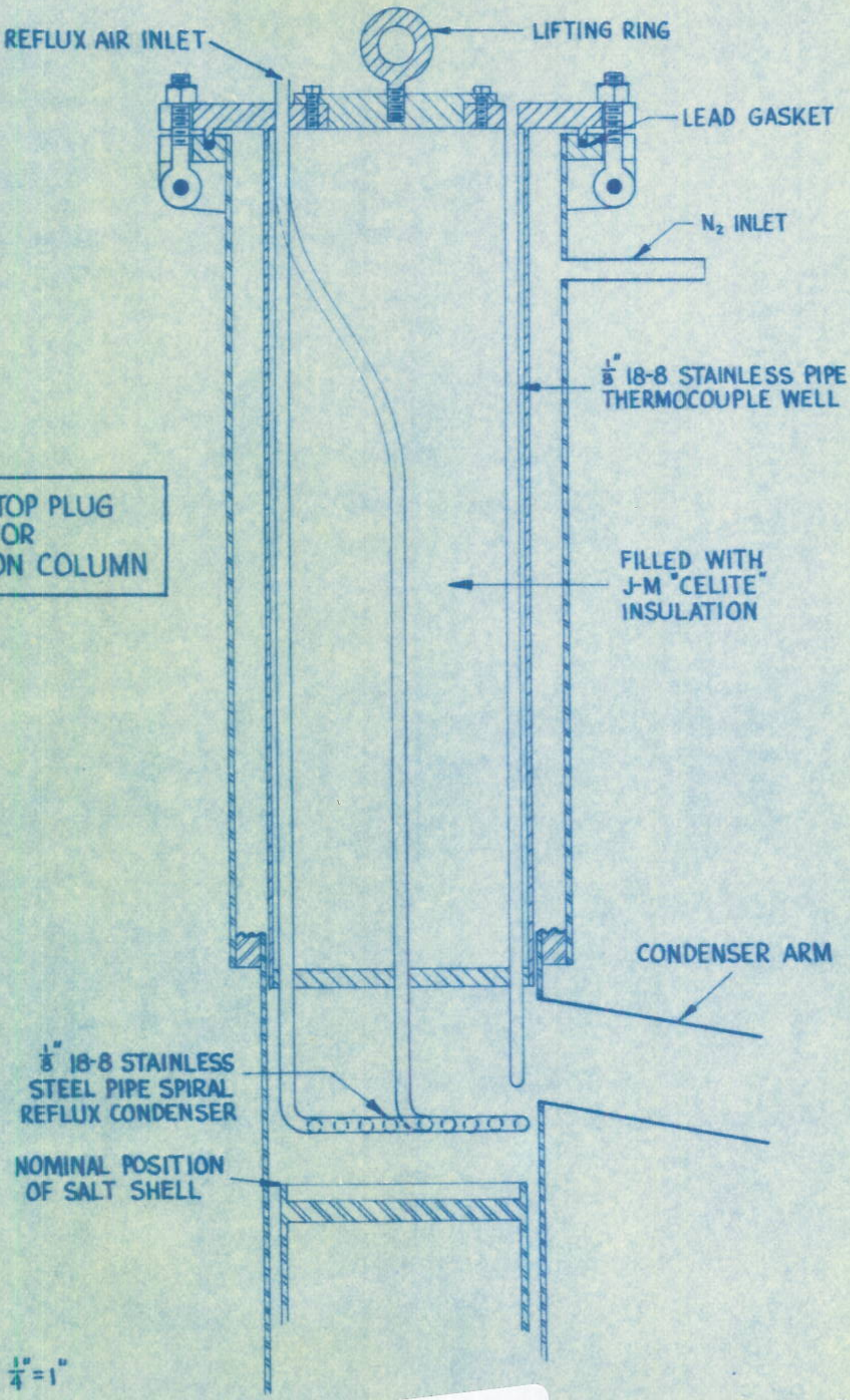

T_{13}
T_{12}
T_{11}
18-8 STAINLESS STEEL (TYPE 302)
A
A
18-8 STAINLESS STEEL SCREEN
SPHERICAL SEAT
SODIUM BOILER
SODIUM INJECTION PIPE
T_{10} - BOILER THERMOCOUPLE
ARMSTRONG A-23 FIREBRICK
$\frac{3}{4}$" GLOBARS
CAREY HI-TEMP INSULATION
DRAIN PIPE
TO NITROGEN SYSTEM
TO CLOSED MANOMETER
BY PASS VACUUM VALVE
TO VACUUM LINE
LEAD GASKET
TO VACUUM PUMP
KINNEY VACUUM VALVE
GLASS WOOL FILTER
POTASSIUM RECEIVER
RECEIVER DRAIN PIPE
$\frac{1}{2}$" STEAM COCK

PLATE 6



NEW TOP PLUG
FOR
REACTION COLUMN

SCALE $\frac{1}{4}$" = 1"

PLATE 7

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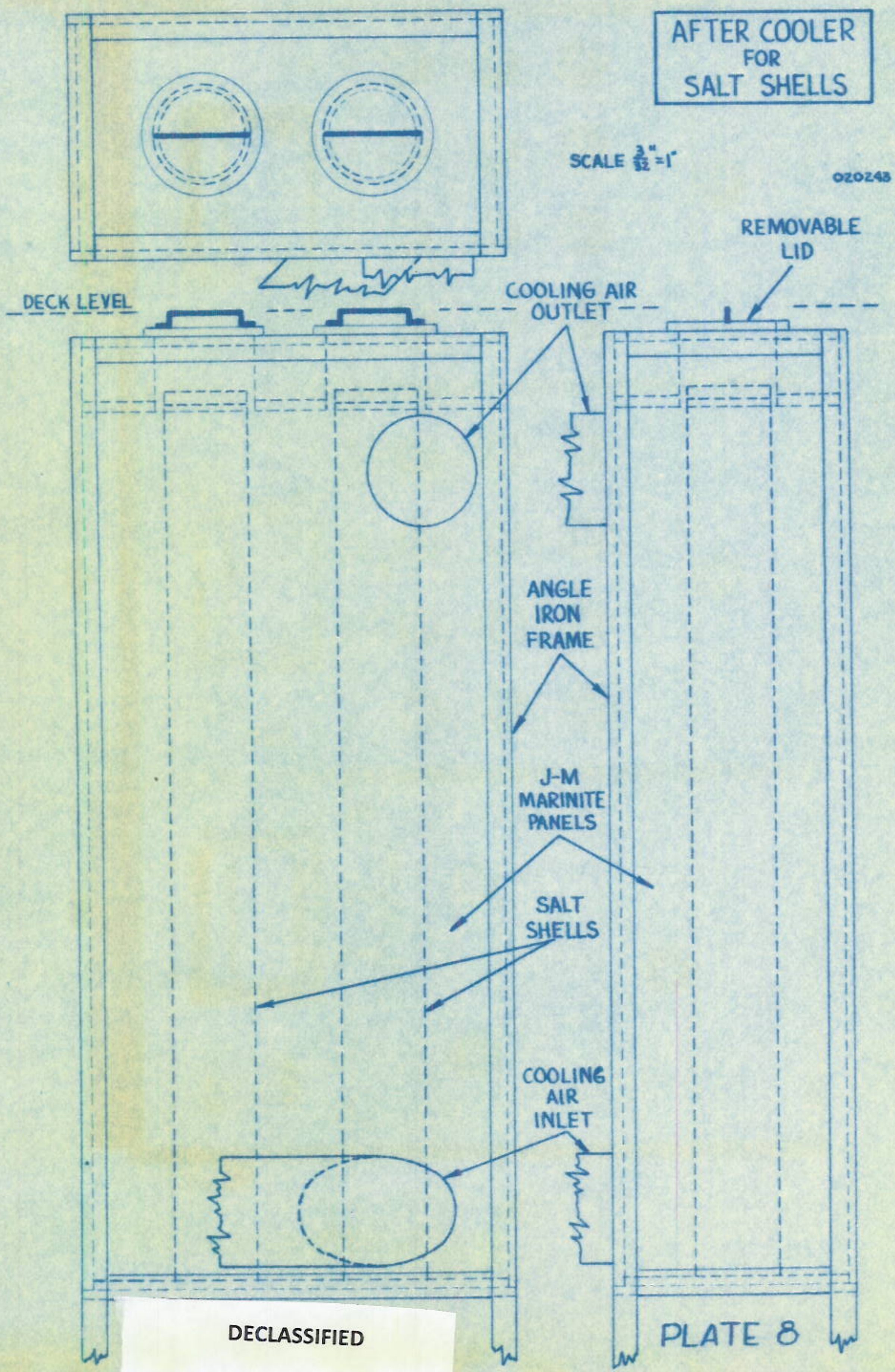

AFTER COOLER FOR SALT SHELLS

SCALE $\frac{3}{32}$" = 1"

