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ENGINEERING STUDIES ON POT CALCINATION FOR ULTIMATE DISPOSAL OF NUCLEAR WASTE FROM THE TBP-25, DAREX, AND PUREX PROCESSES.

PART I: THE SYSTEM EVALUATION

C. W. Hancher
J. C. Suddath
M. E. Whatley

OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
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U.S. ATOMIC ENERGY COMMISSION

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ABSTRACT

Experience with the operation of an experimental engineering-development pot-calcination facility, including two types of evaporators with associated control systems and a calciner pot up to 8 in. in diameter and 90 in. high, formed the basis for the recommendation of equipment and operating procedures for the hot-pilot-plant phase of the waste-calcination program. The performance of each component in the system is discussed.

This report does not present the detailed data from the experimental development program which it covers, but rather gives the evolutionary justification for choices of equipment and operational techniques which are recommended. Expected and unexpected trouble spots which became apparent during the development program, and which were overcome by successive trials, usually over only a few runs, comprise an important part of pot calcination technology. Detailed data and analysis will be presented in ORNL-TM-619.

The pot calcination process reduces radioactive liquid waste solutions to calcined solids, which are permanently stored in the calcination pot. It is a simple and direct approach for preparing high-activity liquid wastes for ultimate storage. It is versatile enough to handle wastes of a variety of fuel processes, for example, Purex, TBP-25, and Darex, and it produces little off-gas to be processed for recycle or release to the environment.

Rehandling and packaging of the calcined solids are not required since the pot used for the final evaporation and calcination serves as the permanent storage vessel. The prolonged high temperature in the pot decreases nitrate in the solids to less than 500 ppm under ordinary conditions, leading to greater stability during storage. Ingredients for improving the physical or thermal properties of the solids or for fixation of isotopes can be incorporated in the system if desired.

Performance information is presented for equipment and system behavior based on engineering scale studies of simulated waste from Purex, Darex, and TBP-25 plants. Two flowsheets (batch evaporation and continuous) were demonstrated satisfactorily after trial and error modification of the equipment and control systems. Average rates of 5 gal/hr were processed in the evaporator-calciner complex for all three types of waste (based on 40 gal/ton, Purex waste; 45 g Al/liter, TBP-25 waste; and 38 g Fe/liter, Darex waste).

1. INTRODUCTION

This report describes a process for reducing radioactive liquid waste solutions to calciner solids, which are permanently stored in the calcination pot.[1,7,8] This is an alternative to the present practice of storing highly radioactive waste solutions from fuel processing in tanks which holds the potential danger of inadvertent release of such liquids to the environment. This program was started to determine the cost of storage of waste as solids and its feasibility, and attempted to determine a safer and more economical method of waste disposal than tank storage.

The pot-calcination process is a simple and direct approach to preparing high-activity liquid wastes for ultimate disposal. It is versatile enough to handle a variety of fuel processing wastes, for example, Purex, TBP-25, and Darex (Table 1), and it produces a small volume of off-gas to be processed for recycle or disposal to the environment.

Rehandling and packaging of the calcined solids are not required since the pot serves as the permanent storage vessel. The prolonged high temperature in the pot decreases nitrate in the solids to less than 500 ppm under ordinary conditions, leading to greater stability during storage in sealed containers. Ingredients for improving the physical or thermal properties of the solids or for fixation of isotopes can be incorporated in the system if desired.

Potential limitations still to be resolved are capacity and confidence in mechanical operations. By its nature, being a batch operation, pot calcination is a lower-capacity process than a continuously operated fluidized bed, for example. Although initial results indicate a satisfactory calcination rate, it has yet to be demonstrated that such factors as foaming,

Table 1. Major Constituents of Simulated Waste

Ion	Concentration (M) of Constituents in the Wastes Purex	TBP-25	Darex
H^+	5.6	0.5	3.0
NO_3^-	6.1	5.4	6.0
SO_4^{2-}	1.0	0.03	
Cl^-			0.1 (300 ppm)
Fe^{3+}	0.5	0.002	0.68
Al^{3+}	0.1	1.6	0.12
Ni^{2+}	0.01		0.08
Cr^{3+}	0.01		0.16
Na^+	0.6		

entrainment, or subsequent difficulties in off-gas handling are not the limiting factors. The techniques and equipment to make and break process lines to the calciner vessel, seal the vessel, remove it from the operating area, and ultimately transport it to a permanent disposal site, possibly many miles distant, must be developed and demonstrated.

Twenty-four calcination tests were made using continuous- and batch-evaporation-and-calcination equipment to determine the necessary data in order to proceed with pilot-plant design. Prior to these tests, about 42 calcination tests were made. A large quantity of valuable operating experience was gained, some of which is primarily descriptive in nature. All of the successes and failures of various components of the system and auxiliaries will be discussed in this paper. Recommendations will be given for equipment for use in a pilot plant to be built at Hanford.

In this report we will describe the ideas and equipment tested, and try to assign a value to their worth to the project. Detailed data and analysis including 191 figures and 100 tables are presented in ORNL-TM-619.* Remembering this, the reader must excuse the summary treatment of operating experience in this report. Included in this report at the end is a special bibliography of background information.

The authors would like to acknowledge the following people and service groups for their help and encouragement in this project: J. S. Taylor, R. O. Payne, D. A. McWhirter, W. L. Collins, Martha M. Dawson, Paula P. Mays, H. W. Godbee, W. E. Clark, J. T. Roberts, J. M. Holmes, E. J. Frederick, G. R. Wilson, W. R. Laing, L. R. Sullivan, and J. D. Flynn, Jr.

2. GENERAL FLOWSHEET

The general flowsheet is shown on Fig. 1. In the pot calcination process, the radioactive liquid waste is evaporated to reduce the volume as much as possible without precipitating solids. It is then pumped to a calcination vessel in which it is evaporated to dryness and thermally decomposed to metal oxides at 900°C. The calcination overhead is recycled to the evaporator. The evaporator, then, eventually boils off all of the nitric acid fed as nitrate to the system, and provides practically all of the decontamination. After the evaporator overhead is passed through a de-entrainer to remove entrained fission products, the nitric acid is recovered by distillation for reuse, and the water, which should contain very little radioactivity[2] is discharged to the environment or recycled as makeup water. The volume of off-gas discharged is small, being only that

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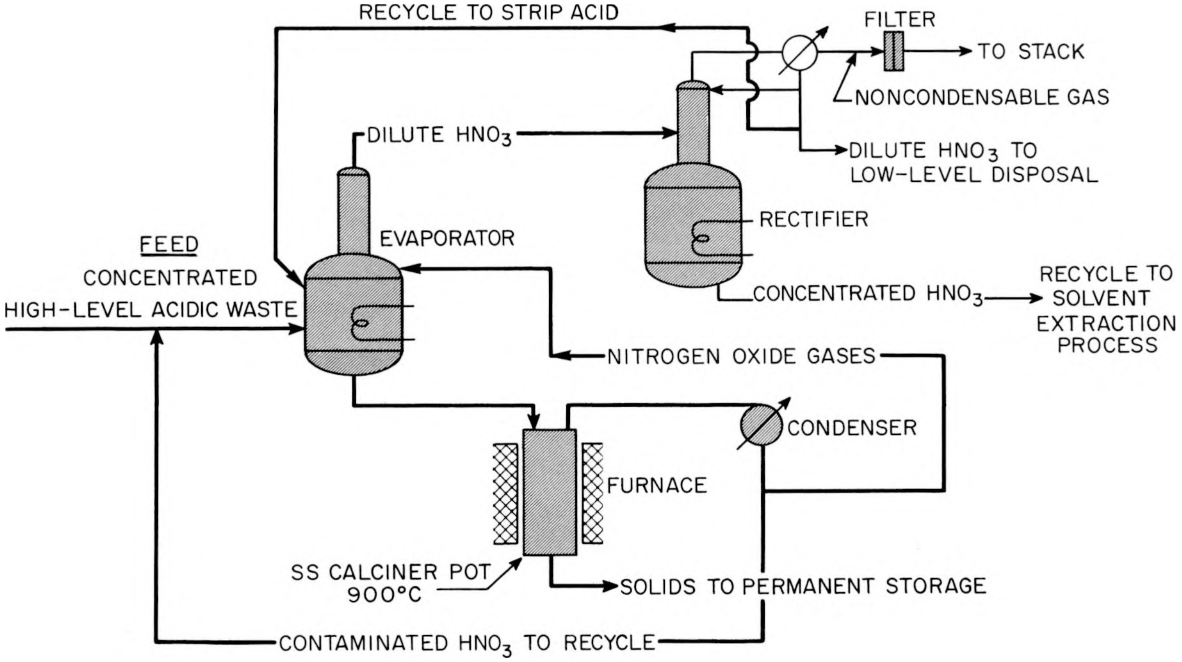

Fig. 1. Flowsheet for Converting High Activity Wastes to Solids by Pot Calcination.

from instrument bubblers and system in-leakage. The system can approach continuous operation by using many calciner pots operating in an ordered sequence.

The heat generated in the calcined solids by radioactive decay must be removed at a rate sufficient to keep the temperature of the solids during storage below some limit.[3] At excessive temperature or in the presence of radioactivity, residual nitrate or sulfate in the calcined solids may decompose causing a dangerous internal pressure, or other unknown undesirable effects may occur.[4] The calcination temperature is presently being used as the working maximum allowable centerline temperature. The diameter is then limited by the necessity to lose decay heat (6 to 24 in. diam), and the length (12 to 15 ft) by the size and weight of a shipping cask permitted on public carriers.

3. CONSTITUENTS OF THE MAJOR TYPES OF WASTE

The major types of waste now stored or being produced are Purex (high sulfate) and TBP-25 wastes. A third type, Darex waste, will also be discussed in this report. Darex waste results from the dissolution of stainless steel fuel in HNO_3-HCl and removal of HCl or from electrolytic dissolution of stainless steel fuel. Table 1 shows the general composition of the wastes (simulated) studied.

The reduction of volume from stored waste to calcined solids are eightfold for Purex and sevenfold for TBP-25 waste. The overall reduction in volumes are shown graphically (to scale) in Fig. 2.

4. ENGINEERING DEVELOPMENT RUNS: INTRODUCTION

Two types of flowsheets were studied on an experimental engineering scale: continuous and batch. Both flowsheets, batch and continuous,

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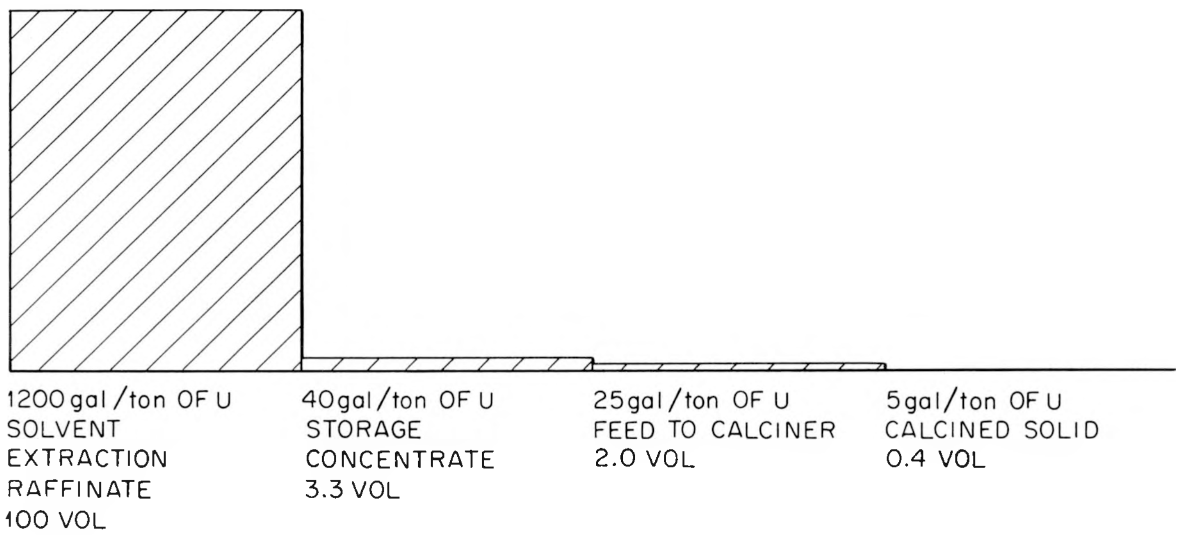
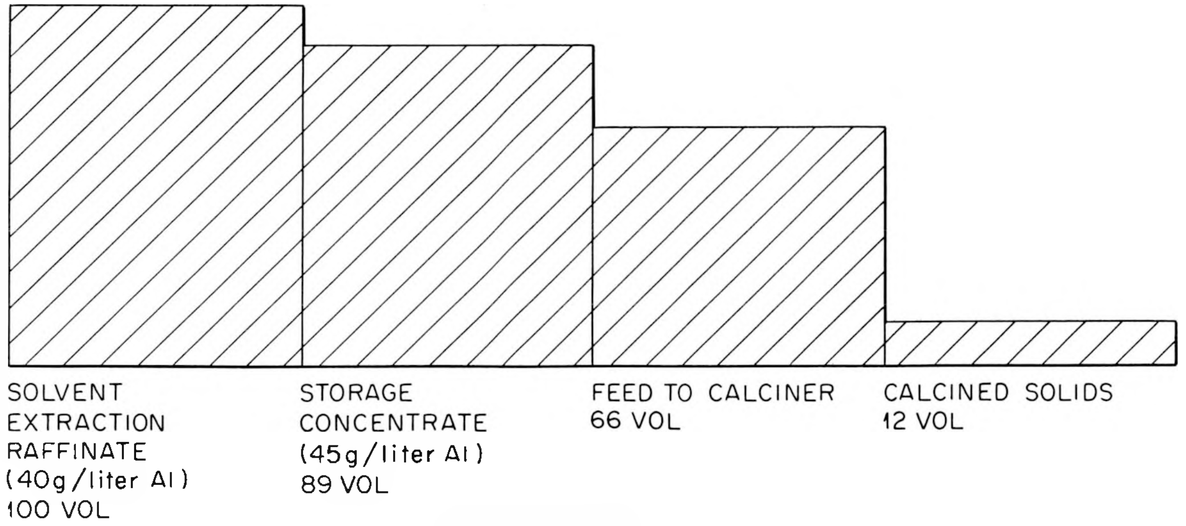


