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DESIGN OF A PILOT PLANT FACILITY FOR RADIOACTIVE
DEMONSTRATION OF THE POT CALCINATION PROCESS

MASTER

B. R. Wheeler and J. A. Buckham

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION

**NATIONAL REACTOR TESTING STATION
US ATOMIC ENERGY COMMISSION**

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A B S T R A C T

Based on process development work at the Oak Ridge National Laboratory, a facility has been designed by the Atomic Energy Division of Phillips Petroleum Company for demonstration of the pot calcination process with a variety of high-level radioactive wastes. In this facility, operational and control problems associated with an integrated process can be identified and solved, procedures to improve characteristics of the calcine can be studied, and important aspects of calcine temperature distribution and fission product behavior can be observed. Specialized equipment and procedures have been developed for this facility.

This 1.45-million dollar facility was originally scheduled for installation at the Idaho Chemical Processing Plant and for initial operation early in 1964. However, its installation is now planned for the Hanford Atomic Products Operation. The designed facility, with minor modification, can be installed in any cell or cells having certain basic features.

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DESIGN OF A PILOT PLANT FACILITY FOR RADIOACTIVE DEMONSTRATION OF THE POT CALCINATION PROCESS

by

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I. SUMMARY

A facility has been designed in collaboration with the Oak Ridge National Laboratory (ORNL) for demonstration of the pot calcination process with various high-level radioactive wastes from processing aluminum alloy, stainless steel alloy, and unalloyed fuels. With this facility, procedures can also be developed for production of low-porosity materials with a low-water solubility by adding suitable reagents to the calciner feed. This pot calcination process, which consists of concentrating wastes by evaporation and thermal decomposition in situ in cylindrical pots, which also serve as the final disposal containers, was evolved principally at ORNL and was developed there through both bench-scale and pilot plant-scale studies with nonradioactive and tracer materials.

The radioactive demonstrational facility was designed with the intent that it would be adequate to obtain the necessary data for design of a production plant. In this facility, operational and control problems connected with the calciner or its closely associated feed preparation and condensate and off-gas decontamination systems can be identified and solved; many of these problems are associated with self-heating and volatilization of fission products. The density, water solubility, melting point, volatile material content, heat release, and thermal conductivity of the calcine and factors affecting the pot loading can be determined. In addition, transient calcine temperature distributions, fission product behavior during calcination, effectiveness of de-entrainment in various parts of the process, degree of decontamination of all liquid and gaseous effluent streams, need for venting pots, optimum method of sealing the pots remotely, and economical methods to minimize the volume of noncondensable off-gas can be studied.

The design basis, a maximum pot feed rate of 100 liters per hour, sets in turn the capacity of all auxiliary equipment. A unique evaporator

design was evolved to permit operation either with close-coupled continuous feed preparation or with batch feed preparation. Provisions were made to circumvent possible explosions due to organic material in feed solutions and other suspected hazards.

A realistic target date, November 1963, for the completion of construction and installation of this facility in the Hot Pilot Plant at the Idaho Chemical Processing Plant (ICPP), was established by means of a network analysis planning diagram. Spike run tests were to take place during the period February through April 1964. In April, 1962, however, the Division of Reactor Development of the Atomic Energy Commission transferred responsibility for the construction and operational phases of this radioactive demonstration to the Hanford Atomic Products Operation. As of that date, the engineering process flowsheet had been fully developed and detailed design of essentially all major equipment for this facility had been completed; the estimated capital cost for the complete facility is 1.45 million dollars.

Although this facility was designed for installation in the ICPP Hot Pilot Plant, it is believed that the design can be utilized with minor modification at any AEC installation. Minimum cell requirements for this facility include 4-1/2-foot-thick shielding walls, a 20-foot cell height, viewing windows located about 9 feet above the cell floor, and a dolly mounted on a track for movement of the pots. A small crane, heavy-duty manipulators, and light-duty manipulators are required for various remote handling operations.

It is believed that a pot calcination facility designed as described herein, a result of extensive ORNL-ICPP exchange of technical ideas, has a high probability of satisfactorily demonstrating an integrated pot calcination process.

II. INTRODUCTION

Intensive effort is being expended at all major atomic energy installations to develop a method for permanent storage of radioactive wastes resulting from the processing of spent nuclear fuel elements. Current storage of these wastes in the form of corrosive liquid in large underground tanks is temporary and potentially hazardous. By converting

the liquid waste into suitable solids, a significant reduction in volume can be achieved, and at the same time the potential hazards can be minimized. Among calcining techniques being developed to accomplish this are the fluidized bed, the pot, the radiant spray, and the rotary kiln calcination processes. The pot calcination process is particularly promising for very high-level wastes if storage in sealed containers is adopted.

The pot calcination process has been under intensive development for several years at the Oak Ridge National Laboratory^(1,2,3,4,5) and has also been studied at the Hanford Atomic Products Operation^(5,6,7,8,9,10). A similar process has been studied intensively at Harwell⁽¹¹⁾ in the United Kingdom. In the pot calcination process, aqueous wastes are evaporated and thermally decomposed; the resulting solids are calcined at a temperature level of approximately 900°C in situ in portable stainless steel pots which also serve as the final storage container. Preparation of feed and decontamination of both condensed and noncondensable portions of the overhead stream from the calciner are associated process steps. Development of this process in the laboratory and cold pilot plant equipment to date has indicated that the process is feasible and can be applied to a wide variety of aqueous wastes.

In February 1961, the demonstration of this process on a high-level radioactive basis was assigned to the ICPP, working in conjunction with ORNL on the process and pilot plant design. Broad objectives of this demonstration were: to demonstrate the feasibility of the pot calcination process for converting a variety of high-activity, solvent-extraction raffinate wastes into solids in a safe operation; to show that such solids are thermally stable at temperatures up to those of calcination; to show that such solids are suitable for permanent disposal in the pot calciner; and to show that operation of the process could be done at rates and costs suitable for possible application to power-reactor fuel processing. Specific information expected from this demonstration and not obtainable with cold or tracer level work includes identification and solution of operational and control problems that are associated with self-heating and volatilization of fission products. These problems are related to the pot calciner and also to its feed preparation and off-gas and condensate cleanup systems. Furthermore, a demonstration with

radioactive material will indicate whether such factors as foaming or entrainment are limiting capacity factors and will also serve to develop equipment and handling techniques required to connect and disconnect calciner vessel process lines, to seal the vessel, and to remove it from the operating area for permanent disposal.

To expedite the radioactive demonstration of this process, responsibility for various phases of the project was assigned to each of the participants. At ORNL, the chemical, material, and energy-balance flow-sheets were to be prepared, and certain uncompleted development work was to be finished. At the ICPP, the process flowsheet was to be developed, the equipment and installation designed, and experiments for the facility designed and performed.

It became apparent during the early phase of the program that additional cold and possibly tracer level work was required on factors such as inhibition of the volatility of various fission products and corrosive sulfur compounds, handling of mercury vaporized during calcination of certain wastes, the nature of the hazard from possible organic contaminants in waste solutions, and the use of additives to form superior solid products. This development work, along with testing of certain devices, equipment control schemes, etc., was to be performed at ORNL. In addition, it was agreed that ORNL would subcontract (to Lockheed Nuclear Products Company at Dawsonville, Georgia) testing of specialized mechanical equipment designed by ORNL for remote operation of the pot calcination facility.

Since the Division of Reactor Development initially stated that an early radioactive demonstration of the pot calcination process was imperative, the detailed design of the demonstration facility at the ICPP was undertaken concurrent with laboratory and pilot plant development work at ORNL. Obviously, such a procedure required a significant interchange of ideas and data. By employing this procedure, it was possible to schedule the facility for spike-run tests during the period February to April, 1964.

In April, 1962, however, the Division of Reactor Development transferred responsibility for the construction and operations phases of this radioactive demonstration to the Hanford Atomic Products Operation. At that time, the engineering process flowsheet had been developed and

detailed design of essentially all major equipment had been completed. This report summarizes the scope, and the objectives of this radioactive demonstration program, discusses in detail the engineering process flow-sheet, presents the design basis for the major process equipment, and presents cost and schedule information for the intended installation at ICPP.

III. SCOPE OF THE RADIOACTIVE DEMONSTRATION

It was intended that this demonstration provide design data for all components of an integrated process similar to one that may be used in a production plant including, in addition to the calcination step itself, feed preparation, off-gas decontamination, and liquid-effluent decontamination systems. While the feasibility of pot calcining radioactive wastes could be established without operating the latter three systems, it was felt that operation of a completely integrated process was required to generate adequate data and operating experience, to establish and evaluate the safety and feasibility of the integrated process, and to make detailed comparisons with other waste disposal processes.

The attainment of complete and meaningful data was an overriding consideration throughout the design of this facility. Every effort was made to insure that each run in the facility could be essentially independent of all others so that a maximum amount of useful data could be obtained from a minimum number of runs.

During the course of the demonstration, several types of radioactive wastes having various compositions and activity levels are to be processed, including enriched uranium-aluminum alloy wastes, Darex or electrolytic dissolution process wastes, high-sulfate Purex-type wastes, and sulfate-free Purex-type wastes. Pot calcination of stainless-steel sulfate wastes and the fluoride-containing wastes from STR, Zirflex, barium fluozirconate, and similar processes is a desirable goal and may be accomplished at a later date, depending on the state of technology; however, calcination of these wastes depends on finding a suitable additive to prevent equipment corrosion from compounds of sulfur or fluorine, or on the replacement of certain key pieces of equipment with special corrosion-resistant items. 9

In a second phase of this demonstration, appropriate additives will be used with various wastes to form impermeable, glass-like materials. Modification of the process in this manner appears attractive as a means of converting liquid wastes to solids having low-water solubility, low porosity, and high thermal conductivity. Such material could be stored in larger diameter pots because of improved heat dissipation with attendant reduction in process costs.

Provisions were made in the design of this facility for eventual sequential demonstration of other calciners. The feed preparation, off-gas decontamination, and process-liquid decontamination systems of this demonstration facility can be used also for the recycled-gas fluidized bed, the rotary kiln, and the radiant-heat spray-calcination process. The use of common auxiliary equipment to demonstrate several calcination processes sequentially will make possible an early comparative evaluation of these contending processes with considerable monetary savings. The maximum size of the other calciners necessarily would be governed by the capacity of the auxiliary equipment designed for the pot calciner, but this appears to be an appropriate size in each case for an effective demonstration. Space is to be left in the cells of the pilot plant which houses the pot calcination demonstration facility for installation of a second type of calciner.

