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NAVY DEPARTMENT

The Twelfth Partial Report  
on  
Oxygen Source Material

Burning of Potassium Metal to Potassium Tetroxide  
with a Devilbiss Spray Gun

NAVAL RESEARCH LABORATORY  
ANACOSTIA STATION  
WASHINGTON, D. C.

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Work Done and  
Prepared by:

L. M. Foster, Contract Employee

Reviewed by:

R. R. Miller, Senior Chemist

P. Borgstrom, Superintendent,  
Chemistry Division

Approved by:

A.H. Van Keuren, Rear Admiral, USN,  
Director

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ABSTRACT

This report concerns the production of potassium tetroxide by burning essentially pure potassium in the liquid state by means of an aspirator type spray gun.

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## AUTHORIZATION

1. Bureau of Ships Project Order 243/42, S77-2(275) dated 30 September 1942 and Bureau Ships' letter S77-2(275) dated 16 October 1942.

## STATEMENT OF PROBLEM

2. Partial report on production and investigation of oxygen source materials. --- Producing potassium tetroxide from potassium metal.

## INTRODUCTION

3. The burning of potassium-sodium alloys was attempted at the Naval Research Laboratory to see if the pure potassium and high potassium alloys being produced here could be converted successfully to pure potassium tetroxide or potassium tetroxide-sodium peroxide mixtures of high available oxygen content. The Mine Safety Appliances Co. of Pittsburgh, Pa., has been successfully burning potassium-sodium alloys of lower potassium content for some time and on one occasion burned essentially pure potassium to give a good yield of oxide which liberated nearly the theoretical amount of oxygen on analysis. It was suggested by Mr. Jackson of that concern that this method be tried at the Naval Research Laboratory with modifications necessary to adapt the process to the method of potassium manufacture being developed.

4. Two methods of burning potassium-sodium alloys have been employed. The metal can be burned in the vapor state by vaporizing it in a suitable container enclosed in a furnace. The vapor is carried in a stream of nitrogen into a large hopper where it is burned in an air stream. This method has the disadvantages of requiring a high temperature to vaporize the metal and of involving a considerable loss of metal in the form of a "drip" of molten oxide which forms on the end of the vapor nozzle and is a total loss. Also the hard lumps of this material must be removed from the product before it is catalyzed and pressed. This is a bothersome procedure and necessitates handling the oxide more than is desirable. The loss of metal in this manner may amount to as much as ten percent.

5. The other method of producing potassium tetroxide, that which is reported here, consists of burning the metal in the liquid state in a stream of air. An aspirator type paint spray gun, manufactured by the DeVilbiss Co. of Toledo and modified at this Laboratory, was found very satisfactory for this purpose.

6. The apparatus used, results obtained and difficulties encountered in this process are reported.

## DESCRIPTION OF APPARATUS

### 7. Alloy Container

The metal to be burned was held in the container shown in Plate 1. It is cylindrical with a conical bottom to facilitate emptying. It was fabricated from 1/8 inch black iron and is of welded construction throughout. The lid is a 9 inch disk of 3/16 inch plate equipped with necessary fittings as shown. The container has a capacity of approximately 225 pounds of metal and can be charged by means of the filling hole shown while burning is in progress.

8. An inert atmosphere of nitrogen is kept above the metal at all times to prevent oxidation on the surface. Normally, no pressure is applied above the metal as sufficient vacuum is produced at the gun tip to suck the alloy into the gun (see Table 1). This vacuum decreases, however, as a tip becomes worn back and it has been found that nitrogen pressure about equal to this decrease, when applied to the metal, will insure smooth burning for a longer period of time. It is important that the pressure in the container should never drop below atmospheric as stoppage in the gun will occur.

9. The change of height of the metal in the container, from which the rate of burning can be followed, is determined by lowering the contact wire until the surface is touched as shown by the indicator light.

10. Since high potassium alloys are solid at room temperature (see Plate 9, Appendix), the metal must be melted. This is done by means of a "Chromalox" cartridge heater (R1-1500, 500 watts), inserted through the bottom. In addition, a low flame from a Bunsen burner is kept under the lead-out tube to prevent the metal from solidifying before reaching the gun. Plate 2 shows the container in place.

### 11. Burning gun

The gun used to burn the alloy is the same as that used by Mine Safety Appliances Co. It is manufactured by the Devilbiss Company of Toledo. The parts as listed in their catalog are as follows:

Fluid spray head Type P-MBC-422 (The air distributing ring and copper-asbestos gasket are removed to permit maximum air flow)

AV-15-E Fluid tip.

No. 116 Air cap

Fluid cut-off cock

The assembled gun is shown diagrammatically in Plate 3 and a photograph of a diametrical section is shown in Plate 4.

12. To adapt the gun to this use, a 90°, 3/8 inch Lunkenheimer fitting was soft soldered into the air inlet in place of the 1/8 inch pipe fitting supplied. One must be careful not to let the fitting extend beyond the point indicated at "A" in Plate 3, so as not to close the first air port.

13. One-eighth inch tight-wound asbestos cord is used for the packing material in the needle packing gland. It is essential that this gland be very tight as any air sucked in here will cause immediate stoppage of the gun.

#### 14. Burning Hopper

Diagrammatical front and side views of the complete hopper are shown in Plate 5. This consists of a cylinder rolled from 12 gauge black iron; length 48 inches, radius 15 1/2 inches. A rear plate of the same material is welded on and a front plate is screwed in place. A reinforced asbestos gasket makes this latter air-tight.

15. The front plate is equipped with the following attachments:

- (a) Two cubical burning boxes, 4 inches on a side. Each has a 1 1/2 inch hole cut in the center of the front where the gun is inserted. This makes a snug fit and requires no further packing. A 1/4 inch hole is cut in the side of each box to serve as a viewing port. The flame can be viewed through this and adjusted to the proper burning speed without danger to the operator.
- (b) An eight inch hole leading to the vent stack for the exit gas. This hole is partially blocked by a circular 8 inch baffle which is supported 2 inches inward from the front plate. This serves to prevent large particles of oxide from being carried into the stack with the exit gas.
- (c) A Weston bi-metallic thermometer, 0-300°C., which extends into the hopper 12 inches in a position approximately between the two gun ports.
- (d) Two turned bearings, one in the center of the plate which takes the shaft of the scraper (to be described below), and the other to support the shaft of a helical screw located along the bottom of the hopper.

16. The entire front can be removed if desired. The front plate with attachments is shown diagrammatically on plate 5 and is photographed with guns and alloy container in place in plate 6.

#### 17. Emptying Device

The hopper is provided with a three vane scraper (see Plate 5) which can be hand rotated from the rear. The vanes are of 1 x 1/8 inch angle iron and run the length of the hopper. There is approximately 1/8 inch clearance between the blades and the hopper wall.

18. A 5 inch cast helical screw is located in a "U" shaped trough which runs along the bottom of the hopper for its entire length and into an enclosed box located at the rear of the hopper which is directly over a collecting barrel.

19. For the first experiments with this apparatus, this screw was operated by means of a motor and reduction unit. It was later found that the hopper could easily be emptied by hand in a few minutes and the motor unit was eliminated.

20. A plunger device consisting of a thin plate, 3 inches in diameter, fastened to the end of a 1/4 inch rod, is located in the stack at a position in line with the exit stack (see Plates 5 and 6). Any oxide which collects in the stack, as evidenced by pressure being built up around the gun ports, can be pushed back into the hopper with this device.

21. Immediately below the plunger is located a cleaning pipe -- a short length of 8 inch pipe with a cover -- which can be opened if the plunger fails to remove all the oxide.

#### 22. Oxide Collecting System

From previous experiments carried out at this Laboratory and at Pittsburgh, it was found that the oxide formed was so light and fluffy that a large percent of it was carried out of the hopper with the exit gas. A rather elaborate collecting system had to be devised to collect it. With this in mind, the present system was constructed to provide for one additional collecting barrel with long lead pipes and a small exit hole in the stack to retain as much of the oxide as possible. However, it was found on burning the metal with this setup that almost all of the oxide produced, which could be collected at all, was retained in the hopper itself and could be readily emptied.