PUREX WASTE



TBP-25 WASTE

Fig. 2. Volume Reduction from Liquid to Solids by the Pot Calciner.

were studied to determine the advantage, disadvantage and operating characteristics of both. The continuous system uses a thermosiphon evaporator whose holdup volume is small ($< 5\%$) compared to the total volume of feed required for a run. A recirculating pump loop from the evaporator feeds the calciner pot on demand. The calciner-pot vapor is condensed and returned continuously to the evaporator which continuously strips the nitric acid from the system by the addition of water. The evaporator overhead, in this experimental system, is condensed, sampled, and dumped.

The batch system consists of a batch hold tank, a batch evaporator, and the other equipment mentioned for the continuous system. A batch of feed is charged to the batch evaporator. The calciner is fed from a batch previously evaporated and held in the batch hold tank. The raw feed (for the next run) in the evaporator is continuously mixed with the calciner condensate, water is added to remove nitric acid, and it is being concentrated while the calciner is operating. Between runs, the prepared batch is pumped to the hold tank, and a new batch of raw feed is charged to the evaporator.

While the batch flowsheet is somewhat simpler because of the large more simply controlled evaporator, the authors favor the continuous flowsheet. The continuous evaporator, with its more reasonable size, was easily and reliably operated in the runs in this study and is ideally suited for serving a multiple pot plant, which the batch evaporator is not.

4.1 Engineering Studies with the Continuous Flowsheet

The natural-circulation evaporator has a 22-ft^2 heat exchanger which uses 100-psi steam (Fig. 3). A pump loop which draws from the bottom of the evaporator flows past the calciner pot and returns to the evaporator.

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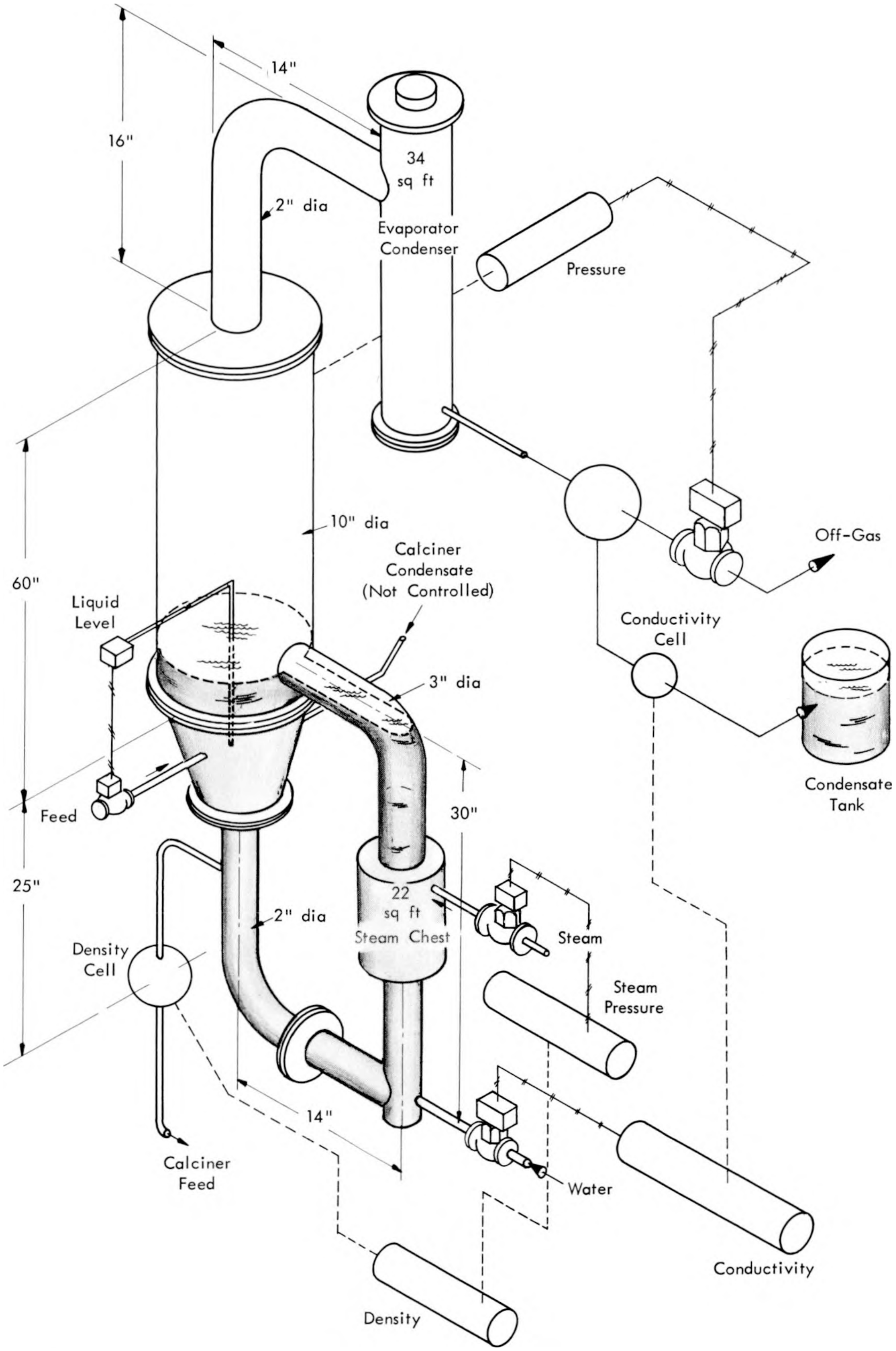

Fig. 3. Waste Calciner Continuous Evaporator.

A small side stream of liquid drawn off through a control valve into the calciner pot (Fig. 4) is kept as short as possible (2 ft), and the control valve is placed as near the operating loop as possible. A water purge bled into the feed stream downstream of the control valve at 1 liter/hr decreased the frequency of plugging. To maintain a constant feed pressure to the calciner, 1 to 10 gpm of flow is maintained in the pump loop.

The calciner vapor is passed to a 30-ft^2 downdraft condenser which discharges directly to the evaporator. The liquid is supercooled to recover the nitric oxides to decrease the gas volume for better decontamination. Water and feed are added to the evaporator through their individual control valves from head tanks. The evaporator vapor passes through a de-entraining section and then to a 34-ft^2 downdraft condenser. The condensable fraction of the vapor goes to a condensate receiver where it is measured and discharged through a pressure seal. The noncondensable vapor goes to the off-gas system, where it is filtered, and the volume is measured by a wet test meter.

The off-gas from a typical 30-hr Purex waste run was 1100 ft^3, or 36 ft^3/hr, of which 32 ft^3/hr was instrument purge and in-leakage gas. The system operated under a 1-psi vacuum. The 4 ft^3/hr of noncondensable gas averaged 3 ft^3 of oxygen per hour. Excess oxygen is always present in the off-gas from the decomposition of nitrate to NO_2 and O_2[5] and at times was as high as 34 vol %. This small amount of off-gas can be easily decontaminated and discharged to the environment.

4.2 Engineering Studies with the Batch Flowsheet

The batch flowsheet tested consisted of a large batch evaporator (1200 liters) with 34 ft^2 condenser and a 1200 liter heated hold tank.

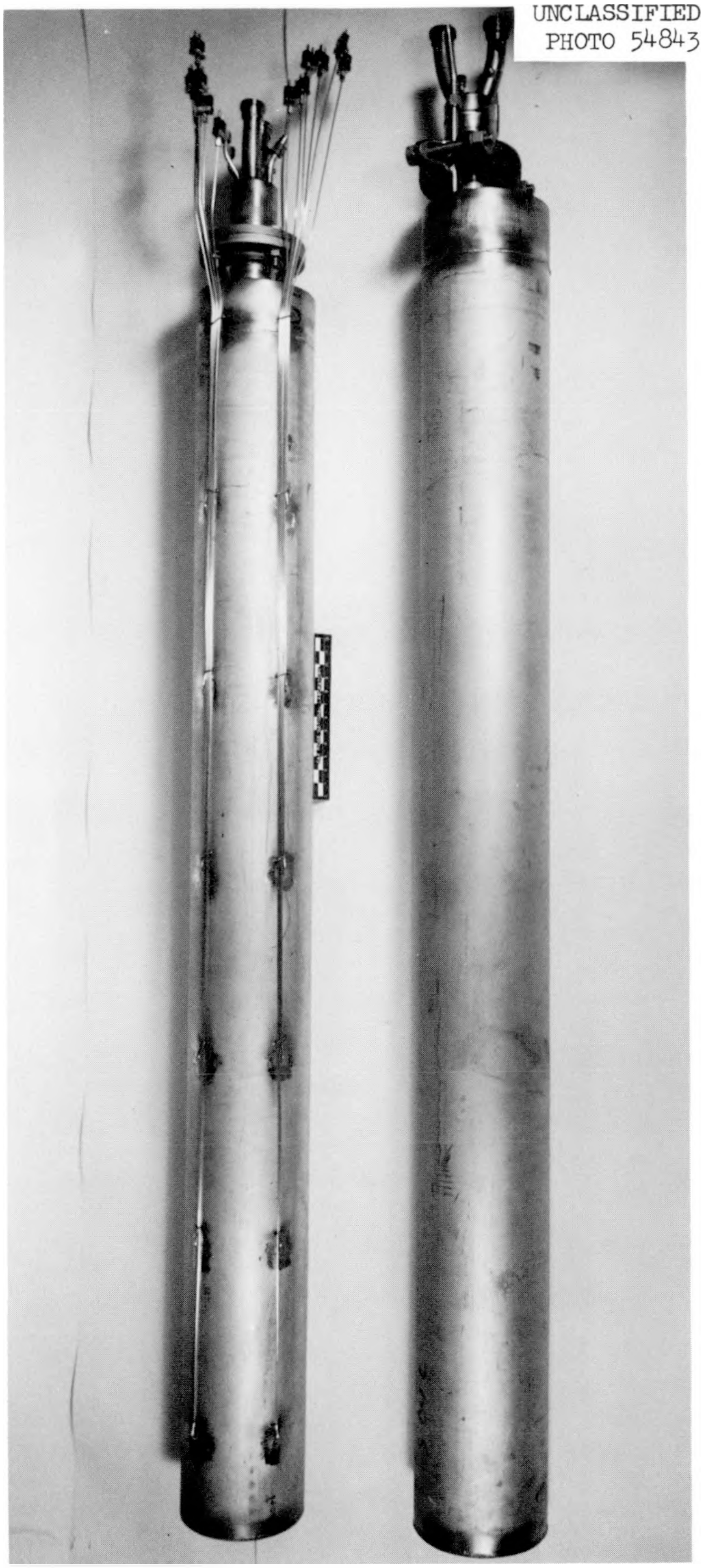
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Fig. 4. Waste Calciner Pot.