IV. OBJECTIVES OF THE RADIOACTIVE DEMONSTRATION FACILITY

Specific objectives of this demonstration have been based upon careful consideration of all pot calcination development work at ORNL and the design studies at the ICPP. As operating experience is gained with this facility, the need for modifying or supplementing these objectives may become evident. For this reason, versatility of operation and of data collection was emphasized in the design. The following specific objectives have been established for the demonstration:

1. Demonstration of Processing Rates.

Operating conditions which influence calcining costs through their effects on processing rates are to be identified and thoroughly explored. The processing rate is affected by average feed rates attained, by down-time required to change pots, and by the pot volume effectively

used. All of these factors will be examined in the radioactive demonstration, but the latter factor is the one most likely to be affected by operation with radioactive material. The optimum fill volume of a pot depends primarily on vessel and plant operating costs; Perona⁽¹²⁾ predicted, on the basis of cold pilot plant work, that this volume varies from 99.8 per cent of the usable pot volume for a 6-inch-diameter pot to 92.5 per cent for a 24-inch pot. These values should be verified or corrected after operation of the radioactive facility.

2. Demonstration of Suitable Feed Preparation.

The feasibility of simultaneously concentrating high-level radioactive raw waste and decontaminating the acidic condensate from the calciner is to be demonstrated. To accomplish this, it must be demonstrated that volatilization of ruthenium, from blended waste and condensate, can be minimized by employing equilibrium vaporization during operation of the evaporator.

3. Determination of Calcine Properties.

The water solubility, porosity, melting point, and volatile constituents of the calcine are to be determined on samples of calcine. Such factors as heat release rate, calcine thermal conductivity, radiolytic decay rate of residual nitrate compounds, and creation of localized hot spots in the calcine are to be determined by appropriate temperature and pressure measurement during interim storage of the pot.

4. Establish Optimum Pot Size for Various Waste Solutions.

The upper limit on pot diameter is determined by the heat generating and dissipating characteristics of the waste solids, and by the maximum allowable centerline temperature of the stored product. Rejection of the generated heat from the calcine without exceeding this maximum centerline temperature is primarily dependent upon the nature and porosity of the calcine. Such factors as waste composition, geometrical configuration of the pots, use of additives, and operating conditions employed may have a significant effect on the calcine porosity and, hence, upon the optimum pot size. Process optimization will be attempted by employing various modes of introducing liquid feed to the pot (continuous or intermittent), and the use of a variety of temperature-time control combinations.

5. Determine Effects of the Use of Additives.

Various chemical reagents may be added to the calciner feed with the expectation of suppressing the volatility of ruthenium and of corrosive sulfur compounds or forming glass-like calcine. Effects on fission product behavior and on other factors from the use of additives are to be determined.

6. Determine Effects of Fission Product Heat Release.

Overheating of the calcine may occur near the end of a run unless adequate controls are devised to switch from pot heating to cooling as the evolution of heat from the fission products exceeds the residual heating requirements. Overheating could result in the release of corrosive sulfur compounds from some wastes or excessive volatilization of some fission products from other wastes. Overheating also endangers the structural integrity of the pot itself. Underheating of any portion of the calcine must also be avoided because excessive residual nitrates may create a pressure problem if the pots are eventually sealed. Operable temperature limits must be evolved for each waste, and the development of suitable operating procedures to detect and control addition and removal of heat from various portions of the pot is necessary.

7. Demonstrate Adequate De-entrainment of Gas Streams.

Effective de-entrainment of mists from the gas streams is required if effluents from the process are to be released to the surroundings. This is of especial importance in the feed evaporator since it not only concentrates the raw waste, but also separates volatile fission products from the effluent stream from the pot calciner. Equipment in the process is designed to minimize entrainment, and the effectiveness of de-entrainment is to be determined during operation with high-level waste solutions.

8. Demonstrate Adequate Decontamination of Effluent Streams.

The integrated pot calcination process produces four effluents-- calcine, off-gas, decontaminated water, and decontaminated concentrated-nitric acid. Reduction of the activity level of the last three of these effluents to a point permitting disposal to the surroundings is to be demonstrated.

9. Develop Suitable Pot Closure Techniques.

For interim storage, the pots are to be closed with a temporary,

reliable, mechanical seal of a type³ which will permit opening them for sampling or venting. Before the pots are moved to a repository for permanent disposal, a high-integrity permanent pot closure, possibly a welded closure, may be necessary. The feasibility of closure without a vent is to be established by observations made during interim storage, and a satisfactory closure is to be identified and demonstrated during remote operation.

10. Observation of Pots During Storage

Temperature and pressure transient behavior of all filled pots are to be observed during an interim period of storage. Should a pot develop an excessive internal pressure, that pot will be connected to a manifold for pressure relief and for analysis of the off-gas. The extent of pot corrosion occurring during or following calcination is to be determined. Data obtained during interim storage will be used to establish permissible conditions for permanent storage of the pots.

A permanent repository for pots must be found. Such factors as possible pot rupture, pot corrosion, the water solubility of the calcine, possible ground contamination, and required pot handling facilities are to be considered when selecting the repository. Factors and potential hazards associated with transporting pots containing radioactive materials must be identified and considered.

V. GENERAL PROCESS DESIGN CONSIDERATIONS

A. Capacity of Demonstration Unit

This process is to be demonstrated using pots of three sizes--six, eight, and twelve inches in diameter, each with an over-all length of 7-1/2 feet and an active pot length of six feet. During most runs, eight-inch-diameter pots will be used; six-inch-diameter pots will be employed only for extremely high activity-level wastes, while the 12-inch-diameter pots will be used to determine the effect of pot diameter on such factors as calcine deposition rate, bridging, and porosity.

The design basis, a maximum pot feed rate of 100 liters per hour, sets in turn the capacity of all auxiliary equipment. While it would be possible with 12-inch diameter pots to exceed this feed rate slightly during a short initial period of operation, the basis selected will not

limit the throughput during any other period of operation. The capacity of a pot for feed tapers off rapidly after the first hour of processing, as solids deposit on the pot walls and create a large resistance to heat transfer into the pot. The minimum of final feed rate at which a batch run is terminated must be established on an economic basis, but it is estimated to be about two liters per hour.

Because preliminary ORNL experience has indicated that the characteristics of the deposited solids are dependent upon the length-to-diameter ratio of a pot and that the over-all average processing rate during the filling period is directly proportional to pot length and independent of pot diameter⁽¹⁴⁾, the process will be demonstrated on the longest pots practical in order to obtain data directly applicable to a production unit. Pots no longer than ten feet are likely to be used in a production plant, because of the difficulty in removing longer pots from the plant and because of cask weight limitations for shipping pots to permanent storage facilities. The length of pots to be used in this pilot plant demonstration will be 7-1/2 feet, a limitation imposed for convenience of handling. The upper 18-inch length of the pot is required for disengaging space and for a baffle to reduce carryover of solids from foaming into the off-gas line. The usable portion, or active length, of the pots in this demonstration will be six feet.

The design flow rates of all main streams in the process are shown in the simplified material balance on the engineering process flowsheet, Figure 1*, and are based on information developed at ORNL.⁽¹⁵⁾

B. Run Procedures

During initial operation of this facility, the procedures developed during cold pilot plant studies at ORNL will be followed. However, other modes of operation may prove desirable in advanced studies, and therefore, sufficient versatility was provided in the design to allow for alternative operating procedures. In current ORNL operation, feed is continuously introduced to a pot, maintaining a constant liquid level, until the feed rate drops to a low value; at this point, feed introduction is stopped and the calcine is cooked at a temperature in the range of 900-980°C. In a variation to this procedure, a second feeding

* All figures referred to in this report are in the Appendix.

and cooking period could be employed for the same pot; additional liquid feed may thermally shock and shatter bridged, porous calcine and make considerable volume available for additional material. In another innovation, a steep temperature gradient across a pot could be employed during processing to melt solids in the lower portion of the pot while calcination and evaporation are carried out in the upper regions of the pot. Still another possibility involves starting a run with a feed concentration period in which high feed rates are employed while operating the calciner at relatively low temperatures, 400-500°C; this could be followed by a period of calcining and melting the solids at a high temperature in the 900-980°C range. The two-step cycle could be repeated as often as desired. These and other promising techniques may be employed to improve certain characteristics of the calcine product, to improve the basic process economics, or to insure a higher degree of safety.

C. Type of Wastes for Demonstration

The pot calcination process is to be demonstrated on several wastes of various activity levels and compositions. These include aluminum nitrate wastes currently in storage at the ICPP, Darex or electrolytic dissolution process wastes yet unavailable, and on high-sulfate Purex wastes currently in storage at Hanford. Median values of the principal constituents (excluding fission products, transuranic elements, and unrecovered uranium) of each of these wastes are given on Figure 1; however, the composition of specific wastes may be considerably different from those shown due to operation of the processing plant on a flowsheet different from that assumed in preparing the table given in Figure 1. This is especially likely for the Purex and Darex processes.

Darex type waste may first become available at the ICPP from operation of an electrolytic dissolution headend process on highly enriched stainless steel alloy fuels. Also, it is possible that Darex (as well as aluminum nitrate) wastes could be synthesized from sulfate-free Purex wastes if they are generated. Aluminum nitrate wastes probably can be prepared also from existing Redox process wastes at Hanford.

D. Hot Cell Description

Remote filling of pots on a batch basis as required by this process involves a considerable number of mechanical operations which

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usually must be observed by an operator. The pot handling system therefore, should be located in a cell fitted with several viewing windows, manipulators, and a crane. A remotely operated dolly is necessary to transport the pot and furnace to and from the calcining station. Manipulative facilities are also required to perform certain critical functions such as remote replacement of the off-gas filters and remote maintenance of the feed pump. A direct maintenance philosophy was employed wherever possible in the design of this facility.

This demonstration facility originally was designed for installation in Cells 1 and 2 of the ICPP Hot Pilot Plant as shown in the cell layouts on Figures 2 and 3. The exterior cell walls are 4-1/2 feet thick with a 2-foot-thick wall of high-density concrete between the two cells. These cells, identical in size, are 10 feet wide, 22 feet long, and 20 feet high. A 6-foot-wide trench, 18 inches lower than the main cell floors, connects the two cells and has tracks for the dolly. Three windows in Cell 2 and two windows in Cell 1 (minimum size, 18 inches square) would permit adequate viewing during remote operation. Cell 2 has a 15-inch-diameter rotary transfer device and a personnel access entrance 2 feet-8 inches wide and 5 feet high. This cell also has master-slave and console-operated heavy-duty manipulators and a three-ton, 18-foot lift trolley-hoist. Cell 1 has an access entrance 6 feet wide and 7-1/2 feet high, master-slave and console-operated heavy-duty manipulators, and a three-ton, 18-foot lift trolley-hoist. The opening between Cells 1 and 2 can be closed by a one-foot-thick steel door. Numerous pipe sleeves in each cell wall can be used for lines to connect the pot calcining facility to utilities located in the operating corridors. Drains in the cells and the hot pilot plant are connected to one of three 500-gallon tanks for interim storage of high-, intermediate-, and low-level wastes. The contents of each of these tanks can be sampled or monitored and then discharged to existing ICPP waste disposal facilities, depending on the activity level of solution in the tanks.