PLATE 8

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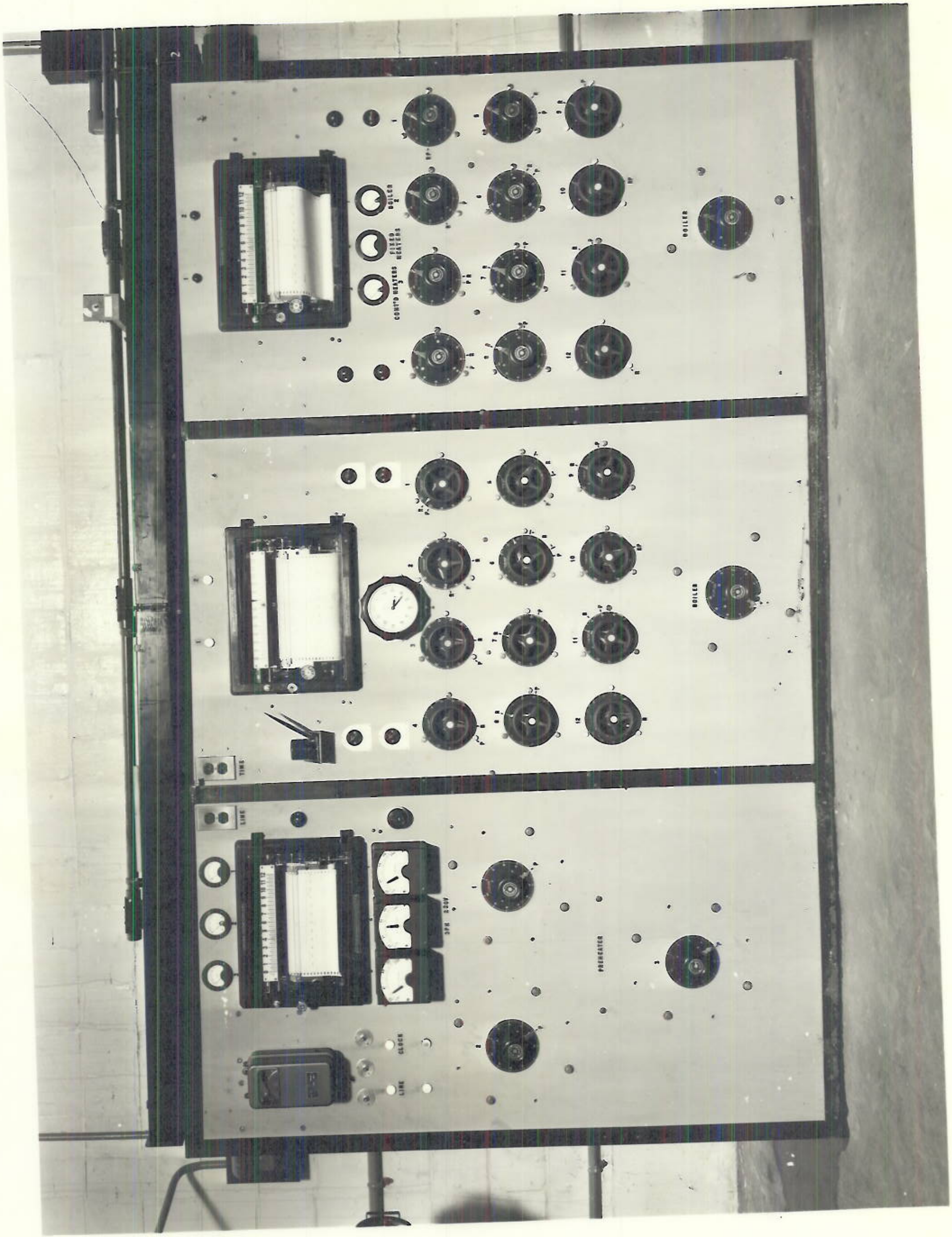

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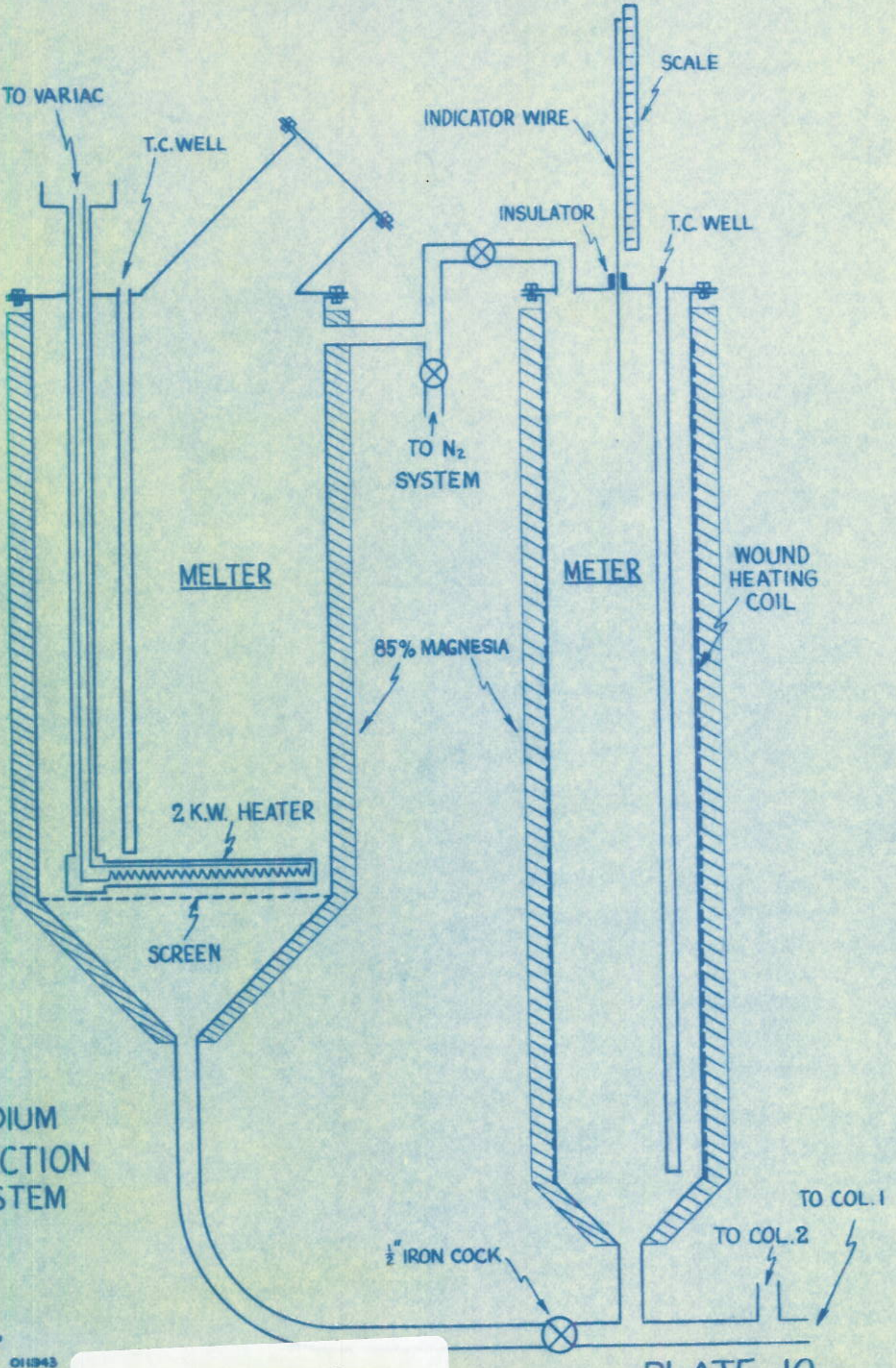
PLATE 9



SODIUM
INJECTION
SYSTEM

SCALE 3/16" = 1"