A Chempump was used for the circulation pump. The system piping was changed so that the batch evaporator and hold tank were connected to the system in the same manner as the small continuous evaporator (Fig. 5).

A batch of dilute feed was pumped to the batch evaporator and boiled down to the desired feeding concentration. Then it was transferred to the hold tank. A second batch of dilute feed was then added to the batch evaporator. The calciner was fed from the hold tank by the recirculating feed pump. The condensate from the calciner with the dilute feed of the second batch was concentrated while the first batch was fed to the calciner.

5. SOLIDS RESULTING FROM CALCINING OF WASTE

Both Purex and TBP-25 waste are calcined, with the solids depositing radially (Figs. 6 and 7) leaving a diminishing liquid core down the center of the pot unit until the end of the feeding period. At the end, this liquid highly saturated with salt, is also calcined to a solid.

The bulk density of calcined Purex waste was 1.1 to 1.4 g/cc,[6,8] while that of TBP-25 waste was 0.4 to 0.8 g/cc (Table 2). The bulk density is calculated from the weight of solids in the calciner pot after the run and the volume of the calciner vessel to the liquid level operating point, and reflects the fraction of the calciner vessel unfilled and voids in the calciner cake. The fraction unfilled varied from 0 to 20% based on visual measurement, depending on the filling time. The tests were usually terminated when the feed rate to the calcining pot dropped below 5-10 liters/hr. An economic evaluation of the procedure requiring better cost data than are now available will be needed to determine the best cut-off time. A scoping study[9] was made, but a more detailed study should be made.

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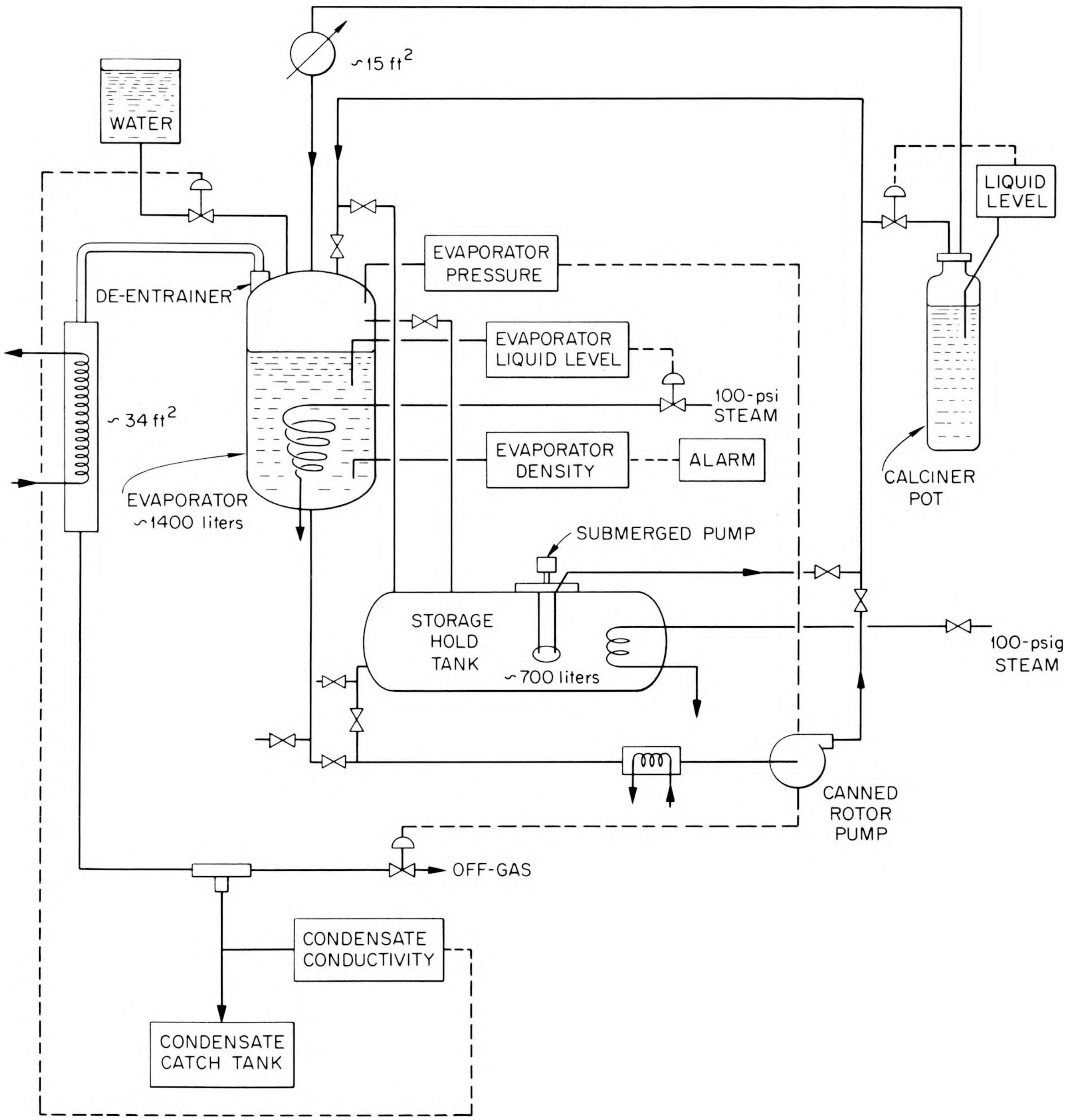

Fig. 5. Flowsheet of UNOP Batch Evaporation Tests for Pot Calcination.

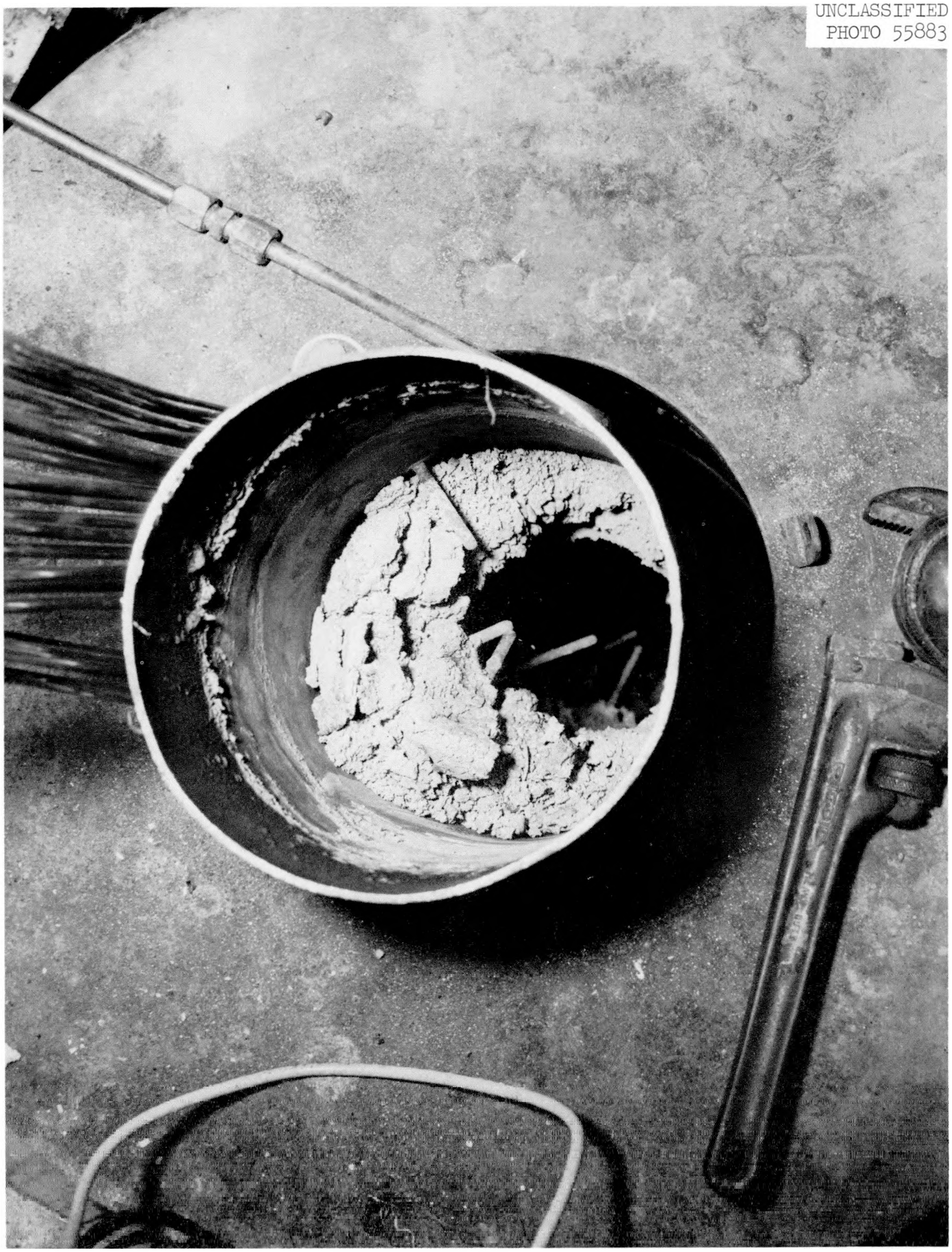

Fig. 6. Purex Calcined Solids

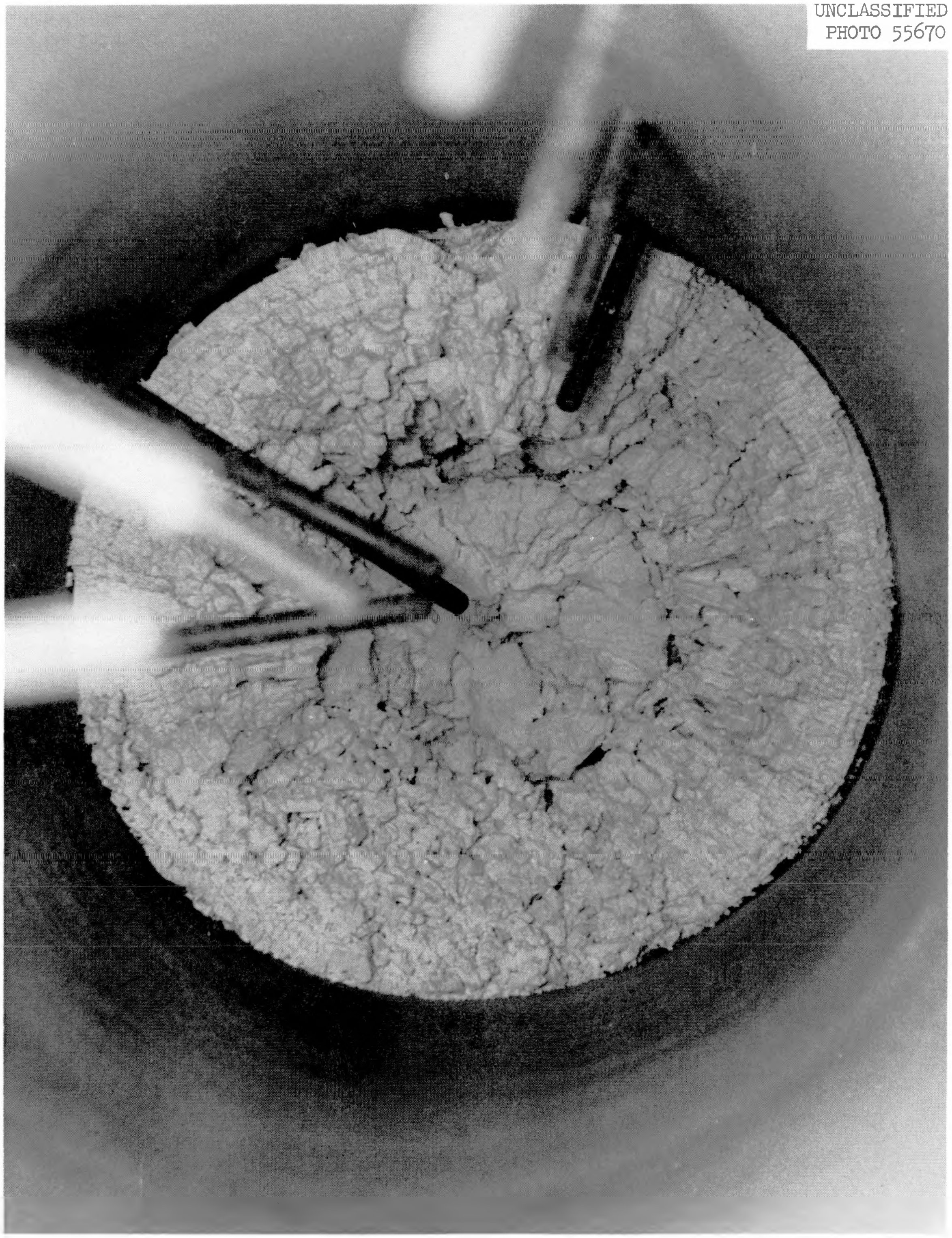

Fig. 7. TBP-Calcined Solids.

Table 2. Characteristics of Typical Calcined Solids

Type of Waste	Density (g/cc)	Thermal Conductivity at 600°F (Btu hr^{-1} ft^{-1} $°F^{-1}$)	Residual Nitrate[a] (ppm)	Appearance of Solid
Purex	1.1-1.4	0.2	300	Brown, hard
TBP-25	0.4-0.8	0.1	500	White, cellular

[a] Best results of calciner that was heated to greater than 600°C for eight hours.

6. CONSTRUCTION OF THE CALCINER POT

The calciner pots used in the development program have been 4, 6, and 8 in. in diameter and a maximum of 90 in. long. The length varies some with flange design and other experimental variables. The pots, in general, were made from Sched. 5 stainless steel, either 304L or 347, depending on availability. The bottoms of the pots were 1/8-in. flat plate with a butt weld, and all the welds were argon-purged Heliarc welds.

Two top connections for the calciner pots were tested as follows: (Figs. 8 and 9)

1. Standard 3-in. four-bolt flange with corrugated metallic gaskets, both solid metal and asbestos filled with stainless covering.
2. Grayloc* flanges. A number of variations of Grayloc flanges were tested. The most satisfactory was model S1K-NO-60-390, which consisted of a 3-in. flange modified with a 1-1/2 in. opening and two separate 1/4-in. openings for feed line and auxiliary feed line.

A baffle was installed in each of the calciner pots to prevent direct splashing or foaming of the liquid at the operating level into the 3-in.-

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*Grayloc is the trade name of the Gray Tool Company, Houston, Texas.

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Fig. 8. Waste-Calciner Vessels.

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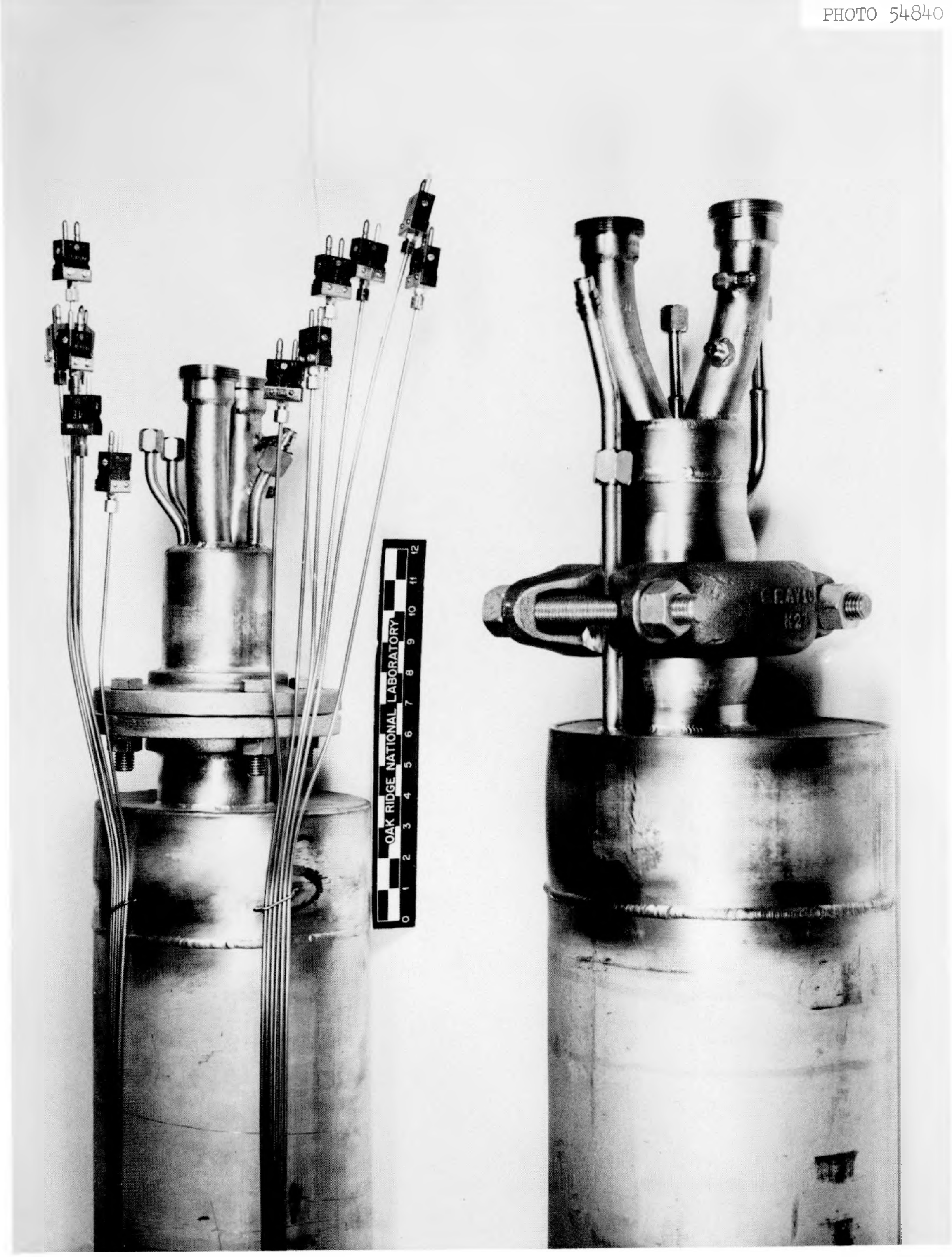

Fig. 9. Waste-Vessel Feed Flanges.

diameter pot head that connects to the off-gas line. The 3-in. off-gas header on most of the pots was offset from the center so that a baffle extending from one side of the calciner to 1 in. past the center line would deflect any direct carryover.

The calciner pot had a nominal 1 liter/min water boilup rate at the full 50-kw heat input. However, as the solids are radially deposited on the walls of the vessel, the heat input to the pot is decreased. The external wall of the calciner is held at a maximum of 900°C by the furnace, which was divided into six sections, each about 13 in. high. The furnace temperature could go to 1050°C before danger of burnout.

6.1 Waste-Calciner Furnace

The waste-calciner furnace has an inner diameter of 10 in. and consists of three sections, each 26 in. high. It is a Heavy-Duty Company, model MK-10724-S, 220-v furnace. Each of the three sections has two controlled zones. Each zone has maximum power input of 8.3 kw, which is equal to 3.2 kw/ft^2. Each zone has a factory-installed Chromel-Alumel thermocouple of approximately 16-gage wires with ceramic bead insulation. Two thermocouple leads came into the center of each furnace section and extended about 4 in. up and down into their respective zones. The thermocouples were unsatisfactory for two reasons:

1. They were easily bent when the liner between the refractory furnace and the calciner pot was removed.
2. When they were bent, the ceramic insulation cracked off and they became shorted. These thermocouples were removed, and 1/8-in. sheathed thermocouples in 1/4-in. type 347 stainless steel wells were installed in each heat zone and have performed satisfactorily.

The reliability of the furnaces has been very good. Some of the replaceable sections of the top furnaces were damaged from overheating when a control thermocouple malfunctioned; they were easily replaced. The total operation time of the bottom sections is over 2000 hr, and they still appear to be in good operating condition. The usable expected life may be 4000 hr.

6.2 Calciner-Pot Thermocouples

Since the calciner furnace was equipped with six independently controllable heat zones, a complete set of thermocouples was used at the midplane of each of the six zones, and each set had four thermocouples installed in the following locations:

1. In or on the furnace element to measure temperature and to monitor for overheating. The furnace elements should not be operated above 1050°C.
2. A dual thermocouple attached to the exterior of the calciner pot to measure and control its temperature at the specified set point. Two thermocouples are necessary because the Brown recorders and the Leeds and Northrup controllers could not be operated compatibly from one thermocouple signal. The operating temperature of the exterior of the calciner pot was to be maintained at about 900°C within ± 25°C.
3. Inside the pot 1 in. from the wall.
4. On the center line of the calciner pot.

During the middle and latter part of the calciner feed period, the thermocouple 1 in. from the wall was often covered with solid, while the center thermocouple was still in the liquid.

Three different methods were used for installing the interior calciner pot thermocouples:

1. Both 1/8-in. and 1/4-in. swaged thermocouples (sheathed in 310 SS) inserted at right angles to the wall and welded directly to the calciner vessel were tried. This procedure was unsatisfactory because the process liquid corroded the thermocouples and attacked the weld between the thermocouples and pot calciner. It was found to be bad practice to weld or tack the thermocouples at any point along their length except for the end closure, and this should be done only with extreme care and followed with x-ray and dye penetration tests.
2. One-quarter-inch type 347 stainless steel or 304 stainless steel thermocouple wells were installed through the wall, and the 1/8-in. swaged thermocouples were slipped into them and mechanically secured. This procedure was satisfactory.
3. The pilot plant pot design (designed by C. W. Hancher, J. M. Holmes, and R. M. Beckers of ORNL) calls for the six thermocouples to be bundled and inserted in a 3/4-in. well, placed at the centerline of the pot. This configuration was not tested here but should be satisfactory (Fig. 10).

6.3 Control of Liquid Flowing Through the Calciner Feed Valve

The calciner feed is prepared either in the continuous evaporator or in a batch evaporator and is circulated in a 1- to 10-gpm pump loop past the calciner pot in order to maintain an adequate supply of feed to the calciner at a reasonable pressure (about 10 to 15 psig). The feed for the calciner is drawn off from the circulating loop at a rate controlled by the calciner liquid level.

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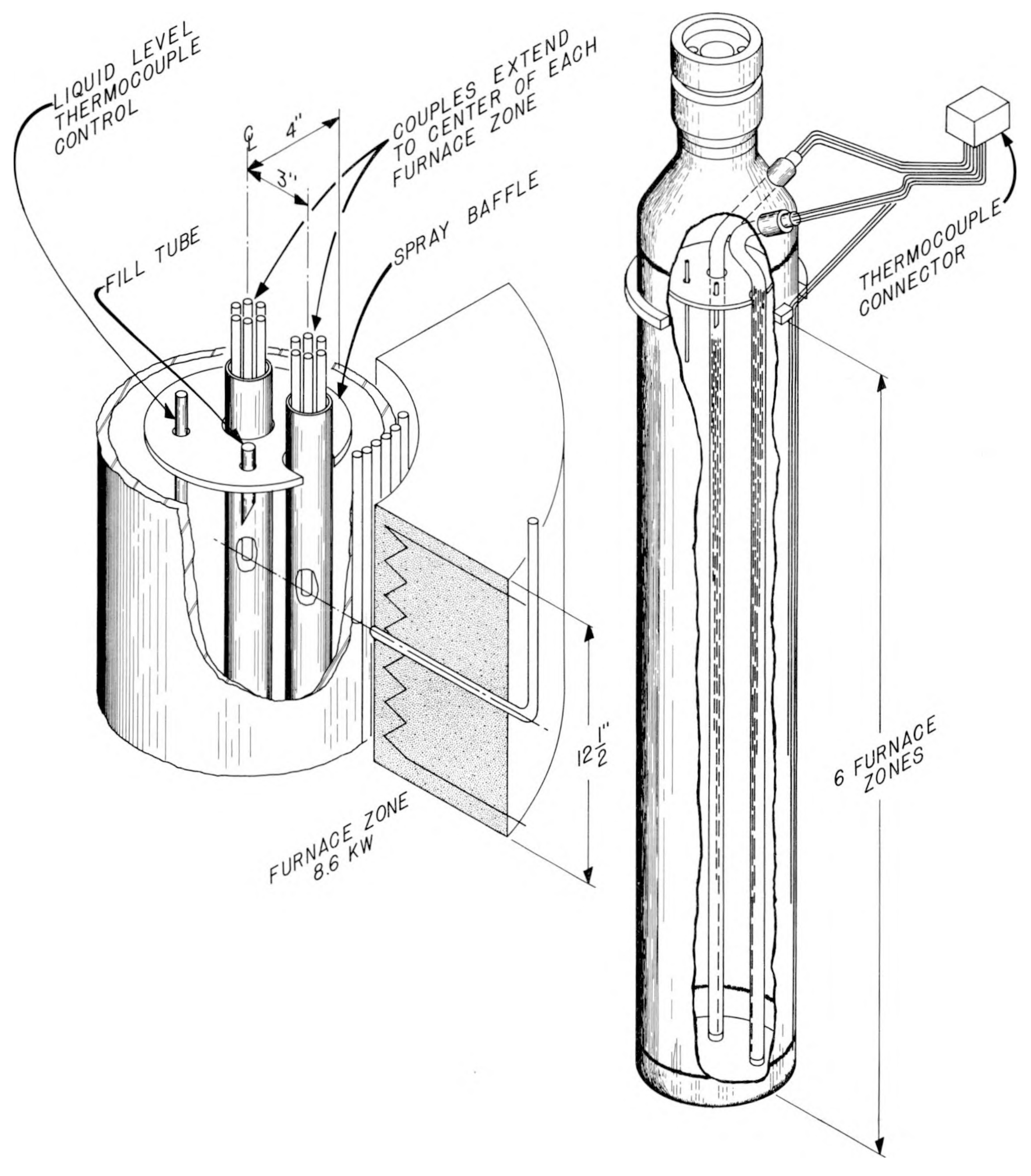

Fig. 10. Proposed Pilot Plant Calciner Thermocouple Detail.