Minor modifications to Cells 1 and 2 were intended for this demonstration because the ICPP Hot Pilot Plant was not designed and built explicitly for the pot calcination process. These included lowering the dolly trench floor about two feet in both cells and replacing the Cell 1

hatch cover with one having a small opening for eventual removal of pots from the pilot plant. Lowering of the floor would have been necessary for complete visual observation of the pot head through existing windows during remote coupling, uncoupling, and capping of pots and for transfer of pots between cells in a vertical position on the dolly. With a modified hatch on Cell 1, decontaminated pots could be pulled directly from the cell into a shielded cask, thereby minimizing any probability of contaminating the cask or pilot plant operation area during this operation.

Although the nature of the existing ICPP facilities influenced the design of the process and certain vessels, it is believed that this design, with only slight changes in the drawings, can be employed in other facilities having the following special features:

1. External shielding equivalent to at least 4-1/2 feet of concrete will be necessary.
2. Viewing windows will be required on at least two adjacent walls to permit observation of the many required operations; the windows in the vicinity of the pot feeding head and the capping stations must be about nine feet above the tracks on which the dolly rides.
3. A cell height of at least 20 feet will be required to retain the use of gravity flow instead of potentially troublesome pumps for various streams.
4. A vertical distance of at least 16 feet between the crane hook and the dolly platform will be required to remove a pot from the furnace, unless a clam-shell furnace is employed.
5. Heavy-duty and light-duty manipulators will be needed, in addition to the crane, to perform the required remote operations.
6. Provisions are required for facility maintenance. Maintenance, either direct or remote, must be provided for in a facility such as this which contains a relatively large amount of mechanical equipment. Isolation of interim storage facilities will be desirable to facilitate any direct maintenance which becomes necessary. While some of

the equipment could and should be repaired remotely, cell entrance for direct maintenance or provisions for complete remote removal of all equipment will be an eventual necessity. All equipment has been designed to permit external and internal decontamination by standard remote techniques employed in direct maintenance plants.

E. Materials of Construction

Complete and effective corrosion protection is difficult if not impossible for equipment in this radioactive demonstration facility which is to process, at high temperatures, a wide variety of solutions. From a corrosion standpoint, an anticipated 20-300 ppm of chloride in Darex waste and as much as 115 ppm of fluoride in some Purex wastes may be particularly troublesome. The best material for each piece of equipment was selected on the basis of the processing conditions and consideration for the order in which wastes are to be tested. Type 304-L stainless steel was selected as the material of construction for most of the equipment, while titanium was selected for the evaporator, the calciner condenser, the four-inch-diameter line between the evaporator and fractionating column, and the fractionating column. These vessels are the ones subject to prolonged contact with process solutions at high temperatures. Factors influencing this selection were:

1. Available corrosion data^(16,17) indicate that stainless steel has a much shorter lifetime expectancy than titanium for aluminum nitrate and stainless steel nitrate wastes,
2. Cr(VI) and Fe(III) in Darex and electrolytic dissolver wastes promote excessive intergranular corrosion⁽¹⁸⁾ of any of the stainless steels,
3. Although certain stainless steels can be made resistant to fluoride corrosion by proper annealing, this is difficult to accomplish completely and satisfactorily on vessels as large as some of those designed for this facility.

Provisions have been made for adding a complexing agent such as aluminum nitrate, boric acid, or zirconyl nitrate to the condenser and fractionating column, if necessary, to keep vessel corrosion from fluorides to tolerable limits. (It is possible that potential corrosion of

stainless steel vessels from chlorides in Darex waste could be minimized by oxidizing the chloride to free chlorine with an air-ozone sparge; however, this is more difficult than complexing fluorides in the Purex wastes and would cause a larger volume of off-gas on runs during the pot calcination demonstration.)

Nozzles on titanium vessels below the normal liquid level in the vessels are to be connected to stainless steel pipe, either with special quick-disconnect couplings or with a tubular, stainless steel-titanium transition joint, commercially available and prepared by tandem extrusion. Ring joint flanges were not selected for this service because even moderate rates of corrosion could result in leaks across their very small contact area.

VI. PROCESS AND EQUIPMENT DESCRIPTION

The four elements of the pot calcination process intended for use in this demonstration are: (1) the calcining operation itself, including connecting the pot to and disconnecting it from the process equipment; (2) feed preparation, including preliminary decontamination of the calciner condensate; (3) decontamination of the noncondensable off-gas for discharge to the atmosphere; and (4) decontamination of liquid effluents for reuse in other plant operations or for disposal to the ground. These four functions are shown as an integrated process on Figure 1 and are discussed along with pertinent design features of the process equipment in the following sections.

A. Basic Pot Calcination System

Considered as part of this system are the pot calciner, a stationary feed head, an induction furnace, a movable dolly, and miscellaneous equipment and viewing facilities. All of this equipment is necessary to fill pots remotely on a batch basis. Figure 4 shows a few of the important details of this system.

At the beginning of a run, an empty pot will be lowered into a water-cooled induction furnace mounted on a movable mechanical dolly. The pot will be centered beneath a stationary feed head and then the pot (HC-2-100*) furnace, and dolly will be mechanically jacked upward until

* Vessel numbers refer to equipment shown on Figure 1 in the Appendix.

the pot can be connected to the feed head by means of a special connector and a special clamp actuated from outside the cell. At this time a gang thermocouple on the pot will be attached to a mating gang lead, and the furnace heat will be turned on.

Liquid waste will be introduced continuously through the feed head into the pot calciner where it will be evaporated and then calcined at temperatures around 900°C. The feed rate to the pot will be controlled by a differential temperature device which employs a rod extending on the pot centerline, 9 to 12 inches below the normal liquid level in the pot, with a thermocouple embedded in the rod 2 to 4 inches above the normal liquid level. The thermocouple will sense a temperature that is a function of the submerged depth of the rod. This scheme has proved satisfactory in ORNL cold pilot studies⁽⁴⁾.

The temperature of the feed line and an additives line can be regulated in the stationary feed head by using constant temperature water or steam in the jacket in the head and on the feed lines; this may prevent plugging which can result from either overcooling or overheating. The separate additives line was provided for admitting process additives to the pot if this becomes desirable in later stages of the development work.

Decomposition gases from the calciner will pass through a mercury trap, HC-2-303, for removal of volatile mercury compounds, if any are present, and then to the calciner condenser, HC-2-301. A seal pot, HC-2-105, was provided in an alternate off-gas line between the pot calciner and the calciner condenser as a safety precaution to prevent overpressuring of the pot should the primary off-gas line between these two units plug.

Thermocouples placed in the center of the pot and one inch from the inside wall of the pot at elevations corresponding to six outer-wall thermocouples will be used to determine the temperature of the solids in the pot. On initial calcination runs, liquid waste will be introduced to the pot until the feed rate drops to under two liters per hour. The solids will then be heated to drive off volatile components, mainly nitrates, until the temperature in all parts of the pot approaches the intended final temperature, about 900°C.

Heat released from fission products near the end of a run may cause auto-calcination and excessive calcine temperatures. For this reason

internal pot temperatures of the calcine must be carefully monitored so that the furnace heat input to the pot can be stopped and cooling begun at the appropriate stage of a run⁽¹³⁾. Excessive temperatures are to be avoided because overheating may cause undesired volatilization of certain fission products or of corrosive sulfur compounds. Overheating may also endanger the structural strength of the pot. On the other hand, failure to reach a sufficiently high temperature in all portions of a pot may result in retention of excessive quantities of volatile material which may create a pressure problem if pots are sealed during storage.

The furnace is designed for individual temperature control in six equally spaced, vertically oriented zones of the pot. This makes it possible to create steep, vertical temperature gradients during specialized runs such as might be employed for forming glass-like products. The heat input to the pot from each zone of the furnace will be controlled, until the final portion of a run, by a temperature signal from a skin thermocouple located at the center of each zone on the pot surface. An induction furnace was selected for use in this facility, instead of a resistance furnace, to achieve instantaneous control of furnace heat input, and to provide for an automatic means of rapid removal of heat from the pot with water-cooled induction coils. Both of these features are expected to be very necessary at the end of a calcination run for removal of fission product heat. Furnace specifications call for a total heat input rate to the pot of 300,000 BTU/hr with a net heat loss of 30,000 BTU/hr from the pot through the insulation to the cooling medium (in the case of the induction furnace, water-cooled coils). If a resistance furnace were employed, depending on the amount of refractory used and the sensible heat therein at operating conditions, as much as 3000 scfm of cooling air may be required for effective temperature control near the end of a run. Obviously, this excessive quantity of cooling air would present undue design problems, as would the use of a liquid coolant. In this latter case the coolant could cause a thermal shock in the resistance furnace or pot calciner. Additional advantages expected to be gained from use of an induction furnace rather than a resistance furnace are a longer life expectancy at the high operating temperatures to be employed in this facility and less susceptibility to damage from abuse during remote insertion and removal of heavy pots.

A small nitrogen purge will be introduced at the bottom of the furnace to curtail pot scaling during a run. Additional emergency cooling can be provided by introducing approximately 100 scfm of air to the furnace through the nitrogen purge lines.

Additional investment cost is the major disadvantage to the use of an induction furnace; preliminary cost estimates indicate that the price of a suitable induction furnace will be between \$40,000 and \$60,000 compared to about \$20,000 for a resistance furnace of equivalent duty.

At the conclusion of a run, the pot will be disconnected remotely from the feed head. During this operation, a jet will draw air through a shroud surrounding the top of the pot to catch any dust that may be dislodged as the pot is lowered. This dust will be collected on a final off-gas filter. The pot and furnace, after being lowered to their original position by means of the mechanical jacks, will be moved by the dolly from under the feed head to a special station for sampling of the calcine and for temporary pot closure.

Following closure, the pot will be removed from the furnace and stored in racks in an adjacent cell for observation of corrosion, internal pressure and temperature, and evidence of products of decomposition. Following interim storage for periods of up to a year, the pots will be fitted with a permanent cover, possibly sealed tightly, decontaminated, pulled through the top of the cell into a cask, and transported to permanent storage. Data obtained during observation of pots in interim storage will be used to determine the desirability of permanently sealing pots and to establish other requirements for design of permanent pot storage facilities. If permanent pot sealing proves to be desirable, remote welding is the currently preferred method.

Mechanical equipment for the remote handling and coupling of pots during this demonstration has been designed at ORNL and is being tested at Dawsonville, Georgia, by Lockheed Nuclear Products. This mechanical equipment includes a pot, a feed head, a movable dolly, a shroud, a remote clamp, several types of pot heads, a capping device, and equipment for remote closures. A complete description of this equipment is beyond the scope of this report. Upon completion of the test program at Dawsonville, the test results were to be examined to determine which

equipment was usable directly; these test results would also serve as a basis for design of any additional mechanical equipment required for this facility.

