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Thirteenth Partial-- Oxygen Source Materials

The Production of Potassium From Potassium Carbonate.

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NAVAL RESEARCH LABORATORY BELLEVUE, D. C.

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Report P-2026

NAVY DEPARTMENT

Thirteenth Partial Report
on
Oxygen Source Materials

The Production of Potassium From Potassium Carbonate

NAVAL RESEARCH LABORATORY
ANACOSTIA STATION
WASHINGTON, D. C.

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ABSTRACT

The production of potassium metal from potassium salts by reaction with sodium and distillation has been studied in a three inch by six foot length fractionating-reaction column. This investigation is a continuance of work reported in P-2011 on a somewhat larger scale and has been conducted to furnish information for the full scale pilot plant. Potassium 99.2% pure was obtained from this method with a high percentage of the potassium replaced in the carbonate. The purity is affected by the salts used and rate at which the operation is carried out.

Data on the reaction of sodium with potassium salts are given for equilibrium conditions. Literature values on the potassium halides and potassium hydroxide and values obtained at this Laboratory on the carbonate are plotted in plates 2 to 7, inclusive.

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AUTHORIZATION

1. Bureau of Ships Project Order 243/42, S77-2(275) dated 9/30/41, Project 109/43 Aer PR427LRM F49-1 JJ781, 4/16/42 and BuAero ltr. Aer-E-2573ERM JC10, 3/7/42 C-9159.

STATEMENT OF PROBLEM

2. Study of Chemical Sources of Oxygen - The production of potassium metal from potassium carbonate and sodium. This method is reported on the laboratory scale in Report #P-2011 entitled "Investigation of Methods for the Production of Metallic Potassium". The present report covers the carrying of this work to a small pilot plant scale prior to the operation of the pilot plant on this study.

FACTS BEARING ON THE PROBLEM

3. The most promising method for potassium production considering the procurement of materials and the yields of product from the above report was the reaction of sodium metal vapor with potassium carbonate under reduced pressure. This operation was carried out with a column packed with the carbonate through which the sodium was distilled.

4. The reaction between sodium vapor and a long column of solid potassium carbonate, carried out under reduced pressure, yielded sodium-potassium alloys rich in potassium. Distillations of sodium-potassium alloys under reduced pressure in a column packed with iron rings showed that a very complete separation of potassium from these alloys resulted using less than 12 inches of packing.

5. The column described in this report was designed to carry out both the reaction and the distillation mentioned above in one unit. The purpose of the experiments with this column was to determine the purity of potassium which could be obtained on a large laboratory-scale, and also, changes necessary in designing a pilot-plant unit.

APPARATUS

6. The sketch and photograph included (App. I, Plates 1 & 2) show the basic column design and the floor arrangement of the individual units. The pertinent dimensions and a brief description of the parts are given in Appendix I.

EXPERIMENTAL PROCEDURE

7. The column was tested thoroughly to insure the absence of leaks. The salt was then introduced and residual traces of water removed by heating to 300°C. under vacuum.

8. Sodium blocks were melted in the bulk injector and forced, under nitrogen pressure, through a wire screen (to remove surface film impurities) and into the metering injector. A volume of sodium sufficient to react with nearly all the potassium carbonate was introduced to the sodium pot from the metering injector by opening the S.S. valve while the column was under reduced pressure. The surface of the sodium in the metering injector was followed with a movable electrical contact connected to a buzzer. The weight of sodium injected was calculated from the volume of the metering injector (180 cc./in. of length) and the density of sodium at 125°C. (0.92).

9. The column pressure was held at 50 mm. while the salt sections were heated to temperatures ranging from 500-675°C. Sufficient heat was introduced to the sodium to vaporize it at a constant rate (from 1-3 lbs./hr.) by means of the Globars.

10. As the sodium started to boil, the reflux air was turned on and the top heating section (H_6) was turned off. As the reaction progressed, thermocouple T_7 indicated the boiling point for pure potassium (535° at 50 mm. pressure). Near the end of the reaction this temperature rose as the potassium content of the distillate decreased.

11. Thermocouple T_8 indicated the boiling temperature for pure sodium (650°C. at 50 mm. pressure) until the sodium boiler was empty. This point was indicated by a sudden temperature rise as shown by thermocouple T_9 . Due to the resistance to flow of the salt column, the boiling temperature of sodium as registered by T_9 varied from 660-675°C.

12. When all the sodium had evaporated the heat input to the salt column was increased until all sections were above 650°C. The column was then evacuated slowly and kept under vacuum for 30 minutes longer to insure the removal of all residual metal. An atmosphere of nitrogen was introduced, the salt container removed and the column allowed to cool in readiness for another experiment.

13. Metal receivers were changed at intervals during the reaction without changing the pressure in the column by first closing the condenser-arm valve.

14. The freezing point diagram of potassium alloys that are solid at room temperature has been accurately determined by chemical analysis in this laboratory so that the composition of solid alloys could be determined by taking their freezing points with a 0.1° thermometer (see Plate I). The composition of liquid alloys was determined chemically by titrating a weighed sample with standard HCl.

15. Samples of the salt residues were taken at intervals along the column and analyzed for residual potassium carbonate by titrating a weighed sample with standard HCl. Traces of hydroxide were readily determined by the addition of an excess of a BaCl₂ solution to a water solution of the carbonate sample and titrating with HCl, using phenolphthalein as indicator. Mixed chloride samples were analyzed by titrating a known weight with a standard silver nitrate solution.

DIFFICULTIES

16. The most difficulty was experienced with the sodium injection. In spite of all ordinary precautions, some of the metal in the column reacted with oxygen or water from the salt. The resulting oxide or hydroxide fused at the temperature of the reaction, ran down into the cooler portion of the injector pipe, and solidified. Before injecting another portion of sodium, this plug had to be heated above 350°C. to melt it. Sometimes, even heating with a torch did not clear a passage, so that the run had to be stopped and the column washed out. Probing with a wire through the bottom end of the injector pipe tee before a run helped somewhat but even this failed much too often.

17. Finally a 3/8" S.S. pipe was welded to the side of the column shell 3" from the bottom. Occasionally this also had to be probed with a rod before the sodium could be injected but here only a thin film of oxide (or hydroxide) was met with and this could easily be broken.

18. Unless the sodium was extremely clean, some metal stuck to the sides of the metering injector, thus introducing less sodium than calculated. A thin oxide film on the sodium offered poor electrical contact so that the volume of sodium injected was subject to further error. For these reasons, a minimum value for the sodium injection was calculated from the weight and analysis of the distillate. As the salt residues were quite free from metal and hydroxide (less than 1%), this value was more accurate than that obtained from the metering injector. The wall effect should decrease considerably on a larger unit.

19. Silver solder joints and brass cocks seemed to fail if they came in contact with hot metal (hydroxide?) for prolonged periods of time. The exact cause of this was not investigated.

EXPERIMENTAL DATA

20. The results of reactions carried out between sodium and potassium carbonate at 50 mm. pressure are presented in Table I. The results of the single experiment using potassium chloride are also given. This latter experiment had to be

Table I

Results of Reactions Between Sodium and Potassium Carbonate at 50 mm. Pressure

Exp. No.	Average Composition of Total Distillate (Atom % K)	Composition of Initial High Purity Fraction (Atom % K)	Yield of High Purity Alloy (Wt. % Total Distillate)	Boiling Rate of Sodium (#/hr.)		Production Rate of High Purity Alloy (# K/hr.)	Sodium Used (#)
				Na	$1/2 K_2CO_3$		
14	97	99.8	95	0.95	1.48	1.32	2.47
11	97	99.1	93	1.04	1.44	1.33	3.11
3	97	--	--	0.84	1.32	1.24	1.98
16	96	99.8	83	0.98	1.39	1.05	3.02
6	96	99.5	95	0.85	1.58	1.33	2.37
8	96	99.4	89	0.95	1.95	0.95	2.77
9	96	99.3	85	0.77	1.79	1.77	2.69
13	95	99.8	87	1.00	1.32	1.31	2.64
15	95	99.4	87	0.95	1.44	1.52	2.87
5	94	99.6	94	0.89	1.11	0.75	2.42
10	94	99.3	89	0.89	1.78	1.37	2.65
22	93	99.7	66	0.99	2.12	1.90	3.17
12	93	98.9	88	0.92	1.35	1.15	2.17
7	92	99.6	84	0.94	1.28	1.43	2.78
19	83	99.5	50	0.81	2.96	2.12	2.47
				<u>1" K₂CO₃ Pills</u>			
4	86	--	--	0.93	1.34	0.93	2.46
				<u>KCl Pills</u>			
23	(40)	99.1	(35)	0.95	0.95	--	3.8

() Estimate

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Table II

Experimental Conditions for Reactions Between Sodium and Potassium Carbonate

<u>Exp. No.</u>	<u>Average Composition of Total Distillate (Atom % K)</u>	<u>Ring Packing at Top (Inches)</u>	<u>Rate of Reflux Air (cfm)</u>	<u>Temperature of Salt Sections as Sodium Started Boiling (Sections) (Temp.)</u>	
14	97	11	1/2	1-3	650
11	97	11	1/2-1	1-5	600
3	97	11	1/2	1-4	575
16	96	0	2-1-0	1-3	650
6	96	11	1/2	1-5	625
8	96	11	0	1-5	600
9	96	0	0	1-5	600
13	95	11	0	1-3	650
15	95	0	1/2	1-3	650
5	94	11	1	1-5	600
10	94	11	0	1-5	600
22	93	0	0	1-4	650
12	93	11	0	1-5	600
7	92	11	0	1-6	500
19	83	0	0	1-4	675
<u>1" K₂CO₃ Pills</u>					
4	86	11	1	1-5	600
<u>HCl Pills</u>					
23	(40)	12	0	1 2-4	600 550

stopped after all the sodium had evaporated but only approximately 70% of the total metal had distilled because a bad leak developed. The results, however, were sufficiently conclusive to be included.

21. Column 3 of Table I gives the composition of the alloy of high purity that distilled over initially. This high purity alloy comprised the weight percent of the total distillate given in column 4. The equivalent ratios presented in column 5 represent the ratio of the number of atoms of sodium used to the total equivalents of potassium carbonate (or KCl) used. The rates of production of high purity alloys presented in column 7 does not include the time necessary to remove the residual metal from the salt.

22. The conditions of packing, reflux rate, and temperature under which each of the above experiments was carried out are presented in Table II. The reflux air rates presented in column 4 of Table II are integrated values. When two or more are given for one experiment, all were effective approximately equal lengths of time in the order given. Column 6 presents the temperature, as the sodium began to boil, of the lower heating sections given in column 5. The heating sections above those presented in column 5 were all at 500°C. initially.

DISCUSSION

23. Table I shows that the initial distillate fractions, comprising approximately 90% of the total distillate, uniformly analyzed above 99 atom percent potassium. The average composition of the total distillate was somewhat lower (approximately 96 atom percent potassium). By the proper selection of operating conditions, an initial 95% fraction of the total distillate analyzing 99 1/2 atom percent potassium was obtained. The total distillate averaged 97 atom percent potassium under the same operating conditions.

24. The initial distillate compositions varied only between 99 and 100 atom percent potassium and the average compositions of the total distillate between 92 and 97 atom percent potassium under the various operating conditions employed, so that it was difficult to correlate positively the condition and its result. Certain correlations, either positive or negative, which the data does indicate are presented below.

25. No effect on the composition of the distillate could be observed when the equivalent ratios were varied from 0.8 to 1.0 or the boiling rate of sodium from 1.1 to 2.0 pounds per hour. Sodium boiling rates greater than two pounds per hour, however, yielded total alloys considerably poorer in potassium (experiments No. 19 and 22).

26. Experiments No. 14 and 15 were conducted under as nearly identical conditions as possible, with the exception that in No. 15 the ring packing was replaced by additional potassium carbonate which was included in the calculation of the equivalent ratios. With salt packing, the total distillate composition decreased from 97 to 95 atom percent potassium. Experiments No. 8 and 9, however, were also carried out under similar operating conditions except for packing, but in this case the purity of the resulting potassium alloy was unchanged as shown in Table III. These results indicate that the fractionating efficiency of a salt packing was approximately equal to that of a packing composed of iron rings.

Table III

Effect of Type of Packing on the Total Distillate Composition

<u>Exp. No.</u>	<u>Packing Type</u>	<u>Average Composition of Total Distillate</u>
14	Rings	97 M % K
15	Salt	95
8	Rings	96
9	Salt	96

27. To insure good fractionation, some liquid metal was necessarily retained in the column during the reaction and subsequent distillation. This was accomplished by passing an air stream through the reflux condenser (forced reflux) and/or by reducing the heat input to the top salt section (ordinary reflux). The ordinary reflux rate was determined by the heat insulating properties of the column which, in this column, yielded a maximum reflux rate of approximately 1/4 pound of potassium per hour. One cubic foot per minute (cfm) of forced reflux yielded approximately 2/3 of a pound of potassium reflux per hour.

28. The experiments in which a small amount of forced reflux was used in addition to the ordinary reflux appeared to yield slightly better potassium alloys than those experiments in which no forced reflux was used. Experiments No. 16 and 13 were carried out under very nearly identical conditions with the exception that 2-1-0 cfm forced reflux was employed in No. 16 while only the ordinary reflux was effective in No. 13. The composition of the total distillate, however, was changed only from 96 atom percent potassium in No. 16 to 95 in No. 13.

29. Due to the high heat of vaporization of sodium and potassium, the metal condensed on the salt and packing was very difficult to remove. This is shown by the results of experiments No. 16 and 13 presented in Table IV. For No. 16 (2-1-0 cfm forced reflux), 62% of the total distillate was obtained 70 minutes after the sodium boiler had been emptied, while 87% of the total distillate was obtained in the same interval of time for No. 13 (no forced reflux).

Table IV

Effect of Reflux on Time of Removal
of Metal from the Salt

<u>Exp. No. 16</u> <u>(2-1-0 cfm forced reflux)</u>		<u>Exp. No. 13</u> <u>(no forced reflux)</u>	
<u>Yield as Wt.%</u> <u>of Total</u> <u>Distillate</u>	<u>No. of minutes</u> <u>after Sodium</u> <u>Boiler was</u> <u>Emptied</u>	<u>Yield as Wt.%</u> <u>of Total</u> <u>Distillate</u>	<u>No. of minutes</u> <u>after Sodium</u> <u>Boiler was</u> <u>Emptied</u>
46	30	72	30
62	70	87	70
76	110		
83	150		

It was therefore advantageous to reduce the liquid metal hold-up to the absolute minimum necessary to carry out the desired fractionation. This was done by reducing the reflux rate and increasing the initial temperatures of the lower salt sections.

30. No correlation could be observed between the temperature of the lower salt sections initially and the purity of the resulting potassium, provided that sufficient liquid metal remained in the column for fractionation. In experiment No. 13, no forced reflux was used, and the bottom three heating sections were at 650°C. throughout the reaction, yet the initial 87% of the total distillate analyzed 99.8 atom percent potassium and the total distillate averaged 95 atom percent potassium. Thus the fractionation resulting from ordinary reflux alone appears to be sufficient to produce high purity potassium alloys.

31. After the sodium pot was emptied, the hold-up metal was removed from the salt by heating all the column sections above 650°C. and then evacuating completely for 30 minutes. This was done in all experiments except Nos. 7, 12, 19 and 22.

In the latter experiments all the salt sections except the uppermost one were heated to 650°C. and then a vacuum applied. Uniformly poorer total potassium alloys resulted in these four experiments.

32. After the sodium had all evaporated, an additional 1/2-3/4 of an hour was required to remove 80% of the total distillate from the salt, provided that the metal hold-up was not excessive. Approximately 90% of the total metal distilled in a little over an hour after the sodium boiler was empty. The total time necessary for each experiment was 3-4 hours, of which 1/2 hour was required to remove the residual metal from the salt. The time required to heat the column to its starting temperature is not included.

33. Considerably more time was required to remove the residual metal from the salt in some of the experiments than in others because the most efficient procedures had not yet been developed. For this reason the potassium yields per hour of total operating time were inconsistent with the operating conditions. The potassium production rates given in column 7 of Table I, however, are independent of the time necessary to remove the residual metal from the salt because only the operating time required to obtain the yield of high purity alloy given in column 4 was used in this calculation. The production rates given in the table are somewhat higher than the actual over-all production rate but they are dependent only on the conditions of reaction, not on the method of removal of the residual metal from the salt.

34. The 1" K_2CO_3 pills used in experiment No. 4 were too large to react completely as is shown by the salt residue analyses of the inside and outside of the pills given in Table V. The residue analyses for experiment No. 14 are included in Table V for comparison and also to show that the bottom salt sections were completely reacted even though they were kept at 650°C. throughout the experiment.

35. Pure potassium can be obtained in a column of this type from the reaction of sodium with potassium chloride, but the yield of high purity alloy is only a small fraction of the total distillate yield. This is shown by the initial distillate compositions given in Table VI for experiment No. 23. Distillate samples No. 1-4 comprise approximately 70% of the total distillate. The residue compositions given in Table VII show that there was no enrichment along the salt column as takes place with potassium carbonate. The enrichment that did occur was due solely to fractionation at the top of the column.

36. It is interesting to note that in experiment No. 23, although the reaction progressed for five hours at 50 mm.

pressure, less than 0.5 gram of mixed chlorides distilled into the condenser arm. No distillation of mixed carbonates was observed.

Table V

Composition of Salt Residues.

Exp. No. 4			Exp. No. 14	
<u>(K₂CO₃ Pills)</u>			<u>(K₂CO₃ Aggregated by Spraying)</u>	
<u>Inches from Bottom of Salt Container</u>	<u>Mol % K₂CO₃ (Inside of Pill)</u>	<u>(Outside of Pill)</u>	<u>Inches from Bottom of Salt Container</u>	<u>Mol % K₂CO₃</u>
0	0.9	1.4	0	0.0
11	24.2	7.9	10	0.1
22	21.9	15.1	20	0.6
33	28.6	20.9	30	5.5
44	36.3	22.1	40	22.3
50	70.5	41.7	50	51.3

Table VI

Initial Distillate Samples for the Na-KCl Reaction

Exp. No. 23 (KCl Pills)

<u>Sample No.</u>	<u>Wt. of Sample</u>	<u>Atom % K</u>
1	0.28#	99.8
2	1.41	99.0
3	1.39	47.5
4	0.58	30.0

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Table VII

Analyses of Mixed Chloride Residues
from Experiment No. 23

<u>Inches from Bottom of Salt Container</u>	<u>Mol % KCl</u>
0	76
14	71
29	74
53	90

THE EQUILIBRIUM BETWEEN SODIUM METAL AND POTASSIUM SALTS

36. Data published by E. Rinck in the Annales De Chimie, Vol 18, page 395, 1932, on the equilibrium between alkali salts and alkaline metals cover the reactions between sodium and the potassium halides and potassium hydroxide. Plates 2 to 6 inclusive, show the atom percentage of potassium in the alloy in equilibrium with the potassium-sodium salts for the reactions given. The reactions were carried out above the melting point of the salt phase. These reactions take place with a small heat of reaction, some being slightly endothermic and the others being exothermic, which results in no appreciable change in the equilibrium constant with temperature. It is worthy of note that the two reactions giving most favorable results with respect to potassium metal production have the lowest heats of reaction. The KF reaction is exothermic to the extent of 2.1 kilogram calories per mole and the KOH reaction is endothermic by approximately the same amount. The heats of reaction for the chloride, bromide and iodide with sodium at ordinary temperature are endothermic by 6.7, 7.0 and 8.7 kilogram calories per mole respectively.

37. The literature values for the heats of reaction of the K_2CO_3 with sodium indicates this reaction is endothermic to the extent of 4.4 kilogram calories per mole at ordinary temperatures and tends toward a very small heat of reaction or even to an exothermic reaction as the temperature is raised. The shape of the curves obtained bear this out since it is similar to those of KF and KOH. However, the measurements made indicate the reaction to be more favorable than the KF and KOH reactions. The values obtained are plotted on plate 7. Apparatus and method are described in Appendix II and Appendix II, plate 1. Two curves have been drawn which are possibly the most and least favorable curves indicated by these data. The exact values are in doubt due to the difficulty in obtaining

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and analyzing representative samples, yet the data probably indicate the general shape and location of the equilibrium curve. Should the values fall as indicated the potassium carbonate would give the most favorable equilibrium of the salts considered herein.

CONCLUSIONS

38. The production of potassium by the method described results from two separate processes carried out simultaneously. The reaction of sodium with the carbonate produces a mixture of sodium and potassium metal and a corresponding mixture of sodium and potassium carbonate. The mixed metal is then fractionated in the column of the carbonate. Under the present conditions the potassium leaves the column as vapor and the sodium "runs back" or reacts with the carbonate to further shift the reaction in the desired direction. The metal mixture can be fractionated on metallic column packing leaving the sodium to return to the boiler (see also report P-2011).

39. Sodium-potassium alloys averaging 97 atom percent potassium were obtained from the reaction of sodium vapor with solid potassium carbonate at 50 mm. pressure in a fractionating-reaction column. The initial high purity alloy comprising 95% of the total distillate, averaged 99 1/2 atom percent potassium under the same conditions.

40. The packing of iron rings was replaced by additional potassium carbonate without materially affecting the purity of the product.

41. Sufficient fractionation was effected without forced reflux by reducing the heat input to the top salt section. The top two salt sections were at the boiling point for pure potassium (535°C.) at the beginning of the reaction while the remaining salt sections were at the boiling point for pure sodium (650°C.).

42. A 1:1 equivalent ratio of sodium to potassium carbonate and a sodium boiling rate of 2 pounds per hour were used.

43. The removal of metal residues was accomplished most advantageously by heating all the salt section to 650°C. at the end of the reaction and then evacuating for 30 minutes.

44. Approximately 90% of the metal had distilled one hour after the sodium in the boiler had all evaporated. Better than one pound of pure potassium was obtained per hour of total operating time.

45. One inch diameter potassium carbonate pills were too large to react completely.

46. High purity potassium alloys were obtained when potassium chloride was used in place of potassium carbonate but the yield amounted to only 35% of the total distillate.

47. Literature data has been added for the equilibrium for the reaction of sodium metal with the potassium halides and potassium hydroxide. Values obtained at this Laboratory are given with discussion for the reaction of sodium metal with potassium carbonate. These measurements indicate the reaction at equilibrium to be quite favorable to potassium production, the exact values however were not obtained.

WORK IN PROGRESS

48. The production of potassium from the potassium carbonate-sodium reaction in the fractionating-reaction column has been carried up to pilot plant scale. It is operating at present and results will be reported when sufficient data on operations are available.

49. Reactions of sodium with the various potassium salts with simultaneous fractional distillation of the alloy are being investigated. This work is being done at temperatures where the salt phases are liquid and with the metal reflux in the fractionating column returning to the salt container. An inert packing (stainless steel) is being used in this column.

50. Further work is planned on the equilibrium data for reactions to be studied where such data are not available. Good equilibrium data on these reactions will be of considerable value in deciding which method (the fractionating-reaction column with solid salt or the liquid phase reaction with simultaneous distillation) is the more advantageous.

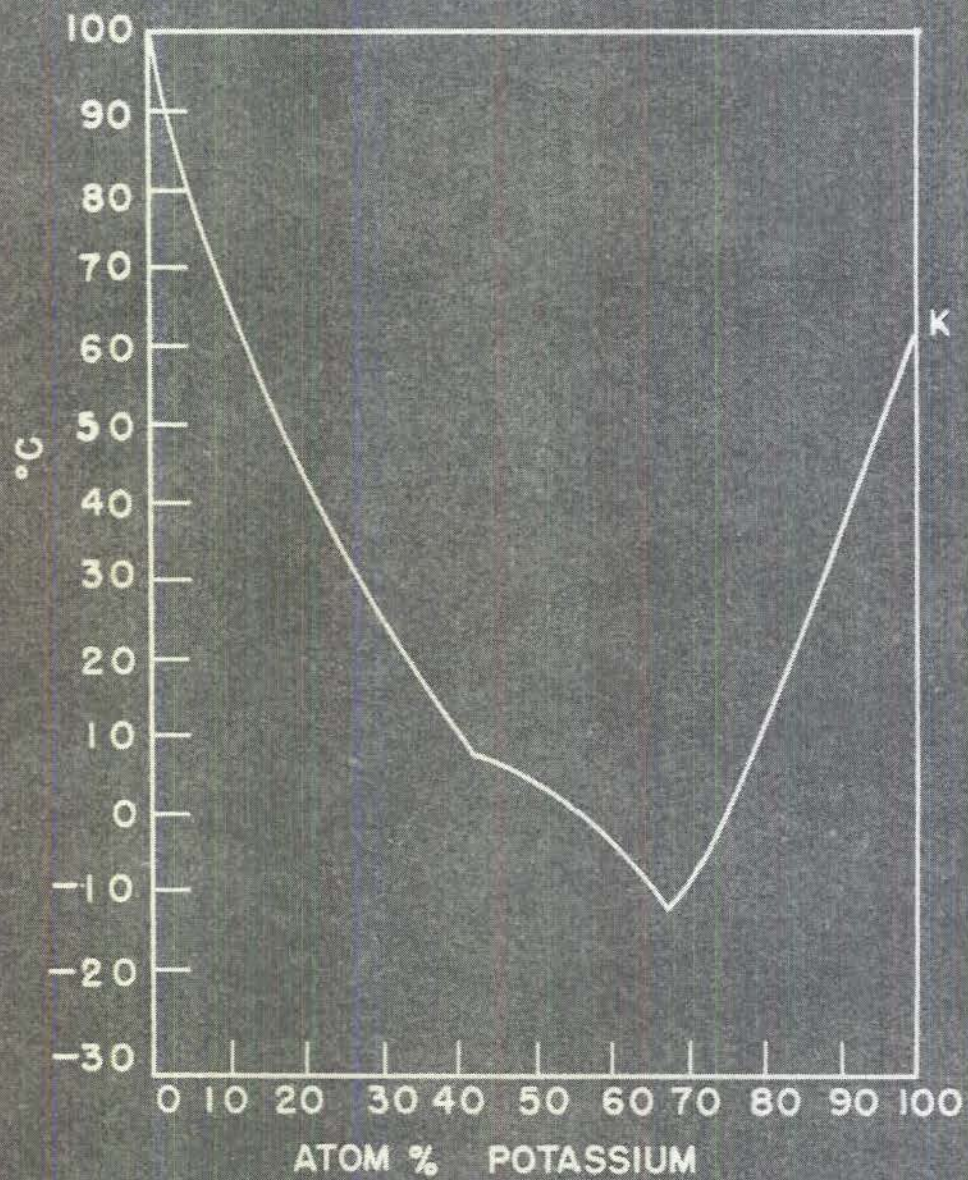


PLATE I

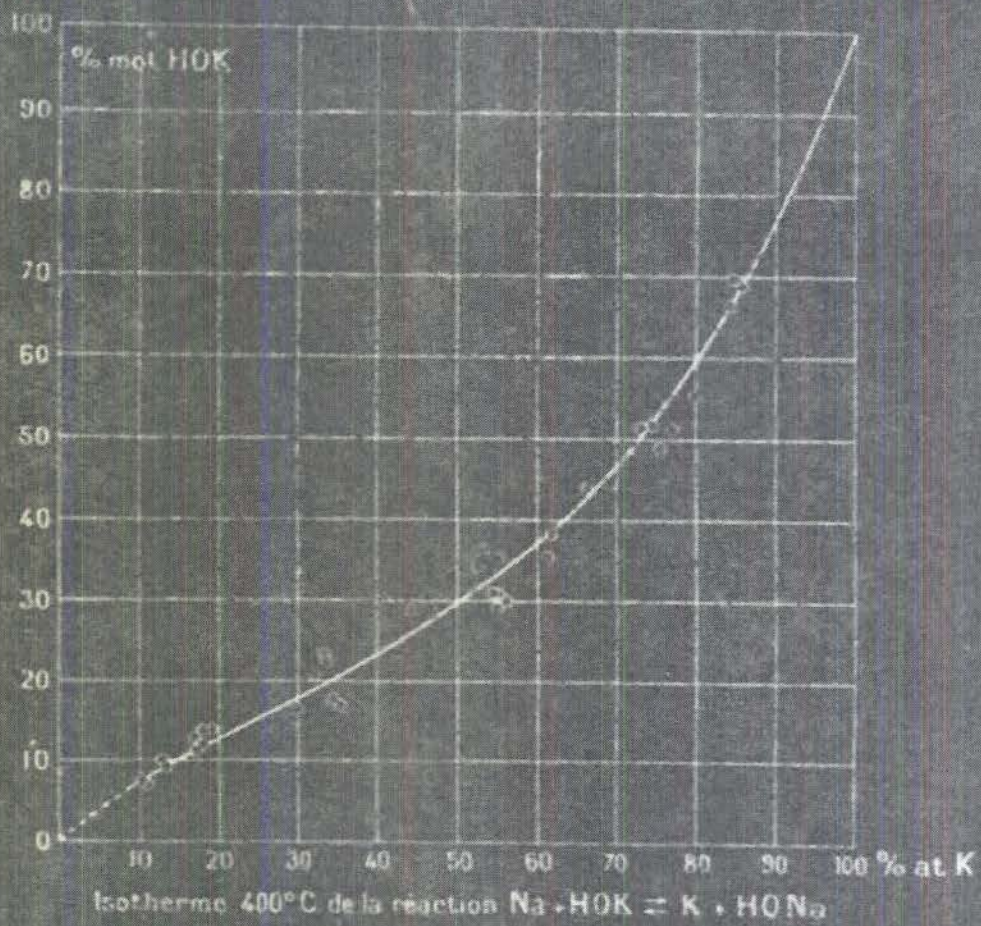
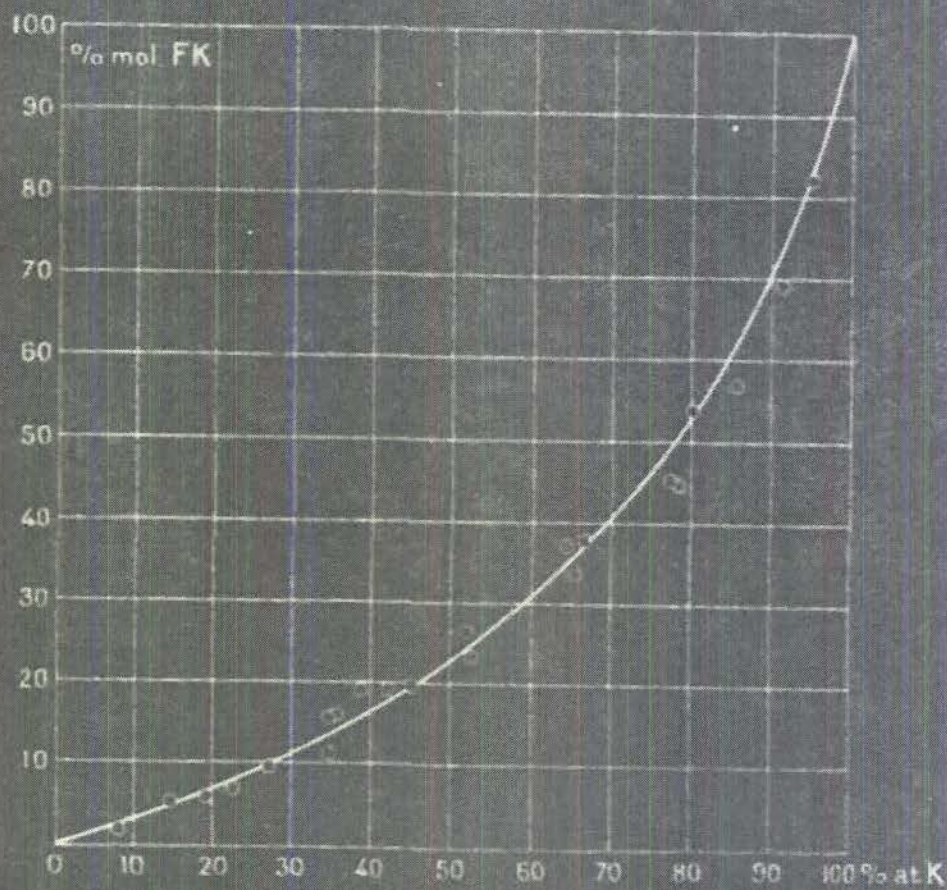
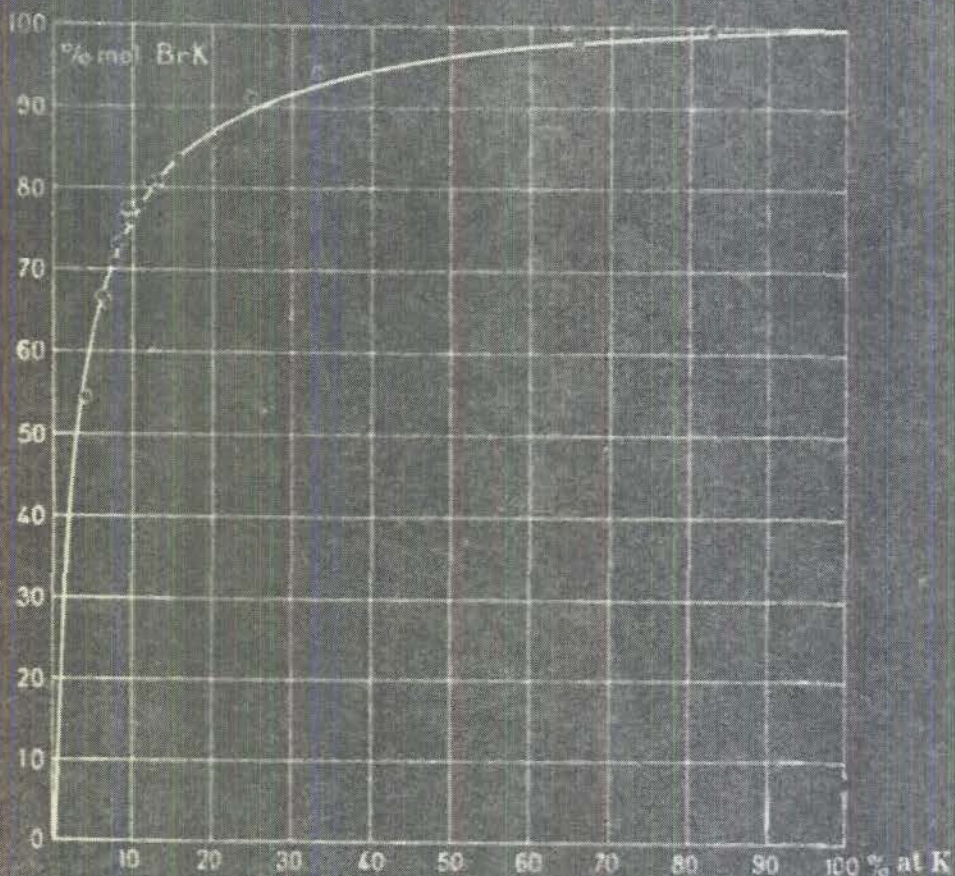


Fig. 3.



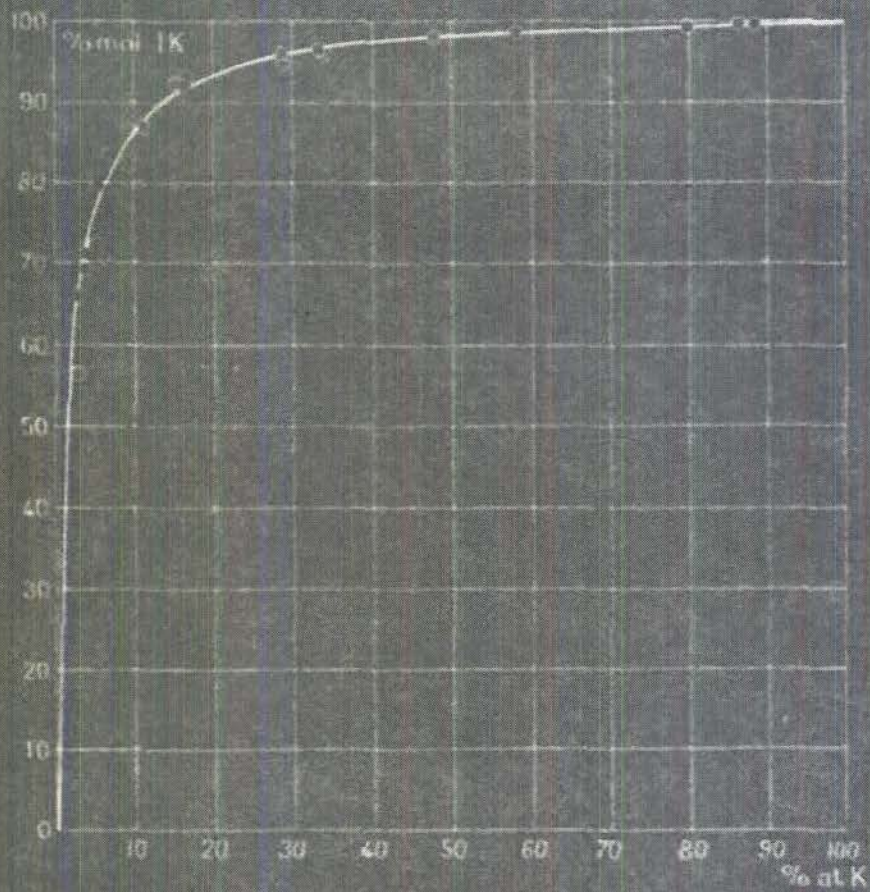
Isotherme 1000°C de la reaction $\text{Na.FK} \rightleftharpoons \text{K.FNa}$

Fig. 11.



Isotherme 800°C de la réaction $\text{Na} + \text{BrK} = \text{K} + \text{BrNa}$

Fig. 7.



Isotherme 800°C de la reaction $\text{Na.IK} \rightleftharpoons \text{K.I Na}$

Fig. 9.

PI ATF 5

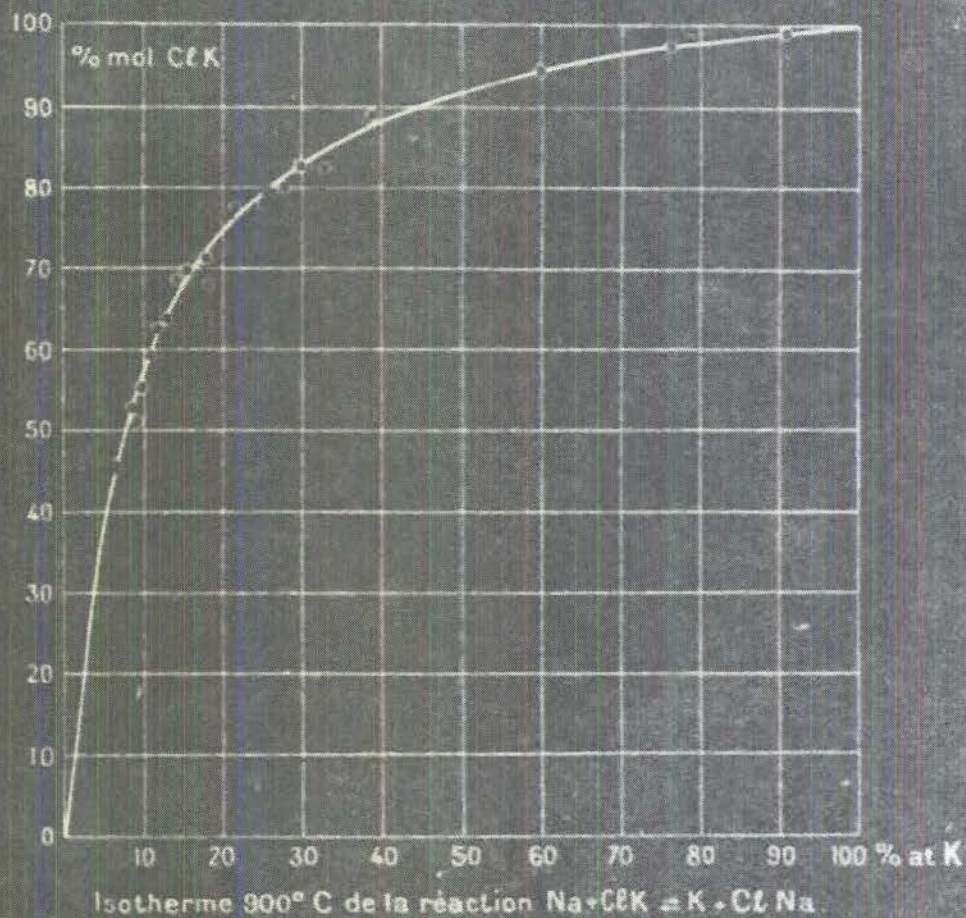


Fig. 5.

$C = \text{CONCENTRATION}$

$$\epsilon = \frac{C_K \cdot C_{Na_2CO_3}}{C_{Na} \cdot C_{K_2CO_3}}$$

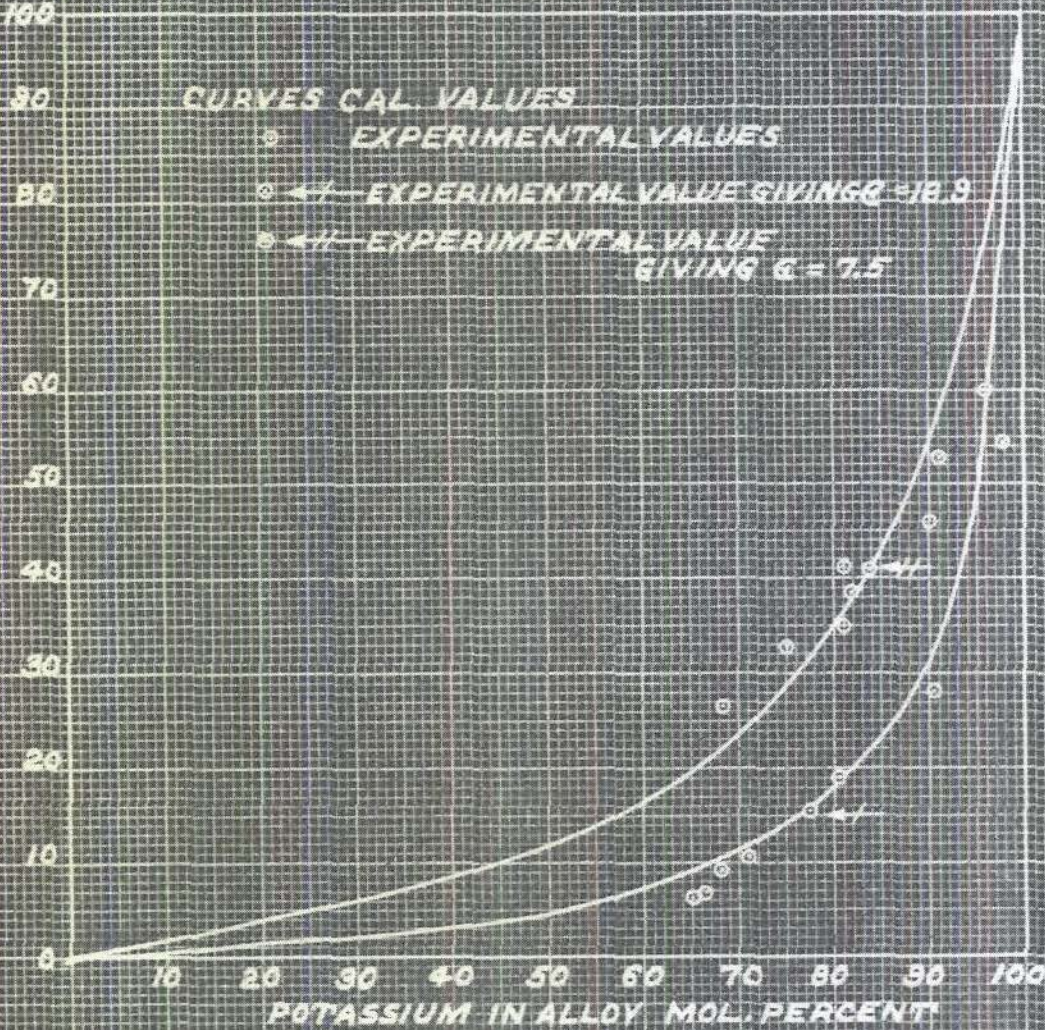
CURVE #1 PLOTTED FOR $\epsilon = 7.5$

CURVE #2 PLOTTED FOR $\epsilon = 10.9$

POTASSIUM CARBONATE IN SALT PHASE - MOL. PERCENT

CURVES CAL. VALUES

- EXPERIMENTAL VALUES
- ← EXPERIMENTAL VALUE GIVING $\epsilon = 10.9$
- ← EXPERIMENTAL VALUE GIVING $\epsilon = 7.5$



APPENDIX I

DESCRIPTION OF THE DISTILLATION-REACTION APPARATUS USED

Stainless steel used was 18-8 chrome nickel steel, type 302.

Reaction Column: 4" O.D. x 3/32" x 96" S.S. tubing.

Condenser: 7/8" O.D. x 1/16" S.S. tubing welded to the column shell 15" from the top. A 1/2" brass steam cock and the female portion of a 1/2" iron union were silver soldered to the exit end of the condenser arm.

Injector Pipe: 1/8" x 11" S.S. pipe welded to a 1/4" S.S. plate at the bottom of the column shell. A 1/8" S.S. tee was silver soldered to the bottom of the injector pipe.

Seat: Two 60° taper, mated, steel conical rings. The female was pinned to the inside of the column shell, 10" from its bottom; the male to the bottom of the salt container.

Sodium Boiler: 3 13/16" diameter x 10" chamber extending from the bottom plate of the column shell to the seat.

Baffles: Thirteen 4 15/16" O.D. x 4 1/32" I.D. x 1/16" steel rings welded to the outside of the column shell at intervals of 6".

Support Flanges: Four 1" x 3" x 1/8" S.S. strips welded horizontally to the bottom edge of the column shell. These rested on brick and supported the weight of the column.

Top Closure: 3 3/4" O.D. x 1/16" x 11 1/2" steel tubing. A closed chamber, 2" O.D. x 1/16" x 5", was welded to the bottom of the closure. This chamber was equipped with a baffle and 1/4" pipe inlet and exit tubes for reflux air.

Top Flanges: A 6" O.D. x 4" I.D. x 1/2" grooved flange welded to the top of the column shell, and a 6" O.D. x 3 3/4" I.D. x 1/2" tongued flange welded to the top closure. These were sealed with a lead gasket.

Potassium Carbonate Container: 3" O.D. x 1/16" x 66 1/2" S.S. tubing with a 3 7/8" guide welded at the top.

Perforated Disc: 3" x 1/16" steel plate, perforated with 115 3/16" holes, and fitted to the bottom of the salt container.

Packing: 3/8" O.D. x 1/16" x 3/8" iron Raschig rings.

Packing Container: 2 3/4" O.D. x 1/16" x 12" tubing closed at the bottom with a perforated plate. This slipped into the top of the salt container and was held in place by means of a 3 1/2" flange welded to the top of the packing container.

Metering Injector: 4" O.D. x 1/8" x 24" tubing, closed at the top with a tongue and groove flange, and at the bottom with a 1/4" Pratt and Cady S.S. angle valve. A union connected the exit pipe from the valve to the injector pipe.

Bulk Injector: 10" x 10" x 3 1/2" steel container (held three 2 1/2# sodium blocks) with a removable top and fitted at the bottom with a 1/2" steam cock and the female portion of a 1/2" union. The male portion of the union was on the top of the metering injector.

Potassium Receivers: Two 6" O.D. x 1/8" x 14" and four 3" O.D. x 1/16" x 6" Iron containers, all fitted at the top with 1/2" male unions and at the bottom with 1/4" unions. A side-arm on each connected with the vacuum line.

Column Heaters: Seven 12" heating lengths (lettered H₀ - H₆ from bottom to top), individually controlled, extended the length of the column. These consisted of 35' of 3/16" x 0.0285" chromel ribbon spirally-wound on 5" I.D. x 3/8" alundum cores.

Sodium Heater: Two 6" x 13" x 1/2" series-connected Globars, placed horizontally 1" below the column shell.

The condenser arm and sodium injector were also heated electrically. Individual control was obtained with ten 2 kvà Variacs.

Temperatures: The salt temperatures (T₁ - T₆) were measured with six thermocouples placed at the mid-points of the six salt-heating elements and extending horizontally through the insulation and alundum core to the outside of the column shell. A 1/8" S.S. pipe extending 1/2" into the sodium pot from the bottom was used to measure the sodium temperature (T₀). Another 1/8" S.S. pipe well extending 1" below the reflux chamber of the top closure contained a thermocouple to measure the metal vapor temperature (T₇) as it left the salt. These temperatures were recorded on three Brown recording potentiometers.

Reflux Air: The rate of flow of the reflux air was measured with a calibrated Hays Flowmeter.

Purified Nitrogen: Residual oxygen and water was removed from the water pump nitrogen by passing it first through copper turnings at 450°C. and then through a potassium hydroxide drying tower.

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Vacuum: A Cenco Mega-Vac pump was of ample capacity to completely evacuate the system in several minutes.

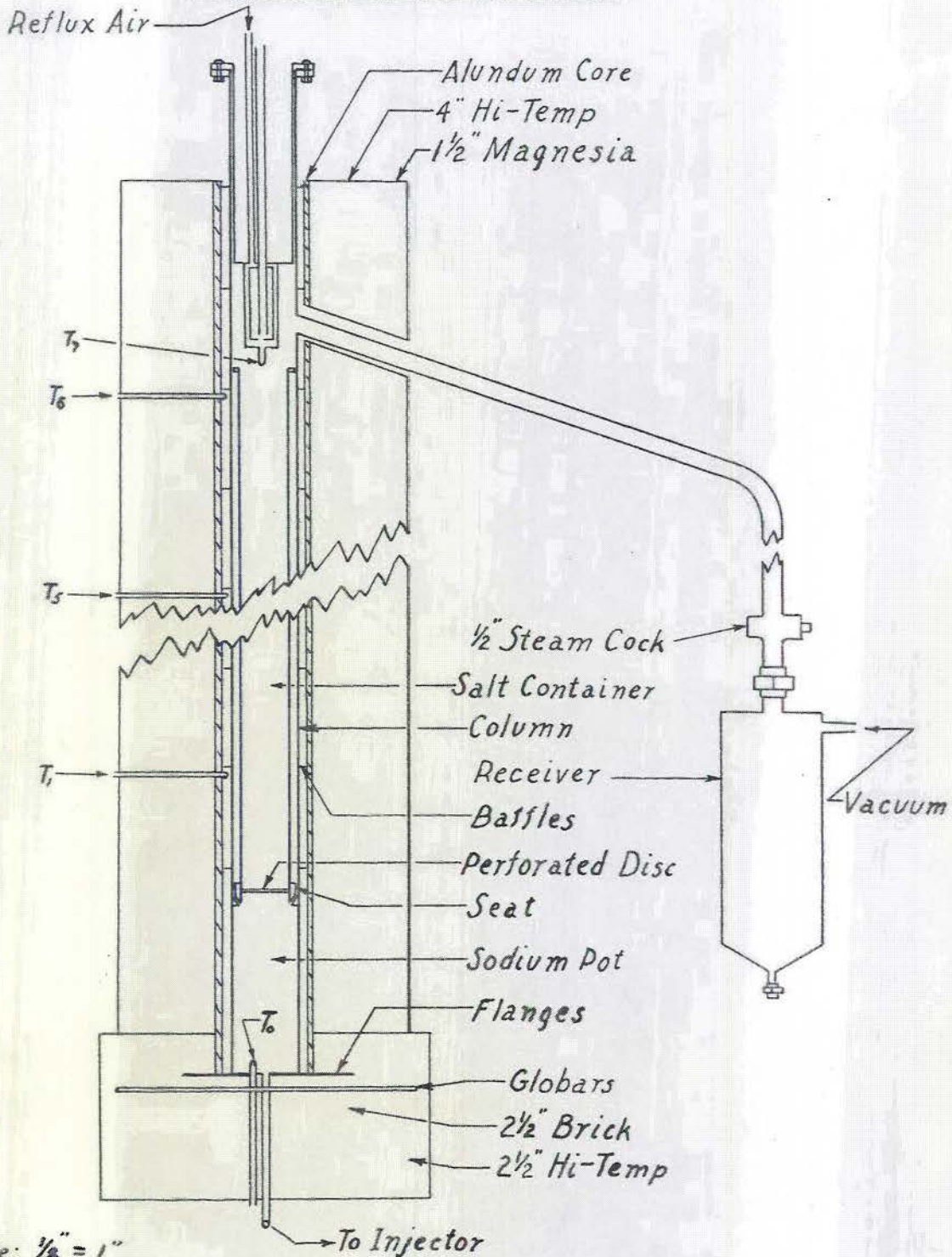
MATERIALS

Niagara Alkali Company granular potassium carbonate was sprayed on trays with 30% by weight of water and dried for six hours at 135°C. It was then crushed, sifted between 1/4" and 3/4" screens and redried until ready for use. Its apparent density (weight divided by the volume of salt container it occupied) was approximately 0.70 g./cc. A commercially prepared sample of aggregated potassium carbonate, furnished by the Mallinckrodt Chemical Works, was used in experiment No. 12.

Dry granular potassium carbonate was pressed into 1" diameter x 1" pills at 5000 psi pressure. The density of each pill was approximately 1.5 g/cc. while the apparent density in a 3" tube was 0.65.