A number of different types of valves were tested: plug valves, both on and off, and throttling valves with both linear trim and square-root trim. On-off control valves, working either directly from the controller or through an on-off ratio sequence controller, caused excessive fluctuation of the liquid level in the calcining pot, which set up a cycling that ruined control. Because of its constant-flow nature a throttling control valve worked better. The calciner liquid level has a characteristic response time which increases to more than an hour as solids build up on the pot wall. The controller which uses this signal to actuate the feed valve must have a very long reset time (about 3 hr), a very wide proportional band (about 200%), and no derivative action. Such a system gave very stable liquid-level control in the calciner vessel and little possibility of solids settling or feed solidifying in the feed line. However, with a feed that has a large amount of solids or is close to its crystallization point, a water purge between the feed valve and the calciner was necessary. A water purge rate of 1 to 3 liters/hr was satisfactory when a 3/8-in. feed tube was used in the calciner. There was no tendency for the feed to calcine in the feed tube inside the calciner vessel when a water purge was used.

6.4 Additives to the Calciner Feed

When Purex waste that contains sulfate is calcined, a chemical must be added to reduce the sulfate volatility from the calciner. Calcium nitrate was chosen because it forms calcium sulfate which is quite stable at calcination temperature. However, there was a problem in how to add it. Were it added directly to the feed tank, the calcium sulfate precipitate would foul the evaporator and the pump loop. Therefore, it was added directly to the calciner to form the precipitate in the boiling liquid of the calciner. When calcium hydroxide was used instead of calcium nitrate, serious foaming occurred.

A solution of calcium nitrate, approximately 3 M, can be handled and pumped into the calciner at a metered rate (not exactly proportional to feed rate) so that the calcium ion is always in excess of the sulfate ion. This procedure prevented the major fraction of the sulfate from volatilizing from the calciner. (Only about 1.0% of the sulfate is evolved from the calciner when calcium nitrate is added in sufficient quantities.) The calcium nitrate was added to the calciner through a secondary feed line that originally was designed to be used as a differential-pressure liquid-level probe. Flow from the gravity head tank was also successfully used in place of a positive displacement pump.

Other chemicals that help form glasses may be added to the calciner pot as secondary feed additives. This has not been tested in large equipment. The advantages of forming glass in the calciner pot are that glasses have a higher thermal conductivity and a lower leaching rate for fission products by environmental water in the event of an unforeseen failure of the pot wall.

6.5 Furnace Liner for the Waste Calciner

A 20-gage stainless steel liner was installed between the furnace elements and calciner pot. The liner was as large as possible to accommodate a slip fit. There was approximately 1 in. radial clearance between the liner and the calciner pot. The calciner pot and liner, after operation at 900°C for extended periods of time, 36 to 48 hr, showed signs of severe surface oxidation and scaling. This was especially true near the top of the furnace where apparently fresh air is thermally cycled when the top furnace zones turn on and off. This was corrected by use of a nitrogen purge of 10 cu ft per hour at the bottom of the calciner between the liner and calciner pot.

7. SYSTEM OFF-GAS

One of the major advantages of the pot calcination process is its small quantity of hot off-gas.[5] The off-gas consists of the following gases:

1. Air due to in-leakage (the system operates with the evaporator at -1 psig) is normally 10 to 15 ft^3/hr but may be as high as 27 ft^3/hr.
2. Instrument purge air; approximately 0.5 cfh per line for 10 lines (5 cfh).
3. Noncondensable gas is formed (NO_2 and excess O_2) in the thermal decomposition of nitric acid and nitrate salts in the calciner. The noncondensable oxides of nitrogen amount to about 5% of the nitrate charged.

7.1 Calciner Off-Gas Handling Equipment

The off-gas from the calciner carries all the volatile constituents fed to the calciner pot plus the purge gas. During the first part of a run, the off-gas is mainly 1 to 2 M nitric acid. After the feeding period is over and the remaining liquid in the calciner pot begins to go to dryness, the nitric acid concentration in this stream increases to more than 16 M. However, the rate at this time is much lower than during the heavy feeding.

The calciner off-gas line from the pot to the calciner condenser is 1-in. IPS, stainless steel, about 6 ft long (as short as practical).

Normally the off-gas is superheated by the hot walls in the top of the pot to a temperature as high as 250 to 400°C. A thermocouple in the off-gas line extending to the center of the 1-in. pipe was used to measure the temperature at this point.

Since the calciner vessel has a thermal expansion of about 2 in. a flexible joint in the off-gas line is required. A 12-in.-long, 1-in.-diameter flexible stainless steel bellows covered with a metallic guard served as a flexible joint. The maximum linear velocity of the vapor through this off-gas line was about 200 ft/sec.

7.2 Secondary Safety Off-Gas Line for the Calciner

In the event that the primary calciner off-gas line plugs, it is necessary to vent the calciner safely. To do this, a secondary off-gas line the same size as the primary off-gas line was installed. The line is sealed from the rest of the system with a hydraulic seal pot requiring 20 ft of water head to blow the seal. We believe this type of seal is superior to a rupture disk because the seal can be tested, and, in the event the seal is blown, it can be recharged during operation.

7.3 Mercury Compounds in Off-Gas from the Calciner and their Effects

When TBP-25 waste which contains 4 g of mercury salts per liter is calcined, volatile mercury compounds tend to plug the off-gas line. These are thought to be a mixture of mercuric oxide and mercuric nitrate. Mercury volatilizes from the calciner pot at a greater rate near the end of the calcination period and condenses on the cold surfaces as it comes in contact with them. The sublimation temperature is about 250°C.

During some tests, the off-gas line above the calciner feed flange plugged almost completely with this yellow mixture. If the off-gas line is heated to > 300°C, deposition does not occur, but the mercury salt is carried to another cold surface and deposited. If the first cold surface for condensation is in the water wetted condenser and it is at a low enough temperature, metallic mercury is formed instead of a solid compound. Several possible modes of operation to exploit this behavior are yet to be tested.

7.4 Evaporator Off-Gas System: Description

The off-gas system to handle noncondensables consisted of a sampling station, a gas-moving device, another sampling station, and two gas-volume-recording devices. The noncondensables were then discharged to the plant off-gas system.

The pressure in this system was controlled to provide -1 psig pressure in the evaporator. Very good regulation of this pressure was necessary to assure that the pressure-sensitive measurements in the evaporator be stable.

Two gas-moving devices were tested: (1) A 1-in. Schutte-Koerting water jet with a water pressure of approximately 60 psig and a flow rate of 15 to 20 gpm. It had insufficient capacity during the high off-gas periods. (2) A Nash-Hytor pump with a control valve in the suction of the pump to control the rate of gas removal from the system. This operated satisfactorily.

After the gas passed through the pump, it went through a sampling station and then through a model 15-A Foxboro differential-pressure (DP) cell with an integral orifice and then through a standard 1-cu-ft wet-test meter. Some of the nitric oxides in the off-gas dissolve in the water of the wet-test meter and corrode it. A corrosion resistant wet-test meter has not been available. The DP cell may be a satisfactory alternate to the wet-test meter for accurate measurement of the off-gas.

8. EVAPORATOR

The evaporator in the waste-calcination system is the main liquid-to-vapor decontaminating device which collects and returns to the calciner fission products from the calciner off-gas. The waste is evaporated here to a high salt concentration while the nitric acid from both the calciner condensate and the raw feed is steam stripped by the addition of water. The evaporator vapor acidity is kept at about 1.6 $\underline{M}$ or below to control ruthenium volatility.

8.1 Configuration of the Evaporator

The experimental continuous evaporator consists of a main section, 10 in. in diameter and 60 in. long. The bottom of the main section is coned to a 2-in. downleg 18 in. long that goes through two 90°-sweep elbow bends to the steam chest. The steam chest consists of a 22.5 ft^2 stainless steel heat exchanger 24 in. long and has 110 tubes, 3/8 in. in diameter. The steam chest discharges into the main section of the evaporator through a long 90°-sweep bend of 3 in. pipe (Fig. 11). The boilup rate of the evaporator, when clean, reaches 12 liters/min. An earlier design used a 2-in. right angle discharge which restricted the boilup rate to about 7 liters/min. The operating liquid-level in the evaporator is about 40 in. from the top of the evaporator.

The top of the evaporator has a de-entraining section. Two different de-entraining materials have been used: 1/4-in. porcelain Berl saddles and stainless steel Yorkmesh. The Berl saddles were supported from a stainless steel plate with 1/4 in. holes drilled at a minimum separation. The saddles, under 6 to 10 liters/min of boilup rate, had a pressure drop of 1 to 2 psig. On one occasion the supporting plate on which they rested was

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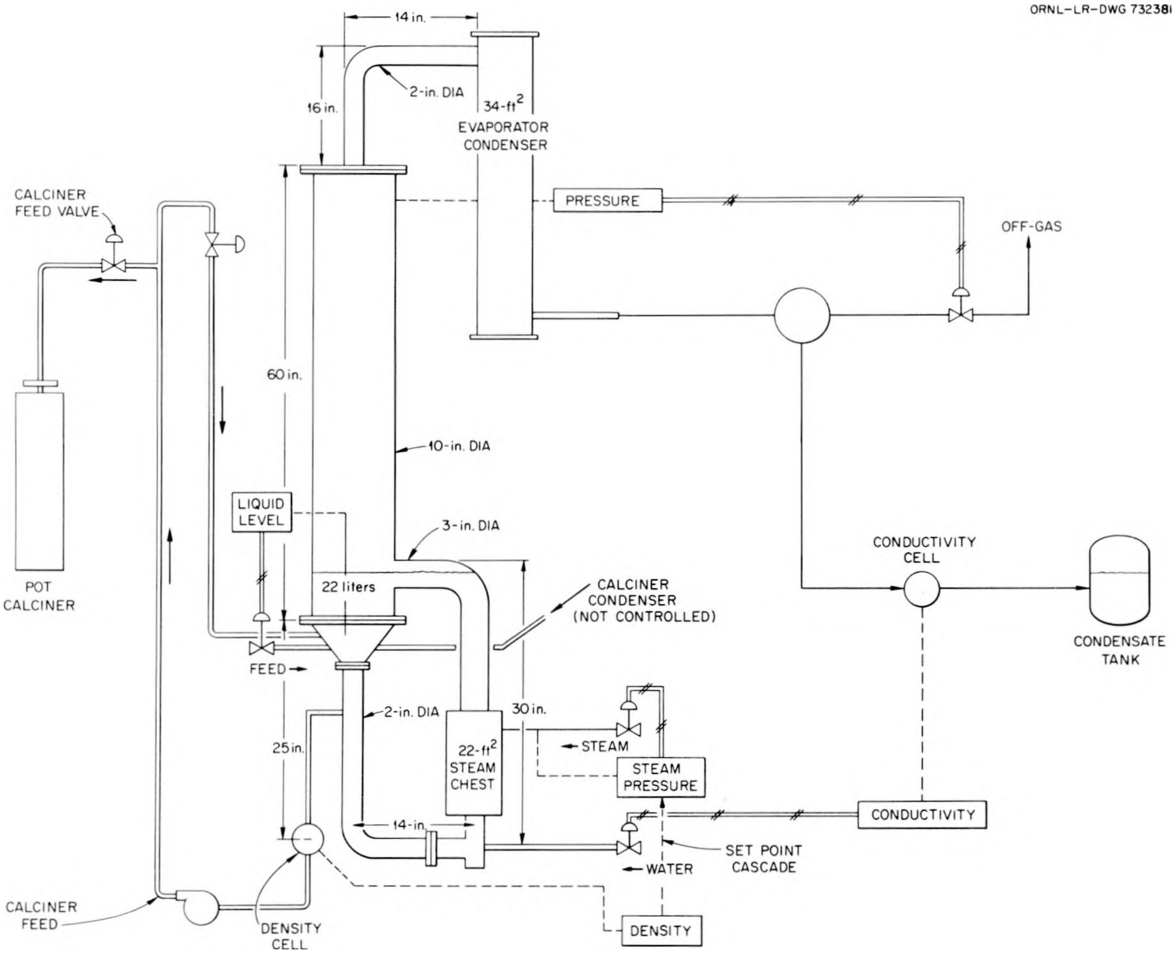

Fig. 11. Waste Evaporator Detail.

lifted, letting them dump into the bottom part of the evaporator. They were distributed throughout the system and ruined one pump by being ground by the impeller. The stainless steel York mesh operated well, with negligible pressure drop. A 2-in. vapor line conducts the vapor to the evaporator condenser.