B. Feed System

1. Process Requirements

Maximum processing rates will be achieved by preconcentrating the calciner feed nearly to the saturation point, thereby imposing on the calciner only the heat duty of final evaporation to dryness and of calcination. Since liquid wastes in storage are far from saturated because of control considerations and partially due to jet dilution during transfer, the use of a feed evaporator was expected to be necessary to achieve a high waste throughput.

The feed evaporator, HC-2-300, in addition to concentrating the raw waste solution, will strip nitric acid from the calciner condensate stream, leaving ruthenium and entrained solids behind for recycle to the calciner. The calciner condensate will consist of water, nitric acid, ruthenium compounds, solids, and perhaps other volatile materials; its composition will depend on the type of waste being calcined, the operating conditions, and any additives used in the calciner feed. In order to avoid volatilization of significant quantities of ruthenium compounds while distilling and concentrating the liquid in the evaporator, it will be necessary to maintain the nitric acid concentration of the boiling liquid below about six molar⁽¹⁹⁾. This will be accomplished by recycling low-activity level water (process water resulting from rectification of the evaporator overhead stream) to the evaporator and stripping nitric acid from the evaporator solution by continuous equilibrium vaporization. The bottoms product from the evaporator will be fed to the pot calciner. The overhead stream from the evaporator will go to the fractionator, HC-2-103, for separation into appropriate streams for recycle or disposal. It is important to note that any fission products vaporized in the evaporator or entrained in the vapor leaving the evaporator must finally end up in a stream intended for disposal. Therefore, a very high decontamination factor must be achieved at this point.

Two methods of evaporator operation have been evolved for this process--close-coupled continuous operation, and batch operation. The

former method of operation employs a low-holdup evaporator to concentrate waste solution and to decontaminate the calciner acid condensate at the same time that the calciner is being fed directly and continuously from the evaporator. Continuous operation in this manner provides the means for recycling the maximum quantity of ruthenium and solids escaping from a pot back to the same pot on the same run. The latter method, batch operation, employs an evaporator having a large holdup to concentrate an entire batch of feed for a succeeding run while decontaminating the calciner condensate from the current run; this requires that all ruthenium and solids escaping from a pot be recycled to a different pot on the succeeding run. Close-coupled continuous operation is preferred so that undesirable interactions between runs might be avoided. However, because of control problems encountered during operation of a close-coupled continuous evaporator at ORNL, special design provisions were employed so that operation by either method could be used if necessary during this radioactive demonstration.

2. Evaporator Design

The design of the evaporator as shown on Figure 5, with the exception of the enlarged vapor-liquid disengaging head, is that of a standard thermosyphon evaporator. This design was selected to obtain high heat transfer rates and a potential low holdup. The 48-inch-diameter vapor head, which will serve only as an oversized gas-liquid disengaging space during continuous operation, will serve as a feed reservoir during batch operation. Cost and cell space limitations indicated the use of this hybrid evaporator rather than the use of two separate evaporators. Design details for the evaporator are given in Table 1*.

The maximum net input rate to the evaporator, 600 liters per hour, includes raw waste, process water, and calciner condensate, and was based on a maximum calciner feed rate of about 100 liters per hour. In addition, 200 to 1100 liters per hour of concentrated calciner feed will be recycled to the evaporator to keep undissolved solids suspended in this stream. (The quantity of recycle used will be dependent on the size of the feed transport pump or on the alternate use of a jet instead of a pump.) The maximum net input rate to the evaporator lasts only for about the first

* Located along with other tables in the Appendix.

hour of each run, after which this rate gradually declines to about 20 liters per hour at run termination. For most of the intended operations, therefore, the evaporator is considerably oversized.

The recirculation ratio of the liquid in the evaporator, determined by standard calculations⁽²⁰⁾, will be about 100 at maximum feed rates during continuous operation; thus, under these conditions, one part of liquid will be vaporized from each 100 parts of liquid circulating during each pass through the tube bundle of the steam chest. The liquid holdup in the tube bundle and downcomer leg of the evaporator during continuous operation at normal liquid level will be about 65 liters.

Process water from the water storage tank, HC-1-101, Figure 6, raw waste from the raw waste storage tank, HC-2-104, Figure 7, and calciner condensate from the calciner condenser, HC-2-301, Figure 8, will be introduced to the evaporator through a common manifold. The feed for the calciner will be removed from an outlet near the top of the downcomer leg to avoid loops in the line (points of possible plugging or excessive contamination) between the evaporator and feed cooler, HC-2-304, Figure 9. Details of the feed cooler and calciner condenser are given in Table 1.

The vapor head is large enough to hold sufficient feed for an entire run with a 12-inch freeboard between the top of the liquid level and the vapor entrance to the impact-type de-entrainment device. This device, detailed on Figure 10, is an ORNL innovation of impingement caps designed by Schlea and Walsh⁽²¹⁾ and is expected to limit entrainment to 1-100 pounds of liquid per million pounds of vapor. The de-entrainment device provided two stages of separation with maximum impact velocities of 35 and 100 feet per second. The concentric drain lines from the bottom of the de-entrainment device extend to a point below the liquid level in the evaporator to effect a seal and prevent vapor by-passing.

The heat loss from the vapor head to the cell was considered in the design because it will cause some condensation and partial fractionation in the evaporator. With a temperature differential of 94°F between the cell and the vapor in the vapor head, there will be a heat loss of about 14,000 BTU/hr. At maximum boil-up rates with 9°F superheat, about 10 pounds per hour of vapor will condense in the uninsulated vapor head. At minimum boil-up rates, approximately 15 pounds per hour of vapor will

condense; even then this condensation will result in a negligible change, about 0.06M, in the exit vapor composition.

The density of the blended solution in the evaporator can be determined by means of density probes in the downcomer leg of the evaporator or from density probes in the concentrated calciner-feed recycle stream to the evaporator. (With no dynamic lag, and with appropriate temperature correction, the solution density at these two points would be identical.) If correct, the measurement in the downcomer leg will be a more accurate indication of evaporator solution density at any instant during continuous operation because of the inherent dynamic lag associated with the other measurement. However, the alternate probes provide a second means of making this important measurement and have been incorporated into the system as a contingency to allow for varied ORNL-ICPP experience. Erroneous density determinations have been obtained occasionally from use of probes on the downcomer leg of an ORNL evaporator. This appears, however, to be a peculiarity of that particular evaporator, since densities determined in the same location on other evaporators at ORNL and at the ICPP have been accurate and satisfactory. A bias in the density reading from the downcomer leg may be experienced (only during batch operation) due to vapor entrainment. Since the solution density can be determined only from the probes in the downcomer leg during batch operation, a slight correction to density values may have to be employed. This factor should not cause any significant problem, however.

Provisions have been made to determine the acidity of the evaporator overhead vapor either by dew point or by electrical conductivity of a totally condensed side stream. Of the two methods, results from use of the conductivity cell will probably be more accurate since acidity determinations by vapor temperature will be dependent on the pressure in the evaporator, a factor which may vary somewhat during a run. The conductivity cell technique will increase the complexity of operation since it requires an auxiliary jet and condenser, equipment not needed for the dew-point technique. Design of the conductivity cell, auxiliary jet, and condenser are pending additional development work at ORNL.

3. Evaporator Operation and Control

During close-coupled continuous operation, the gravity flow of calciner condensate to the evaporator will be unregulated, that of raw

waste solution will be controlled by liquid level in the evaporator, and that of process water will be controlled by the acidity of the evaporator vapor. The heat flux or boil-up rate will be controlled by the density of the blended solution in the evaporator. Control of the continuous evaporator will be complicated by the necessity to concentrate feed solutions within narrow specification limits; since the holdup in a continuous evaporator is low, small changes in the composition or rates of the input streams will cause large changes in the boil-up rate and evaporator liquid composition.

Control of the evaporator during batch operation will be less difficult since the total volume of raw waste required for the succeeding run will be added prior to the start of operation; the effect of small changes in the composition or inlet rates of condensate and process water will be diminished due to the large volume of liquid in the evaporator. During batch operation, as in continuous operation, the flow by gravity of calciner condensate will be unregulated, that of process water will be controlled by the acidity of the overhead vapor, and the heat flux or boil-up rate will be controlled by the density of the blended solution in the evaporator.

Since it has been predicted at ORNL that control of the evaporator during close-coupled continuous operation may prove troublesome, two combination-predictive control systems to supplement that primary control system already outlined have been designed. These systems should be incorporated into the process for trial if the need arises during early process testing; they are as follows:

- a. A combination-predictive control system employing feedforward and feedback principles may markedly improve both operation and stability. In this system, total flow to the evaporator would be computed from individual flow signals and the steam flow would be proportional to this total stream inflow. An output signal based upon the density of the solution in the evaporator would be used to adjust the flow ratio of steam to total feed.
- b. A separate feedforward and feedback system can be used to control accurately the acidity of solution in the evaporator. Changes in the evaporator acid concentration are due largely to changes

in the rate of acid introduction with the calciner condensate. Therefore, the condensate flow rate and acid concentration (as indicated by measurement of its density) may be used to proportion the process water input to the evaporator before a change occurs in the composition of the evaporator overhead to signal for a change in process water addition rate. This predictive control system should improve the uniformity of the acidity of the solution in the evaporator; however, entrained solids could give a false indication of high condensate acidity. Therefore, the acidity of the evaporator overheads, as estimated either by conductivity cell or by dew point, will override the flow ratio by means of a cascade control.

4. Other Equipment in the Feed System

The versatility achieved by providing for both continuous and batch operation of the evaporator requires the installation of much additional piping and of an additional tank, the concentrated feed storage tank, HC-2-101, Figure 11. This tank will be required to store concentrated feed solution during batch operation but is not needed during continuous operation. It is possible that the concentrated feed storage tank may also be used for the admission and mixing of additives to the feed system to control volatilization of fission products or to form glass-like products. (These additives can also be introduced directly to the calciner through the feed head.) The concentrated feed storage tank and the raw waste storage tank each have a capacity of 1200 liters, sufficient to hold the maximum quantity of waste solution that can be converted to a solid and contained in a 12-inch-diameter pot.

Both the concentrated feed storage tank and the water storage tank must be mounted in a horizontal position near the ceiling of the cell to allow sufficient vertical space to permit gravity flow of liquid to the calciner and evaporator, respectively. These tanks have bottom outlets and will be sloped 1/4-inch per foot to effect complete drainage.

Forty-five square feet of the surface of the raw waste storage tank and 30 square feet of the surface of the concentrated feed storage tank will be jacketed to permit the contents of these vessels to be heated. A condenser, HC-2-302, Figure 12, will be mounted on top of the raw waste

storage tank for refluxing purposes should it be necessary to thermally digest raw waste during some runs.