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PLATE 10

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PLATE 11

NITROGEN DEOXIDIZER AND DRYING TOWERS

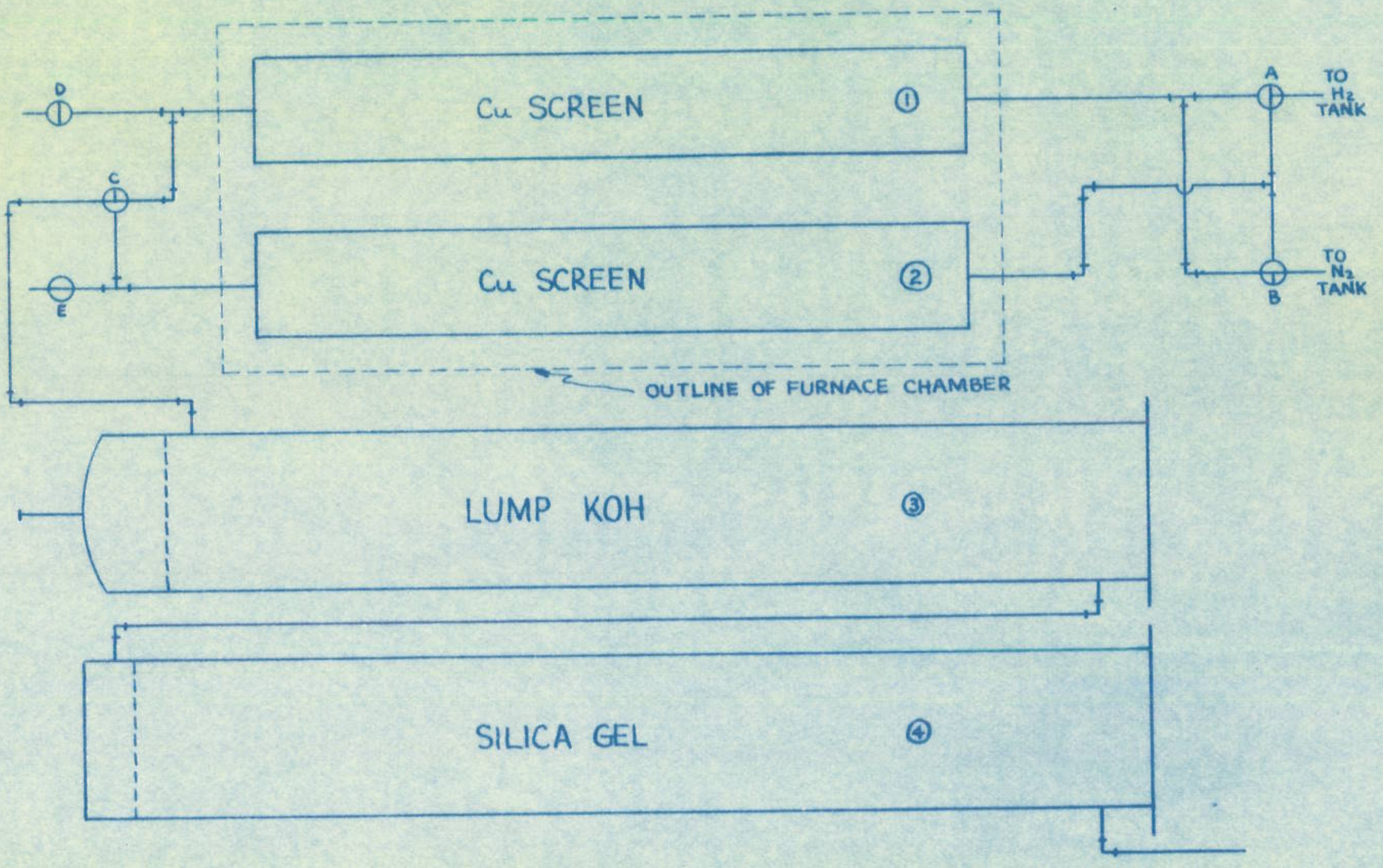

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PLATE 12

SCALE 1/4" = 1"

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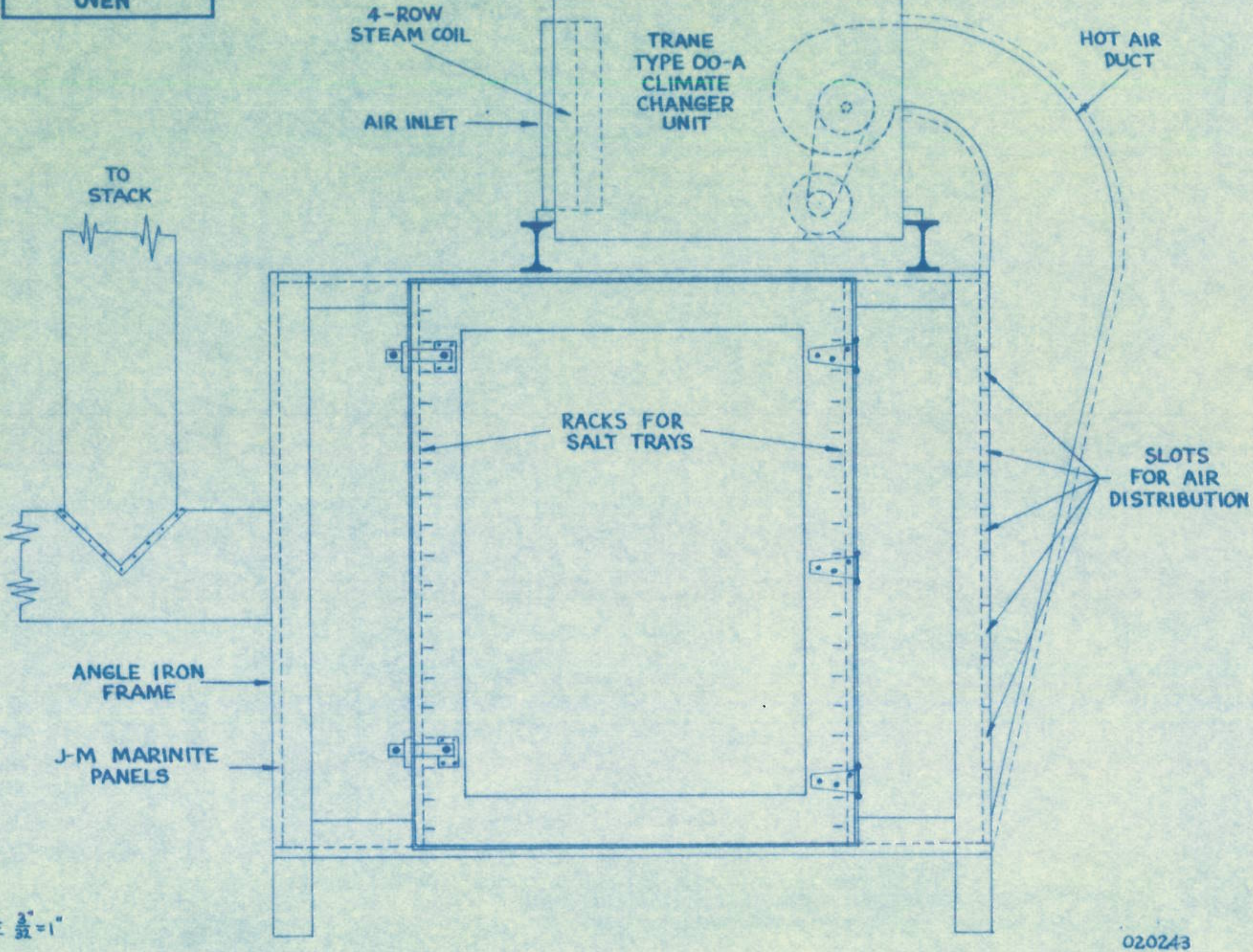
STEAM HEATED SALT DRYING OVEN



SCALE $\frac{3}{32}'' = 1''$

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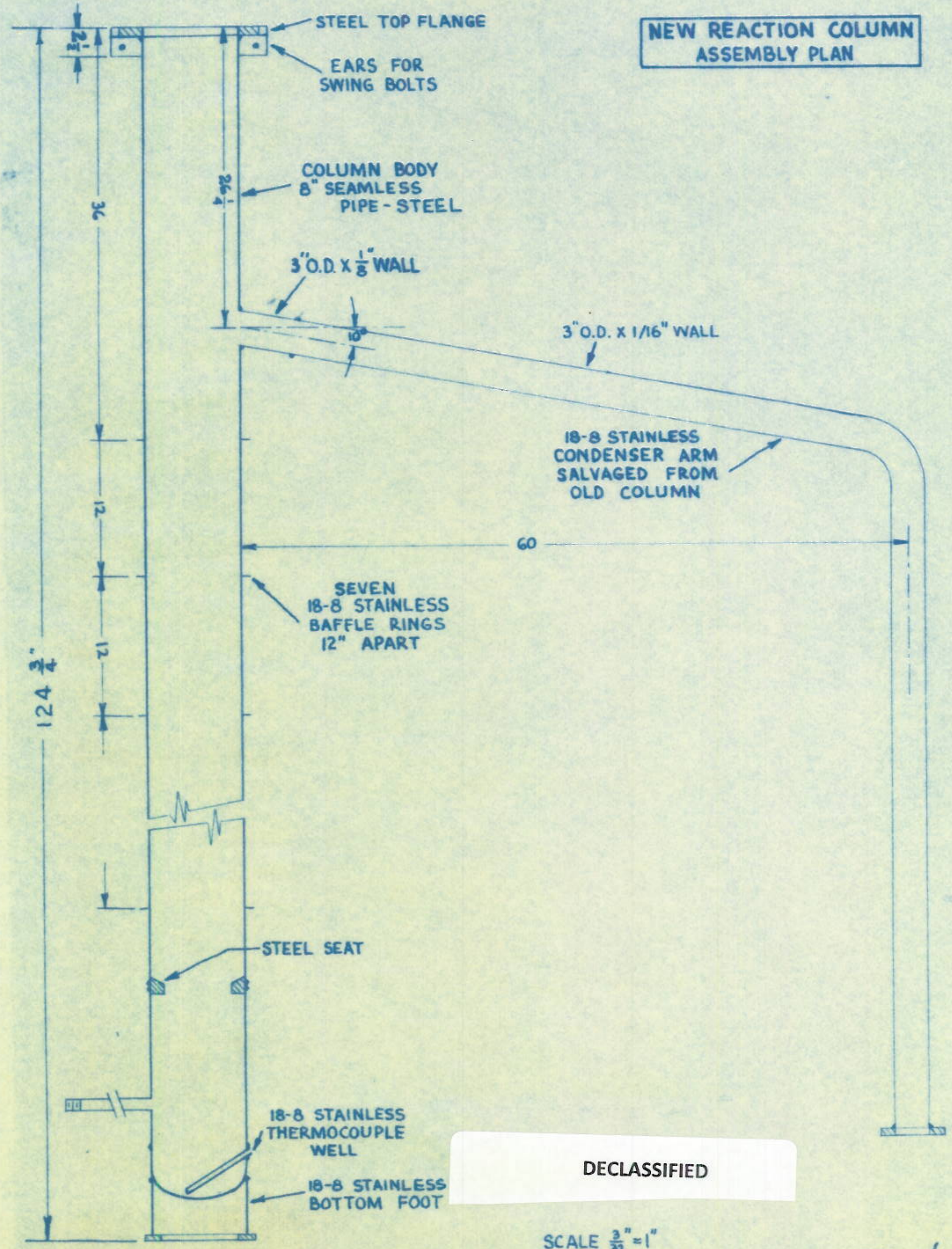
PLATE 13

NEW REACTION COLUMN
ASSEMBLY PLAN



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SCALE 3/32" = 1"

PLATE 14

APPENDIX I

DETAILS OF EQUIPMENT

SALT DRYING OVENS

1. Homemade, see Plate 13 of report. Walls are of 3/4" J-M Marinite panels in a 3" x 3" x 1/8" angle iron frame. Overall dimensions of the box are 58" wide x 48" high x 38" deep. Tray rack in center is made of 1" x 1" x 1/8" angle iron to hold 18 trays 33-1/2" x 33-1/2" x 1" each. Trays are spaced 1-1/2 inches apart. A Trane, Type OOA, "Climate Changer" is mounted on top with appropriate ducts to lead hot air into dryer. This heating unit has 4 rows of coils and delivers 400 cfm of air 130°C to the ovens with a consumption of about 100 lbs/hr of steam. The blower in this unit is powered by a 1375 rpm, 1/8 hp, 120v AC electric motor.

GRINDER FOR SALT CAKES

2. Made by Abbe Engineering Co., New York, N. Y. A pick through comb type grinder with a 9" x 12" feed opening powered by a 1/2 hp, 120v AC motor. Comb is adjusted to deliver 3/4 - 1/4 mesh particles at about 700 lbs/hr.

VIBRATING SCREEN

3. Homemade. The box is made of 3/8" plywood in a 1" x 1" x 1/8" angle iron frame. Provisions are made for two removable 24" x 48" screens and the box is fitted with appropriate exit ducts. The vibrating motion is imparted by an eccentric driven through a variable speed drive by a 1/2 hp, 120v AC motor. This unit handles adequately the output from the grinder.

ELECTRIC FURNACES

4. Made by H. E. Trent Co., Philadelphia, Pa.

5. Column Furnaces. (Two. Their drawing #2285.) Each furnace has a total connected load capacity of 18 kw divided into 9 individually controllable 110v, 2 kw sections. The furnace splits longitudinally and is 105" high, 10" inside diameter; 22" outside diameter. It is insulated by 4-1/2 inches of insulating type fire brick and contained in a 1/8" steel jacket. Suitable openings are provided to accommodate thermocouples in each section and to allow the condenser arm to project through.

6. Preheating Furnace. (Their drawing #2276) The total connected load capacity is 20 kw and it is divided into three 220v sections. The interior contains a stainless steel cylinder 75" long and 8" inside diameter with a hemispherical bottom and a 2" stainless pipe leading out. The outer case of the furnace is 1/8" steel and is 22" in diameter and 86" high. The above furnaces are wire wound with a Nichrome ribbon heating element

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formed into a patented design, and have proved quite satisfactory.

OIL FIRED FURNACE

7. Furnace Box. The inside dimensions of the fire box are 14" wide x 28" high x 24" deep. It is lined with Armstrong insulating fire brick 4-1/2" thick and insulated with 2 inches of Carey Hi-temp insulation. The furnace box is supported 4" off the floor on I-beams in a 3" x 3" x 5/16" angle iron frame with galvanized iron sides. The air heating coil housed in this furnace box is wound from 30 feet of 1/2" standard stainless steel pipe in the form of a helix 12" O.D. and 24" long.

8. Oil Burner. Made by Williams Oil-O-Matic, Bloomington, Ill. This burner is a standard home-heating model of 3 gal/hr maximum capacity on #2 fuel oil. It has proven very satisfactory for the job.

REACTION COLUMNS

9. Stainless, two. Pfaudler Co., Elyria, Ohio. (Their drawing #27929-D) The original columns were fabricated of Type 302 stainless steel of rolled and welded construction. The wall thickness was 1/8", the inner chamber approximately 118" deep and 8" I.D. with an annular ring to support the salt containers about 18" above the bottom of the column. The upper portion was of enlarged diameter (10" I.D.) to accommodate the original top closing device. The condenser arm was 3" x 1/16" wall stainless steel tubing, 120" long. The salt containers are made of 1/8" stainless steel rolled and welded into a cylinder 7" I.D. x 72" high fitted with a lifting bar on the top, a stainless steel screen and machined seat on the bottom.

10. The new top closing device (see Plate 7 of report) is fabricated from steel for the most part, the spiral reflux condenser and thermocouple well being made of 1/8" standard stainless steel pipe. The plug is filled with diatomaceous earth to minimize heat losses from the top of the column. The clearance recommended between top plug and walls of reaction column is of the order of 3/32" on a side.

11. Steel Columns, two. NRL Shop. (See Plate 14 of report) These columns are made of seamless 8" standard pipe (wall about 5/16" thick). The column is of uniform diameter, with the annular ring inserted near the bottom to support the salt containers as in the original columns. Each of these weighs 360 lbs when fitted with a blind flange on top and nothing on the condenser arm. Fabrication of this type column is considerably less expensive, and indications are that their life will be satisfactory. The new steel columns utilize the "new top closing device", the old stainless steel condenser arms, and other parts salvaged from the original stainless columns.

12. Vacuum Pumps, two. Beach Russ Co., Brooklyn, N. Y. Vacuum attained in the system by Type 50-D rotary vacuum pump,

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air cooled, and of 50 cfm free air capacity. Each pump is driven by a 2 hp, 220v, 3 phase motor, connected by V-belts. These pumps are quiet and efficient and have performed in a very satisfactory manner.

13. Vacuum Valves, two. Kinney Mfg. Co., Phila., Pa. These valves are 2" brass body models of the packless type having the bonnet sealed by means of a flexible metallic bellows. They are an excellent vacuum valve.

14. Hoists, four. Shaw Box Crane Division, Maxwell Manning and Moore, Muskegan, Mich. The hoists are of 1000 lbs capacity, electric, chain hoists equipped with chain containers to collect the unused length of chain. They are manually controlled by cords and are light enough to be easily lifted by one man. Each hoist is powered by a 3 phase, 220v AC motor and has a vertical lifting rate of 34 ft/min. These units have proved indispensable around the plant.

15. Globars, four per column. Carborundum Co., Niagara Falls, N. Y. Globars are rods of silicon carbide that make a very effective heating element for high temperature work. The size used directly beneath the sodium boiler is 23" long x 8" over-all heating length by 3/4" diameter. Each rod is rated for 50 amps at 39 volts or approximately 2 kw each, four are used in series and serve as a very convenient means of applying a relatively large quantity of heat to a small area.

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APPENDIX II

PRODUCTION FIGURES

REGULAR RUNS

1. The first run in the new pilot plant was made 16 October 1942. During the first month and a half numerous minor alterations were made to assure smooth operation. On 4 December 1942 operations were speeded by the instigation of a 12 hour day which permitted two runs to be made. The 12 hour day was continued through February, but was discontinued in March when the stainless steel columns began to develop frequent leaks. Throughout, the columns were run one at a time, the idle column standing by so that in the event of a failure, the investigation would not be interrupted. The following table shows the monthly production; note the improvement in the quantity of high quality potassium as research improved operating technique.

TABLE ONE
PRODUCTION FIGURES
REGULAR RUNS

MONTH	NUMBER OF RUNS	POUNDS K_2CO_3	POUNDS SODIUM	TOTAL WT POTASSIUM PRODUCED	POTASSIUM 94-97 wt%K	POTASSIUM GRTR 97 wt%K
Oct.	6	378	95	127	23	1
Nov.	12	728	206	257	116	65
Dec.	23	1372	446	576	112	286
Jan.	32	1883	627	925	259	400
Feb.	30	1771	589	827	50	427
Mar.	12	712	238	330	40	192
Apr.	9	598	191	273	86	115
May	12	683	225	347	0	217
June	16	857	294	412	54	228
TOTALS	152	8982	2912	4074	740	1931

REPROCESSING RUNS

2. The low quality alloy (ave. 83 wt%K) from the final cut of the regular runs is collected until enough is on hand to reprocess. It is injected from a barrel through the sodium injection pipe into the boiler of the reaction column in the same

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manner as the sodium, and is boiled through a salt column exactly as in a regular run. However, the lower heat of vaporization per pound permits a higher evaporation rate, and for that reason four injections (about 80 lbs of alloy) can be processed in a day. For most satisfactory results enough alloy of known composition is vaporized through the potassium carbonate column to bring the equivalent ratio to about 0.7. The following table shows the results of runs of this type.

TABLE TWO
PRODUCTION FIGURES
REPROCESSING RUNS

MONTH	NUMBER OF RUNS	POUNDS K_2CO_3	POUNDS OF ALLOY USED AVE. 83 wt%K	TOTAL WT POTASSIUM PRODUCED	POTASSIUM GREATER 97 wt%K	POTASSIUM AVERAGE 95 wt%K
Dec.	5	316	401	416	300	116
Jan.	5	297	412	429	251	178
Feb.	4	242	329	340	266	74
Mar.	1	72	24	9	0	9
Apr.	3	196	280	286	205	81
May	0	---	---	---	---	---
June	3	167	190	170	76	94
TOTAL	21	1390	1636	1650	1100	550

BURNING POTASSIUM TO THE TETROXIDE

3. Concurrently with the production of potassium shown above, a spray gun burning unit has been in operation making potassium tetroxide as described in Report P-1999 in the amounts indicated in the following table.

TABLE THREE
PRODUCTION FIGURES
POTASSIUM TETROXIDE

MONTH	TOTAL OXIDE PRODUCED	LESS THAN 230 cc/gm	230-232 cc/gm INCL	233-234 cc/gm INCL
Oct. - Dec.	50	50	-	-
Jan.	822	152	401	269
Feb.	141	-	71	70
Mar.	517	71	334	112
Apr.	1237	40	216	981
May	1182	402	592	188
June (25)	685	283	402	-
TOTAL	4634	998	2016	1620

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4. Present burning technique can produce tetroxide within 2 cc/gm of the theoretical value for the alloy burned. Average recovery on the present unit is around 85%. One spray gun can burn from 5-6 lbs of potassium an hour. No difficulty is experienced in obtaining a 33.0 wt% available oxygen (231 cc/gm) product under present burning conditions.

TABLE FOUR
INVENTORY

MATERIAL	JUNE 25, 1943
Calcined Regular K_2CO_3	4120 lbs
Prepared K_2CO_3 (course & medium)	2593
Prepared K_2CO_3 (fine)	1425
Sodium	956
Potassium (total)	1089
Ave 99 atom % K	512
Ave 96.5 atom % K	473
Ave 75 atom % K	104
Potassium Tetroxide (total)	4192
Less than 230 cc/gm (ave 32.5 wt%O_2)	998
230-232 cc/gm incl. (ave 33.0 wt%O_2)	1795
233-234 cc/gm incl. (ave 33.3 wt%O_2)	1399

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APPENDIX III

PREPARATION OF POTASSIUM CARBONATE

1. In order to prepare the potassium carbonate into porous lumps as has proved desirable, the following procedure is followed. "Carbonate of Potash, Calcined Regular" (98% K_2CO_3) is spread on 33-1/2" square trays to a depth of about 5/8". Approximately 25% (of the dry weight) water is then sprayed over the trays. The trays are dried in the hot air ovens at 125°C for about three days. At the end of this drying period a uniform, porous, brittle cake of salt is obtained with a water content less than 1%. The cakes are broken in the Abbe Grinder and screened to the desired size. With the present equipment, 700 lbs of new salt can be processed per batch. Yields after grinding and screening are around 80% usable material, the fines, however, may be readily reworked. The three day drying period may be shortened by breaking and turning the cakes as they start to dry. This practice, however, noticeably increases the quantity of fines per batch, and since salt production is ahead of consumption, the time saved in drying does not warrant the extra labor involved reworking the more abundant fines.

2. Potassium carbonate is quite hygroscopic and care must be exercised where and how it is stored. For this reason, salt for use in the plant is kept in 55 gallon drums with sealing lids.

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APPENDIX IV

ANALYTICAL METHODS

1. In order to study the properties of this reaction, it is necessary to know the composition of the high cuts, the composition of the low cuts, and the distribution of potassium carbonate in the mixed sodium-potassium carbonates in the spent carbonate containers.

DETERMINATION OF COMPOSITION OF HIGH POTASSIUM-SODIUM ALLOYS

2. The most convenient and rapid method of determining potassium and sodium-potassium alloys of greater than 90% K is by their freezing point. Since the published data (Z. anorg. Chem. 74, 155 (1912)) is inaccurate in this region, a more reliable freezing point curve has been determined at this laboratory. By use of this curve and a calibrated 1/5° division thermometer, compositions may be determined with an accuracy of ± 0.1 atom %K which is sufficient for most purposes.

3. The curve may be represented by a straight line in the region 90 - 100 atom %K by the equation

$$x = 0.432y + 72.51$$

Where x equals composition in atom %K, y equals corrected freezing point.

4. A graphical representation of this equation is attached (Plate 1). It should be observed that this gives a freezing point for pure potassium of 63.7°C, this value is 1.2° higher than the usually reported value, and is, no doubt, more accurate. A considerable amount of potassium has been made in this laboratory with a corrected freezing point of 63.7°C. The freezing point method of analysis is not readily extendable below 90 atom %K alloys because of the decreased sharpness of the first thermal arrest on cooling.

DETERMINATION OF POTASSIUM IN LOW QUALITY ALLOYS

5. Alloys of this class are normally liquid at room temperature, and the apparatus (shown in Plate 2) has been developed to obtain a clean, oxide free sample, which is decomposed with alcohol, and titrated with standard acid.

6. Sampling. A sample (about 30 grams) of the alloy in question is drawn into the sample bulb (shown in Plate 2) by evacuating the bulb through the stopcock and drawing the alloy from the original container through a glass tube fitted with a ground joint into the sample bulb. If the alloy is of such a composition that it is not practical to utilize the freezing point method of analysis, the sample bulb is fitted into the

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sampling device (shown in Plate 2). A weighed thin glass ampule is placed in the sampler; the system is evacuated through the stopcock indicated, and filled with dry, oxygen-free nitrogen. The sample bulb and sample are warmed with a flame to assure free flow of the alloy, and tipped so that the alloy will flow from the sample bulb through the ground joint into the capillary. A slight vacuum is then applied to draw the desired weight of sample (about 1 gram) into the alloy ampule, whereupon nitrogen is let in to force the excess out of the capillary and the sampler returned to horizontal. The alloy ampule is removed and quickly sealed in a flame and weighed. In this manner an accurate weight of clean, oxide-free sample is obtained.

7. Decomposition. The weighed ampule of alloy is placed in a 32 x 300 mm test tube, covered with distilled, dry n-hexane and broken with a glass rod. Absolute alcohol is added dropwise to the hexane and decomposition takes place. Care must be exercised not to add alcohol too fast or a loss of sample by entrainment in the hydrogen evolved will result. The decomposition test tube is kept in a water bath to minimize over-heating.

8. Titration. When the sample has been decomposed it is transferred to a 250 cc Erylenmeyer flask, distilled water being used for washing. It is titrated to a phenolphthalein end point with 1 N HCl. The hexane is evaporated and the titration continued to a methyl orange end point. The use of phenolphthalein reduces danger of overstepping the methyl orange end point, and speeds the titration.

9. Calculation. To express the results as atom %K, the following equations must be solved simultaneously.

$$(1)\quad x = \frac{\frac{100y}{39.1}}{\frac{y}{39.1} + \frac{w-y}{23}} \qquad (2)\quad \frac{ME}{1000} = \frac{y}{39.1} + \frac{w-y}{23}$$

x = atom %K
y = wt K in sample
w = wt of sample
ME = milliequivalents of HCl

The solution of equations (1) and (2) gives

$$x = \frac{1000w-23ME}{.161ME}$$

Since both w and ME are known, x (atom %K) may be calculated directly.

10. If there is oxide on the sample, if part of the solution is lost transferring, or if the hexane is impure the results will be high, so due care must be exercised in following this procedure.

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11. The above method was used for alloys from 90 - 100 atom %K in determining the freezing point curve reported here. (Plate 1).

DETERMINATION OF POTASSIUM CARBONATE IN MIXED SODIUM-POTASSIUM CARBONATES.

12. Samples of mixed carbonate as taken from the spent carbonate container at regular intervals of height are crushed and dried at 150°C. Two weighed samples are put into solution. The first is titrated with 1 N HCl to the methyl orange end point to obtain the total alkalinity. The second is used to determine the hydroxide present (normally quite small) by adding an excess of $BaCl_2$ to the sample (precipitates $BaCO_3$) and titrating to the phenolphthalein end point without filtering.

13. The mol % potassium carbonate is calculated by the solution of the following simultaneous equations:

$$(1)\quad \frac{\frac{100\,y}{138.2}}{\frac{y}{138.2} + \frac{w-y}{106}} \qquad (2)\quad \frac{ME}{1000} = \frac{y}{69.1} + \frac{w-y}{53}$$

x = mol % K_2CO_3
y = wt K_2CO_3
w = wt of sample
ME = milliequivalents of HCl

The solution of (1) and (2) gives the following equation from which x (mol % K_2CO_3) may be calculated directly.

$$x = \frac{100w - 5.3ME}{.161ME}$$

14. If hydroxide is found in the sample tested both w and ME are corrected accordingly. The hydroxide present is assumed to be NaOH although it could conceivably be a mixture of KOH, K_2O_4, NaOH, Na_2O_2, K, and Na before the sample is put into solution. However, this factor is usually small so the error introduced is not appreciable.

RECOVERY OF SODIUM CARBONATE FROM SPENT SALT

15. A scheme of leaching and recrystalization has been worked out that appears to be fairly satisfactory for the recovery of the sodium carbonate from the spent salt. However, the low market value of sodium carbonate has discouraged further investigation along these lines.