The batch evaporator was a stainless steel vertical tank 3-1/2 feet in diameter and has a maximum capacity of 1400 liters (Fig. 5) with a liquid level indicator range from zero to 1200 kilograms. The evaporator had top inlets for feed and water, and an impingement plate de-entrainer vapor outlet which was connected to the 34 ft^2 downdraft evaporator condenser. A maximum steam pressure to the 22 ft^2 heating coil was 100 psig. There was appropriate piping so that liquid could be transferred from the batch evaporator to the 700 liter heated hold tank or back to the evaporator from the hold tank.

8.2 The Evaporator Condenser: Description and Operating Characteristics

The present evaporator condenser is a 34-ft^2 stainless steel exchanger with 5/8-in. tubes 6 ft long with the cold water (20°C) in the tubes. It has never accumulated scale nor has it needed to be cleaned. Foam from the evaporator has gone into the condenser but has never caused it to plug. The condenser uses 20 gpm of cooling water, and the surface area is inadequate at condensate rates exceeding 6 to 7 liters/min.

8.3 Control of the Continuous Evaporator

The variables that must be controlled in the process are:

1. Acidity in the evaporator. The vapor from the evaporator is maintained at an acidity of about 1.6 M which allows the liquid

to come to about 6 M NO_3^-, depending upon the salt concentration. The acidity is not permitted to rise above this concentration because of its effect upon ruthenium volatility. This is effected by adding water to the evaporator. The vapor acidity was selected for control over the liquid acidity because it was easier to measure, and because it was felt that, being more directly related to nitrate activity, it was a better index to ruthenium volatility. Two methods of measuring the vapor acidity were employed. Using the vapor temperature was the easiest, but this proved unreliable because of its sensitivity to system pressure fluctuations, superheating, and partial condensation. The latter two effects were surprisingly important because of the high salt concentration in the boiling liquid and the wide variation in boilup rate during a run. The second method was using the electrical conductivity of the evaporator condensate. Both a homemade cell and a commercial unit (Leeds and Northrup flow through type) were used with equal effectiveness. They were placed directly in the line with nominal provision to maintain a flooded condition, bypassing entrained gas, and were found adequately sensitive, accurate, reliable, and rugged. They measure acid concentration directly and are not noticeably disturbed by other operating variables.

It should be noted that a practical limit on reducing the acidity by the addition of water is imposed by the increasing volume of boilup with its small but inevitable entrainment.

2. Metal ion concentration in the evaporator. This concentration is kept as high as practical, consistent with safely avoiding exceeding

the limit of solution stability. Metal ion concentration is inferred from a measurement of the liquid density, which, for this system, is justified by: (1) the nitric acid concentration, which is the only other variable, has a much smaller effect on the density, and (2) the nitric acid concentration is independently controlled. The density signal controls the boilup rate by controlling the steam to the evaporator steam chest.

3. Liquid level in the evaporator. The liquid level in the evaporator is measured and used to control the feed rate to it.
4. Pressure in the evaporator. The pressure in the evaporator is kept at a negative value to prevent outleakage of radioactive off-gas by regulating the off-gas pump.

For details on the control system, see Section 9.

8.3.1 Evaporator Steam Service

A 2-in., 250-psig steam service was made available and brought to a reducing station near the cell for the experimental testing. A Mason-Nielan steam reducing station No. 1-6030 was installed. The steam was reduced from 250 to 100 psi before it went to the Hammel-Dahl control valve, which was modified so that it had approximately linear characteristics. One-inch piping carried the steam from the control valve to the steam chest in the thermosiphon loop. A bucket trap was used, backed up with a 0.5 ft^2 heat exchanger to cool the steam condensate for rate measurement.

8.3.2 Evaporator Steam Chest

The evaporator steam chest was a 2-ft-long, 22-ft^2 heat exchanger (110 3/8-in. tubes), constructed from type 347 stainless steel. At times of malfunction, using Purex waste, the evaporator went dry, leaving a very tough plug of dry Purex waste solids in the evaporator heat exchanger tubes; this had to be drilled out. On one occasion when TBP-25 waste went dry and solidified in the evaporator heat exchanger, the solid was removed from the tubes by applying 15-psi steam directly to the solid and melting it out of the tubes.

8.4 Control of the Batch Evaporator

The batch evaporator is charged with a complete batch of dilute raw feed, about 600 liters. This raw feed is concentrated to about 400 liters. During this time the steam control is operated manually to conform with a desired rate of concentration. Once the desired concentration level is reached, the batch evaporator is operated similarly to the continuous evaporator; that is, the density is used to control the boilup rate, and the vapor temperature or vapor conductivity is used to control the water addition for removing the excess nitrates in order to reduce the possibility of ruthenium volatility. At the end of each test, the concentrated evaporator volume is transferred to a feed hold tank to become feed for the next calcination test.

8.5 Thermosiphon Evaporator Control Rates

Two liquid streams were fed to the evaporator and were controlled with Foxboro control valves catalogue No. Bull-5C-13A. These 1-in. control valves had stainless steel trim of varying sizes (3/16, 1/4, 3/8, and 1/2 in.). When the valves were checked for leak tightness and set correctly, they operated well. The valve characteristic was much improved by adding a pneumatic positioner to the operating complex. The feed valve used had a 1/4-in. trim, and the water control valve had a 3/8-in. trim. The rates varied from 0 to 10 liters/min for water and 0 to 4 liters/min for feed.

8.6 Evaporator Pumps

In this process, liquid from the evaporator is pumped around the recirculation loop at about 10 gal/min, from which the calciner feed is drawn. The liquid has a density from 1.0 to 1.5 g/cc, a maximum temperature of 116°C, 1 to 6 $\underline{M}$ nitric acid and a maximum of 70 g of iron per liter or 80 g of aluminum per liter. The circulation pump must develop an output of about 25 psig in order to be used in the present system.

A Milton-Roy piston pump, tried first for this service, had a rate of only 3 or 4 gal/min and could not accommodate slurry. A 1L2 Moyno pump operated satisfactorily mechanically but required excessive maintenance. A stainless steel rotor and stator were severely abraded when aluminum oxide or calcium sulfate slurry was used. The nitric acid solutions destroyed a rubber stator. The Moyno pump also had a packing gland characteristic that allowed the pump to leak.

A model G canned rotor Chempump operated satisfactorily. The bearings must be flushed with clean water at a minimum rate of 25 cc/min for sufficient protection. When the water-flow rate was decreased to 5 or 10 cc/min, the bearings were damaged.

A Chempump should be protected in the following ways with electrical overrides and interlocks. (1) If the bearing water is not operating, the pump should not operate. (2) If there is not a positive discharge pressure in the pump, the pump should not operate. (3) If the pump temperature exceeds 200°C in the rotor section, the pump should not operate. An impeller on one model D Chempump was ruined because the pump was operated with a suction valve closed, causing the pump to cavitate.

A Byron and Jackson canned **rotor** pump developed from an ORNL design was briefly tested with Coors aluminum oxide bearings and appears to operate well. It is rated at a 50-ft head at 10 gpm. The pump has a bearing-water bleed and a cooling-water coil, both of which must be activated before the pump will operate. It has a motor-winding temperature cutout set for 50°C.

8.7 Disposition of Evaporator Condensate

The evaporator condensate after leaving the condenser goes to a liquid-gas separator and then to the condensate catch tank, which operates on overflow principles through an airlock. The liquid level of the condensate catch tank is monitored and recorded and the flow rate out of the catch tank is metered using an orifice. The condensate is sampled before going into the catch tank.

The evaporator entrainment was very low. From a Purex-waste evaporation, 0.5% of the iron and 0.9% of the sulfate were carried over. From TBP-25-waste evaporation, the per cent of aluminum in the evaporator condensate was 0.2% of the amount fed to the evaporator.

9. INSTRUMENTATION AND CONTROL

Instrumentation and control equipment development was undertaken for the evaporator and calciner. The calciner requires liquid level and exterior temperature control. However, the calciner pressure, inside temperatures, and the furnace temperatures are recorded. The evaporator has four control variables: the nitric acid concentration in the evaporator; the salts concentration in the evaporator; the evaporator pressure, and the liquid level. Acidity and salt concentration are controlled through their counterparts, condensate acidity and liquid density (Fig. 12). See Section 9.3.

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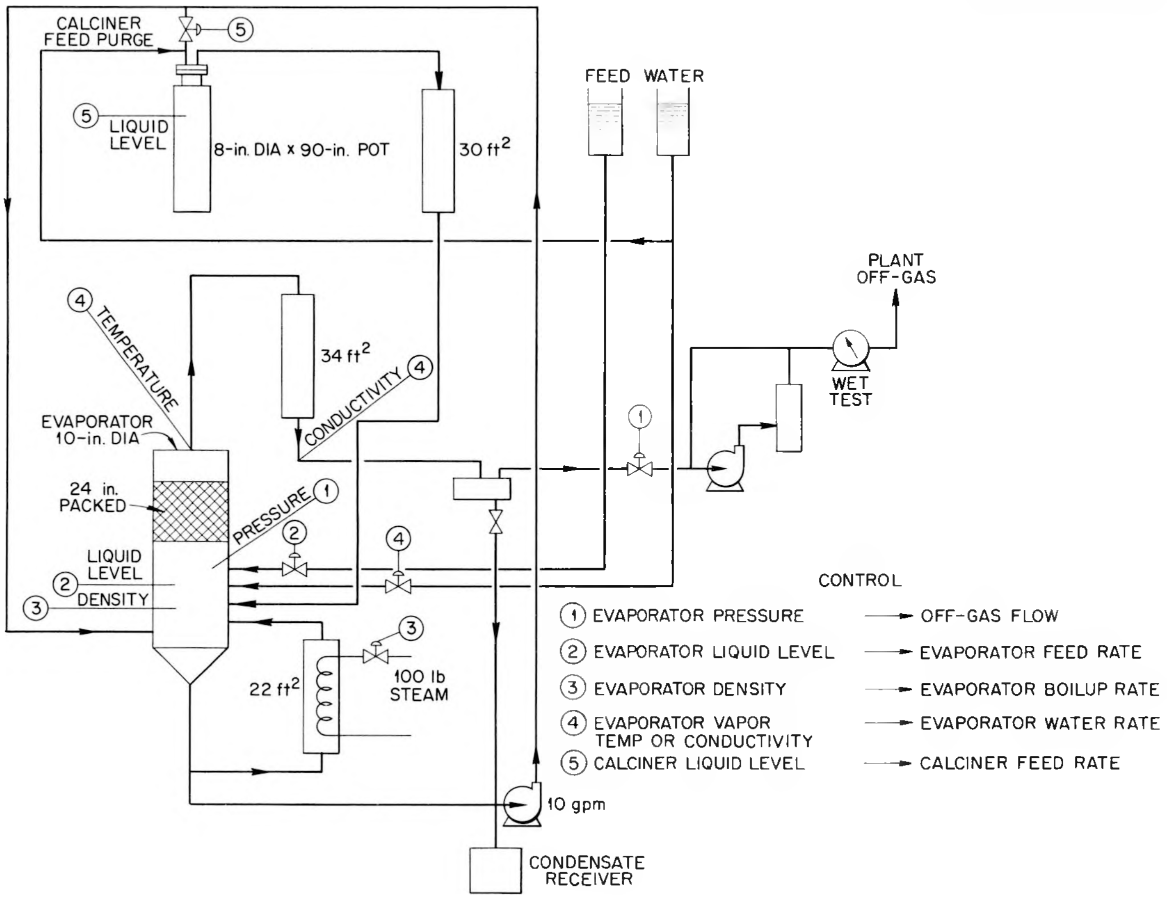

Fig. 12. Calciner-Evaporator Control Flowsheet.

9.1 Calciner Liquid-Level Measurements: Conductivity Probe

The first attempt made to measure and control the calciner liquid level was made with a three-probe conductivity controller. When the bottom and top conductivity probes were short circuited, meaning that the liquid was covering both conductivity probes, the liquid being pumped to the calciner was automatically turned off. When the liquid level dropped below the bottom probe, the liquid was automatically turned on. The third probe was used as a common ground for the system. This arrangement attempted to keep the liquid level of the system between the two probes, and the separation of the probes could be changed to allow for flexibility. Because of the foaming characteristics of the calciner-pot operation, this on-off control did not work.

9.2 Calciner Liquid-Level Measurements: Differential-Pressure Bubbler Probe

A standard differential-pressure liquid-level bubbler, the type commonly used to measure liquid levels in open or pressurized tanks, was tried to measure and control the liquid level in the calciner. This system was set up with a 10-in. difference between the pressure probes. When clean it performed well, but calcined solids built up in the probe, ruining the pressure signal. (The solids can be removed by flushing with air and water; however, a small amount of solids often caused a spurious signal.)

9.3 Calciner Liquid-Level Measurements: Thermocouple Probe

The calciner-pot liquid level is indicated by a thermocouple type of liquid-level probe. Several different types of thermocouple liquid-level probes were tested (Figs. 13 and 14, Table 3) to find the type of proportional response most suitable for control.

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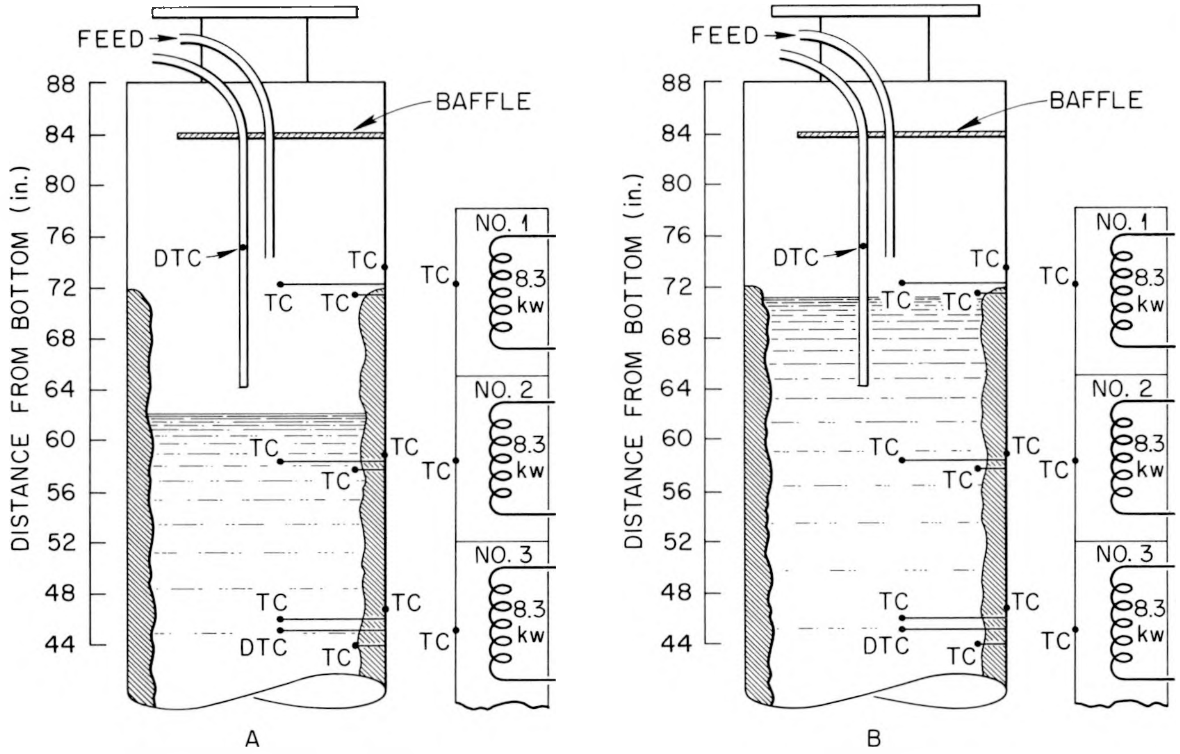

DCT ΔT = 600°C − 120°C = 480°C
TOP BOTTOM

DCT ΔT = 220°C − 120°C = 100°C
TOP BOTTOM

CONTROL RANGE = 50−150°C FOR 0−100% CONTROL OUTPUT

Fig. 13. Dual Thermocouple Temperature Control System.

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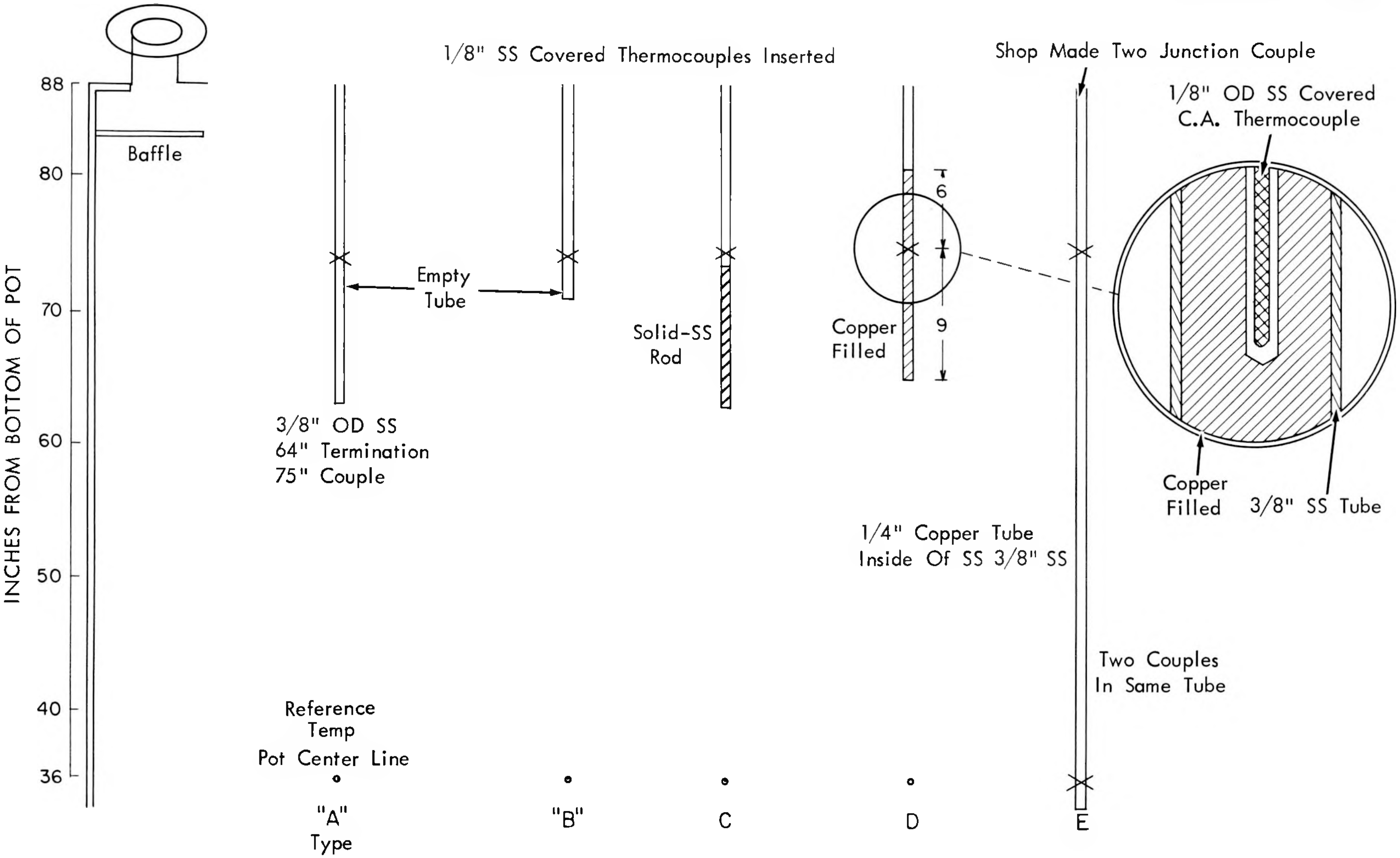

Fig. 14. Control Thermocouple Details.

Table 3. Type of Thermocouple Used for Tests R-43 to R-64

Type of Thermocouple (Fig. 13)				
R-43	A	R-54	C	
R-44	B	R-55	C	
R-45	B	R-56	C	
R-46	B	R-57	D	
R-47	B	R-58	D	
R-48	B	R-59	D	
R-49	B	R-60	D	
R-50	B	R-61	D	No ref.
R-51	B	R-62	E	
R-52	B	R-63	D	No ref.
R-53	A	R-64	D	No ref.

The type selected (type D) is a 3/8-in. diameter 347 stainless steel tube with a closed end. The closed end of the type is filled with a 15-in. piece of solid copper that has a 6-in.-long hole drilled in it for a 1/8-in. diameter stainless covered Chromel-Alumel thermocouple. The 9-in. rod below the thermocouple acts as a variable heat path and allows the thermocouple temperature to change as a function of the liquid height on the rod. It is important that the incoming feed not wash the rod.

This device effectively indicates the level of the foam, which is desirable for avoiding carryover into the off-gas line, it is sensitive to a fraction on an inch change in level, it is insignificantly affected by the solids which deposit on it, and it has a response time, which although not small by absolute standards, is much shorter than characteristic times of the system and adequate for detecting any creditable upset.

9.4 Acid Control of the Evaporator

The preferred method of measuring the amount of nitrate in the evaporator condensate is to measure its conductivity. The homemade conductivity cell for measuring the acidity of the evaporator condensate was made of 3/8-in. polyethylene tubing with insulated stainless steel fittings as electrodes. The center of the tubing was grounded and the two legs were used in parallel as part of a twelve volt, 60 cycle bridge circuit. The cell constant was 100 cm^{-1}, and when applied with proper circuitry produced a signal which was converted to a pneumatic signal which read from 0 to 100% corresponding to an acid variation from 1 to 3 M HNO_3. The conductivity cells are somewhat temperature sensitive, but in this service the condensate normally ran between 25 and 30°C and manual adjustment for temperature was adequate. The total length was 31 inches, and the holdup was 125 cc. A "U" tube arrangement was used with provision for entrained gas to be bypassed across the top. A commercial conductivity cell (Leeds and Northrup flow through type 4803-50-SS without temperature compensation) with a cell constant of 50 cm^{-1} was used in the same loop with the homemade unit, and functioned similarly. The measurements of the conductivity cell were found to follow the nitrate concentration very well. The signal was used to control the rate of water addition to the evaporator in order to hold this nitrate concentration at about 1.6 M.

The use of temperatures of the evaporator liquid and vapor to indicate the nitrate concentration of the evaporator liquid was unsatisfactory. The change of temperature with the changing concentration of the dissolved salts was greater than the temperature change due to the changing concentration of the nitrate, therefore control of the temperature of the liquid could not be used.

The dew point of the vapor leaving the evaporator would make an excellent index to the nitric acid concentration if it could be measured accurately enough. Several attempts to do this were frustrated. The difficulty lies in the fact that the vapor leaves the boiling solution with a 2 or 3°C superheat, which is of the same magnitude as the spread covering the pertinent acid concentration range. If all the superheat is removed and a slight amount of condensation occurs, the temperature rapidly drops below the dew point because of the high relative volatility of the nitric acid-water system. An attempt to draw a sample of vapor out of the evaporator at a constant rate, to allow for a fixed loss of superheat, was unsuccessful. A thermocouple in the vapor line between the evaporator and the condenser usually indicated below the dew point. Two thermocouples installed in the vapor space in the top of the evaporator were tried; one was pointed upward to keep it dry, and the other was pointed downward so that liquid would run over the tip. The degree of superheat was found to be sensitive to the boilup rate, and the wet thermocouple, though hardly satisfactory, was the best indication of dew point. These methods apply equally to the continuous and the batch evaporators.

9.5 Density Control of the Evaporator

The measurement of the evaporator density is a critical variable in the control of the continuous evaporator, and a number of different methods were tried for comparison:

1. internal differential pressure probes
2. differential pressure probes in the external loop
3. a density float cell in the external loop
4. a U-tube—weighing apparatus in external loop
5. a commercial U-tube in the external loop

Two types of internal differential pressure density probes were installed in the continuous evaporator; neither functioned satisfactorily. The first had a 6-in. probe separation, with a shroud around the two probes in the main section of the evaporator. This density instrument was sensitive to boilup and foam. As the liquid boiled at a higher rate, foam was entrained in the liquid which caused the measured density to decrease. A second differential pressure density cell was installed, using two probes (40-in. separation), one up in the main section of the evaporator and the other at the bottom of a thermosiphon leg. This cell was also boilup sensitive because of the pressure drop in the suction side of the thermosiphon leg.

The differential pressure measurement in the external loop functioned well. The measuring device consists of a 2-in. pipe with two 1/4-in. probes (24-in. separation) in the circulating pump loop near the return of the loop to the evaporator.

A density float cell of the "Mackey"[11] design, developed at ORNL, functioned fairly well. This density cell has a stainless steel float chamber containing a piece of iron which is detected by a magnetic-pickup assembly. The magnetic pickup has a 3- to 15-Mv output, which is converted to a 3- to 15-psig pneumatic signal used for control. Although the float cell is somewhat flow-rate sensitive, the flow rate through the cell can be maintained at a nearly constant rate.

A U-tube density-measuring cell was made in the laboratory shops. It consisted of a U-tube (24-in. x 1-in.-diameter) with a flexible bellows on both ends. The deflection of the U-tube by the weight of liquid passing through it produced the signal which was used. This device was unsatisfactory because the bellows expanded when the pressure was changed. A commercial U-tube cell was purchased from the Bell Instrument Corporation of Odessa, Texas, and it performed well on preliminary tests.

External devices for density measurement were not used with the batch evaporator, since simple dip tubes work well and are sufficient.

The liquid levels in the evaporators have been successfully measured with a differential pressure dip tube. In the continuous evaporator the lower tube is in a transition piece between the main section of the evaporator and the beginning of the thermosiphon downleg. A DP cell (50-in. of water) that corresponds to a liquid level of 50% of scale when the volume is 23 liters was used. The signal was somewhat affected by changes in boilup rate and pressure but not to the extent of disturbing control.

The continuous evaporator pressure is measured approximately halfway between the operating liquid level and the Yorkmesh de-entraining section. The distance between the operating level and the Yorkmesh is about 4 ft.

9.6 Recording and Control Equipment

The recording and controlling equipment used in this process were mostly standard commercial items. All temperatures were recorded on Honeywell-Brown temperature recorders, with a range of either 0 to 350 or 0 to 1200°C. These recorders were equipped with Zener power supplies to replace the battery used for cell standardization.

The two- and three-mode pneumatic controllers used for the system were Foxboro Minature control, model-5310. These controllers were good, but there was some trouble in keeping the manual-to-automatic transfer switch from leaking. When the defective switches were replaced, the problem was eliminated. Foxboro EMF-to-pneumatic converters were used when a thermocouple signal or a millivolt signal from a density cell was converted to a pneumatic 3- to 15-psig signal.

The solution control valves and off-gas control valves were Foxboro control valves No. Bull-5C-13A, equipped with positioners. The steam control valve was a Hammel-Dahl valve No. 13230 HD with an ORNL-made proportionating seat that was nearly linear. The conductivity-cell control instrument was fabricated from a number of standard pieces of equipment. The conductivity cell was monitored with a bridge circuit from a modified Honeywell-Brown recorder, the output of which was converted to a pneumatic signal. This 3- to 15-psig signal was then fed to a standard Foxboro controller.

10. RECOMMENDED EQUIPMENT AND OPERATING PROCEDURES FOR PILOT PLANT EVALUATION

In the flowsheet for the reduction of radioactive waste liquids to solids by pot calcination, the advantages lie in the simple operation of evaporating a waste solution to its maximum consistency and being able to pump this waste into a pot calciner that will be used for permanent storage. After the pot has been filled and calcined, it can be removed safely because there is almost no dust produced by this calcination process. Any equipment to be used in this process should be simple enough to comply with the foregoing flowsheet objectives.

10.1 Recommended Equipment to be Tested in Pilot Plant

The recommended equipment for a continuous pot calciner-evaporator flowsheet can be divided into three sections: evaporator, calciner, and off-gas equipment.

The recommended evaporator should be a thermosiphon type with an operating capacity of 25 to 50 liters. A small continuous evaporator is being recommended because of: (1) ease of operation, (2) low holdup volume, and (3) cost less than a batch evaporator system. It should have 25 to 40 ft^2 of heat exchange surface and should have a 50-to-1 minimum recycle flow ratio. The de-entraining volume should be 4 or 5 times the liquid holdup volume, and the de-entrainer should be equipped with either an impingement-plate system for de-entraining or a Yorkmesh de-entraining cartridge.

The evaporator—recirculation-pump system should be installed in such a manner that an adequate supply of calciner feed is available to the calciner. The recirculation pump should be of canned-rotor design with an external bearing bleed so that fresh water can be bled through the bearings at all times. The pump may or may not have an external cooling coil to remove the motor heat.

The recirculation pump should be located as close to the evaporator as possible, with a large, jacketed suction line from the evaporator to the pump. If the pump is located at a level below the operating liquid level of the evaporator such that a net positive suction head of 3 to 4 ft at the pump is maintained for all flow rates, this will eliminate the need for cooling the incoming liquid in order to prevent cavitation. However, if cavitation were common in the operation, cooling the jacketed section of the suction pipe would reduce the temperature sufficiently to correct this. The pump capacity should be 1 to 10 gpm and develop approximately 10 to 15 psig pressure at the calciner feed valve. This large flow rate will minimize the chance of solids settling in the recirculation loop, and the high available pressure drop will tend to remove any tendency of the calciner feed line to plug. The external density measurement should be made on the recirculation stream close to the evaporator. A bubbler type density cell with upward liquid flow proved to be the most satisfactory type of density cell tested.

10.2 Proposed Pilot Plant Calcination System

The recommended calcination equipment can be grouped into two categories: (1) the calcination pot and connecting equipment, and (2) the calciner furnace. The calciner pot should be 8 to 12 in. in diameter, depending on fission product activity and should have a minimum operating level to the liquid-level control point of 6 ft. For a length of operating calciner less than 6 ft, the end effects would seriously affect the worth of the data for scale-up, for pot diameters of 6 inches or greater. The calciner feeding system should have at least two feed addition lines, (1) from the evaporator circulation pump and (2) from the additive feed tank, such as calcium nitrate, to reduce sulfate volatility in Purex waste or the addition of chemicals to make a glassy mixture. The calciner pot should have a baffle approximately 1 ft above the operating liquid level to prevent splashing and the direct carrying of entraining liquid into the off-gas lines. The off-gas lines should have a minimum diameter of 1-1/2 in. (preferably 2.0-in.) and should rise vertically from the calciner to the highest necessary elevation and then should be sloped towards the condenser.

If TBP-25 waste, which contains 4 g of mercury per liter, is calcined there will be a mercury deposition problem in the off-gas lines. A mercury—

nitric oxide mixture, a yellow solid, will deposit in the off-gas lines at a temperature below 250°C and can completely plug the off-gas lines. This can be eliminated by heating the off-gas lines above 300°C. However, the mercury compound must be removed from the system by some method. There are two alternatives, neither of which has been thoroughly tested. One possibility is to install a mercury trap consisting of an expanded section of the off-gas line, which would be cooled and would allow the mercury compound to build up on the walls but which would have a large enough cross sectional area that it would not become plugged during a run. (This mercury compound is very soluble in 6 M HNO_3 and could be removed from the mercury trap between tests.) The other possibility is to heat the off-gas line from the calciner to the calciner condenser, where a stream of water would be injected into the calciner vapor line. It has been demonstrated that the mercury-containing solid will deposit on a film of water in the calciner condenser and can be carried back to the evaporator with the calciner condensate. However, this is only a stop-gap measure because the mercury would build up in the evaporator and then recycle.

A secondary calciner off-gas line is necessary because of the possibility of plugging the primary line. The secondary off-gas line should be of the same diameter as the primary line and should be connected to a liquid seal pot with a hydraulic discharge head of 15 to 20 ft, which would give a 7 to 10 psig positive seal. It may be necessary to use a gas purge and heat the secondary off-gas line to remove all chance of solid deposition in this line when it is not being used.

The calciner condenser should have an adequate surface area and cooling water to reduce the calciner condensate at its maximum rate (which would be 60 to 100 liter/hr) to a temperature below 40°C to encourage the complete absorption of the nitric oxides produced by thermal decomposition of the calciner contents. This condensate should flow by gravity to the evaporator and be introduced into the evaporator at a point that would give the best mixing with the evaporator liquid phase.

Calciner Furnace. The calciner furnace should have a power input of approximately 8 to 10 kw per foot of calciner pot. A resistance furnace with 1-ft controlled zones has proved to be very satisfactory. A thermocouple on the calciner pot surface has been used to control the heat input to the system and a thermocouple on the furnace wall has been used to protect against overheating

10.3 Recommended Off-Gas System

The handling and control of the off-gas from the system have the following functions to perform. The system must be operated under a negative pressure of about 1.0 psig below atmospheric. This is a safety measure ensuring that all leakage of the system will be inward. The pressure inside the evaporator should be controlled at a constant pressure to keep the boiling point of the evaporator constant. The pressure drop from the calciner to the evaporator should be small, about 1/4 psig under normal operating systems. The recommended prime moving device for the calciner off-gas would be a mechanically driven gas pump similar to a Nash Hytor. A jet was tested and was slightly inferior, because of the large volume of water required.

10.4 Recommended Control Procedure

The control of the waste-evaporator—calciner was easily obtained by the use of standard control equipment. Five conditions must be controlled, and evaporator control is the most complex part of the system. However, the control of the calciner itself is the heart of the system.

1. The liquid level in the calciner must be controlled within a fairly narrow limit at a level near the top to maintain the maximum calciner rate. If the level is too high and foaming occurs, a serious plugging problem could develop in the off-gas lines if foaming continues over an extended period. The liquid level in the calciner is measured by the use of a thermocouple encased in a rod. When the tip of the rod touches the surface of the liquid, heat is removed from the rod, thereby reducing the temperature of the thermocouple contained in the rod. Therefore, if the temperatures of the thermocouple decreases, the liquid level in the calciner is increasing. A 100°C change of temperature of the thermocouple in the rod indicates an approximate liquid-level change of 4 to 6 in. This method of liquid level indication in the calciner proved to be superior to the other types of liquid level measurements attempted. The liquid-level thermocouple has been controlled to within ± 50°C, which is approximately ± 3 in. in height.

2. The liquid in the evaporator must be controlled for inventory and safety reasons. The salt concentration must be maintained so that a maximum rate of solids is fed to the calciner. However, if the solids concentration is too high, they will precipitate and may plug the evaporator and recirculation line. The nitrate ion concentration in the evaporator must be below 8 M to reduce the volatility of ruthenium.[10] The pressure in the evaporator must be controlled at a subatmospheric value. Pressure fluctuations would cause unwanted boiling point changes.

To accomplish these necessary control functions, the various measuring devices and appropriate control valves on the necessary streams were tested, and the following scheme is preferred for controlling the evaporator.

(a) The density of the contents of the evaporator should be measured in an external density cell. The density signal should be cascaded and used as a remote setpoint from the steam-pressure controller. The steam-pressure controller then will regulate the amount of steam supplied to the steam chest of the evaporator and therefore regulate boilup.

(b) The liquid level in the evaporator, measured by a bubble-type liquid-level probe, should control the incoming raw feed from the feed storage tank for the system.

(c) The conductivity of the evaporator condensate is an excellent measure of its nitric acid content, and the nitric acid content of the vapor phase of the evaporator is a reasonable measure of the nitrate ion composition of the liquid phase. Therefore, the conductivity of the condensate is used to control the water addition for stripping nitric acid from the evaporator.

(d) The evaporator pressure is sensed in the disengaging section and is used to control the throttling valve on the suction of the gas pump in the system off-gas.

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