Concentrated feed from the evaporator can be transferred directly to the calciner during continuous operation or to the concentrated feed storage tank during batch operation either by means of a pump or a 200-liter-per-hour steam jet operating on a recycle loop. During batch operation the solution in the concentrated feed storage tank may be fed to the calciner by gravity, by steam jet, or by pump. All feed lines will be jacketed to prevent cooling of feed solutions and possible precipitation of salts during feed transfer. The feed cooler, HC-2-304, will be required when using the steam jet for concentrated feed transfer, since steam jets do not function satisfactorily when the inlet liquid temperature is above 60°C.

Plugging from insoluble material (originating from entrainment in the pot and contained in the calciner condensate fed to the evaporator) will be avoided by maintaining continuous solution flow through the feed lines and recycle loop and, in some cases, by continuously bleeding a small quantity of water into these lines and the pot calciner feed head. The quantity of purge water used either to flush the feed lines or to purge the bearings of the feed transfer pump should be minimized to avoid an excessive increase in the amount of process water that must be decontaminated and disposed to ground or low-level waste. This can be accomplished partially by minimizing the flow rate of feed in the recycle loop. If a canned rotor pump is employed, the amount of water used to purge the bearings will be a function of the pump output rate, probably one or two per cent; on the other hand, if a feed transfer jet is employed, a dilution factor of five per cent of the jet output will be encountered. The jet dilution from a feed recirculation rate of 200 liters per hour results in a 50 per cent increase in process-effluent water per run over that resulting from calcination of a typical raw waste. Obviously, high water purge rates are also to be avoided since gross increases in the quantity of process water are very undesirable.

Gravity flow of feed from the concentrated feed tank to the pot will be tested as an alternate means of feeding the pot during batch operation. Although an attempt to gravity-feed a pot during cold testing at ORNL was

not satisfactory, it is believed that the differently designed feed system of this facility may function satisfactorily. The use of a temperature-regulated 1/2-inch feed line and a water-cooled head on the pot should circumvent the plugging problems experienced in the ORNL tests.

5. Explosive Hazards from Organics in the Feed

Organics entering the process with raw feed will not create a potential explosion hazard in the evaporator, the calciner, or fractionating column, if both the temperature and concentration of organics in this equipment are closely controlled. Although experimental work^(22,23) has shown that a system of excess TBP, nitric acid (TBP/HNO₃ ratio of 0.43) and water can react with violence if heated rapidly to some temperature above 150°C, the same study showed that the organic-saturated aqueous phase of a system of TBP, nitric acid, water, and uranyl-nitrate does not contain sufficient organic material to constitute an explosion hazard. To avoid any explosion hazard in experimental operations of this radioactive demonstration facility; therefore, raw wastes should not be taken from the top or bottom layers of storage tanks but rather from the central portion where a single aqueous solution is known to exist. By employing such a procedure only that organic material (presumably kerosene diluents or butyl phosphates) dissolved in the waste can get into the process. As a further precaution, the maximum operating temperature of all vessels except the pot calciner will be limited to 140°C.

With these restrictions, the pot calciner is the only piece of equipment in the process where a rapid reaction conceivably might occur through the combination of high temperature and high organic concentration; however, it is probable that even there small quantities of organic will steam distill before violent reaction can occur.

Foaming tests performed at ICPP on samples of radioactive aluminum nitrate wastes taken from the midportion of the storage tanks, indicate that these wastes contain insufficient organic to cause foaming during calcination. These tests were performed after it was discovered at ORNL that degradation products of tributyl phosphate, i.e., dibutyl phosphate and monobutyl phosphate, caused synthetic waste solutions to foam during calcination.

Gross quantities of organic could inadvertently get into a pot calciner if one is ever coupled directly to a chemical processing plant.

This could cause an explosion, and is an inherent problem with this process which must be recognized and circumvented by adequate controls.

C. Process Off-Gas Decontamination System

Gases will be evolved from the pot calciner at temperatures varying between 100°C and 900°C, and they will consist of nitric acid, water vapor, nitrogen oxides, nitrogen, oxygen, ruthenium tetroxide, and perhaps volatile mercury compounds; also, solids may be entrained in this stream. The gases first will be introduced to a cooler, HC-2-303, to trap mercury compounds and then to the 148-square-foot calciner condenser, HC-2-301, Figure 8, in which nitric acid and water vapor will be condensed and where partial absorption of nitrogen dioxide will occur.

(Details of this condenser are given in Table I. The mercury trap design was deferred pending additional data from tests underway at ORNL.)

A large percentage of the solids entrained from the calciner will be removed with the condensate from the calciner condenser. This condenser was designed to be an effective absorber of nitrogen dioxide as well as to condense nearly all of the water vapor, in line with the intent to operate the experimental unit with a minimum production of off-gas.

The effluent gas from the calciner condenser will be routed through the thermosyphon evaporator, HC-2-300, to take advantage of any additional decontamination of this stream which may be obtained by scrubbing in the evaporator or by passing through its overhead de-entrainment device. It is possible that some particles escaping the calciner condenser may grow sufficiently large in their passage through the evaporator--by means of condensation on the particulate nuclei--that the particles will impinge on the de-entrainment device.

The off-gas, along with vapors from the evaporator, will be introduced then into the fractionator, HC-2-103, Figures 13 and 14, where the dilute nitric acid vapor will be separated into water and concentrated nitric acid, and where the noncondensable gases will be further decontaminated. Separation of additional particulate matter from the off-gas in the fractionator may occur by particulate attenuation, a mechanism similar to that which occurs in a condenser; during repeated condensation, solid particles can act as nuclei for condensation until they grow in size sufficiently to impinge on the column packing and be collected in the

liquid phase. Perona⁽⁵⁾ has obtained decontamination factors in the range 10^3 - 10^4 in a packed tower for aerosol particles whose volumetric mean diameter was 10 microns. Since the diameter of particulate matter entering this fractionating column will probably be considerably smaller than 10 microns, a much lower decontamination factor is expected.

The overhead stream from the fractionator will be introduced to a 149-square-foot condenser, HC-1-302, Figure 15, where condensation, additional nitrogen dioxide absorption, and further decontamination of the gas stream can occur. The noncondensibles leaving the fractionator condenser will pass through a small heat exchanger, HC-1-301, Figure 16, to vaporize any residual liquid droplets, and finally through one of a pair of small replaceable AEC-type filters to which are attached one cubic foot silica-gel cartridges for adsorption of any residual ruthenium^(24,25,26). Details of the condenser and heat exchanger are given in Table 1. Silica gel will be used during early runs primarily as a safety precaution. It is possible that adequate ruthenium decontamination can be achieved in the process equipment without silica gel; however, this must be established.

Off-gas motivation will be accomplished by means of a steam jet located downstream of the AEC-type filters. The rate of gas recycle to the suction side of the jet will be used to control the negative pressure in the pot, or alternatively that in the evaporator. The steam from the jet will be condensed in the final off-gas condenser, HC-1-303, details of which are given in Table 1. The noncondensibles will be discharged to existing plant facilities.

A blower was considered as the prime mover for maintaining a negative pressure difference of one psig in the pot calciner system; however, it was rejected in favor of a 1/2-inch-diameter steam jet. A steam jet has no moving parts, will be simpler to operate, and the control problems of a jet will be no more difficult than those of a blower.

The quantity of noncondensable gas leaving the process will be accurately determined before discharge, possibly with an integrating flow meter, as will the quantity of instrument and purge airs introduced to the process. The off-gas leaving the process will be primarily air from instrument bubblers in the process vessels. (All air used to air-lift

process solutions will be kept separate from the process gas and most of the instrument air and will be discharged to vessel off-gas facilities.) By material balance, the net quantity of off-gas generated in the pot calcination process can be calculated.

Both the calciner condenser and the fractionator condenser are oversized at least two-fold for the condensing requirements since NO_2 absorption was limiting to the design. Both condensers are the largest size practical for vertical installation for use with other process equipment in the hot pilot plant.

Installation of the calciner condenser so that the tubes are vertically oriented is especially desirable; condensate flowing down the tubes will therefore continuously flush away any solids carried over from the pot calciner. In addition, some increase in wetted area for nitrogen dioxide absorption during low processing rates should be obtained with vertically mounted condensers since the tube walls therein will be more uniformly wetted than would those of horizontally mounted condensers. In addition, provisions have been made to introduce steam to the tube side of the calciner condenser during periods when the processing rate is low to insure wetted tube walls and high absorption of nitrogen dioxide at all times to minimize the net volume of process off-gas.

D. Process Liquid Decontamination System

The vapor leaving the de-entrainment device on top of the thermosyphon evaporator will undergo slight partial condensation on its way to the fractionator because of heat loss to the surroundings; since the condensate might contain additional particulate matter, the line leading from the evaporator to the fractionator will be sloped back towards the evaporator. The vapor from the evaporator will be rectified in the fractionator, HC-2-103, to produce a .02M nitric acid stream (generally referred to herein as process water), and a concentrated nitric acid stream, both of which are expected to be sufficiently free from radioactivity that they may be safely discharged to the ground or reused in a chemical processing plant. The relative quantities of water and concentrated nitric acid resulting from pot calcination will be dependent upon the composition of the waste solution fed to the process; for many wastes, approximately equal quantities of these solutions will be generated.

If any radioactive material enters the fractionator along with the vapor from the evaporator, it will probably accumulate in the bottoms product from the fractionator, the concentrated nitric acid stream. For this reason, the activity level of the concentrated nitric acid effluent might be greater than that of the water, and there will, therefore, be a greater incentive to reuse that stream than the water stream in a chemical processing plant.

The fractionating column was designed to separate 1.0M nitric acid, fed as a vapor, into an essentially water overhead stream consisting of 0.02M nitric acid and a bottoms product consisting of 13M nitric acid. Five theoretical plates are required for the separation, and a reflux ratio of twice the minimum value was selected. A packed column was selected, rather than a bubble-cap plate column, in order to increase particulate attenuation. Pertinent details of the fractionating column and its reboiler are summarized in Table 1 and are shown on Figures 13 and 14.

The 16-inch-diameter fractionating column has ten feet of one-inch Raschig Ring packing and is equipped with two packing support plates and two liquid distributor plates, one of each for each section of packing. In accordance with recommended practice⁽²⁷⁾, the support plate free area exceeds that of the packing; also, the distributor plates have at least five points of feed introduction per square foot of tower cross section. The vapor feed will be introduced to the column at the midpoint.

Flooding calculations based on standard correlations⁽²⁸⁾ indicate that the maximum vapor rate to the column, 1200 pounds per hour, is 90 per cent of the flooding velocity.

Product quality from the fractionating column will be controlled by adjusting both the boil-up rate in the reboiler and the reflux rate. The boil-up rate will be controlled by the density of the acid in the reboiler, while the reflux rate will be controlled by the vapor temperature at the top of the column which regulates the flow of cooling water to the reflux condenser. The vapor temperature must be accurately controlled in about a 2°C-wide band to achieve the desired nitric acid-water separation; however, no difficulty is expected. With this mode of operation, rates of all streams are variable, but product quality is maintained constant.