#### 23. Cooling System

From previous experience at this Laboratory and at Brown University, it was known that after an hour or so of steady burning, the hopper got very hot (200-300°C.). With this in mind,

the present hopper was equipped with a cooling shell. This consists of a concentric cylindrical shell of 18 inch radius, fabricated from 18 gauge galvanized iron. It completely encloses the burning hopper at the sides and rear but ends flush with the front plate of the latter. It is provided with a 9 inch standard pipe air intake at the rear and exit at the top (see plates 5 and 7). The cooling air is supplied by a 900 cfm centrifugal blower.

#### 24. Burning Air Supply

During the first part of these experiments, air was supplied by the reservation from a 100 cfm compressor. Later, a separate 100 cfm unit was installed which would maintain a higher pressure with less variation.

#### 25. Air Dryers

Since any moisture in the burning air will immediately react with the oxide, it is desirable to reduce the water content as much as possible. This was accomplished by using a potassium hydroxide pre-drying tower followed by an activated silica gel tower to remove the last traces of moisture. The potassium hydroxide tower was constructed of 4 inch pipe, 3 feet long, with a blind flange and gasket on the top to facilitate filling and cleaning. The silica tower was of 10 inch pipe, 5 feet long, likewise equipped with a blind flange top. The potassium hydroxide tower had a capacity of approximately 10 pounds of walnut size material (supplied by the Niagara Alkali Co.). The silica tower held approximately 100 pounds. The drying system was constructed in duplicate so that either or both sides could be used or one side could be regenerated and/or recharged while the other was in use. The silica tower could be regenerated by passing a slow stream of air, electrically heated to 170°C., through it until the exit air temperature reached at least 105°C.

#### 26. Air Heaters

It was necessary to heat the air above the freezing point of the alloy being burned to prevent its solidification in the gun tip. Moreover, it was found by Mr. Jackson of Mine Safety Appliances Co. that heating the burning air improved the product considerably. It was found that heating the air to 100-110°C. gave satisfactory results. Further heating greatly reduced the quantity of air flowing through the tip. In any event, 175°C. could not be exceeded without danger of melting the soldered parts of the gun. The effect of the heated air on the flame temperature was not investigated, but it is probable that the heat put into the air tends to balance the heat lost when it expands at the tip of the spray gun.

27. The air was heated by passing it through a 9 foot section of 1 inch pipe, insulated and wound with Chromel wire

(Chromel A, 3/16 x .0285 inches, .102 ohms per foot). About 350 watts was found sufficient to maintain the air at 110°C. during burning.

#### 28. Pressure Control

The air is supplied at 90-100 pounds per square inch. There is about a 20 p.s.i. drop in the drying system per gun. A Fisher lever type reducing valve (Globe Pattern) was installed in the line near the burner in order to reduce the air pressure to the optimum for burning. It was found, however, that this valve did not function properly and the downstream pressure fluctuated markedly with the rate of flow. By fastening a threaded rod to the adjusting lever of the valve and securing it to a support through which it could be raised or lowered, a fine adjustment could be obtained and, moreover, the pressure could be changed at will. The pressure drop across this device was about 15 pounds.

#### 29. Air Flow Determination

It is desirable to know fairly accurately the quantity of air flowing through the gun. A standard orifice was installed for this purpose. This is a standard pipe flange orifice supplied by the Pittsburgh Equitable Meter Company. An eight foot length of 2 inch pipe led from the orifice on both the upstream and downstream side to insure non-turbulent flow. The orifice was 3/8 inch diameter on the leading edge and the specifications supplied by the Pittsburgh concern were closely followed in its construction. The flanges were equipped with pressure taps which led to a mercury manometer. Downstream pressure was read from a gauge located at the downstream pressure tap. It is estimated that the air flow could be determined in this manner to within 2%.

### ANALYSIS OF THE AVAILABLE OXYGEN CONTENT OF THE OXIDE

30. The purity of the oxide produced was determined according to instructions given in Memorandum to the Director SS/S38(RRM:hsl) of January 26, 1942. In brief, this method consists of adding water to the oxide in the presence of a catalyst such as manganese dioxide and measuring the volume of oxygen liberated.

### PRODUCTION OF POTASSIUM TETROXIDE

#### 31. Source of Metal

All of the potassium-sodium alloy which has been burned and reported here was produced at the Naval Research Laboratory in a semi-commercial apparatus making use of the reaction



This process is described in a later report.

### 32. Purity of Metal

The only constituents of the alloy burned during this investigation were sodium and potassium. By the nature of its preparation the alloy contained no volatile salts, scale from the reacting vessels or hydroxides. Because of the method of feeding the guns during the burning, any oxide film formed on the surface of the metal is retained in the alloy container.

33. The alloy analyzed 94-96 mol percent potassium (97-98 weight percent). This composition alloy was used during the present investigation as a large supply of it was on hand and it was thought advisable to obtain necessary data with it rather than the more valuable pure potassium. The results presented herein can be reasonably applied to the burning of pure potassium.

### 34. Suction Available for Feeding Guns

It was suspected that the determining factor in the life of the burning tips was the decrease in the suction which is produced by aspirator action and by means of which the liquid alloy is drawn into the tip. Table 1 presents data which was obtained in order to correlate this information with the serviceability of the tips. Plate 7 shows three tips of different degrees of wear. Tip A is a new tip; B has been used approximately 16 hours (80 pounds of alloy); and tip C has been completely burned down by forcing metal to the tip with pressure on the alloy container. This last tip was used at the beginning of the investigation before it was known how to use the gun properly for satisfactory burning.

Table 1 (Note 1)

<u>Tip</u>	<u>Air Pressure</u>	<u>Cm. Hg. Suction</u>
A	40 lbs.	12
	45	14
	50	18.5
	55	31
	60	42
B	40	9
	45	14.5
	50	15.5
	55	16
	60	16.5
C	(note 2)	

Note 1 - It is to be noted that these data were obtained by this Laboratory, using guns modified to meet the requirements of this investigation. They may or may not be consistent with data of a similar nature obtained by the Devilbiss Company under more exacting conditions.

Note 2 - Tip C was burned so badly that there was actually a high pressure area at the orifice and air blew into the alloy chamber.

35. Description of Burning

Before starting to burn, it is advisable to rotate the scraper to such a position that neither vane will be directly in the path of the flame from the gun.

36. The alloy is heated to about 100°C. and the copper lead tube is warmed above the melting point of the alloy with a Bunsen burner. After opening the gate in the exit stack, the heater is turned on and a slow stream of air is heated to about 100°C. The air pressure is then increased to 60 p.s.i. for burning.

37. The burning commences immediately on pulling back the control needle of the gun. A fast hot flame is desired at first until the hopper gets up to the desired temperature.

38. The height of the alloy in the container is measured with the contact wire and recorded with the time at the beginning of the burning period.

39. Determination of the Optimum Burning Temperature

As noted in a previous section, a Weston 300°C. thermometer is located at a position approximately between the two guns and extends 12 inches into the hopper. This location is of course arbitrary and the temperature read at this point is meaningless in absolute terms. It has been found, however, that this is the best means of following the rate of burning.

40. The flame is maintained large and hot for approximately 15 minutes during which time the hopper will have reached about 200°C. It is then usually necessary to cool the flame down by closing the control needle until an equilibrium temperature of 190-210° is attained. It is very important to maintain this temperature throughout the burning period to give satisfactory results. Following are the results of several burnings periods which will serve to illustrate this point:

December 31, 1942

"The gun started smoothly and burned for 1/2 hour. After this time we noticed that air was leak-

ing out around the retaining ring so we closed down to repair this. In the meantime the hopper cooled down to 135°. Starting again we burned for another hour when we noticed the potassium hydroxide air dryer was bubbling and required changing. We stopped again to refill this and again the hopper cooled to about 100°. Over a burning period of 6 1/2 hours we were never able to keep the hopper temperature up to 200° for more than a few minutes because of interruptions of one sort or another."

Analysis of Oxide - - 227 cc./g.  
Theoretical - - - - - 234 cc./g.

41. It was noticed during this day's operations, and likewise on other days when for some reason it was necessary to stop the gun giving the hopper a chance to cool, that on starting the gun again, the first oxide formed was brown in color. It was previously thought that this material was a hydrate of the oxide. This seems unlikely in the light of this investigation, however, as the brown oxide would be formed regardless of whether the hopper had been left unused for several days previous or was half filled with high quality oxide from burning just completed. Its formation seems to depend only on the temperature of the burning flame. The brown color would turn to yellow after standing in the presence of dry air, this change being accelerated by heat. Invariably the brown oxide would analyze considerably below theoretical if removed from the hopper immediately as formed.

January 6, 1943

"Eleven and four tenths pounds of alloy was burned over a period of 1 3/4 hours (rate of 6.5 lbs/hr.) with the equilibrium hopper temperature of 250°C. Seventeen pounds of oxide was removed from the hopper. The oxide was quite badly sintered and contained large spongy lumps partly broken by the emptying screw."

Analysis of Oxide - - 229 cc./g.  
Theoretical - - - - - 234 cc./g.

42. Obviously the optimum burning temperature is in the neighborhood of 200°C. Table 2 summarizes the data gathered over a period of approximately two weeks of burning. The individual burning periods ranged from 2 to 11 hours. The process had to be interrupted frequently because of failure in the air supply and because of lack of tip replacements. At no time during this period was it necessary to close down due to failure in any part of the apparatus nor was there any indication that this method could not be used for continuous twenty-four hours per day production.

43. A practice was made of cranking the oxide into the receiving barrel every two hours and emptying the barrel every six hours. This could be done while burning was in progress.

44. This investigation showed that the cooling system which was constructed was unnecessary. The internal hopper temperature depended on the rate of burning only and could not be greatly influenced by moderate external cooling. On the other hand, it is quite possible that the dead air space between the hopper and the outer shell provided insulation for the former so that it could be heated up rapidly. If this shell were to be removed it might be advisable to substitute some other means of insulation. This would require further investigation.

Table 2

Weight of Analysis cc./g. Equilibrium

<u>Oxide</u>	<u>(Theo. 234)</u>	<u>Hopper Temp.</u>	<u>Remarks</u>
407 1/2	232-234	190-200	From long burning periods
205 1/4	230-232	190-200	From short burning periods
68 1/2	227	250	Quite badly sintered.

Total Oxide - 681 1/4 lbs.

Total metal burned (95 mol % K) - 441 lbs.

Ratio of metal burned to oxide made - 1.54

Total time burned - 86 1/2 hours

Rate of burning - 5.2 lbs./hour

45. The Determination of Air Flow and Oxygen Content of Exit Gas.

It is important that a quantity of air considerably in excess of that required to react with the alloy be supplied to the gun. Following are air flow measurements made on the burning air and oxygen analysis of the exit gases:

Composition of alloy burned - 95 mol % K (97 wt. %)

x = mols of K burned per hour

y = mols of Na burned per hour

$x + \frac{y}{2}$  = mols of oxygen consumed per hour

Q = total air flow in, in mols per hour

z = mols of oxygen in burning air per hour (using the experimental value of 20.6% for room air)

$z - (x + \frac{y}{2})$  = mols of oxygen left in exit gas per hr.  
(calc'd)

$$Q - (x + \frac{y}{2}) = \text{total mols of exit gases per hour}$$

$$\left[ Q - (x + \frac{y}{2}) \right] (\% \text{ oxygen found in exit gas}) = \text{mols}$$

of oxygen left in exit gas per hour  
determined by analysis

46. The oxygen found in the exit gas by chemical analysis should equal the amount calculated to be there by measuring the flow in and subtracting the amount used in the reaction. Four measurements were made over a period of four hours of steady burning during which time 18.6 pounds of metal was burned and 28 pounds of oxide was produced. The oxide analyzed 234 cc/g which is theoretical for this composition alloy. The results of these measurements are presented in Table 3 below.

Table 3

<u>Pressure on gun</u>	<u>Burning rate, #/hr.</u>	<u>Q mols</u>	<u>%O<sub>2</sub> in exit gas</u>	<u>mols O<sub>2</sub> in exit gas (calc'd)</u>	<u>mols O<sub>2</sub> in exit gas (found)</u>	<u>% deviation</u>
1 60 lbs.	4.65	1400	17.8	233	240	2.9
2 60	4.65	1400	17.4	233	234	0.4
3 60	4.65	1350	17.4	223	225	0.9
4 60	4.65	1350	16.8	223	218	2.3

These checks are excellent considering the conditions under which the measurements were made. It is seen that about 1400 mols of air per hour (18.3 cfm at S.T.D.) is sufficient to burn completely 4.65 pounds of potassium per hour.

47. Appearance of the Oxide

The oxide produced by this method of burning is of excellent quality. It contains no traces of free metal, hard lumps, specks, scale or foreign particles of any kind. It is of uniform texture and color and would be ideally suited for either canister or ship use.

48. Apparent Density of Oxide

The density was determined by weighing an amount necessary to just fill a cylindrical can 11 inches long and 3 inch radius. Some packing undoubtedly takes place during the emptying process so it is difficult to compare the observed density with that obtained by other methods. The average density the oxide produced in the manner described is 0.2 grams per cc. It can be hand packed to 0.4 grams per cc. It is of interest

that this product is about twice as dense as the oxide made at this Laboratory by the vapor method and also is considerably more dense than that being produced by Mine Safety Appliances Company. The reason for this has not been determined. Previous work has indicated that pure potassium sinters at a considerably lower temperature than oxide containing a considerable proportion of sodium peroxide. It is quite possible that the burning temperature approaches close enough to the sintering temperature to start the gathering of the particles. The fact that nearly all of the oxide is retained in the hopper instead of going into the stack bears out this fact.

## CONCLUSIONS

49. The spray-gun method is a clean, safe and economical means of burning essentially pure potassium to potassium tetroxide which will release theoretical available oxygen content on decomposition.

50. The metal can be burned at a rate of about 5 pounds per hour per gun. The total rate of production is limited only by the number of guns which can be put into operation at once.

51. The product is of excellent quality. It is of uniform color and texture, free from lumps and foreign particles and can be easily catalyzed, pressed and ground for canister use or used as produced for submarines.

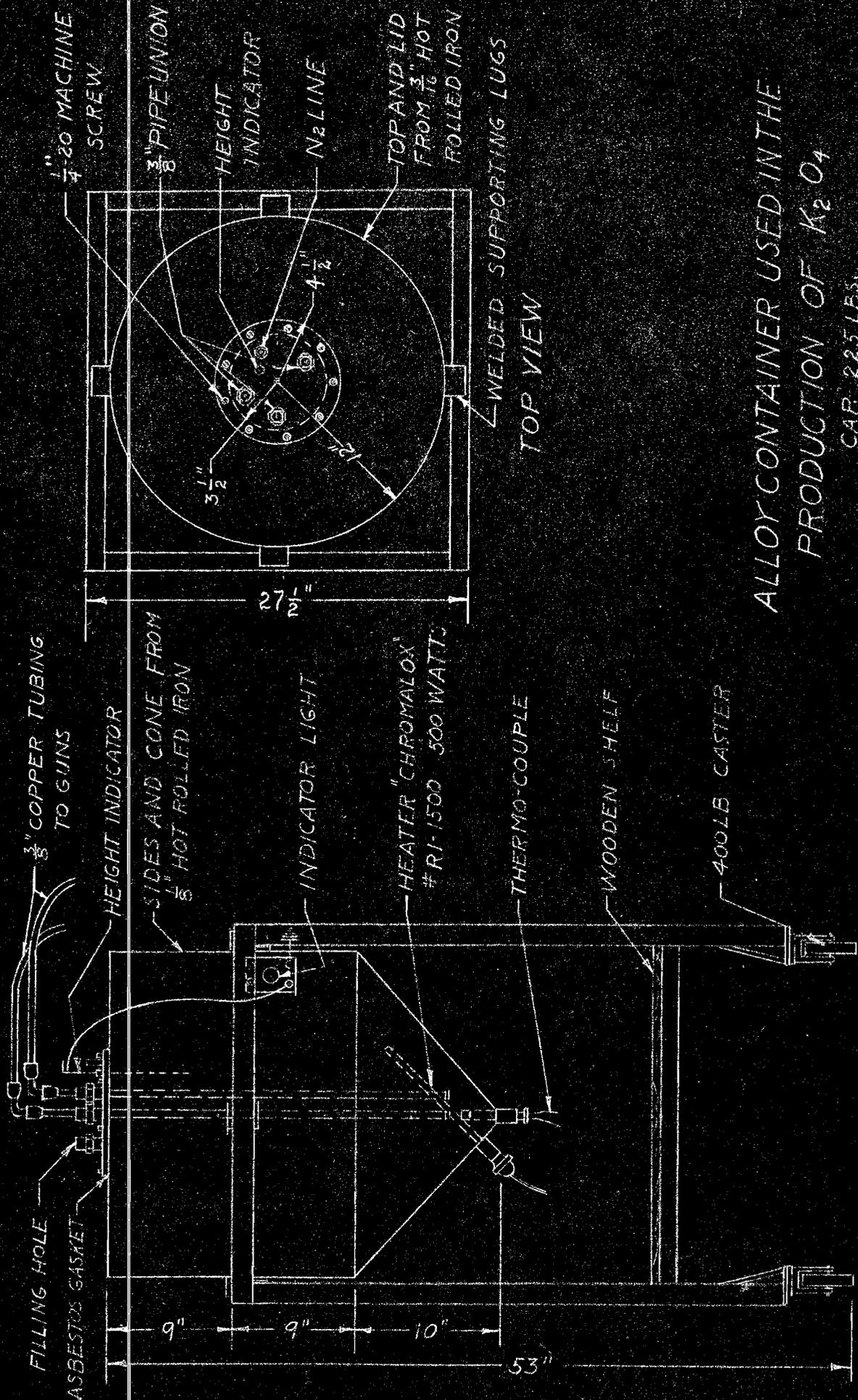
52. A yield of 86% has been obtained burning sporadically. This can be increased considerably by continuous burning.

## LINES FOR FURTHER INVESTIGATION

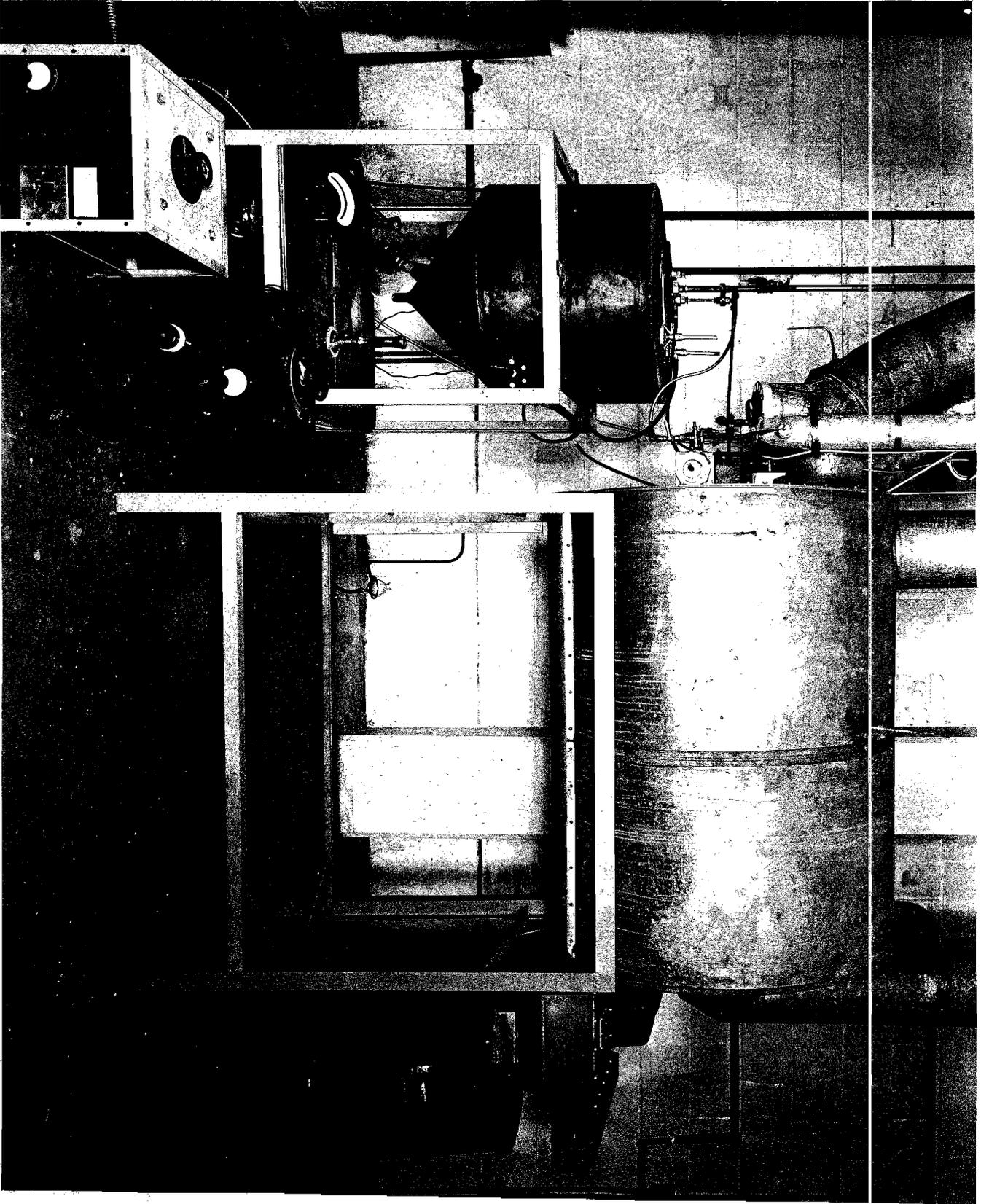
53. Although two guns have been set up to be used simultaneously, the air supply has not been sufficient to supply both guns at the required pressure. The principal factor determining how many guns can be used per hopper is the hopper temperature. It is quite reasonable to assume that an equilibrium temperature will be reached with two guns which is not too different from that with one as the air flow through the hopper is doubled.

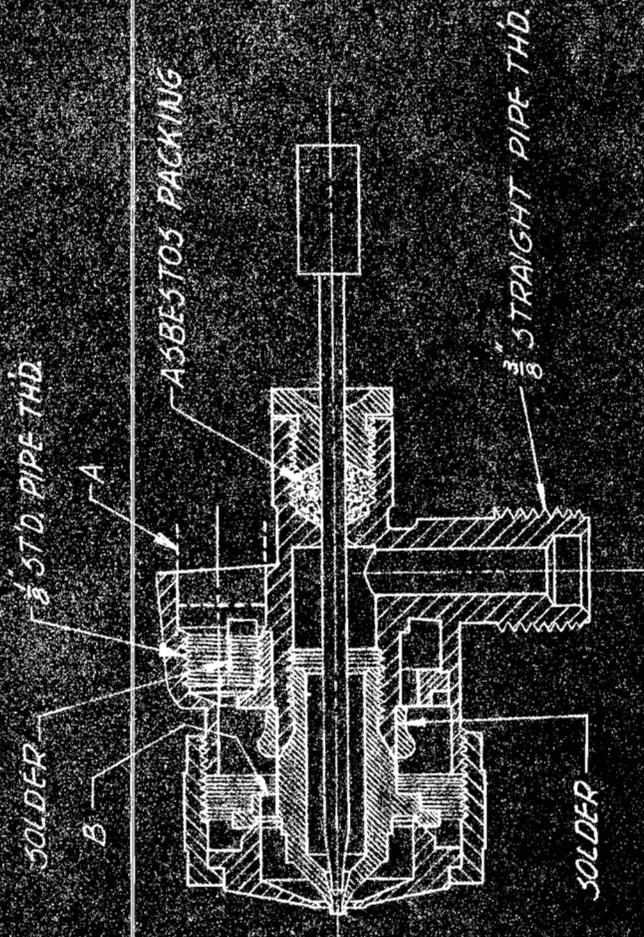
54. It might be desirable to increase the yield beyond the point which can be realized with mechanical collecting devices. A Cottrell precipitator has been tried at this Laboratory and found very satisfactory for retaining the oxide ordinarily lost as smoke. This device presented several mechanical difficulties however, and was abandoned. Further investigation might result in an efficient workable unit.

55. Of theoretical interest would be the study of the nature of the brown material formed by slow, cold, sporadic burning.



ALLOY CONTAINER USED IN THE  
 PRODUCTION OF  $K_2O_4$   
 CAP. 22.5 LBS.



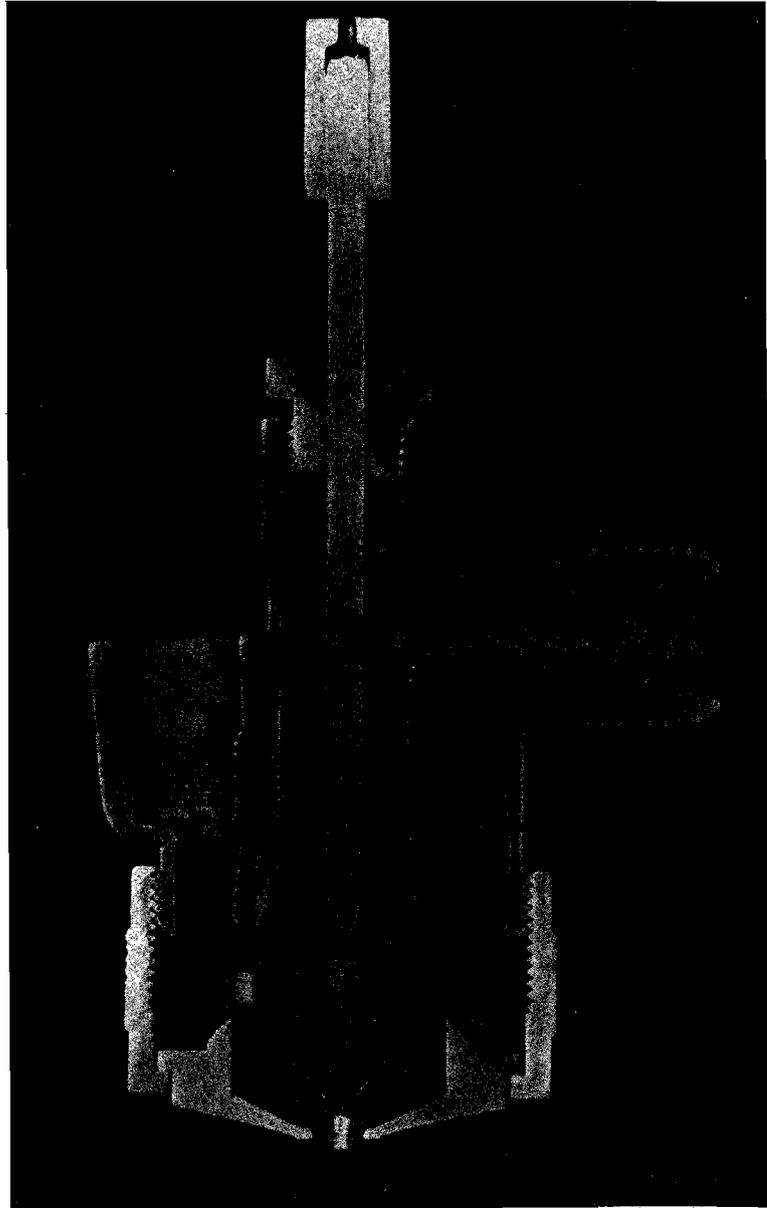


DIAMETRICAL SECTION OF DEVILBISS SPRAY HEAD

TYPE P-MBC-422

# AV-15-F FLUID TIP

# 116 AIR CAP



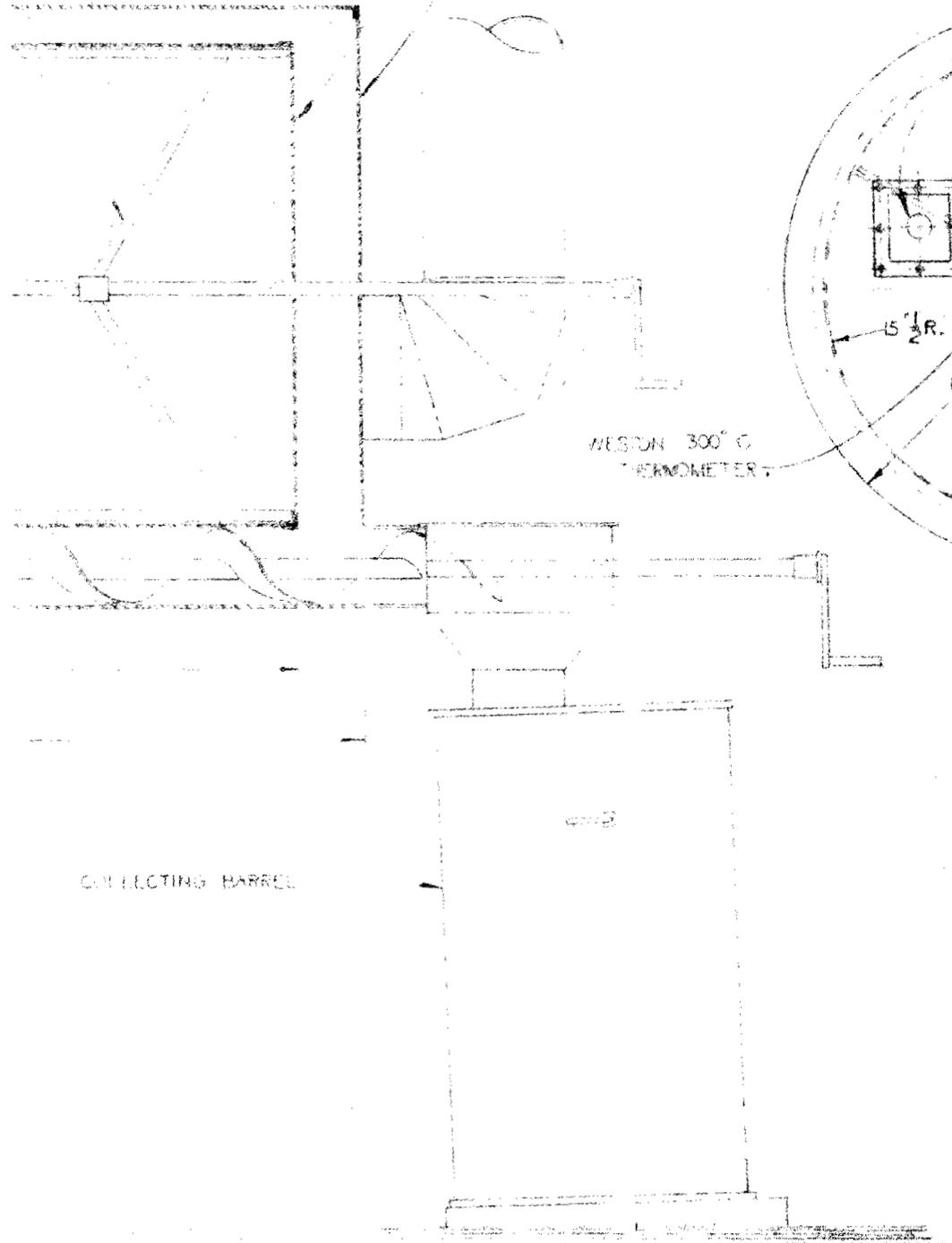
VER.

1/4" SHELL THERMOMETER (SCRAPER)

COPPER, #12 USS GAUGE IRON.

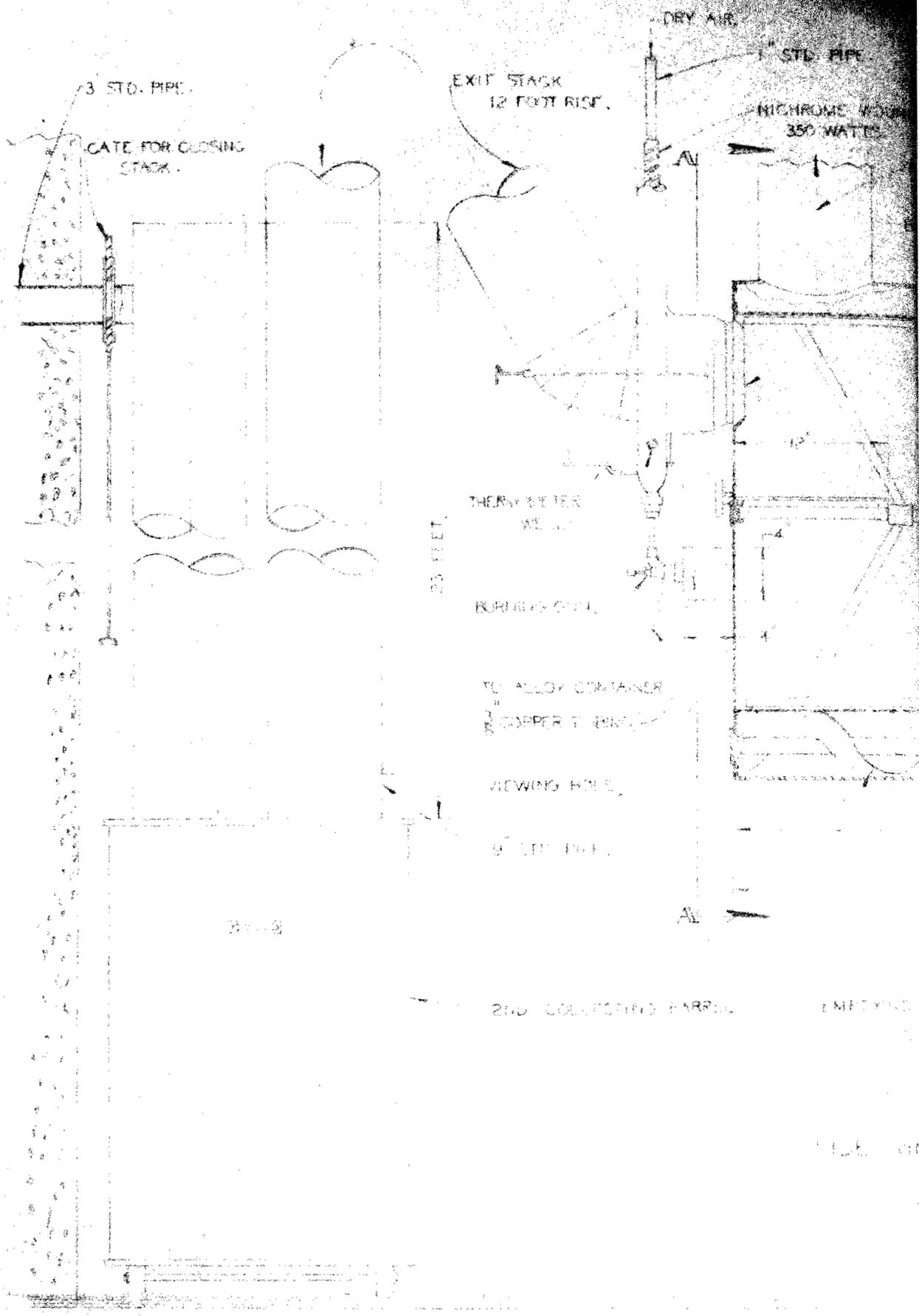
SHELL, #18 USS GAUGE GALV. IRON.

GUN BOXES.



VIEW A-A

SCRAPING VANE.



3 STD. PIPE.

GATE FOR CLOSING STACK.

EXIT STACK  
12 FOOT RISE.

DRY AIR

1" STD. PIPE

NICHROME WIRE  
350 WATTS

COUPLER

THERMOMETER  
WELL

BURNING CHAMBER

TU ALLOY CONTAINER  
3/8 COPPER THERM.

VIEWING HOLE

9" STD. PIPE

ALL

2ND COLLECTING BARRIL

EMPTYING

BURNING  
FOR  
1000 GRS OF  
SCALE

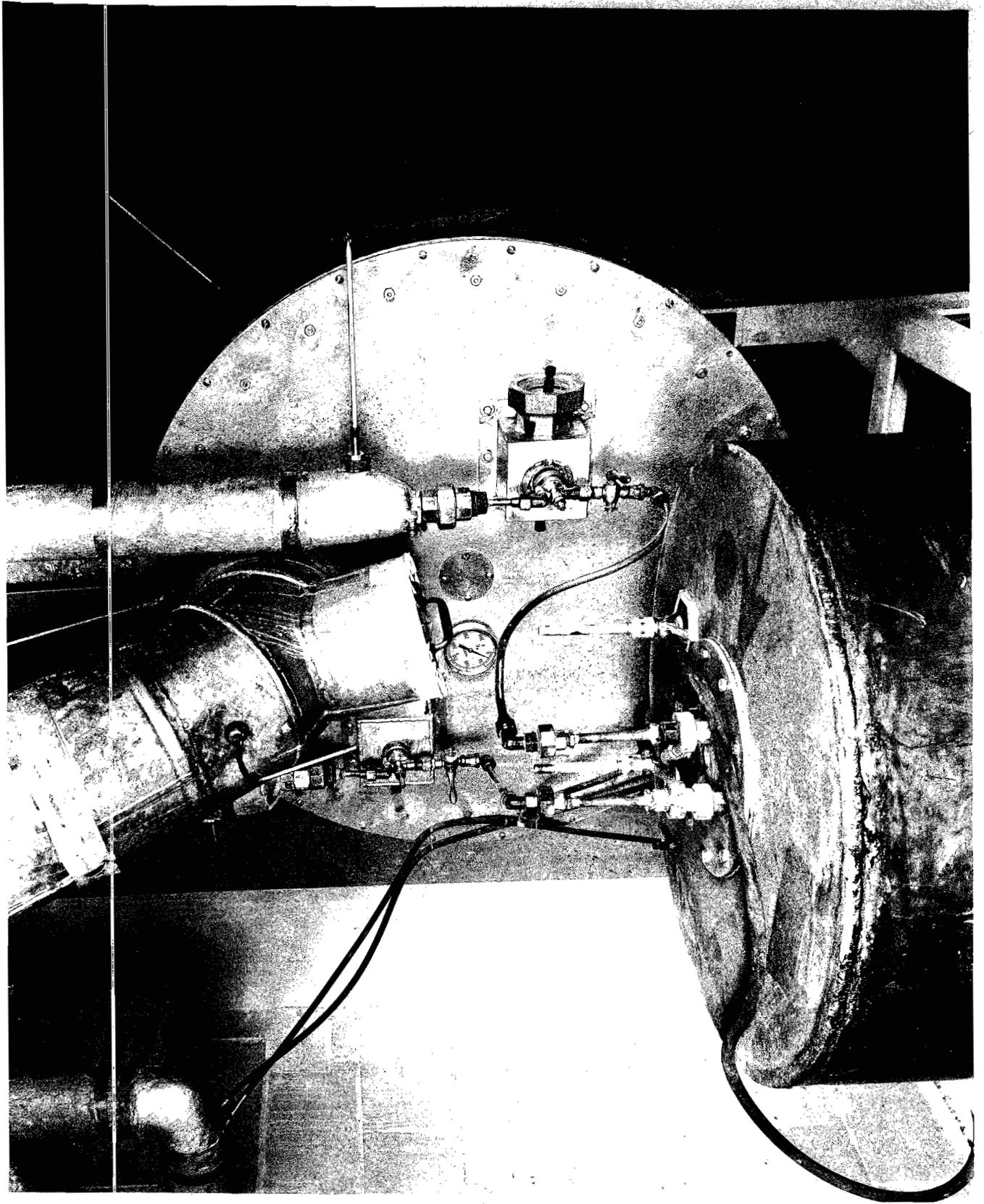


PLATE 6

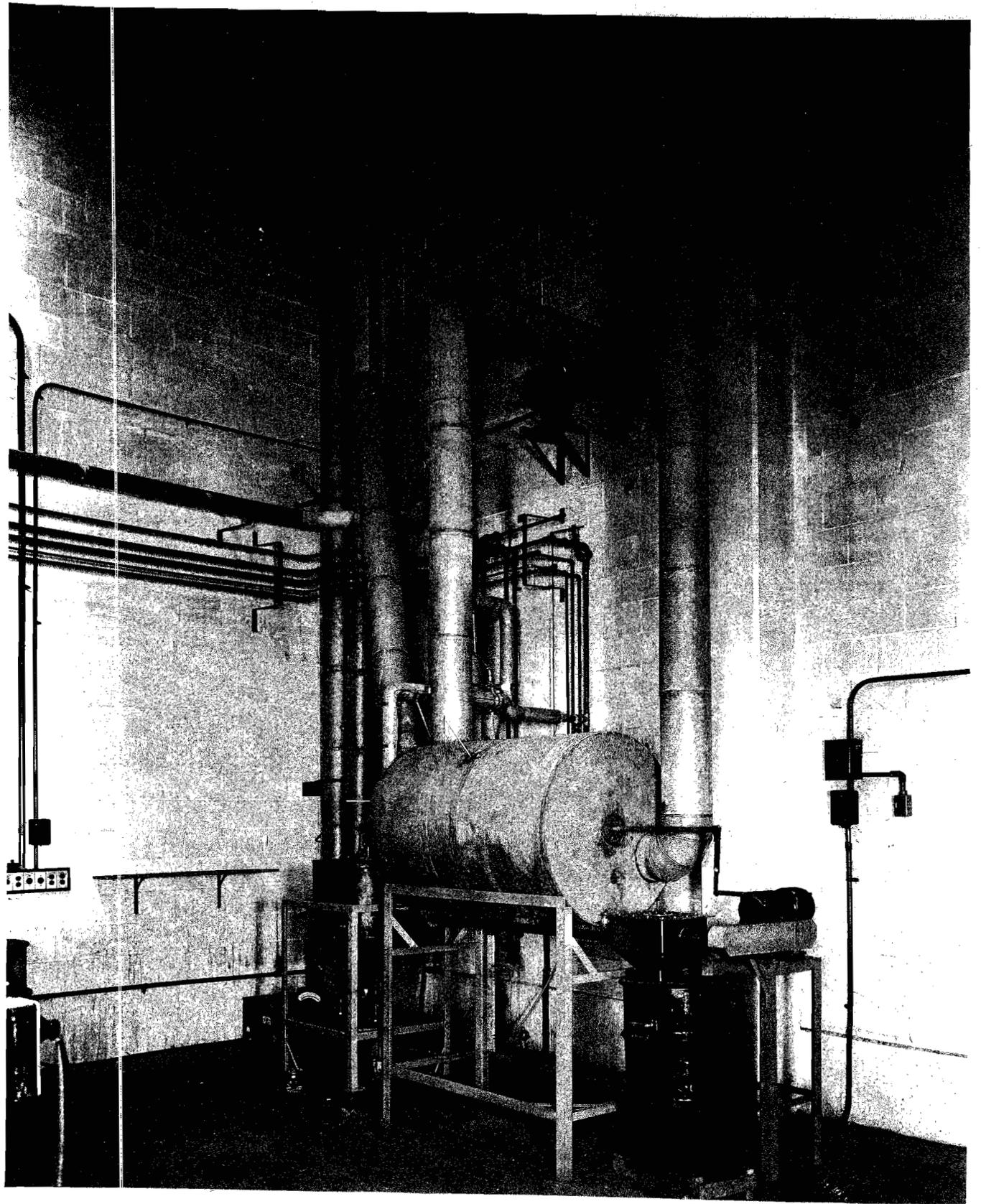
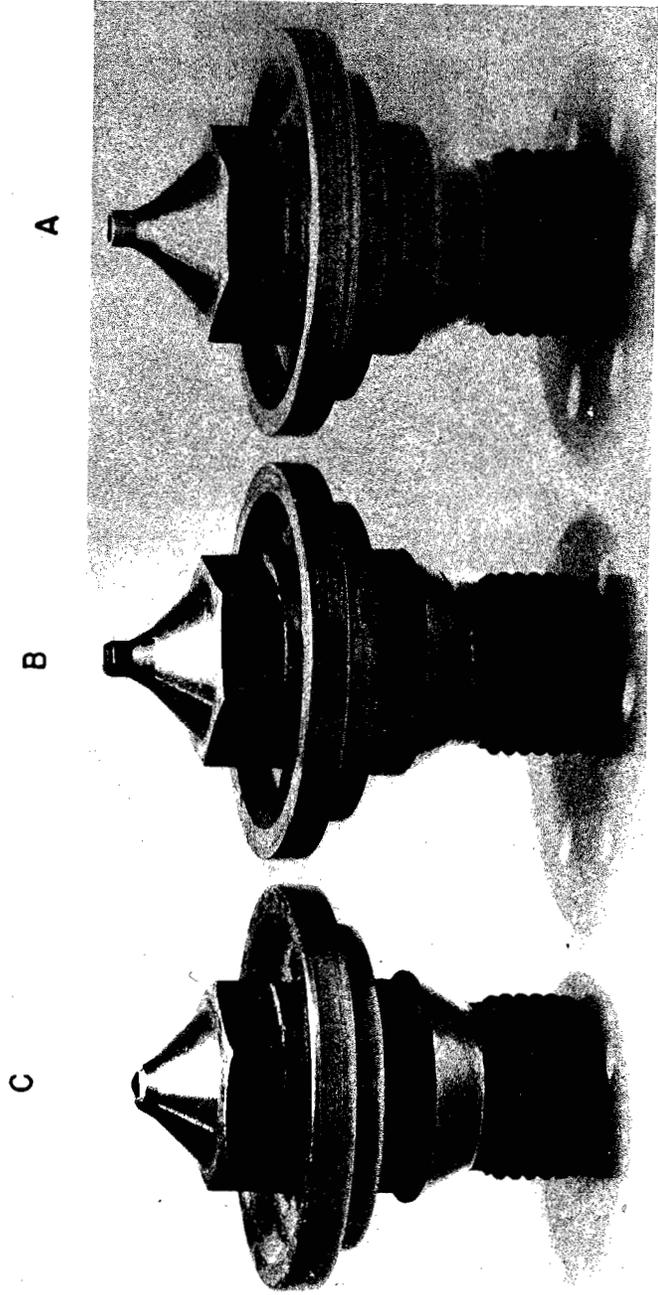
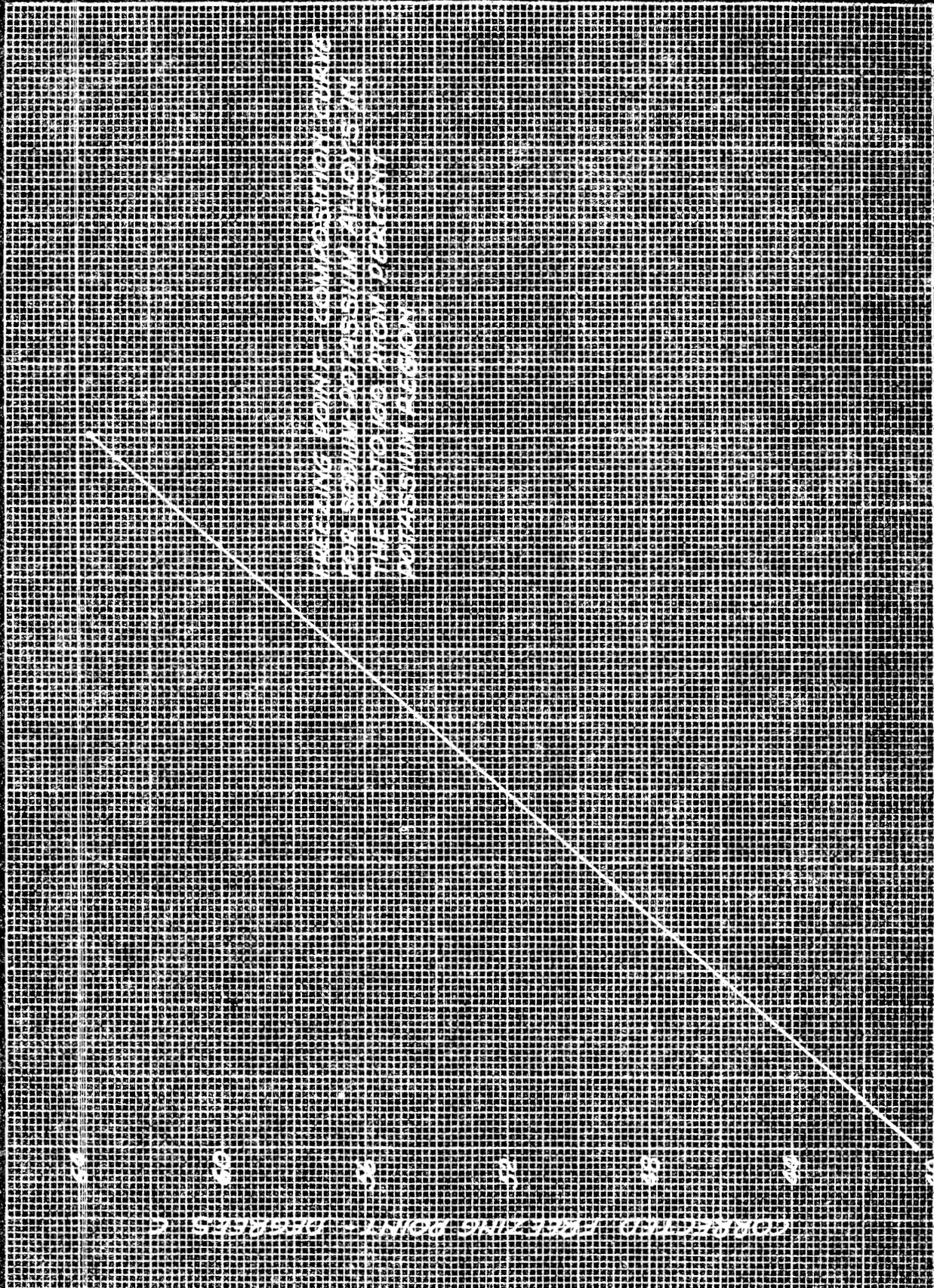


PLATE 7

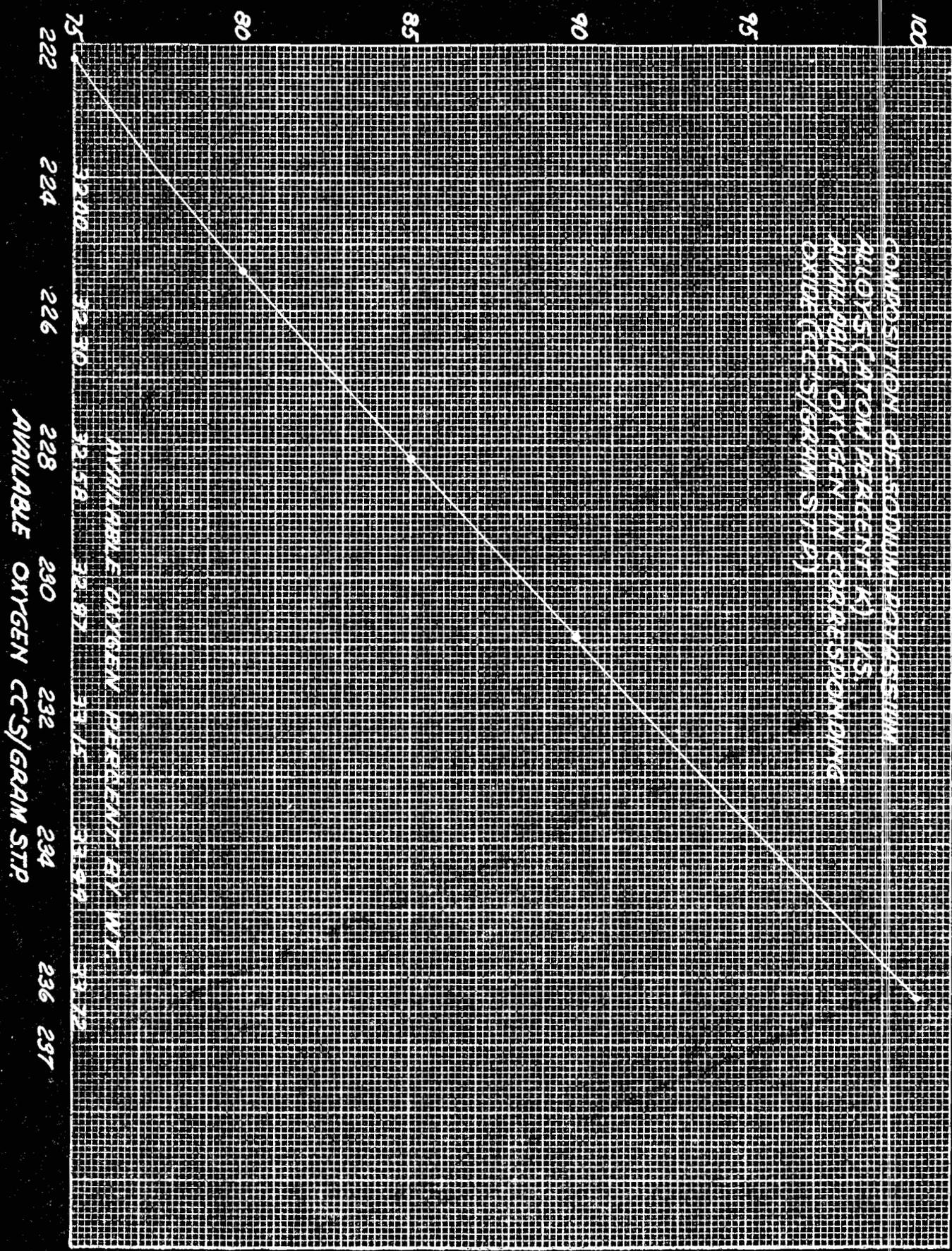




100  
COMPOSITION-ATOM PERCENT POTASSIUM  
90

COMPOSITION OF ALLOY-ATOM PERCENT K

PLATE 10



COMPOSITION OF SODIUM-POTASSIUM ALLOY (ATOM PERCENT K) IS AVAILABLE OXYGEN IN CORRESPONDING OXIDE (CC'S/GRAM STP)

AVAILABLE OXYGEN PERCENT BY WT.