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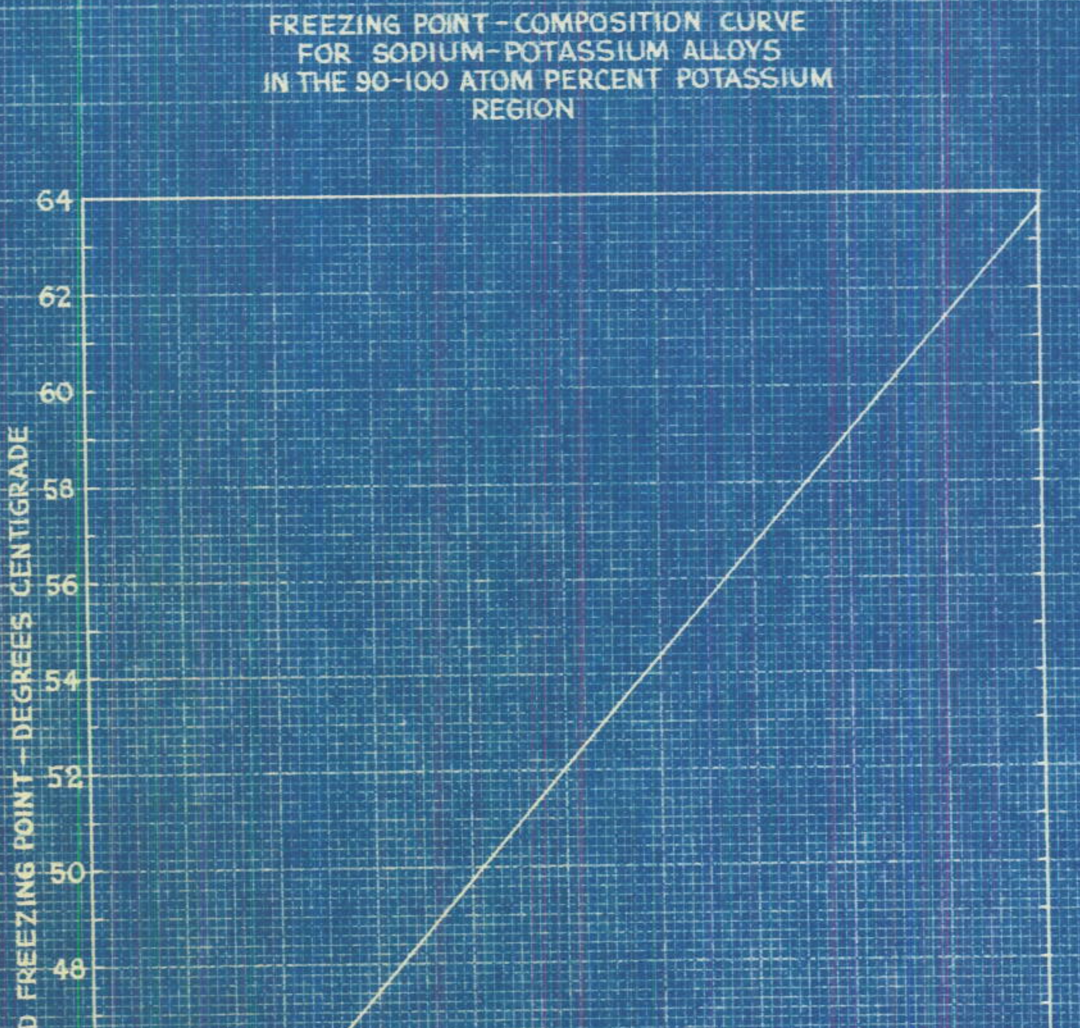

PLATE I
APPENDIX 4

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SAMPLER FOR ALLOY ANALYSIS

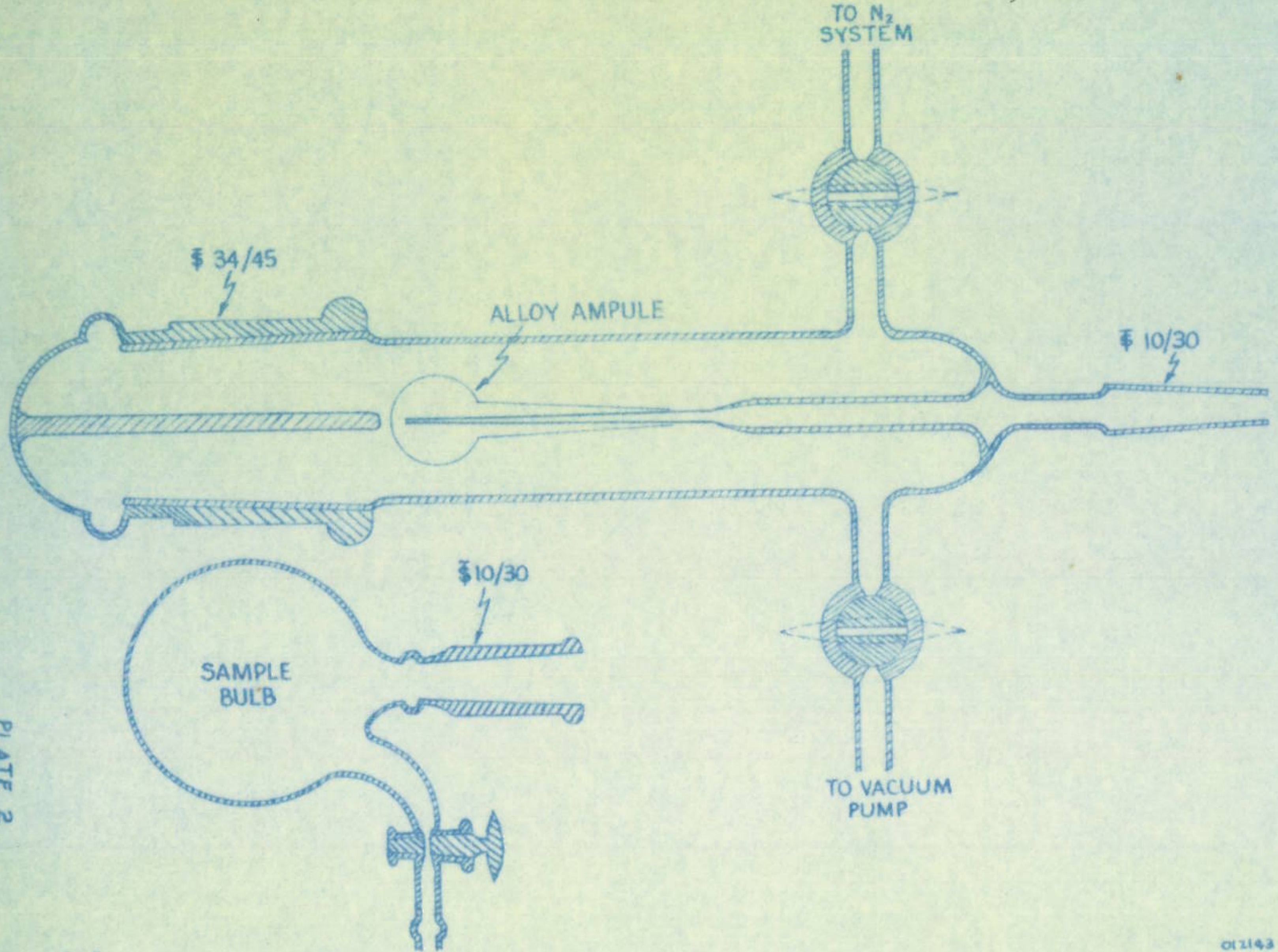

PLATE 2
APPENDIX 4

SCALE 1"=1"

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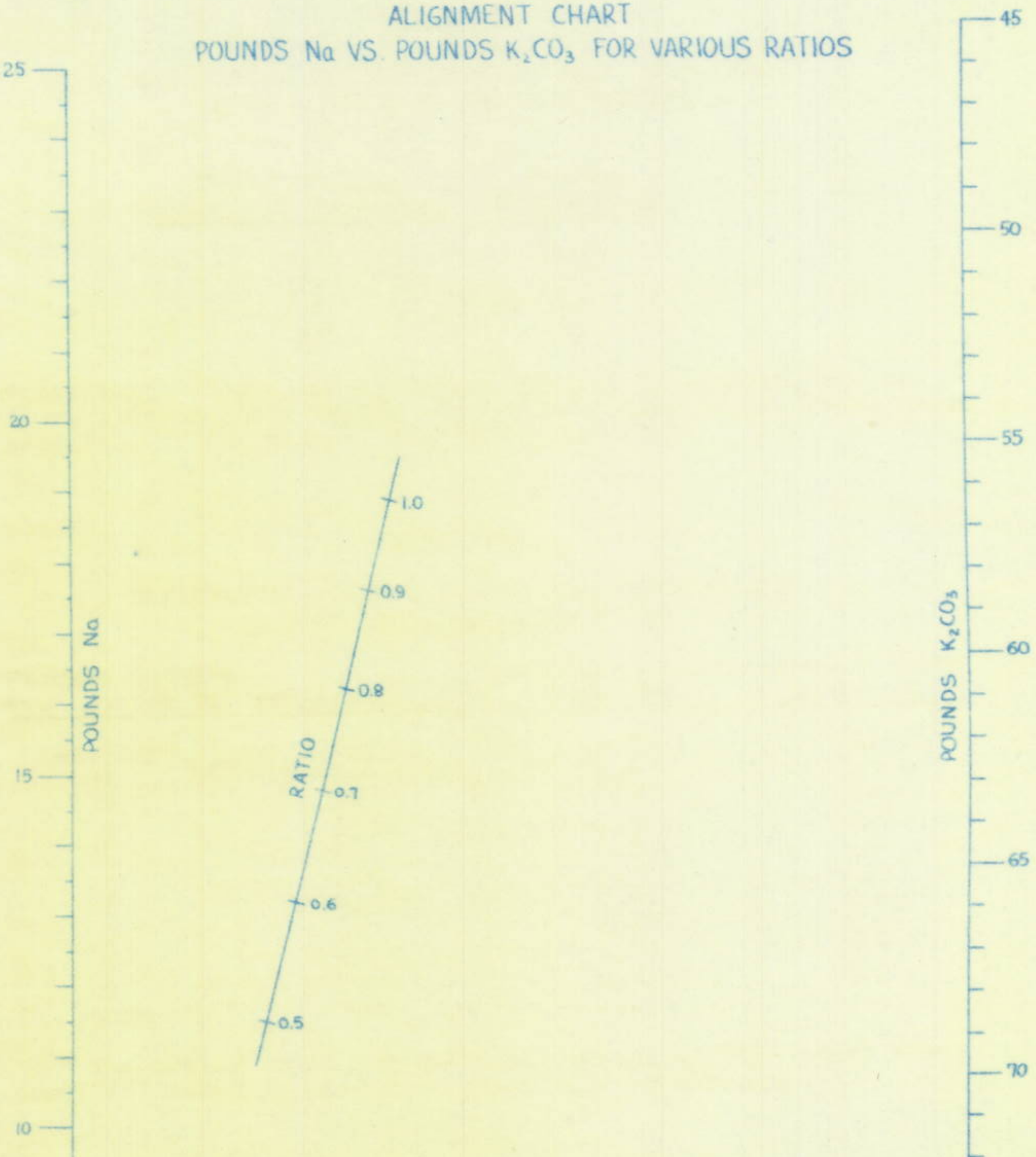

ALIGNMENT CHART
POUNDS Na VS. POUNDS K_2CO_3 FOR VARIOUS RATIOS

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PLATE I
APPENDIX 5

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APPENDIX VI

TABLE ONE

TEMPERATURE DISTRIBUTION IN SALT CONTAINER AFTER 2 HOURS IN PREHEATER-DRYER

Distance from Bottom of Container	Average Temperature
0'	490°C
2'	470
4'	450
6'	430

These temperatures can be varied readily by altering the air flow, time, or power input. The above values proved to be quite satisfactory for making consecutive runs.

TABLE TWO

TEMPERATURE-POWER DATA FOR REACTION COLUMNS 4 HOUR HEAT FROM COLD START

Furnace Section From Bottom Up	Temperature °C	Stainless Columns Power kw	Steel Columns Power kw
*Boiler	640	3.0	3.0
1	650	1.6	1.6
2	650	0.9	0.9
3	650	0.8	0.8
4	615	0.8	0.8
5	550	0.7	0.7
6	500	0.5	0.6
7	470	0.2	0.5
8	425	0.15	0.5
9	400	0.1	0.4
Vapor	390	---	---

* The boiler is on intermittant control at 640° hence the power indicated is used only about half of the time.

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TABLE THREE

TEMPERATURE-POWER DATA FOR REACTION COLUMNS REQUIREMENTS DURING RUN (50 mm pressure)

Furnace Section From Bottom Up	Equilibrium Temperature °C	Stainless Columns Power kw	Steel Columns Power kw
Boiler	660	5.1	5.1
1	650	1.6	1.6
2	650	0.9	0.9
3	650	0.5	0.5
4	615	0.4	0.4
5	550	0.3	0.3
6	540	0.3	0.3
7	535	0	0.15 *
8	535	0	0.15 *
9	535	0	0.15 *
Vapor	535	0	0

* More power required by steel columns in upper sections to compensate for heat losses due to their higher thermal conductivity.

TABLE FOUR

THEORETICAL WEIGHT RELATIONS FOR THE REACTION

$$K_2CO_3 + 2Na \rightarrow 2K + Na_2CO_3$$

K_2CO_3	1.00	3.00	1.76
Na	.33	1.00	.59
K	.56	1.70	1.00
Na_2CO_3	.77	2.31	1.35

This table is most easily used by selecting the material of interest, moving horizontally to the column containing unity, reading vertically the weight of the other components necessary to satisfy the reaction equation.

[redacted]

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TABLE FIVE

PHYSICAL PROPERTIES

	K_2CO_3	Na_2CO_3	K	Na
Molecular wt.	138.2	106.0	39.1	23.0
Density			.83 (63°C)	.93 (97°C)
Apparent Density as used	.55 - .65	.7 - .8		
Melting Point	891°C	851°C	63.7°C	97.5°C
Boiling Point				
(atm pressure)			760°C	880°C
(50 mm pressure)			537°C	649°C
Heat of Vaporization				
kg cal/gm			.517	1.09
kw-hrs/lb			.273	.577
Specific Heat cal/gm	.32(600°)	.26(45°)	.23 (200°)	.33 (100°)

The minimum melting mixture of the Na_2CO_3-K_2CO_3 systems of mixed crystals is 60% Ne_2CO_3 - 40% K_2CO_3 at a temperature of 705°C.

[redacted]

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VAPOR PRESSURES OF
SODIUM AND POTASSIUM

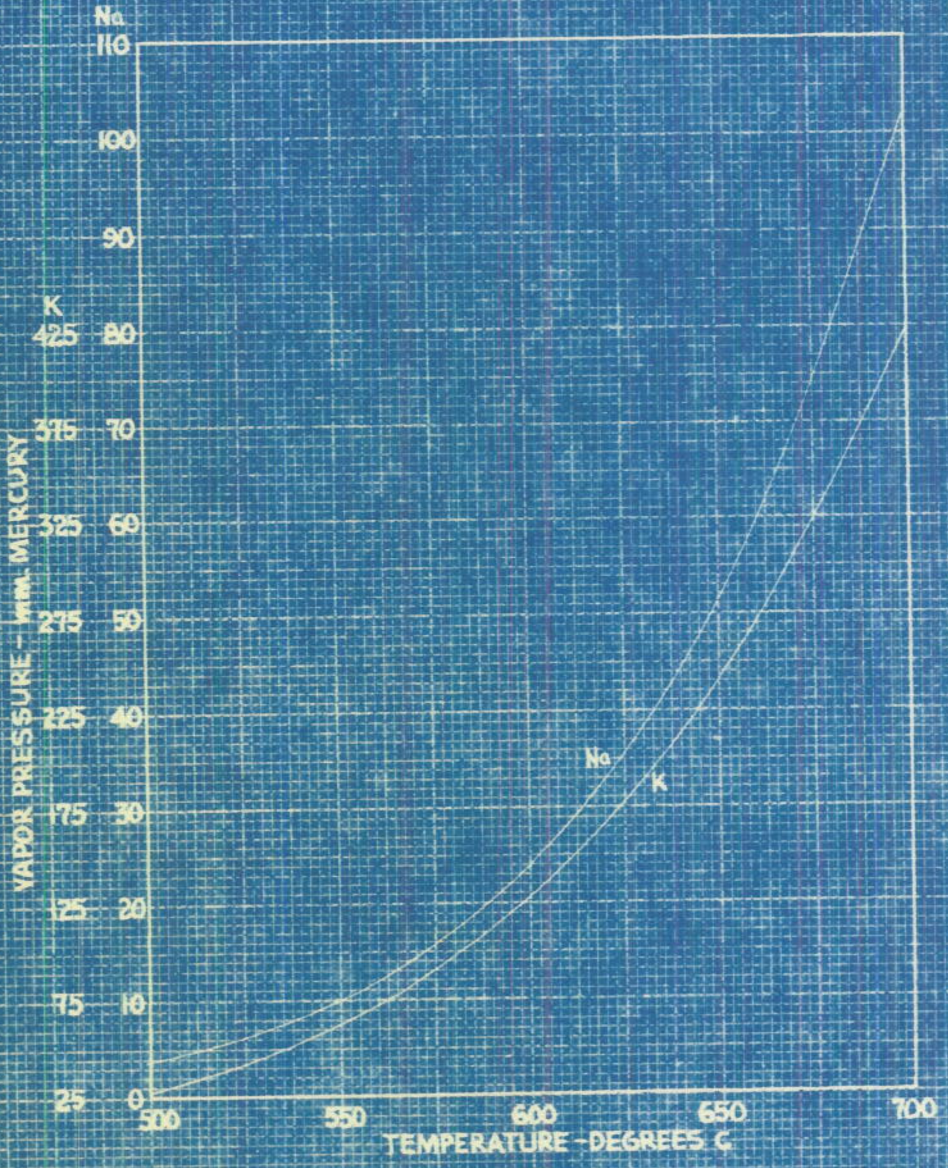

PLATE I
APPENDIX 7

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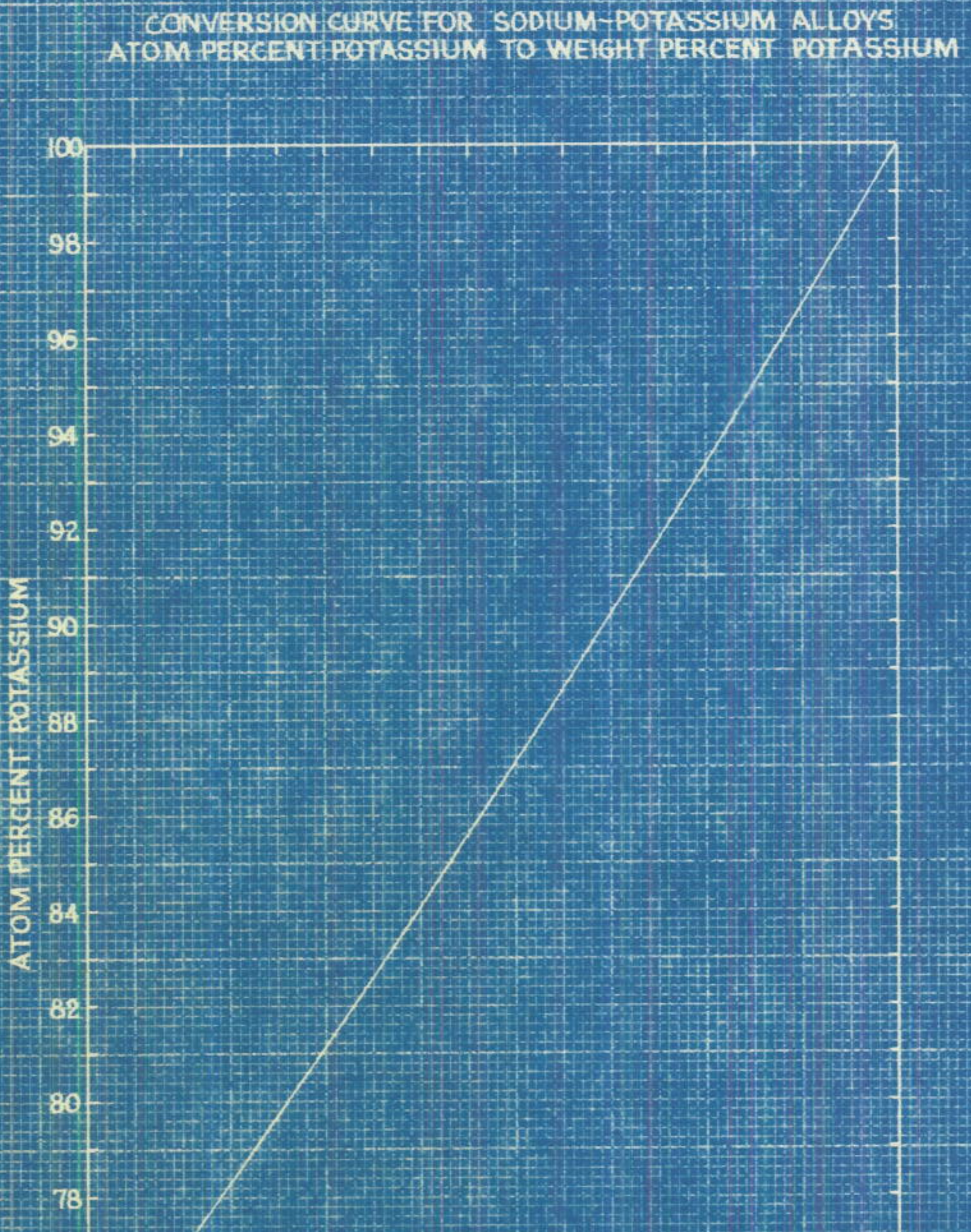
N. R. L. 81A



CONVERSION CURVE FOR SODIUM-POTASSIUM ALLOYS
ATOM PERCENT POTASSIUM TO WEIGHT PERCENT POTASSIUM

N. R. L. 84A

PLATE 2
APPENDIX 7

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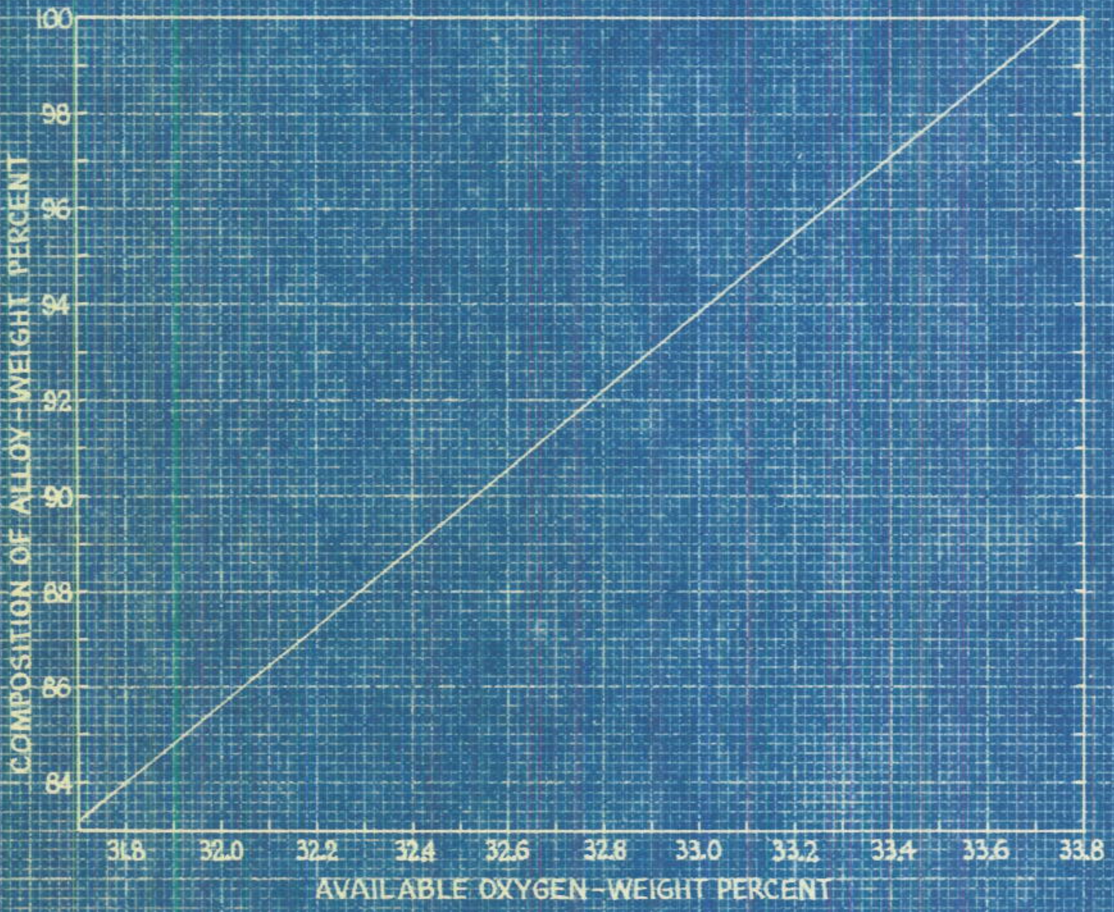
COMPOSITION OF SODIUM-POTASSIUM ALLOYS VS. AVAILABLE OXYGEN IN CORRESPONDING OXIDES



N. R. L. 24A

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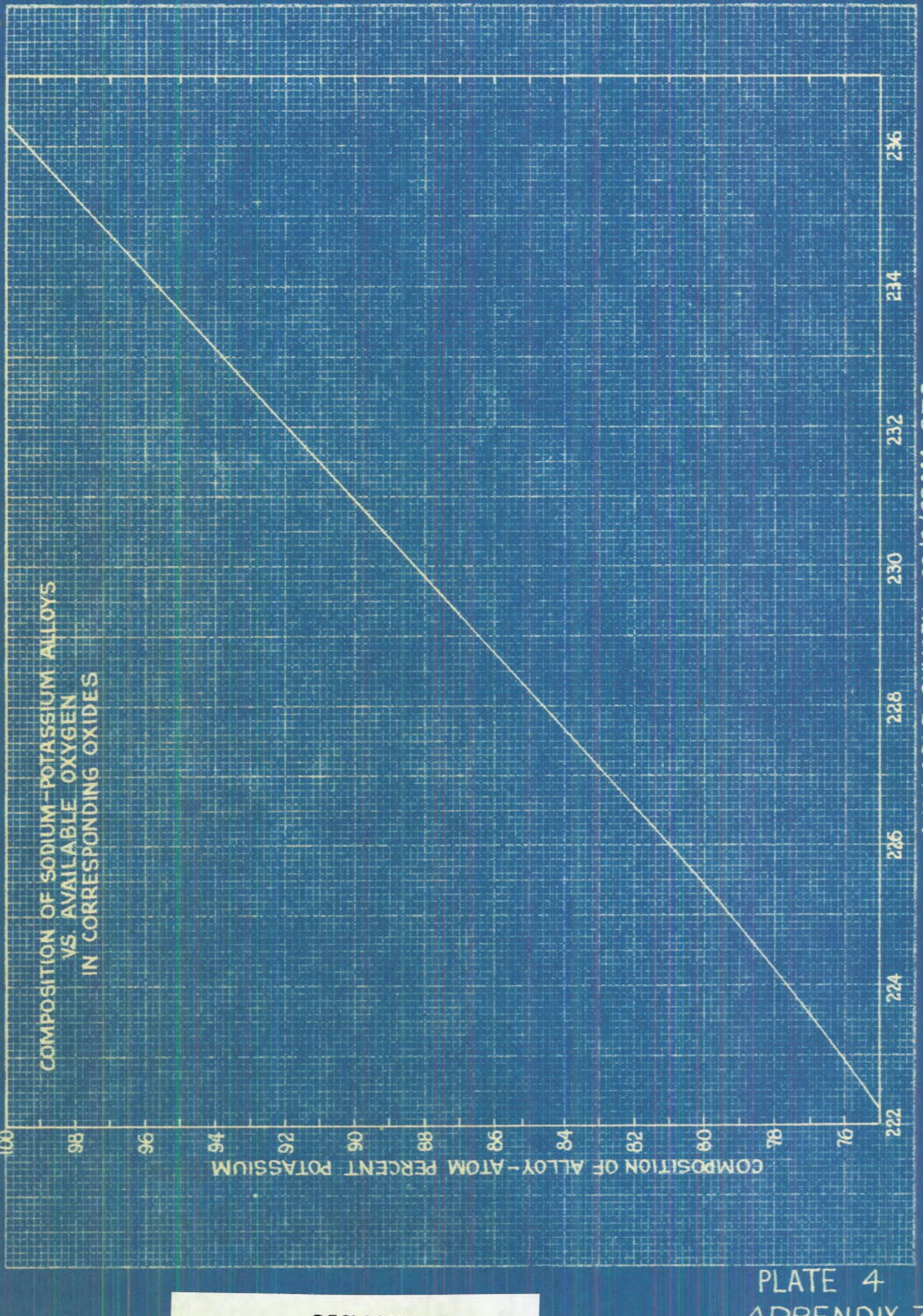

COMPOSITION OF SODIUM-POTASSIUM ALLOYS
VS. AVAILABLE OXYGEN
IN CORRESPONDING OXIDES

PLATE 4
APPENDIX 7

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