An alternate method considered for achieving the desired fractionation involved holding both the reflux rate and the volume of liquid in the reboiler constant, and allowing the quality of the product streams to vary slightly. For this mode of control, where liquid level in the reboiler would control the boil-up rate, a constant reflux rate would be employed. Operation can be converted to this method if unexpected problems are incurred.

Concentrated acid accumulates in the reboiler until the end of each run when it will be sampled and, if its activity level permits, jetted to an acid storage tank for reuse or discharged to a low-level waste well; it will be jetted back to the evaporator for additional processing if its activity level exceeds specifications for reuse or disposal.

Vapor from the top of the fractionating column will be condensed in condenser HC-1-302 and transferred to the water storage tank, HC-1-101, by a two-stage air lift. This stream, except that portion recycled by gravity flow to the evaporator, will be accumulated in the water storage tank until run termination when it will be sampled and jetted to disposal facilities. The air from the second stage of the air lift will be separated from the process water in the water storage tank and will be discharged to the existing vessel off-gas facilities. The water storage tank has a capacity of 1200 liters and is not jacketed since the contents of this tank need never be temperature controlled.

The first stage air-lift pot, HC-1-100, Figure 17, is six inches in diameter and eight feet high; it will contain when full, all water flowing from the fractionating column condenser during a five-minute interval at maximum processing rates. The second stage air-lift pot, HC-1-102, will be a smaller vessel six inches in diameter and 12 inches long. (The drawing of HC-1-102 was not completed.) The first stage pot will have a liquid level recorder which will alarm during operation if liquid starts to overflow back into the fractionating column condenser and off-gas line. An overflow line on the second stage air-lift pot normally will not be used; it will be employed, however, if it becomes desirable to control the rate of the inlet stream to the water storage tank.

When calcining aluminum nitrate wastes, which contain significant quantities of mercuric nitrate, an additional effluent stream will be

produced, namely mercuric nitrate solution resulting from removing mercury and mercury oxides collected in the mercury trap. This stream will not be returned to the calciner because mercury cannot be retained in the calcine at the temperature employed in this process. It will be stored, therefore, as a radioactive liquid.

Process vessels will be interconnected so that solutions can be stored in various vessels or reprocessed in the event of maloperation. The acid condensate hold tank, HC-2-102, Figure 18, will be normally empty although it can be used as a storage tank for acid condensate from the calciner condenser, HC-2-301. Acid stored in this vessel can be jetted to the evaporator for further processing, as can concentrated acid in the reboiler of the fractionating column.

The acid condensate hold tank has a capacity of 1200 liters, will be jacketed for temperature control, and will be floor mounted. It will have a reversed dished head to effect complete drainage.

E. Auxiliary Equipment, Sampling and Decontamination Provisions

1. Process Makeup Tanks

The tanks in the process makeup area (PM area) of the Hot Pilot Plant have not been designed in detail. The feed additive tank, the nitric acid tank, and the complexing agent tank were to have a minimum capacity of 400 liters, have mixing facilities, and be jacketed vessels. The decontamination tank, similar in design to the other vessels, was to be a multipurpose vessel large enough to serve all processes that would eventually be housed in the Hot Pilot Plant. All tanks and vessels in the PM area were to be utilized for a variety of purposes in connection with the pot calcination demonstration; therefore, the design of these vessels was to be versatile.

2. Piping

All process lines as shown on the process flowsheet, Figure 1, were sized to permit satisfactory flow rates under all conditions of intended operation. With the exception of the line between the evaporator and fractionator, lines will be sloped in the direction of flow so that undissolved or entrained solids will be carried to the succeeding vessel.

All calciner feed lines will be jacketed; constant temperature water or steam will be used to maintain solution temperatures or to heat plugged

lines. The 1-1/2-inch off-gas line between the pot calciner and mercury trap will be electrically heated to prevent any condensation of mercury and line plugging upstream of the trap. The line connecting the mercury trap to the calciner condenser will be insulated to minimize vapor condensation in the line; any liquid droplets that do form in this line will flow back to the mercury trap and require later disposition. The one-inch off-gas line between the off-gas heater and the AEC filters also will be insulated to prevent vapor condensation and consequent premature plugging of the final off-gas filters.

3. Instrumentation

Recorder controllers will be used on those process streams that must be regulated as shown on the engineering process flowsheet, Figure 1. Signals indicating flow rates, pressures, densities, liquid levels, and temperatures for those streams to be monitored in accordance with the flowsheet will be fed to a data logger. All data will be periodically logged both on a typewriter and on a punched tape. The punched tape can be used with a computer to make material and energy balances for the process, and to perform other routine calculations after computer programs are written.

4. Sampling Provisions

Samples of solution in the raw waste storage tank, the concentrated feed storage tank, the acid condensate hold tank, the reboiler, the water storage tank, and the seal pot may be taken with an air jet at any time during a run. (A steam jet was not selected for this service since the accompanying sample dilution would incorporate an unnecessary variable to analytical results which would have to be eliminated by calculation.) Flowing stream samples of evaporator solution and calciner condensate may be taken. Each sample station (some of which may be common to two or more sample points) will have a sample line and a return line to permit circulation of solution through the sampler and to prevent overflow of radioactive solution in the sampling station.

Gas samples may be taken downstream of the condensers and the AEC-type filters through a sample bomb or filter in a gas sampling station and then discharged back into the process off-gas line upstream of the filters by means of the vacuum created by the main off-gas jet.

A sampling point between the silica-gel cartridge and AEC-type filters is necessary to establish the need for silica-gel cartridges in the off-gas train. Gas sampling lines are to be heated as a precaution against condensation of the small amount of vapor present in the gas. Compositions of those streams having gross quantities of water vapor will be determined best from material balance since condensation in these streams will be difficult to prevent and will cause difficulty in interpreting analytical results. Decontamination factors across each major piece of equipment can be determined either from analytical data or by simple material balances.

Samples of calcine in pots should be taken routinely. Equipment designs and procedures for this have not yet been established.

5. Decontamination Provisions

Whenever possible, spray nozzles were provided in vessels for decontamination purposes. In some instances it may be necessary to flood a vessel or line to decontaminate the equipment properly; remote control valves are to be installed in the drain lines of the vessels to permit this.

All vessels that may contact high activity level solutions will be jacketed or have heating coils so that decontaminating solutions therein can be heated. Also, for this purpose, provisions were made for use of steam on the shell side of the condensers.

Water and steam purges can be introduced to each process vessel through decontamination nozzles. All vessels are to have air or steam sparging facilities.

VII. ESTIMATED COST AND SCHEDULE FOR THE RADIOACTIVE DEMONSTRATION FACILITY

A. Estimated Cost

The estimated investment cost for the radioactive demonstration facility, including required minor cell modifications and purchase of 100 pots for use during the demonstration, is \$1,454,000. A detailed break-down of this cost estimate follows:

ESTIMATED COST OF RADIOACTIVE DEMONSTRATION FACILITY
FOR THE POT CALCINATION PROCESS

<u>EQUIPMENT AND MATERIAL</u>	<u>COST</u>
Instrumentation	\$ 69,500
Piping	27,500
Remote Control Valves	5,500
SS Vessels (fabricated)	39,000
Pots	120,000
Shielded Carrier, Waste (fabricated)	30,000
Shielded Carrier, Pots (fabricated)	34,000
Titanium plate, tubing and flanges	29,000
Titanium dished heads	6,000
Titanium vessels (fabricated)	38,000
Induction Furnace	45,000
Special Equipment	113,000
Supports and Miscellaneous	4,000
Subtotal	\$ 560,500
<u>LABOR</u>	
Installation and Testing	157,000
Minor Cell Modification	56,000
Subtotal	\$ 773,500
<u>INDIRECTS @ 31%</u>	240,000
Subtotal	\$1,013,500
<u>DESIGN @ 15%</u>	150,000
Subtotal	\$1,163,500
<u>CONTINGENCIES @ 25%</u>	290,500
TOTAL	<u>\$1,454,000</u>

B. Schedule Information

Construction and installation of this facility for calcining radioactive wastes was scheduled for completion during November, 1963, with cold testing and spike runs to start immediately thereafter.

A summarized schedule of the proposed development, design, construction and operation of this facility is shown in the bar chart on the next page. Obviously, this schedule for operation with radioactive wastes is dependent upon availability of particular raw wastes and upon satisfactory solution of the inevitable problems encountered during early testing.

SUMMARIZED PROJECT SCHEDULE* FOR RADIOACTIVE DEMONSTRATION OF POT CALCINATION PROCESS AT ICPP

* BASED ON COMPLETION OF PROCESS FLOWSHEET BY MARCH, 1962

At the inception of the ORNL-ICPP cooperative program expeditious demonstration of this process was deemed highly desirable. Therefore, the many facets of development, design, procurement, and installation necessary for the completed facility installation were incorporated in a project planning diagram to permit maximum coordination. This diagram, shown in detail on Figure 19, is based on a network analysis method⁽²⁹⁾. This method, referred to by many as critical-path scheduling (CPS) and, in a modified form, as program evaluation and review techniques (PERT), is finding increasing use in industry. It is believed that this is one of the earliest applications to a coordinated development and construction project.

The intended functions of this project planning diagram were to identify and schedule the many jobs involved, to identify and expedite those jobs whose completion would affect the early completion date of the entire project, and to avoid unnecessary expediting of less-important jobs. On the planning diagram, Figure 19, this entire project has been broken down into 264 steps or jobs, each of which is represented by an

arrow and identified by job title and the estimated job duration in working days. These arrows connect boxes or nodes which represent points in time. The implication of connecting nodes by arrows is that all jobs entering a node must be completed before a job leaving that node may be started. Two key terms "early-start date" and "total-float time" are used in connection with each of the various jobs and nodes. The "early-start date", shown in each node on the planning diagram, is defined as the earliest date on which a succeeding job may start, and it is calculated on the basis that all prior jobs are done in the most expeditious manner. The early-start date is not particularly significant until it is coupled with "total-float time", which is defined as the number of working days by which the start of a job can be delayed beyond the early-start date without delaying the estimated project completion date. Thus, each job should be started on a date somewhere between its early-start date and the date equivalent to the early-start date plus total-float time.

Key jobs which have a total-float time of zero are, by definition, on the "critical path" and must be expedited to the maximum possible extent. Almost as important are jobs with very low total-float times, since these may become critical when estimating errors are corrected. Frequent up-dating of estimates and recalculation of early-start dates and total-float times is necessary for the judicious use of a project planning diagram. At the ICPP, an IBM-650 computer program is used to calculate these factors, and a routine monthly recalculation appears desirable for a project of this nature.

The estimated construction completion date of November, 1963, was based on the project planning diagram; it is believed that this date was realistic and that with appropriate use of this diagram, project completion could have been reached with a minimum expenditure of time and money and with a maximum probability of incorporating all pertinent factors.