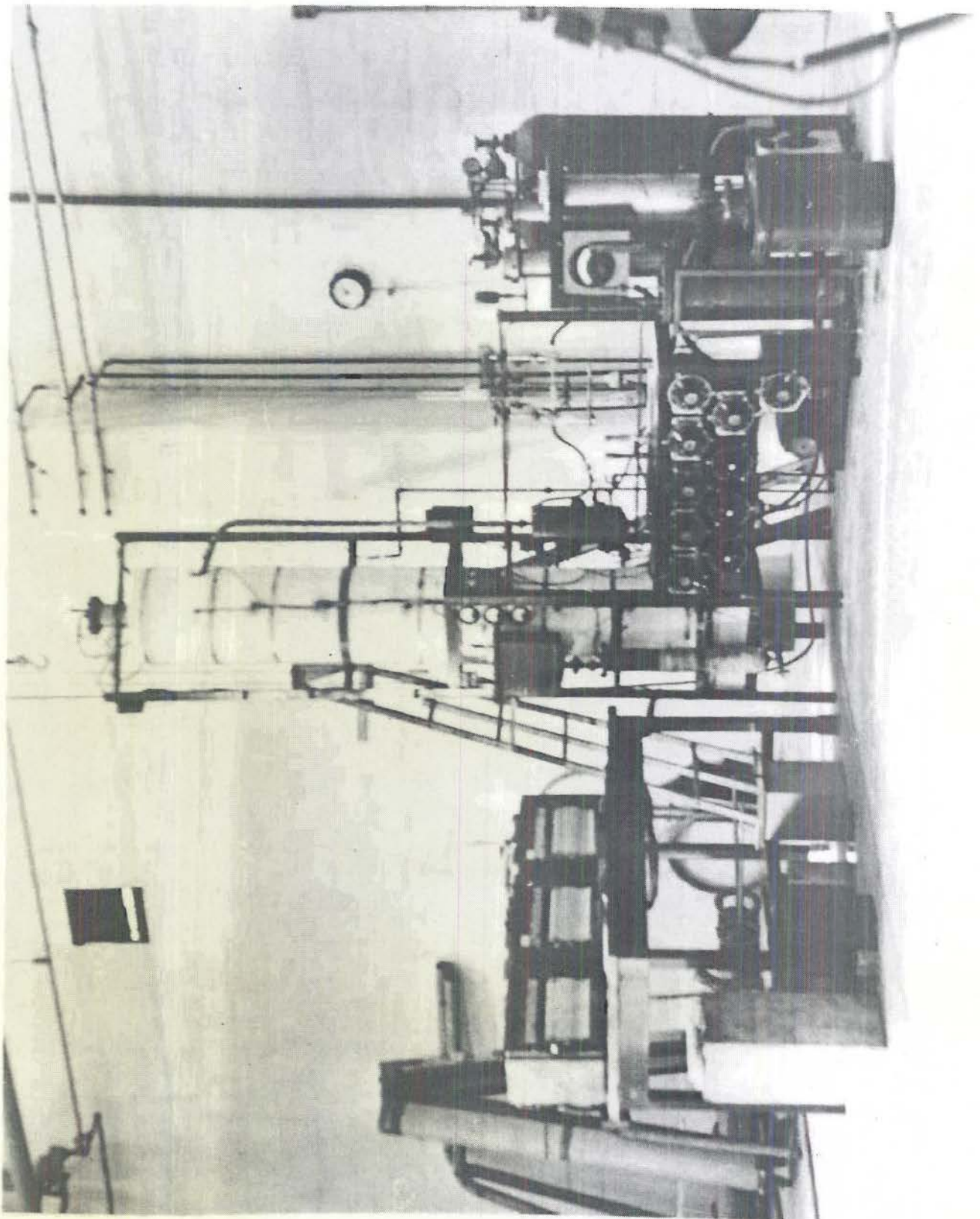
Dry potassium chloride was pressed in a pill machine to 3/8" diameter x 1/8" pills. The apparent density was 1.1 g./cc.

6' REACTION COLUMN



Scale: 1/8" = 1"
Witschonke
010843

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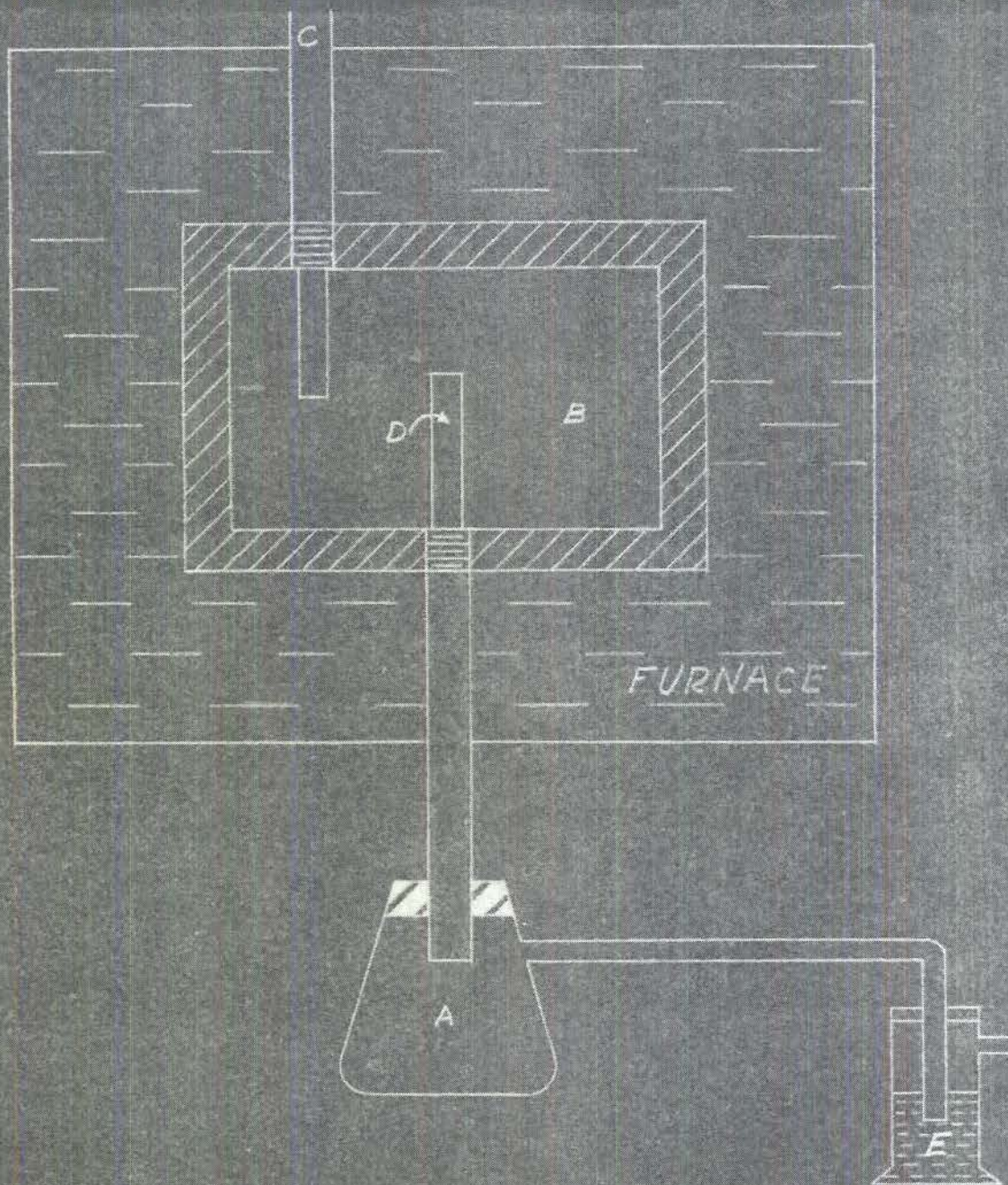
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APP. 1

PLATE 2

removing of vapor which differs from the composition of the alloy will shift the equilibrium as the distillation proceeds. Since the metal sample collected was a small fraction of the total metal present (approx. .025 out of 25 grams) the shift of the equilibrium in these experiments should not be greater than the experimental error in the analytical determinations or calculations based on these analyses.

The equilibrium measurements made were always with temperatures below the melting point of the salt phase.



REACTION POT AND TRAIN FOR COLLECTING
ALKALI METAL SAMPLE

- A - GLASS SAMPLE RECEIVER
- B - IRON REACTION POT
- C - FILLING TUBE AND N_2 INLET
- D - CONDENSATE TUBE
- E - MERCURY SPILLING TRAP FOR N_2