VIII. EPILOGUE

Much of the design work and many of the vessel drawings for a radioactive facility to demonstrate the pot calcination process with a variety of high-level radioactive solutions were complete when the Division of Reactor Development (DRD) informed Phillips Petroleum Company that

responsibility for construction and operation of this facility would be transferred to the Hanford Atomic Products Operation. Reasons given for this transfer were:

1. "The cancellation of the aqueous Hot Pilot Plant Demonstration Program has eliminated the primary source of Purex-type waste at the ICPP. DRD feels that shipment of these wastes from Hanford to the ICPP is an unnecessary hazard.
2. Hanford has indicated that they will have suitable hot cell space in the Fuel Recycle Pilot Plant. This facility is expected to be complete by August, 1964.
3. Hanford will be demonstrating their radiant spray calciner in the same facility. Their operating costs will be less than Phillips', since the Pot Calcination Program would be required to support the entire operating costs for the Hot Pilot Plant."

At the time of project transfer on April 30, 1962, as indicated in Table 2, drawings and specifications for the titanium vessels were complete and ready for submission to the fabricators for bids and the drawings of all other major vessels needed only minor revision for completion. The near-finished drawings of the vessels have since been completed for this report; copies of these drawings are in the Appendix along with other project drawings.

The status of other ICPP drawings related to this project is shown in Table 3. Although six of the detailed drawings were at least 70 per cent complete, they were not finished for this report, since some of the design details in these drawings will need to be modified for installation of this facility at Hanford. However, these nearly-finished drawings are included in this report--as Figures 20 through 27--since they indicate the project complexity and serve as a guide to the piping and instrumentation requirements of this facility.

The design of the pot calcination radioactive demonstration facility incorporates the combined ideas and efforts of many ORNL and ICPP persons connected with the project and was completed only after extensive discussion and careful consideration of many ideas and schemes. It is believed that the versatile process and equipment described in this report have the highest possible probability of satisfactorily demonstrating the many facets of an integrated pot calcination plant.

The facility design was initiated on the basis of laboratory and pilot plant work at ORNL, essentially all of which was with nonradioactive simulated wastes. Later, during the course of the design effort, the need for additional definitive paper and experimental studies was indicated; as a result, significant simplification and improvement of the process was effected.

It is believed that several of the equipment choices made as a result of these studies will significantly improve the operability of the radioactive demonstration facility. Included among these are:

1. Use of an induction furnace rather than a resistance furnace will provide the means for adequate control of the calcine temperature and will permit operation with a greater variety of control methods.
2. Selection of a temperature measuring device for sensing liquid level in the pot will eliminate reliance on bubbler probes which are prone to plugging.
3. Temperature control of pot feed solutions by jacketing the lines adjacent to and in the pot feed head should minimize plugging of these lines.
4. The use of a shroud on the pot feed head to minimize dusting during pot changing operations should ease decontamination problems.
5. Employment of a low-holdup evaporator for continuous feed preparation will minimize undesirable interactions between runs.

In addition, the original process was simplified during the design studies. Extensive off-gas holdup and recycle facilities were eliminated, as were other non-essential process components. Equipment was arranged for minimum use of pumps and maximum use of gravity flow for solution transfer. Safety hazards were evaluated throughout the course of the design effort, and possible hazards were circumvented by employing appropriate design features.

A target date for radioactive operation of the pilot plant facility at Hanford has been established as early in 1965. Data from radioactive operation of this facility are expected to provide a basis for design of a production facility employing the pot calcination process.

IX. ACKNOWLEDGMENTS

The authors wish to express their gratitude to Messrs. F. L. Culler, R. E. Blanco, W. E. Clark, M. E. Whatley, J. C. Suddath, C. W. Hancher, and especially to Messrs, J. O. Blomeke and J. M. Holmes, all of the Oak Ridge National Laboratory, for the valuable advice and assistance rendered during the facility design.

X. BIBLIOGRAPHY

- (1) Godbee, H. W., and J. T. Roberts, Laboratory Development of a Pot Calcination Process for Converting Liquid Wastes to Solids, ORNL-2986, September, 1961.
- (2) Culler, F. L., Jr., et al., Annual Progress Report, Chemical Technology Division for Period Ending May 31, 1961, ORNL-3153, September, 1961.
- (3) Hancher, C. W., H. W. Godbee, J. T. Roberts, and J. J. Perona, Trans. Am. Nuclear Soc., 3, 363, December, 1961.
- (4) Hancher, C. W., and J. C. Suddath, Pot Calcination of Simulated Radioactive Waste with Continuous Evaporation, Paper presented at National Meeting of American Institute of Chemical Engineers, Los Angeles, California, February 4-7, 1962.
- (5) Morgan, J. M., Jr., D. K. Jamison, and J. D. Stevenson, Fixation of Radioactivity in Stable Solid Media, Report of the Second Working Meeting at Idaho Falls, Idaho, Sept. 27-29, 1960, TID-7613, Two Books, February, 1961.
- (6) Pearce, D. W., Quarterly Progress Report, Research and Development Activities, Fixation of Radioactive Residues, Oct.-Dec., 1960, HW 68193, January, 1961.
- (7) Pearce, D. W., Quarterly Progress Report, Research and Development Activities, Fixation of Radioactive Residues, Jan.-Mar., 1961, HW 69176, April, 1961.
- (8) Irish, E. R., Quarterly Progress Report, Research and Development Activities, Fixation of Radioactive Residues, April-June, 1961, HW 70517, July, 1961.
- (9) Irish, E. R., Quarterly Progress Report, Research and Development Activities, Fixation of Radioactive Residues, July-Sept., 1961, HW 71371, October, 1961.
- (10) Irish, E. R., Quarterly Progress Report, Research and Development Activities, Fixation of Radioactive Residues, Oct.-Dec., 1961, HW 72324, January, 1961.
- (11) Elliot, M. N., J. R. Grover, W. H. Hardwick, and K. D. B. Johnson, The Disposal of Fission Product Wastes by Incorporation into Glass, AERE-R3610, January, 1961.
- (12) Perona, J. J. ORNL Chemical Technology Division, Private Communication, November, 1961.
- (13) Perona, J. J., The Effects of Internal Heat Generation on Pot Calcination Rates for Radioactive Wastes, ORNL-3163, October, 1961.

- (14) Perona, J. J., R. L. Bradshaw, J. T. Roberts, and J. O. Blomeke, Evaluation of Ultimate Disposal Methods for Liquid and Solid Radio-Active Wastes. Part II. Conversion to Solid by Pot Calcination, ORNL-3192, October, 1961.
- (15) Holmes, J. M., ORNL Chemical Technology Division, Private Communication, October, 1961.
- (16) Cadwell, J. J., HW-30896, March, 1954 (Secret).
- (17) Bower, J. R., Chemical Technology Quarterly Progress Report, January-March, 1961, IDO-14560, August, 1961.
- (18) Bower, J. R., Chemical Technology Quarterly Progress Report, April-June, 1961, IDO-14567, November, 1961.
- (19) Wilson, A. S., Ruthenium Behavior in Nitric Acid Distillation, HW-45620, September, 1956.
- (20) Kern, D. Q., Process Heat Transfer, McGraw-Hill Book Company, 1950.
- (21) Schlea, C. S., and J. P. Walsh, De-entrainment in Evaporators, Preprint No. 38. Presented at National Meeting of American Institute of Chemical Engineers, February 21-24, 1960.
- (22) Colven, T. J., G. M. Nichols, and T. H. Siddall, Interim Technical Report--TNX Evaporator Incident, January 12, 1953, DP-25, May, 1953.
- (23) Nichols, G. S., Decomposition of the Tributyl Phosphate-Nitrate Complexes, DP-526, November, 1960.
- (24) May, C. E., et al., The Absorption of Ruthenium from Nitric Acid-Air Mixtures by Solids Absorbers, IDO-14458, December, 1958.
- (25) Stevenson, C. E., Technical Progress Report, Jan.-March, 1958, Idaho Chemical Processing Plant, IDO-14443, August, 1958.
- (26) Stevenson, C. E., Technical Progress Report, April-June, 1958, Idaho Chemical Processing Plant, IDO-14453, November, 1958.
- (27) Treybal, Robert E., Mass Transfer Operations, McGraw-Hill Book Company, 1955.
- (28) Perry, John H., (Editor), Chemical Engineers Handbook, 3rd Edition, McGraw-Hill Book Company, 1950.
- (29) Kelley, J. E., Jr., and M. R. Walker, Proceedings of Eastern Joint Computer Conference, Boston, Mass., December, 1959.

Table 1

PROCESS DESIGN SPECIFICATION OF POT CALCINATION HEAT EXCHANGE EQUIPMENT

Component	Vessel No.	Figure No.	Maximum Process Stream Throughput (1/hr)	Maximum Heat Duty (BTU/hr)	Heat Transfer Area (ft ²)	Over-all Design Heat Transfer Coefficient (BTU/hr(ft ²)(°F))	Maximum Steam or Water Requirement (lb/hr)	Approximate Shell Diameter (inches)	Tubes		
									No.	Diameter (inches)	Length (inches)
Evaporator Reboiler	HC-2-300	5	600*	1,280,000	98	250	1,380	12	111	3/4	54
Fractionating Column (Reboiler)	HC-2-103	13, 14	53	380,000	33	250	410	**	**	3/4	56
(Partial Condenser)	HC-2-103	13, 14	154	345,000	12	300	6,900	Coils	2	3/4	--
Calciner Furnace	HC-2-100	--		300,000			Induction Furnace				
Calciner Condenser	HC-2-301	8	100	240,000	148	100	4,800	12	111	3/4	82
Fractionating Column Condenser	HC-1-302	15	473	1,110,000	149	150	15,800	12	113	3/4	81
Raw Waste Storage Tank Reflux Condenser	HC-2-302	12	35	75,000	5	250	1,500	4	10	3/4	32
Feed Cooler	HC-2-304	9	200	60,000	32	25	1,200	8	74	1/2	40
Off-Gas Preheater	HC-1-301	16	175 SCFH	2,500	10	2.5	3	6	22	3/4	28
Final Off-Gas Condenser	HC-1-303	--	90***	208,000	33	100	3,000	6	31	3/4	66

* Includes 100 1/hr feed, 100 1/hr, calciner condensate, and 400 1/hr process water.

** Arranged as a 10-inch-diameter bundle of 18 individual U-tubes (3/4-inch-diameter) submerged in a 27-inch-diameter vessel.

*** Condensed jet-motivation steam.

Table 2

VESSEL DESIGN STATUS FOR RADIOACTIVE DEMONSTRATION FACILITY ON DATE OF PROJECT TRANSFER

Vessel	Figure Number	Vessel No.	CPP Drawing No.	Design Calculations	Drawing	Vessel Specifications	Remarks
Fractionating Column and Reboiler Assembly	13	HC-2-103	D-3567	100	100	100	The drawings of these vessels were to be submitted to fabricators for bid on April 30, 1962.
Fractionating Column and Reboiler Details	14	HC-2-103	E-3568	100	100	100	
Thermosyphon Evaporator	5	HC-2-300	E-3563	100	100	100	
De-entrainment Device	10	HC-2-300	D-3564	100	100	100	
Calciner Condenser	3	HC-2-301	D-3570	100	100	100	Drawings of these vessels have been completed for this report since April 30, 1962.
Concentrated Feed Storage Tank	11	HC-2-101	D-3556	100	98	100	
Water Storage Tank	6	HC-1-101	D-3561	100	98	100	
Acid Condensate Hold Tank	18	HC-2-102	D-3562	100	98	100	
Raw Waste Storage Tank	7	HC-2-104	D-3565	100	98	100	
Fractionating Column Condenser	15	HC-1-302	D-3566	100	98	100	
Raw Waste Storage Tank Reflux Condenser	12	HC-2-302	D-3571	100	98	100	
Off-Gas Preheater	16	HC-1-301	D-3572	100	98	100	
Concentrated Feed Cooler	9	HC-2-304	D-3595	100	98	100	
First Stage Air Lift Pot	17	HC-1-100	D-3583	100	98	100	
Second Stage Air Lift Pot		HC-1-102	-----	100	0	100	Drawings of these vessels were not completed for this report.
Seal Pot		HC-2-105	-----	100	25	100	
Final Off-Gas Condenser		HC-1-303	-----	100	25	100	
Mercury Trap		HC-2-303	-----	0	0	0	
Conductivity Cell Condenser		HC-2-305	-----	0	0	0	
Density Pot		HC-2-107	-----	0	0	0	
Process Make-Up Tanks		-----	-----	0	0	0	

Table 3

STATUS OF INSTALLATION, INSTRUMENTATION, AND MISCELLANEOUS DRAWINGS
FOR RADIOACTIVE DEMONSTRATION FACILITY ON DATE OF PROJECT TRANSFER

Title	Figure Number	CPP Drawing No.	Purpose of Drawing	Per Cent Complete
Engineering Process Flowsheet	1	E-3555	Detailed process design	100
Functional Layout Elevation, Cells 1 and 2	2	D-3558	Show orientation of major vessels in cells	100
Functional Layout Plan, Cells 1 and 2	3	D-3557	Show orientation of major vessels in cells	100
Project Planning Diagram	19	F-3028	Coordinate and expedite all phases of project	100
Line Index Drawing, EM Area	20	D-3573	Identify and index all process vessels, piping, and valves in process makeup area	70
Line Index Drawing, Cell No. 2	21	E-3574	Identify and index all process vessels, piping, and valves in Cell No. 2	70
Line Index Drawing, Cell No. 1	22	E-3573	Identify and index all process vessels, piping, and valves in Cell No. 1	70
Utilities Index Flowsheet	23	E-3553	Identify and index all other piping, valves, and utilities for process	70
Instrumentation Schematic, Cell No. 2	24 25 26	E-3577 E-3578 E-3579	Identify and show schematic connection for all process instrument components in Cell No. 2	70
Instrumentation Schematic, Cell No. 1	27	E-3580	Identify and show schematic connection of all process instrument components in Cell No. 1	70
Piping Prints, Cells 1 and 2 and EM Area	--	-----	Show orientation of all piping, valves and vessels for installation	10

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CPP-S-2256

Figure 4.

Pot Calciner and Furnace Details

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FITTING SCHEDULE			
NO.	NO. REQD.	DESCRIPTION	REMARKS
A	1	1/2 SCH. 40 PIPE STUB	STEAM INLET
B	1	1/2 SCH. 40 PIPE STUB	CONDENSATE OUTLET
C	1	1 SCH. 40 PIPE STUB	PROCESS OUTLET
D	1	1 SCH. 40 PIPE STUB	PROCESS INLET
E	1	1/4 SCH. 40 PIPE STUB	DECONTAMINATION

DESIGN NOTES:	
CONSTRUCTION:	FUSION WELDED INERT GAS SHIELDED ARC OF RADIOGRAPH QUALITY.
CODE:	ASME BOILER AND PRESSURE VESSEL CODE, SECTION VIII.
SPECIFICATIONS:	PHILLIPS PETROLEUM SPECS. 11.01, 11.02-3, 11.02-4, 11.03-1, 11.03-3, 11.04, 13.01-2, 13.07, 13.08.
OPER. CONDITIONS:	TUBE @ 125 PSIA @ 220°F WITH SHELL @ 50 PSI @ 290°F.
DESIGN CONDITIONS:	TUBE @ 125 PSIA @ 220°F WITH SHELL @ 50 PSI @ 290°F.
MATERIALS:	TUBES ASTM-A-213 304L S/S. PIPE ASTM-A-312 304L S/S. WELDING ROD ASTM-A-371 ER 308L S/S.
STRESS RELIEF:	NOT REQD.
INSPECTION:	EXTERNAL AND INTERNAL INSPECTION REQUIRED BY BUYER. CODE INSPECTION NOT REQD.
STAMPING:	VESSEL ITEM NO. AS PER SPECIFICATIONS.
NAMEPLATE:	NONE PERMITTED.
PAINT:	NONE
TESTING:	DYE PENETRANT, FREE IRON, RADIOGRAPH, HYDROSTATIC LEAK DETECTION IN THIS ORDER AS PER PHILLIPS DET. CO. SPEC. 13.08.
ESTIMATED CAPACITY:	4 GALLONS
ESTIMATED WEIGHT:	EMPTY - 92 LBS. FULL OF WATER 126 LBS.

GENERAL NOTES:	
1.	THIS DWG. FOR GENERAL ARRANGEMENT ONLY. SEE VENDORS DWG. FOR AS BUILT INFORMATION.
2.	VESSEL SHALL BE SO TESTED THAT EACH PRESSURE CHAMBER RECEIVES HYDROSTATIC TEST WITHOUT PRESSURE IN THE OTHER.
3.	ALL PIPE NOZZLES TO BE BEVELED 3/16" WITH 1/16" LAND.

REF. DRAWINGS:	
CPP-E-3588	PROCESS FLOW SHEET
CPP-E-3573	PROCESS LINE INDEX FLOW SHEET
CPP-E-3580	INSTRUMENT SCHEMATIC
CPP-D-3557	FUNCTIONAL LAYOUT
CPP-D-3538	

ISSUED FOR CONSTRUCTION	
ISSUED FOR CONSTRUCTION	NO.
REVISION	

PHILLIPS PETROLEUM COMPANY	
ATOMIC ENERGY DIVISION	
IDAHO FALLS, IDAHO	
CPP-640	HAP
POT CALCINATION PILOT PLANT	
OFF-GAS PREHEATER	
HC-1-301	
SCALE NOTED	DATE
DESIGNED BY	DATE
DRAWN BY	DATE
CHECKED BY	DATE
APPROVED BY	DATE
DRAWING NO.	REV.
CPP-D-3572	A

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FIGURE 17.

PHILLIPS PETROLEUM COMPANY
ATOMIC ENERGY DIVISION
IDAHO FALLS, IDAHO

CPP-640 **HPP**
POT CALCINATION PILOT PLANT
FIRST STAGE AIRLIFT POT
HC-1-100

SCALE: LISTED **DATE: 4-10-67** **DRAWING NO.:**
DESIGNED: W.K. HALL **4-10-67** **CPP-D-3583**
DRAWN: P.A. SMITH **4-10-67**
CHECKED: W.K. HALL **4-10-67**
APPROVED: MKG **4-14-67**

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REVISION

NO.	REVISION	DATE	BY
1	ISSUED FOR CONSTRUCTION	4-10-67	W.K. HALL

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FIGURE 19.
(LEFT SIDE)

FIGURE 19.
(RIGHT SIDE)

TOLERANCES UNLESS OTHERWISE SPECIFIED FRACTIONS: DEC
BREAK ALL SHARP EDGES UNLESS OTHERWISE SPECIFIED.
FINISH MARKS ARE STANDARD.
DO NOT SCALE THIS DRAWING.

ISSUED FOR DATE	APPROVED DATE	PHILLIPS PETROLEUM COMPANY ATOMIC ENERGY DIVISION IDAHO FALLS, IDAHO
ISSUED FOR APPROVAL		POT CALCINATION PILOT PLANT PROJECT PLANNING DIAGRAM
ISSUED FOR CONTR.		SCALE: 1" = 1' 0" 1/2" DATE: 1-7-63 CHECKED: 1-7-63 APPROVED: 1-7-63
CHECKED REV. BY	REV. APPD DATE	DRAWING NO. CPP-F-3028
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- LEGEND:
- ELECTRICAL LINES
 - INSTRUMENT LINES
 - PROCESS LINES
 - ELECTRIC PLUG DISCONNECT OR SWITCH
 - 50 PSIG INST. AIR
 - 50 PSIG INST. AIR
 - PURGE ASSEMBLY UNIT
 - TRANSMITTER
 - RECORDER
 - MANUAL CONTROL STATION REMOTELY OPERATED EQUIPMENT
 - CONTROLLER
 - THERMAL ELEMENT
 - AMPLIFIER
 - INSTRUMENT ITEM NO.
 - SELF-OPERATED PRESSURE CONTROLLER
 - VISUAL & AUDIBLE ALARM POINT

REFERENCE DWGS:

- CPP-E-3555 POT CALCINATION PILOT PLANT PROCESS FLOWSHEET
- CPP-E-3573 HAP POT CALCINATION PILOT PLANT LINE INDEX FLOWSHEET
- CPP-E-3574 " " " " " " " " " " " "
- CPP-E-3575 " " " " " " " " " " " "
- CPP-E-3576 " " " " " " " " " " " "
- CPP-E-3577 " " " " " " " " " " " "
- CPP-E-3578 " " " " " " " " " " " "
- CPP-E-3579 " " " " " " " " " " " "
- CPP-E-3580 " " " " " " " " " " " "
- CPP-E-3581 " " " " " " " " " " " "

TOLERANCES UNLESS OTHERWISE SPECIFIED FRACTIONS:
BREAK ALL SHARP EDGES UNLESS OTHERWISE SPECIFIED.
FINISH MARKING AS STANDARD.
DO NOT SCALE THIS DRAWING.

FIGURE 26.

PHILLIPS PETROLEUM COMPANY	
ATOMIC ENERGY DIVISION	
IDAHO FALLS, IDAHO	
CPP-640	
POT CALCINATION PILOT PLANT	
INSTRUMENT SCHEMATICS	
CELL #2 PART 3	
SCALE	DATE
DESIGNED	DATE
CHECKED	DATE
APPROVED	DATE

ISSUED FOR APPROVAL	
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REVISION	
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**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION