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June 1966

OPERATION OF THE WASTE CALCINING
FACILITY WITH HIGHLY RADIOACTIVE AQUEOUS WASTE

Report of the First Processing Campaign

R. E. Commander, G. E. Lohse, D. E. Black, E. D. Cooper

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

**PHILLIPS
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ATOMIC ENERGY DIVISION

**NATIONAL REACTOR TESTING STATION
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ABSTRACT

The world's first major breakthrough in treating high-level radioactive aqueous waste has been achieved at the Idaho Chemical Processing Plant where the Waste Calcining Facility converts radioactive waste solutions to solids. From December 1963 to October 1964, about 512,000 gallons of waste containing up to 40 curies of various radionuclides per gallon were calcined in an air-fluidized bed heated to 400°C by means of a liquid-metal heat transfer system. An operating factor of 99.2 percent was achieved during this campaign. Decontamination of the off-gas from the fluidized bed was excellent.

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I. SUMMARY

The fluidized bed calcination process for the conversion of aqueous radioactive wastes to solids was successfully demonstrated in the Waste Calcining Facility (WCF) at the Idaho Chemical Processing Plant (ICPP). From December 1963 to October 1964, over 512,000 gallons of waste containing up to 40 curies per gallon of various radionuclides were converted to solids which occupied 7500 cubic feet of storage. The heart of the process is a four-foot-diameter fluidized bed calciner. Radioactive waste solutions are injected through pneumatic atomizing nozzles into a heated (to 400°C) bed of granular solids which is fluidized with air. The feed for this 312-day operating period originated from the reprocessing of aluminum-uranium alloy fuel, which was dissolved in nitric acid, and contained a full range of aged fission products.

Off-gas which consisted of fluidizing air, atomizing air, water vapor, and nitrogen oxides was successfully decontaminated in a series of devices including a spray quench tower, a venturi scrubber and associated entrainment separators, silica gel adsorbers, and high-efficiency filters before being discharged to the atmosphere.

Significant results of the first processing campaign are summarized as follows:

- (1) The net rate at which the aqueous waste solutions were converted to solids averaged 68.8 gallons per hour. This is nearly 15 percent above the design rate.
- (2) An on-stream time of 99.2 percent was achieved during the 312 days of operation. The feed to the calciner was shut off for only 57 hours -- 51 hours of this resulted from two NaK leaks which occurred from the same fitting located in the nonradioactive portion of the facility. The remaining downtime was caused by power outages.
- (3) The product from the calciner was essentially amorphous alumina which was transported pneumatically to solids storage bins. (The solids transport line is about 120 feet long and has a vertical rise of some 30 feet.) Though numerous bends are present in the system, no transporting problems were encountered during the campaign.
- (4) The estimated bulk density of the solids in storage varied from 53.7 to 60.9 lb/ft³ and averaged 57.1 lb/ft³ overall. The average volume reduction factor for the waste processed was 9.3 (liquid-to-solid volumes as stored).
- (5) The estimated total number of curies in solids storage as of October 1964 was 1.13×10^7 , and the average heat generation rate was 0.52 Btu/hr-lb. The maximum temperature reached with no flow of cooling air over the bins was 225°C at the center line of the bin which contained the hottest waste.
- (6) The off-gas cleanup was so efficient that no hazards resulted from direct discharge of gas to the environment. The average

decontamination factor for strontium-90 was 1.27×10^7 for the entire campaign, while that for ruthenium-106 was 1.9×10^3 .

- (7) The average release rates of strontium-90 and ruthenium-106 were 0.62 and 730 millicuries per day from a 250-foot stack. Monitoring of the air and of the vegetation surrounding the site indicated that the point of maximum integrated concentration occurred at a distance of one mile from the stack. Continuous exposure at this point during the entire campaign would have been 0.1 rad, attributable solely to ruthenium-106 as other radionuclides were not detected.

II. INTRODUCTION

Aqueous methods for nuclear fuel processing generate large quantities of highly radioactive wastes which generally are stored as aqueous solutions and/or slurries in large underground tanks to prevent the release of radionuclides to the surroundings. Although storage of aqueous wastes in this manner has been adequate, the need for safer, cheaper, and more reliable containment has long been evident.

Numerous approaches to achieve better storage by converting the aqueous waste to stable solids have been and are being investigated^[1]. Among these are continuous calcination (in a fluidized bed, in a rotary ball kiln, in a radiant heat spray calciner, and in pots), adsorption of fission product in clays or sponge-like media with subsequent sintering, fixation of the wastes in glass, and burial of the wastes in geological structures. For most wastes, conversion to the solid phase offers the advantages of a large volume reduction, immobility, and potentially a reduction in storage costs.

The most advanced of these schemes is continuous calcination in a fluidized bed. This process was originally conceived at Argonne National Laboratory^[2] and was first applied to simulated radioactive waste by the Technical Branch at the Idaho Chemical Processing Plant^[3]. Development efforts were primarily concentrated on the conversion of waste resulting from aqueous reprocessing of aluminum-uranium reactor fuel in six- and twelve-inch-diameter units, a two-foot-square unit, and a four-foot-diameter plant scale unit. These efforts culminated in December 1963 with the successful operation of the Waste Calcining Facility, the world's first production facility for converting high-level radioactive waste to solids. The conceptual design of the WCF was originated by Phillips Petroleum Company, and the architectural design and construction was completed by Fluor Corporation. The facility is operated for the Atomic Energy Commission by Phillips Petroleum Company.

Early development efforts conducted in the pilot plant units^[4, 5, 6, 7] established best values for use in the WCF for such variables as bed temperature (400°C), superficial fluidizing velocity (0.75 to 1.0 foot per second), and feed atomization (volume ratio of nozzle atomizing air to liquid feed of 500 to 650). During about 3700 hours of operation of the WCF with nonradioactive simulated wastes over a period of nearly three years, many deficiencies in the original design were uncovered. These had to be corrected before acceptable performance was achieved. While these improvements^[8] were being incorporated into the plant, an extensive safety analysis^[9] was undertaken to assure that potential hazards had not been overlooked. A final test run^[10] was then made to confirm the expected adequacy of the modifications, to discover if additional limitations for the ranges of the operating variables were required, and to simulate remote operation of the facility. This final run showed conclusively that radioactive waste could be processed successfully.

The purpose of this report is to describe the process and equipment performance of essentially all components during the first WCF processing campaign with high-level radioactive waste and to describe the shutdown, decontamination, and subsequent inspection of the facility. This campaign was begun on November 30, 1963, using nonradioactive simulated waste. By December 8 equilibrium conditions had been reached, and the first radioactive feed was introduced. The run continued virtually uninterrupted until October 15, 1964,

at which time the solids storage facility was full. The chronology of the hot run is shown in Figure 1. About 512,000 gallons of liquid waste from three

Fig. 1. Chronology of the first WCF processing campaign with radioactive aqueous waste.

separate tanks were converted to solids which occupied 7500 cubic feet of storage during the 312 days of operation. The net feed rate averaged about 15 percent above the design rate of 60 gallons per hour. The volumes in two of the three tanks were essentially depleted. Process performance was excellent, and only 57 hours of downtime occurred throughout the campaign. About 51 hours were lost because of two NaK leaks in a nonradioactive area of the facility. The remaining downtime was caused by power outages. Failure of the bellows of two valves rather early in the run resulted in leakage of solution to the off-gas cell and the calciner cell. However, by altering methods of operation, overall performance of the WCF was not impaired because of the valve failures.

After the termination of the first highly successful processing campaign, most of the facility was decontaminated so that inspection of numerous components, repairs, and minor modifications could be accomplished. This decontamination proved to be much easier than was anticipated, and essentially complete inspection, even of inside surfaces of the vessels, was possible.

III. PROCESS EVALUATION

The primary purpose of the WCF is the conversion of corrosive aqueous radioactive wastes to noncorrosive solids, but any conversion process ultimately generates gaseous by-products which must be treated to remove, then contain, radionuclides which may be present either as particulate matter or volatile species. The WCF, therefore, contains equipment which directly or indirectly supports two functions -- calcination and off-gas cleanup.

1. CALCINATION

The heart of the WCF process is the four-foot-diameter fluidized bed calciner shown in Figure 2. Radioactive waste solutions are injected through pneumatic atomizing nozzles into a heated (to 400°C) bed of granular solids which is fluidized with air. The feed, an aluminum nitrate solution with a full range of aged fission products, is divided equally among three nozzles. The feed flows at a nominal total rate of 92 gallons per hour. This feed rate includes

Fig. 2. WCF calciner vessel and auxiliary equipment.

batch recycle of off-gas scrubbing solution at an equivalent rate of 23 gallons per hour.

Thermal decomposition of the nitrate salt solutions to metallic oxides (primarily alumina), water vapor, and nitrogen oxides is the principal reaction occurring during calcination. Solid products build up in layers on the bed particles while gaseous products and some particulate matter are swept from the vessel with the fluidizing gases. The average bed particle size is controlled at the desired value by controlled attrition of bed particles achieved by varying, as necessary, the volumetric ratio of atomizing air to liquid feed. The bed level is maintained six to eight inches above the feed spray zone at all times (by adjusting the rate of product removal) so that the spray contacts bed particles in a fluidized state.

Heat for the decomposition is supplied to the fluidized bed by an internal heating bundle through which sodium-potassium eutectic alloy (NaK) at temperatures up to 760°C is circulated. The NaK is pumped by an electromagnetic pump and is heated by an oil-fired furnace. Preheated fluidizing air is introduced through 14 orifices equally spaced in an air distributor plate which supports the bed. Each orifice is surmounted by a cap which helps to distribute the air more uniformly in the bed and to minimize backflow of solids if the fluidizing air flow stops. Operation at a superficial fluidizing velocity between 0.75 and 1.0 ft/sec has been satisfactory over the usual mean-bed-size range of 0.4 to 0.7 mm. This size range was chosen as the best compromise between the diametrically opposed considerations of high heat transfer rates and low rates of elutriation of fine solids from the bed.

Solid product is transported from the calciner to the storage bins from two sources by means of a pneumatic transport system. About 60 percent of the product is removed from the bottom of the calciner through the takeoff line connecting the calciner and the product transfer system. Dilution air, added to the takeoff line near the junction with the calciner, regulates the rate of product withdrawal; the product rate varies inversely with the dilution air flow rate. About 40 percent of the product is elutriated from the calciner with the off-gas and is separated from this gas in the calciner cyclone. These solids are transferred by air jet into the solids transport line where they combine with the solids removed from the bottom of the calciner. The product is transported to a second cyclone above the storage bins where it is separated from the transport air. The solids fall by gravity into the bins, and the transport air is returned to the overhead space of the calciner to be decontaminated with the process off-gas. Since no other motive force is normally applied, the driving force for the transfer system is limited by the pressure drop between the product takeoff point and the top of the calciner where the transport gas reenters.

In the subsections to follow, the purpose and operating performance of each of these systems will be discussed. Detailed designs will not be included, as these are presented elsewhere^[9].

1.1 NaK System

The heat required for decomposition of the calciner feed is supplied through an indirect heat-transfer system which is depicted schematically in the WCF process flowsheet shown in Figure 3. The eutectic mixture of sodium and potassium (NaK) is pumped through a three-inch stainless steel

Fig. 3. ICPP Waste Calcining Facility process flow sheet.

pipe to an oil-fired furnace where the temperature is raised to a maximum of 760°C, past expansion tanks which allow for an increase of 25 percent of the cold volume, to the fluidized bed heater where it gives up heat and is cooled approximately 115°C, then back to the pump. The average daily NaK temperatures are shown in Figure 4. Variations were attributable to feed rate or composition

Fig. 4. Temperatures at various points in the WCF process.

changes or to bed temperature changes. The NaK flow is normally kept at a constant rate by manual adjustment of the power input to the electromagnetic pump. The average NaK flow during the campaign was 45,000 pounds per hour. During the run, the system was started up from room temperature three times, two of these following repair of leaks in NaK piping. It operated at a furnace outlet temperature below 600°C for 52 hours and for 7622 hours above 600°C. Since the initial startup of the system, there have been 33 startups, 836 hours of operation below 600°C, and 11,817 hours above 600°C. With the exception of the NaK leaks which were responsible for 51 of the 57 hours of downtime during the run, the system performed smoothly and reliably. NaK temperature responded to adjustments in the furnace firing rate to maintain the calciner bed temperature (also shown in Figure 4) within $\pm 5^\circ\text{C}$ of the setpoint. This variation was due to feed rate fluctuations, to changes in bed heat transfer properties, and to the inertia of the NaK system.

1.2 Fluidizing Air System

Air for fluidizing the calciner bed is pumped from one of two positive-displacement blowers through a metering orifice and control valve to an air coil in the top of the NaK furnace where it is heated to 400°C before being introduced into the calciner vessel. The heated air passes through 14 distributor caps^[11] mounted on the bed support plate. These caps are of a novel design which was developed to greatly reduce erosion of the caps as well as to provide adequate pressure drop across the distributor plate.

Control of the fluidizing air generally was adequate throughout the campaign. Following the test runs^[8], modifications were necessary to convert the system

from indirect to direct control of the air rate. Sensitive pressure instrumentation was installed to control the pressure upstream of an orifice meter by venting excess air to the atmosphere. A throttling valve was installed downstream of the orifice meter for direct control of the fluidizing air rate. Variations in the recorded flow rates normally were within two percent of the desired rate (superficial fluidizing velocity of 1.0 foot per second) and were independent of the variations in feed rate or transport air rate. In all probability the recorded fluctuations were caused by a flutter of the transmitter diaphragm that resulted from pulsations from the blower, and were not actual variations in the fluidizing air rate. The only problem (see Section IV-3.1) with this system was observed when the alternate blower was placed in service, but this was compensated for by manual adjustment of instrument settings immediately before and subsequent to switching the blowers.

The temperature of the fluidizing air was maintained within $\pm 10^{\circ}\text{C}$ of the specified 400°C temperature. Fluctuations were usually cyclic in nature and varied through a period of at least six hours. However, these variations produced no undesirable process effects and no attempts were made to eliminate the variations during the campaign.

The pressure drop across the calciner distributor plate generally varied from 19 to 21.5 inches of water, and that across the fluidized bed varied from 51 to 55.5 inches of water. The observed variations were very gradual and these produced no noticeable process effects. A small percentage of fluidizing air (30 scfm in 225 scfm) was added through the calciner decontamination ring above the distributor plate to improve fluidization near the walls of the calciner vessel.

The temperature profiles in the bed were used to indicate the adequacy of fluidization. Separation between temperature points invariably indicated insufficient fluidization or caking in the bed. Throughout the campaign, even during process fluctuations, temperatures at the bottom, center, and top of the bed were within a 10°C spread. Thus adequate fluidization was maintained.

1.3 Feed System

Waste feed to the calciner was controlled by three remotely operated throttling valves actuated by pneumatic controllers which received signals from electromagnetic flow transmitters installed upstream of the valves. The feed was sprayed into the fluidizing bed through three pneumatic-atomizing spray nozzles. The average gross feed rate achieved during the campaign is plotted in Figure 5. For the first 70 days of the run, the gross feed rate averaged 83.5 gph; thereafter, the rate was increased and averaged 94.7 gph for the remainder of the run. The air rate to the nozzles was adjusted to obtain a volumetric ratio of air to liquid (NAR) of 625 for the first part of the campaign, but after the feed rate was increased, the NAR averaged 580.

The gross feed to the calciner originates from two sources: the first from the liquid waste tank plus dilution from steam jet transfer (jet dilution averaged about five percent); the second from the off-gas scrubbing solution. The solutions are transferred batchwise to the calciner waste hold tanks at the approximate ratio of three waste volumes to one volume of scrubbing solution.

The volumes of waste calcined, obtained from five different liquid storage tanks, are shown in Table I and in Figure 1. Initially, waste from WM-185 tank

Fig. 5. Feed rate and scrubbing solution data.

(five- to seven-year aged) was processed until WM-185 tank was emptied. Then waste from WM-187 tank (three-year aged) was processed until that tank was emptied. About two-thirds of a batch of WM-188 waste was processed before switching to WM-183 (eight-year aged), and approximately 5000 gallons of WM-180 waste was processed about midway through WM-183 processing. Operation with feed from WM-188 and -180 tanks was suspended after process upsets were observed. For WM-188, depletion of the calciner bed occurred due to low aluminum concentration. For WM-180, an increase in the undissolved solids content in the scrubbing solution dictated switching back to WM-183. Approximately 512,000 gallons of radioactive waste were reduced to solids which occupied 7500 cubic feet of storage in 312 days of operation.

The chemical and radionuclide concentrations in each of the liquid storage tanks, the gross and net feed rates, and the volumes processed from each tank are shown in Table I. The heat generation rate which is also shown was calculated from the radionuclide concentrations using the energy estimates for each decay chain.

The compositions of the wastes from the three main tanks were similar. The waste from WM-185 had higher aluminum-ion and lower acid content than did WM-183 and -187. The heat generation rate (0.69 Btu/hr-gal) for WM-187 was due to the high cerium-144 content.

Performance of the feed control system generally was satisfactory. The feed rates were normally maintained within ± 10 percent of the desired value. Variations of this magnitude have no effect on the calciner-performance; the effect, if any, on the off-gas system performance is unknown.

TABLE I
FEED RATES AND COMPOSITIONS OF
RADIOACTIVE WASTES PROCESSED THROUGH THE WCF

	Source of Waste					
	WM-185	WM-187	WM-188	WM-180	WM-183	Overall
Gross operating hours	2,671	2,796	10	74	1,948	7,499
Hours downtime	54.0	2.0	0	0	1.0	57.0
Gross feed rate (gph)	87.2	95.6	---	92.8	93.8	91.7
Net feed rate (gph)	65.1	74.5	---	66.6	66.9	68.8
Net recycle rate (gph)	22.1	21.1	---	26.2	26.9	22.9
Net gallons reduced to solids	170,365	208,245	≈ 600	4,930	129,000	512,540
Chemical composition						
Al(NO ₃) ₃ , <u>M</u>	1.58	1.49	1.08	1.63	1.49	---
HNO ₃ , <u>M</u>	0.60	0.88	0.53	0.08 N ^B	0.91	---
NaNO ₃ , <u>M</u>	0.04	0.06	---	0.01	0.05	---
Hg(NO ₃) ₂ , <u>M</u>	0.013	0.011	---	0.013	0.012	---
H ₃ BO ₃ , <u>M</u>	0.01	0.01	---	0.01	0.01	---
NH ₄ NO ₃ , <u>M</u>	0.06	<0.10	---	1.12	<0.05	---
Radiochemical composition, μ Ci/ml						
Cs-137	1,760	1,040	1,510	740	1,635	---
Sr-90	1,460	940	---	690	1,470	---
Ce-144	570	3,545	255	Nil	260	---
Ru-106	75	350	---	2	25	---
Cs-134	170	86	40	5	45	---
Zr-Nb-95	Nil	84	---	Nil	Nil	---
Pm-147 (estimated)	1,460	2,000	---	350	960	---
Heat generation rate, Btu/hr-gal	0.30	0.69	---	0.11	0.25	0.52 ^[a]

[a] As of August 1964.

1.4 Product

Calcined product from the WCF is granular, largely spheroidal solids and consists of two portions -- that removed directly from the bottom of the fluidized bed and that collected in the primary cyclone. Neither of these streams can be sampled directly during radioactive operation; therefore, absolute rates and analyses are not available. Samples of the fluidized bed were obtained and analyzed daily, and the compositions of the two streams were assumed to be equal to that of the bed. Analyses of bed material resulting from calcination of solutions from WM-185, -187, and -183 wastetanks are presented in Table II, and day-to-day analyses of physical properties are shown in Figure 6. The mass median particle diameter of the solids in the bed generally varied between 0.55 and 0.60 millimeter and averaged near 0.57 throughout the campaign. The fluidized bed density increased from an initial value of 0.65 g/cc to an average value of 0.75 g/cc after three months of operation. The bulk density of the bed material averaged 1.02 g/cc during calcination of WM-185 waste, 1.00 g/cc for WM-187 waste, and 1.10 g/cc for WM-183 waste. The height

TABLE II
CALCINER PRODUCT DATA

	<u>WM-185</u>	<u>WM-187</u>	<u>WM-183</u>
<u>Physical Properties of Bed Material</u>			
Mass median particle diameter (mm)	0.57	0.57	0.57
Bulk density (g/cc)	1.02	1.00	1.10
<u>Total Solids Rate to Storage Bins (lb/hr)</u>	55.2	55.1	54.1
<u>Chemical Composition (weight percent)</u>			
Aluminum as Al ₂ O ₃	89.1	88.2	88.2
Sodium as Na ₂ O	1.3	2.1	2.0
Nitrogen as N ₂ O ₅	4.1	4.0	3.9
Mercury as HgO	2.9	2.8	2.9
H ₂ O	2.0	2.0	2.0
Gross fission product oxides (stable and radioactive)	0.6	0.6	0.6
<u>Radioactive Properties</u>			
Heat generation (Btu/hr-lb)	0.34	0.85	0.28
Principal radionuclides (curies/lb)			
Sr-90	7.2	4.9	6.6
Cs-134	0.3	0.4	0.2
Cs-137	8.0	5.2	7.0
Ce-144	2.4	17.3	1.3
Ru-106	0.1	0.5	0.04
Zr-Nb-95	Nil	0.5	Nil
Pm-147 (estimated)	7.2	10.6	3.3
Total curies in solids storage bins	3.3 x 10 ⁶	5.8 x 10 ⁶	2.2 x 10 ⁶

of the bed was held between 72 and 74 inches (six to eight inches above the feed nozzles) by adjusting the product removal rate.

Approximately 55 lb/hr of solids from the cyclone and fluidized bed were transferred to the storage bins. The estimated total number of curies in solids storage at the end of the campaign was 1.13×10^7 , and the calculated heat generation rate was 0.52 Btu/hr-lb (based on radiochemical analyses as of March 1964 for WM-185 and as of August 1964 for WM-183 and -187). X-ray analysis of the solids from the bed indicated that the product was amorphous. The solids contained 88 to 89 percent alumina, two to three percent mercury, about one to two percent sodium as sodium oxide, four percent nitrogen as N₂O₅.

Fig. 6. Bed and product data.

about two percent water, and less than one percent total fission products. The liquid-to-solid volume reduction factor achieved was about nine to one.

The particle size of the bed solids was controlled by adjusting the nozzle air-to-liquid volume ratio (NAR). A decrease in the NAR caused an increase in the particle size. During startup, when the particle size was small (0.3 mm), both the fluidizing air and the NAR were kept at reduced values to prevent bed breakup by attrition as well as to reduce possible spray drying and elutriation of solids from the bed. As the particle size increased to the normal range (0.5 to 0.6 mm), both the fluidizing rate and the NAR were increased until the fluidizing air rate reached 1.0 ft/sec and the NAR reached about 600. The fluidizing air rate was held constant for the rest of the run, but the NAR was increased or decreased as required to halt upward or downward trends in particle size.

1.5 Product Transport System

As mentioned previously, the solids transferred to storage originate from two sources, that from the calciner cyclone and that from the bottom of the calciner bed. Both product streams combine below the calciner from whence they are transported pneumatically to a cyclone above the solids storage bins. There the bulk of the solids is removed and falls by gravity to the bins. The air, which contains a small amount of very fine dust, is returned to the top of the calciner to be decontaminated with the process off-gas. The motivating force for the solid transport is provided by the calciner vacuum with a maximum limited by the pressure drop across the bed during usual operation. Air from the fluidizing air blower is reduced to below atmospheric pressure by a throttling valve which is actuated from a pressure controller in the transport line downstream of the calciner. This air flows through a heater where its temperature is raised to 100 - 120°C, then past the bottom of the cyclone hopper where solids are transferred to the line by means of an air jet (the pressure in the cyclone is lower than the pressure in the transport line), then below the calciner

where solids from the calciner bed are conveyed into the line by a special product takeoff scheme, then to the solids storage cyclone.

The flow rates, temperatures, and pressures at various points in the transport air system during the campaign are shown in Table III. The total

TABLE III

TRANSPORT AIR SYSTEM DATA

	Flow Rate (scfm)	Temperature (°C)	Pressure
Inlet air	54	50	4.0 psig
Transport air heater outlet	54	100	12-in. water vacuum
Primary air to fines ejector	15	80	40.0 psig
Secondary air and purges	27	30	----
Total transport air to solids storage	96	60 to 77	15- to 19-in. water vacuum
Total transport air from solids storage	96	----	27- to 30-in. water vacuum
Total transport air to calciner	96	68 to 72	42-in. water vacuum

transport air rate averaged 96 scfm and originated from the following sources: the inlet air from the blower averaged 54 scfm; the primary air to the cyclone fines ejector was 15 scfm; secondary air from the cyclone and purge and control air for the product takeoff lines averaged 27 scfm. The vacuum in the transport air line downstream of the bed takeoff was maintained between 15 and 19 inches of water (average of 17) depending upon the operating conditions. The vacuum at solids storage was between 27 and 30 inches of water and that in the calciner vessel was 42 inches of water. Thus, the measured pressure drop in the transport air line was 25 inches of water, which corresponded to the calculated value. The controlled vacuum was normally within ± 1.0 percent of that desired.

The transport air was heated to prevent condensation of vapors drawn from the cyclone by the fines ejector. During the first part of the campaign, the solids storage area was thermally cold and the transport air temperature at the inlet to the calciner vessel was approximately 50°C. After about two months, the heat generated by the stored solids raised the ambient temperature from 0 to 40°C. Thereafter, the temperature in the transport air return was maintained in the range from 68 to 72°C. The variations from the setpoint were about $\pm 1.0^\circ\text{C}$.

Solids were transported to storage at an average rate of 55 pounds per hour. The rate is equivalent to 0.1 pound of solids per pound of air. The mass ratio of solids to air was relatively constant throughout the campaign. Only during

unloading of the bed at shutdown (described in Section IV) was the ratio increased drastically.

2. OFF-GAS CLEANUP

One of the major problems associated with operation of radioactive waste solidification processes is the decontamination of the off-gas from the calcination vessel. Off-gases from such processes contain both particulate and volatile species which are radioactive. In the WCF, the off-gas from the calciner cyclone is decontaminated as it passes through a series of devices which include, in sequence, a spray quench tower, a venturi scrubber and associated entrainment separators, silica gel adsorbers, and high-efficiency filters, before it is discharged to the atmosphere. These systems are shown schematically in Figure 3. The performance of each of these systems is described in sections which follow.

2.1 Humidity Control and System Stoichiometry

A main function of the WCF off-gas is to carry the water content and nonradioactive gaseous species from decomposition of the feed to the atmosphere. Approximately 80 percent of the water in the calciner feed is removed with the off-gas by adjustment of the gas humidity. The remaining 20 percent is condensed and used for scrubbing particulate matter from the off-gas as well as for control of the off-gas temperature. The off-gas leaves the calciner cyclone at a temperature near 300°C and is cooled to 70 to 75°C by scrubbing solution sprayed countercurrently by four pressure-atomizing nozzles as it passes through the quench tower. The gas then enters the venturi scrubber and is cooled an additional 10°C as it contacts scrubbing solution. The average daily temperatures of the gas downstream of the venturi scrubber are shown in Figure 4. Through control of the scrubbing-solution temperature, the rate of condensation (20 percent of that in the off-gas) is established to maintain the concentration of the largely soluble solids in the solution at a level safely below saturation, and the rate of recycle to the feed is set equal to the condensation rate. The solution is recirculated through the scrubbing system and is recycled to the feed tank by a vertical, centrifugal pump. The flow rate of scrubbing solution to the quench tower averaged 230 liters/min (61 gpm) at a temperature of 60 to 65°C. The flow rate to the venturi scrubber averaged 46 liters/min (12.2 gpm) at a temperature of 60 to 65°C. The scrubbing solution was cooled from about 65 to 70°C to 60 to 65°C as it passed through a water-cooled heat exchanger, WC-307.

In order to prevent condensation in the off-gas lines or vessels downstream of the venturi scrubber, the gas is superheated 10°C in a steam-heated shell and tube heat exchanger. This temperature difference was automatically maintained by "feeding" the output of an electronic controller for the temperature upstream of the heat exchanger to the setpoint of the heat exchanger steam controller. The setpoint was biased 10°C above the input signal. The controlled off-gas temperature was normally within $\pm 1^\circ\text{C}$ of the setpoint.

2.11 Methods of Recycle. The pre-run specified feed rate to the calciner included 60 gph of radioactive waste from the liquid storage tanks, plus 20 gph of recycled scrubbing solution. The scrubbing solution was recycled in

about 360-gallon batches approximately every 18 hours. During the first two months of operation, the gross feed rate averaged 83.5 gph which included 20.8 gph of recycled scrubbing solution. Failure of the bellows in one of the scrubbing system valves (see Section V-4.1) prevented normal operation of this system for the remainder of the campaign. Solution leaked continuously at a rate between 25 and 29 gph through piping to the cell floor drain, then to the hot sump tank, WC-119, from which it was transferred through an evaporator, then to the liquid waste tanks before being returned to the calciner. During this time the temperature of the scrubbing solution, and hence the off-gas temperature, was adjusted so that a constant level was maintained in the scrubbing solution tank. The evaporator compensated for the excessive leak rate and for the steam jet dilutions which occurred during transfers. The net recycle rate after evaporation was about 21 gph.

In late August, during operation with waste from WM-180, the leakage from the valve located in the off-gas cell was stopped by accumulation of solids in the bellows vent line. A second valve in the recycle system, but located in the calciner cell, also contained a defective bellows, and solution leaked to the calciner cell, then to the hot sump tank, during recycle of the solution to the feed. For the remainder of the run, the recycle rate averaged 25.6 gph and originated from both the scrubbing solution and hot sump tanks.

2.2 Particulate Solids Removal

2.21 Scrubbing System. Scrubbing of particulate solids from the WCF off-gas is accomplished both in the quench tower and in the venturi scrubber. The extent to which each contributes to off-gas decontamination is not certain because the solutions from each vessel drain to a single scrubbing solution tank. Analyses of liquid samples from this tank and of off-gas samples downstream of the venturi scrubber were used to determine both the dust input and the efficiency of the scrubbing system. Figure 7 shows an overall material balance for alumina and major fission products during operation with WM-185 feed. The gross alumina input during this period was 2.4×10^4 grams per hour (52.6 lb/hr) of which 4.1×10^3 grams per hour (9.0 lb/hr) was recycled from the scrubbing system after having been scrubbed from the off-gas. Only 6.9 grams per hour (0.15 lb/hr) passed through the scrubbing system with the off-gas. Hence, the scrubbing system was 99.85 percent efficient in removing particulate matter from the off-gas. This efficiency was determined from the radio-nuclide balance because of the obvious analytical advantages of radiochemical techniques.

The carry-over rate of alumina to and concentrations of aluminum and nitric acid in the scrubbing solution are shown on Figure 5. The average carry-over rate during the initial operation with WM-185 waste solution was 8.4 lb/hr when the average gross feed rate was 83.5 gph. When the feed rate was increased (average = 94.7 gph), the carry-over rate increased to 10.4 lb/hr. During operation with waste solutions from WM-187 and -183 tanks, both of which had lower aluminum feed concentrations than in WM-185, the alumina carry-over was about one pound per hour higher at the same average gross feed rate. During operation with WM-187 waste solutions, the calciner bed temperature was intentionally lowered from 400 to 375°C to reduce the operating temperature of the NaK heat transfer system. The alumina carry-over rate increased to an average value of 15.3 lb/hr, and the rate of release of ruthenium to the atmosphere also increased (for further discussion see Section III-3.1).

Fig. 7. WCF solids and radionuclide balance during operation with WM-185 waste solution.

The carry-over rate of the alumina is also indicated by the nitric acid concentration in the scrubbing solution. Because the amorphous alumina generated in the calciner is soluble in nitric acid, a higher carry-over rate expends more acid; hence, the acid concentration decreases. This is evident from the decrease in the molarity of the acid, as shown on Figure 5, during operation with WM-187 waste. The aluminum concentration, however, did not change because of a different mode of operation during that interim.

2.22 Adsorbers and Filters. The operating conditions for the off-gas system are shown in Table IV.

The total gas flow through the adsorbers and filters was 1000 to 1100 scfm, depending on the calciner feed rate. The temperature of the off-gas passing through the adsorbers was 66 to 74°C and depended on the method of recycle of scrubbing solution as well as on the feed rate. The pressure drop across the adsorbers, which was initially six inches of water, increased gradually throughout the run to nine inches of water. This was probably due to an accumulation of solids on the silica gel.

After it passed through the adsorbers (three operated in parallel, one on standby), the off-gas was heated in a jacketed pipe exchanger to 82 to 83°C

TABLE IV
OFF-GAS CLEANUP SYSTEM OPERATING CONDITIONS

	WM-185	WM-187	WM-183
<u>Scrubbing System</u>			
Scrubbing solution rate to the quench tower (gpm)	60	61	60
Pressure drop across the quench tower (inches of H ₂ O)	2.9	3	3
Scrubbing solution rate to venturi scrubber (gpm)	12.5	11.5 to 13.0	11.5 to 13.0
Pressure drop across the venturi scrubber (inches of H ₂ O)	50	54 to 68	54 to 68
<u>Ruthenium Adsorption and Filtering</u>			
Total gas flow (scfm)	1005	1100	1000
Dew point of gas (°C)	64	54 to 60	59 to 64
Adsorber operating temperature (°C)	60 to 74	60 to 73	69 to 74
Pressure drop across adsorber (inches of H ₂ O)	6 to 7	7 to 8	8 to 9
Final filter operating temperature (°C)	82	83	83
Pressure drop across the final filters and adjacent piping (inches of H ₂ O)	2.5 to 3.5	3.5 to 6.0	3.4 to 10.0

before entering the filters. The pressure drop across the three parallel filters and their associated piping increased from an initial value of 2.5 to 3.5 inches of water during operation with WM-185 tank feed. It varied from 3.5 to 7.0 inches of water during operation with WM-187 feed, and then varied from about 3.4 to 10 inches of water during operation with WM-183 feed. The pressure drop obviously was dependent upon the solids loadings on the filters and on the integrity of the filter medium. The filters were replaced three times during the campaign, and the pressure drop would increase gradually from a minimum following each change. Further discussion of the filter changeouts is presented in Section V.

Samples of the off-gas upstream and downstream of the adsorbers and downstream of the filters were obtained to determine off-gas loadings and cleanup efficiency in the system. Based on the radiochemical analyses for Cs-137 in the off-gas samples, about 88 percent of the particulate matter (essentially 6.9 grams of alumina/hr) entering the adsorbers during operation with WM-185 waste was collected on the adsorbers. Only 0.8 grams/hr of alumina entered the final filters which then trapped at least 99.7 percent of the input solids. Thus, only 2.3×10^{-3} grams/hr of solids were discharged to the atmosphere.

The performance of various components in the WCF off-gas cleaning train during operation with WM-185 feed is shown in Table V. The decontamination factors are the ratios of the input rates to the effluents rates.

After repeated off-gas sampling confirmed the reliable performance of the off-gas components, operation of the samplers at the various process points was suspended because information gained therefrom was not necessary for plant operation. Performance of off-gas components is not available for operation with feed from WM-187 and -183. Only the off-gas discharged to the atmosphere was sampled routinely. A metered side stream was passed through a Millipore type HA[a] filter to trap particulate solids, and then through a polyethylene

[a] Millipore Filter Corporation, Ashby Rd., Bedford, Massachusetts.

TABLE V

DECONTAMINATION FACTORS FOR PRINCIPAL
RADIONUCLIDES DURING WCF OPERATION WITH WM-185 FEED

	Component Performance ^[a]				Overall Performance of Process, Gross Feed to Stack
	Calciner and Cyclone	Scrubbing System	Adsorbers	Filters	
Cesium-137	6.0	690	9	280	10.0×10^6
Strontium-90	6.5	600	12	160	7.5×10^6
Ruthenium-106	2.5	11	240	[b]	2.8×10^3

[a] Based on contents of feed tank, including recycled scrubbing solution.

[b] Analyses indicated a lower rate of Ru-106 input to the final filters than that going to the stack.

adsorber cartridge to remove gaseous ruthenium compounds. Details of sampler design and operation are presented elsewhere^[1].

The day-to-day release of strontium-90, the most critical particulate radionuclide in the waste, is shown on Figure 8. The average daily release for this isotope was 0.62 millicurie for the entire run. This release rate was

Fig. 8. Strontium-90 and ruthenium-106 activity in the WCF off-gas stream.

about 0.024 percent of that value which would have caused a ground level concentration in the atmosphere equal to that allowable in the Radioactivity Concentration Guide[12] for occupational exposure. A diffusion parameter^[9] of 10^{-4} was used to establish the release limits shown in this figure when the off-gas is discharged from the 250-foot stack under moderately adverse weather conditions. The average decontamination factor (dF) for strontium-90 was 1.27×10^7 for the entire campaign. The dF varied from 2.5×10^6 during initial operation with WM-185 feed to as high as 1.5×10^8 during portions of operation with WM-183 feed.

It is obvious that filter performance will greatly affect the release rate of particulate matter to the atmosphere. In mid-March the originally installed off-gas filters were replaced. The efficiency of the new filters, as determined by DOP testing^[13], was considerably better than that of the original filters. A considerable decrease in the strontium release rate was observed, as is evident in Figure 8. Similarly, when a poor equipment vent filter was installed in mid-July (the equipment vent system ties into the main off-gas line upstream of the sample takeoff), the strontium release rate increased by about a factor of 20. Also during the last month of the run, when the main off-gas filters were replaced with units which contained fibers subject to acid vapor attack, the strontium release rate increased rapidly as the filters deteriorated. These observations point to the necessity of having to install and maintain filters with consistently high efficiency and integrity in the process environment.

2.3 Ruthenium Removal

Removal of ruthenium from the WCF off-gas presents a unique problem because certain compounds of this element are volatilized in the calcination process^[14]. These species then act similarly to water vapor and can be carried with the off-gas to the atmosphere. Thus, special unit operations are required to reduce the ruthenium concentrations to tolerable limits as prescribed in the Radioactivity Concentration Guide. The techniques used to remove ruthenium are described in the subsections which follow.

2.31 Scrubbing System. During operation with WM-185 feed, approximately 2.7×10^5 millicuries per day of ruthenium-106 entered the WCF off-gas system from the calciner cyclone. About 91 percent of that ruthenium was removed in the scrubbing system. The decontamination factor, as shown in Table V, was about 11. The portion of ruthenium in the scrubbing solution averaged about 42.9 percent of that introduced in the feed, regardless of the feed concentration. Hence, it appears that the efficiency of the scrubbing system for removing ruthenium was constant throughout the campaign.

2.32 Adsorbers and Filters. Because off-gas samples from intermediate process locations were taken routinely only during the first two months of operation, the efficiencies of the adsorbers and filters for removing ruthenium are available only for the operating period with WM-185 waste. As indicated by Table V, the adsorbers removed 99.6 percent of the ruthenium (2.41×10^4 millicuries/day) from the entering off-gas (dF = 240).

Though ruthenium was detected qualitatively on the off-gas filters, a precise measure of their efficiency for removing ruthenium from the off-gas stream cannot be made. Off-gas was sampled from two locations: immediately downstream

of the filters and downstream of the junction of the main off-gas and equipment-vent-gas streams. The latter sampler (the off-gas monitor) frequently gave results for ruthenium which were higher than those for the former. Because of the consistency of the results from the off-gas monitor, results from the former, which showed considerable variation, are questionable. Higher ruthenium concentration obtained with the off-gas monitor may have been due to volatilization in the equipment vent system, but because this system could not be sampled separately, this conclusion is uncertain. The overall decontamination factor for ruthenium-106 throughout the campaign was 1.91×10^3 . The dF's during operation with feed from the three waste tanks were as follows:

WM-185 -- 2.8×10^3

WM-187 -- 1.65×10^3

WM-183 -- 460

The reason for the low decontamination factor during operation with WM-183 feed is unknown. There does not appear to be an increasing trend in the day-to-day release rate, as shown on Figure 8, which would indicate saturation of the silica gel. To the contrary, the trend decreased until the last two weeks of the campaign, during which deterioration of the filter material is known to have occurred.

During the 312 days of operation, 215 curies of Ru-106 were discharged to the atmosphere (the average daily release was about 700 millicuries). This average was about 1.4 percent of that allowable in the Radioactivity Concentration Guide. (For further discussion, refer to page 20.) During the next four days, beginning on the day of calciner shutdown, 90 curies were released to the atmosphere. This latter release originated from the silica gel adsorbers and was probably attributable to changes in the temperature and off-gas humidity and/or the nitrogen oxide concentration in the off-gas, all of which resulted after the feed to the calciner was stopped. Two of the four adsorber beds were washed during this period, and this operation undoubtedly caused some of the release, though 55 of the 90 curies were discharged within four hours of the feed stoppage.

2.33 Adsorber Regeneration. One of the adsorber beds was washed during routine operation between October 11 and 14. The release rates of strontium and ruthenium increased, but both rates were within tolerable limits. A rapid increase in pressure drop across the equipment vent filter necessitated change-out of that filter. Previously, operation with a poor equipment vent filter showed a 20-fold increase in the strontium-90 release rate and probably a higher than normal ruthenium-106 release. Hence, increases in both strontium and ruthenium releases during the adsorber regeneration cannot necessarily be attributed solely to the regeneration. Further experience with adsorber regeneration will be required before the effects of such operation on the release rates can be fully assessed.

2.4 Mercury Removal

In the reprocessing of aluminum-uranium alloy nuclear fuel, mercury is added to the nitric acid to catalyze the dissolution of the aluminum. Hence, ionic mercury is present in all aluminum-type raffinates. Because of the low-decomposition temperature of the majority of mercury compounds and because of its high toxicity, mercury can present unique problems during calcination processes for solidifying these aqueous wastes. In the WCF, however,

mercury presented no problem. About 95 percent of the mercury in the feed appeared with the product transferred to storage. The wet scrubbing systems removed about 80 percent of the mercury in the calciner off-gas, and the adsorbers and filter efficiently trapped the remainder.

2.5 Special Off-Gas Sampling

Problems inherent with sampling off-gas streams by filtration or adsorption may arise because only the particles or gases trapped by the system can be analyzed. Consequently, if a species were to "pass through", only those quantities collected by the samples would be accounted for in the final analysis unless special techniques were available to measure the untrapped species. Considerable effort was expended during the first campaign to ascertain if unmeasured species were escaping to the atmosphere. The first of these incorporated a five-stage polyethylene adsorber (in series with a filter) for detecting ruthenium. Three separate tests were made and the following results were obtained:

RUTHENIUM ADSORBED IN EACH 10-GRAM STAGE (Disintegrations per minute)			
	Test 1 [a]	Test 2	Test 3
Stage 1	2.70×10^6	9.95×10^6	1.27×10^7
Stage 2	1.43×10^5	2.32×10^6	3.30×10^6
Stage 3	4.95×10^4	6.25×10^5	8.80×10^5
Stage 4	2.05×10^4	9.10×10^4	1.49×10^5
Stage 5	1.25×10^4	---	2.75×10^4

[a] ASTM Type 1 polyethylene used in Test 1, Type II used in Tests 2 and 3.

These results strongly suggested that all of the ruthenium in the off-gas was accounted for by the existing filtration-adsorption techniques. There still remained, however, the possibility that some species were not being adsorbed. Therefore, gas samples were taken initially in 425-milliliter bottles, then in 22-liter spheres to determine the concentrations of ruthenium and mercury. The gas bottles were counted directly, but high concentrations of ruthenium were not found. Strong caustic was added to the bottles to adsorb ruthenium (and mercury) and the resulting solutions were analyzed. The ruthenium concentration calculated from the analyses gave a concentration approximately two-thirds of that measured by the filtration-adsorption technique. Direct counting of the gas bottle after four caustic washes indicated background radiation. Mercury concentration in the stack off-gas was below the analytical detection limit (one part per million). Cesium-137 and cerium-144 radionuclides were present but their concentrations were too low (due to the much higher ruthenium concentration) to obtain accurate values. These results indicate that the filtration-adsorption technique was adequate for sampling the WCF off-gas.

2.6 Environmental Monitoring of the Off-Gas Discharge

In assessing the hazard from releases of radioactive material into the atmosphere, atmospheric concentrations and subsequent deposition in the immediate environment of the WCF were determined from air and ground samples taken prior to, during, and after the campaign. Complete details of this monitoring are presented elsewhere^[15], and only a summary of the findings will be presented here.

A complete grid was established around the ICPP, extending as far as 16,000 meters downstream in the directions of the prevailing winds, to establish isopleths of the air concentrations and depositions on vegetation. During the first two months of the campaign, high- and low-volume air samples were obtained which indicated that about 12 curies of Ru-106 were released. Calculations based on the high-volume samples indicate an average concentration of about 2×10^{-12} $\mu\text{Ci/cc}$ at 1600 meters, the average distance to maximum concentration downwind. This is about one percent of that allowed by the Radioactivity Concentration Guide for insoluble Ru-106 in the atmosphere for continuous exposure in an uncontrolled area.

For a period of seven weeks beginning on May 12, 1964, high-volume air samples were collected for 24 hours duration once per week. The average concentration of Ru-106 was 8.2×10^{-12} $\mu\text{Ci/cc}$ with a maximum concentration of 1.4×10^{-11} $\mu\text{Ci/cc}$ recorded. This latter value is about ten percent of that set by the Radioactivity Concentration Guide for continuous exposure in an uncontrolled area. In these samples, Sr-90 was looked for but not detected. Fallout plates were used concurrently to determine deposited activity, but measurements were too low to determine deposition velocities.

Finally, deposition velocities (average between 0.4 and 0.5 cm/sec) were determined by measuring the deposited activity on green leafy parts of sagebrush. The resulting measurements indicated detectable activity as far as 16,000 meters in the prevailing wind direction, with a maximum deposition per square meter of surface of $0.22 \mu\text{Ci}$ at 1600 meters. These samples were also analyzed for mercury, but this element could not be detected by the available analytical techniques.

Results of this environmental monitoring indicate that (a) only the oxides with a very low boiling point were released in significant amounts, and (b) there was no significant hazard from operation of the WCF. Calculations indicate that the total dose which would have resulted from continuous exposure at the point of maximum integrated concentration was about 0.1 rad.

3. SPECIAL TESTS

Certain operational changes were instigated during the campaign to improve process performance or to attempt operation with previously untested feed compositions. The results of these tests are discussed in the subsections which follow.

3.1 Operation at a Calcining Temperature of 375°C

About the same time that leaks were detected from ruptured bellows of valves in the off-gas scrubbing system, the calciner feed rate was increased in order to determine the processing rate limitations of the facility. The average gross feed rate to the calciner was increased from 83.5 to 94.7 gph. The heat for the decomposition of the additional feed was supplied by increasing the NaK temperature at the furnace outlet (Figure 4) from 713 to 748°C. Approximately 21,000 Btu/gal are required to maintain the calciner bed temperature at 400°C. After switching to WM-187 feed, which had a low aluminum nitrate concentration, the heat demand decreased and the NaK temperature correspondingly decreased to 730°C. It was postulated that further reduction in NaK temperature could result by decreasing the bed temperature to 375°C. Pilot plant tests^[5] previously had indicated the likelihood of an increase in bulk density (hence, lower volume of stored solids per gallon of liquid processed) at the lower bed temperature.

On June 1, the bed temperature was decreased to 375°C and the NaK temperature at the furnace outlet decreased to about 685°C. After two weeks of operation, the anticipated increase in the bulk density was not observed, but an increase in the alumina carry-over rate to the scrubbing system (from 11.1 to 15.3 lb/hr as shown in Figure 5) as well as an increase in the release rate of Ru-106 to the atmosphere (from 875 to 1500 millicuries/day as shown in Figure 8) was observed. Therefore, the bed temperature was returned to 400°C on June 16 and the process parameters returned essentially to their previously observed values for 400°C operation.

3.2 Bed Level Test for Fines Rate Determination

As stated previously in Subsection 1.4 of this section, product from the WCF consists of two portions; that removed directly from the bottom of the fluidized bed and that removed by the primary cyclone. Because the rates of these streams were unknown, two tests were conducted to obtain approximate values and improve material balance estimates for various radionuclides. The technique consisted of stopping the flow of solids through the product takeoff line from the bottom of the bed, thus allowing the bed height to increase. By assuming the rate of bed buildup to be equal to the rate of solids which would normally be removed via the product lines, the rate of solids removed from the cyclone could be determined from the bed buildup rate, the feed rate, and the scrubbing solution buildup rate. Samples of the feed and scrubbing solutions were obtained and analyzed to determine the equivalent solids content.

Results of the two tests indicated that over the normal bed operating depth of 72.5 to 73.5 inches, approximately 39 lb/hr of alumina were removed via the bed takeoff line and 11 lb/hr were collected by the cyclone. About 11 lb/hr passed through the cyclone to the off-gas scrubbing system.

Material balances for the radionuclides, determined from these results and the assumption that the fines concentrations were "equivalent" to the scrubbing solution concentration, were approximately the same for cesium, cerium, and strontium (86- to 101-percent range). This range agreed closely to that calculated when the fines concentrations were assumed equal to the bed concentration (89- to 98-percent range). For ruthenium, however, the latter method gave a 68 percent material balance; whereas, the results of these tests gave a 104 percent material balance.

3.3 Operation with Dilute Feed

Near the end of the processing of WM-187 waste solution, 870 gallons of liquid from WM-188 tank were transferred to the calciner for processing. This latter tank contained about 26,000 gallons of diluted first-cycle raffinate. Shortly after this feed was introduced, difficulty in maintaining the calciner bed level was experienced. Because a flow sheet for the diluted feed had not previously been worked out, no attempt was made to adjust operating parameters to correct for the decreasing bed level. The difficulties were overcome by blending the remaining dilute solution in the calciner hold tank with solution remaining in WM-187 tank.

3.4 Operation with Waste Solution Containing High Ammonium Ion Content

Approximately 5000 gallons of waste solution from tank WM-180 were processed through the WCF from August 27 through August 30. Operation of the calciner was smooth and no difficulties were encountered throughout this period. However, on August 30 the scrubbing solution became milky due to a buildup of insoluble material in that system. Operation with this feed was suspended because the increase in the insoluble material might have accelerated erosion-corrosion of the piping and because sufficient solution was available in tank WM-183 to complete the first campaign. Further, presence of the insoluble material might have rendered decontamination of the scrubbing solution tank much more difficult. Operation with WM-183 feed was reinstated and the pseudo-equilibrium previously experienced was reestablished within a few days.

Composition of the solution from WM-180 tank is shown in Table I. An exploratory run in the pilot plant calciner, using simulated waste of this composition but without mercury, previously had indicated that no difficulties with this waste would be expected.

Analysis of the undissolved solids from scrubbing solution samples has indicated that the milky appearance was probably due to the formation of a mercury-hydroxyamine complex, the exact formula of which is unknown. The complex is probably a derivative of Millon's Base or of mercuri amido nitrate. This substance may have formed in the scrubbing solution or in the off-gas line between the calciner and the off-gas system. The complex was not detected in the calciner, although large concentrations would have been required to override the "background effect" of the amorphous alumina on the X-ray diffraction pattern. Recent tests in the pilot plant calciner suggest that the formation of the complex can be prevented by proper adjustment of the nozzle-air ratio. Since high ratios cause considerable spray drying, it is probable that ammonia does not remain in the fluidized bed long enough to undergo complete decomposition at the condition employed during the WCF operation with WM-180 feed. Hence, the mercury-hydroxyamine complex can form in the off-gas line or scrubbing system.

IV. SOLIDS STORAGE PERFORMANCE AND EVALUATION

Storage for calcined waste is provided by four units located underground in a concrete vault. Each unit, as shown in Figure 9, is made up of a central cylinder and two progressively larger concentric chambers with an annular space between each chamber. The outermost chamber is enclosed by a carbon steel shell which leaves a 1-1/2-inch annular space between the chamber and the shell. Further details about the design and operation of these units are presented elsewhere^[9].

Fig. 9. Individual solids storage bin.

Solids are conveyed pneumatically from the calciner to a cyclone in which they are separated from the air. They then fall by gravity into the storage bins. A schematic drawing of this system is included in Figure 3. The containers filled progressively from inner to outer chambers, and from unit to unit, until all four were filled. The entire filling operation was automatic and required no motive equipment or maintenance.

1. BED TRANSFER TO STORAGE

Approximately 4150 pounds of solids remained in the calciner after the feed was stopped. The feed nozzles were flushed with water to remove salts which could dry and cause plugs to form. Thereafter, the bed was allowed to cool for about an hour before its transfer to storage was begun. Because the solids loading in the transport air line during bed transfer is much higher than normal, an air jet (WC-520) is required to provide sufficient air flow. The air jet compensated for the increase in kinetic energy required to move the solids and for the decreasing pressure drop as the bed level in the calciner decreased. Initially, the transport air flow rate past the calciner increased from about 55 to 88 scfm, but after the transport of solids was begun, the air rate dropped back to about 55 scfm. The temperature in the transport line downstream of the calciner increased from 75 to 150°C. The vacuum at the solids storage cyclone increased from 20 inches of water to 40 to 45 inches of water about one-half hour after the transfer was started. The pressure drop across the cyclone was relatively constant at about 1.5 inches of water. The transfer of solids to storage was essentially completed in 4-1/2 hours at an average rate of about two pounds of solids per pound of air, though a few additional hours were expended in trying to remove the last traces of solids from the calciner.

2. SOLIDS INVENTORY

The solids storage bins were fabricated with flat heads with the inlet pipes installed in the heads equidistant from the vertical walls. Because the angle of repose of the solids is approximately 34 degrees, void spaces exist at the top of each chamber. The entire volume of the bins was calculated to be 8378 cubic feet, but the usable volume was estimated as 7800 cubic feet. Approximately 170 cubic feet of the usable volume was filled with solids generated from non-radioactive feed during the week of operation before radioactive waste was introduced.

2.1 Estimated Bulk Density of Solids in Storage

Based on the average net feed rates and the equivalent weight of solids in each feed, the weights of solids transferred to storage were calculated for each waste calcined. These data are presented in the following listing:

Source	Volume ^[a] (Gallons)	Solids Weight (Pounds)	Total Volume Occupied by Solids (Cubic Feet)	Estimated Actual Volume (Cubic Feet)	Estimated Bulk Density (lb/cu ft)
WM-185	170,365	144,457	2608	2452	58.9
WM-187	208,245	169,990	3369	3167	53.7
WM-183	129,000	104,318	1823	1714	60.9
Total	507,610	418,318	7800	7332	57.1

[a] Volume from WM-180 tank was omitted.

From the temperature profiles in the bins obtained throughout the campaign, the volume of solids in each chamber was estimated. The estimated void volumes in the chambers were subtracted from the total volume occupied and then the bulk density calculated. The estimated bulk density varied from 53.7 lb/cu ft for WM-187 waste to 60.9 lb/cu ft for WM-183 waste, and averaged 57.1 lb/cu ft for the entire run. The volume reduction factor for the entire campaign averaged about 9.3.

3. HEAT GENERATION IN AND REMOVAL FROM THE BINS

The heat generation rates of wastes calcined during the run are shown in Table II (page 12). These ranged from 0.28 to 0.85 Btu/hr for each pound of solids in storage. At the end of the campaign the total heat generation rate was 222,800 Btu/hr, of which 65 percent was due to calcined solids from WM-187 feed solution. The temperatures at the center line of the various chambers obviously were dependent upon the heat generation rate of the solids in the specific chamber. Temperature profiles at various elevations in the middle chamber of storage bin WC-115-2 are shown in Figure 10. These profiles are typical of all the chambers, except for the magnitudes of the temperatures and the times at which the elevations became covered with solids. The bottom half of this chamber contains solids from "WM-185" operation, and the top half contains solids from "WM-187" operation. The temperatures during November 1964 (about 225°C at 15.5-foot elevation) were the highest observed during and following the campaign.

3.1 Removal of Heat from the Bins

The solids storage bins were designed to accommodate solids with a heat liberation level of 2.5 Btu/hr per pound of solids. Forced convection by means of a cooling air blower was considered necessary to prevent the solids temperature from exceeding 400°C. Because the heat generated per pound of solids averaged only 0.52 Btu/hr, operation of the blower was found to be unnecessary. The bins were cooled by natural convection of fresh air as the air rose past the bin surfaces. With cooling air flowing, the maximum temperatures reached

Fig. 10. Center line temperatures of solids at various elevations in the middle chamber of WC-115-2.

in the chambers were about 185°C. By September 1964, it became apparent that a maximum temperature had been attained and that declining temperatures (see Figure 10) were following the decrease in the ambient atmospheric temperature resulting from the seasonal changes.

In mid-September, air flow rates and temperature differentials through the cooling air system were measured for determining the amount of convective heat transfer to the fresh cooling air. From these data, conservative estimates indicated that only 34 percent of the heat generated was removed by the cooling air stream. Cooling air valves were then closed to obtain the equilibrium temperature with no inflow of cooling air. The maximum temperatures approached 225°C after two months, then decreased with the decay of the radionuclides. After an additional 3-1/2 months, the cooling air inlet valves were re-opened (to prevent high-temperature deterioration of the concrete), and the temperatures reached a new pseudo-equilibrium within one month, the maximum temperatures being 145 to 150°C. Outside concrete wall temperatures at the soil-concrete interface midway up the wall surface reached a pseudo-equilibrium value of 45 to 50°C. Though a rigorous mathematical solution is not yet possible, it is apparent from the above measurements that considerable heat is being transferred (perhaps by a combination of radiation, convection, and conduction) to the soil surrounding the solids storage vault.

V. UTILITIES AND EQUIPMENT PERFORMANCE

1. UTILITIES

Air, water, and steam are supplied to the Waste Calcining Facility by underground lines, and each originates from the Service Building, CPP-606. Fuel oil for the NaK furnace is supplied by an independent system which supplies the calciner only. The fuel oil supply and return lines are also underground and the circulation pumps are in a pump house west of CPP-633. Service waste (condensate and cooling water) leaves the calciner building in an underground line and eventually joins the main CPP service waste header. Power is supplied by the CPP electrical distribution network, and emergency power is supplied by a diesel-generator set at the Service Building. Details of the utility systems are available elsewhere[9].

1.1 Air

Air is supplied to the WCF at 90 to 100 psig, but part of it is reduced in pressure to provide air for the 50- and 20-psig systems. The air flow to the WCF is metered, and the air consumption during the run averaged about 800 scfm with peak rates as high as 1200 scfm. Generally the header pressure ranged from 88 to 92 psig, but on a few occasions it dropped as low as 80 psig. Except for one occasion, the supply of air was adequate, but at times the moisture content of the air was much too high. Whenever overall usage of air at the WCF was less than 1000 scfm, the air was reasonably dry. However, at higher air rates, the capacity of the air dryers was exceeded, and in order to maintain pressure, part of the air had to be bypassed around the dryers. As a result, the dewpoint of the air at such times was much higher than is desirable.

A complete loss of air took place on December 2, 1963, while the WCF was still operating on cold feed. During a routine load test of the emergency generator, the air compressor was placed on emergency power to provide the load needed for the test. When the emergency generator was shut down, automatic switching to normal power did not activate. The air pressure gradually dropped to zero and was off for about 15 minutes. Although there was no loss of electrical power at the WCF, shutdown of the process was necessary because there was no air for instrumentation and valves. When air pressure was restored and instruments were functioning again, the off-gas and fluidizing air blowers were restarted, and within a short time, the process lined out and the feed was restarted. The calciner bed refluidized without any difficulty, and there was virtually no plugging of air-purged instrument lines. The nonradioactive feed was off for only about one hour and fifteen minutes. There were no stoppages due to air failure for the remainder of the campaign.

1.2 Steam

There was an ample supply of steam all during the WCF run, and the supply pressure never dropped below 127 psig. Most of the process steam at the WCF is taken at the header pressure and throttled manually to the desired use pressure. However, steam to control valves for four gas superheaters, WC-305, -352, -354, and -356, and the heating and ventilating system was supplied through pressure reducers at about 35 to 55 psig. Steam consumption

at the WCF is rather low. Consumption averaged 3700 lb/hr during the three coldest months (January, February, and March) and 1700 lb/hr during the three warmest months (July, August, September) when none was used for building heat. Many of the lines and valves which supply steam to transfer jets and heating coils are oversized. The oversized valves made it difficult to control steam pressure for applications which require relatively small amounts of steam. This was a minor inconvenience, however, and the overall efficiency of the WCF operation was not affected.

1.3 Water

Water for the WCF is supplied by the firewater system and is not treated or softened. As a result, hard water deposits tend to foul surfaces where heating or evaporation takes place. The equipment items most affected were the hot water heater, the spray coil in the heating and ventilating system, and water-purged instrument tubing, fittings, and purge rotameters. During the run, fouling of some of the water-purged instrument lines and fittings became so bad that complete replacement of the hardware was necessary. Investigation of the problem revealed that the deposits were not hard water salts as such, but reaction products of the water with aluminum tubing and fittings. Copper has been substituted for aluminum and should greatly reduce the fouling in these instrument lines. Eventually it would appear wise to supply the WCF with treated water if it can be done without excessive cost.

Water requirements for operation of the WCF are not very high (about 100 gpm). The main process uses are for cooling the off-gas blower bearings, for the scrub system cooler, WC-307, and for the demister, WC-130. All other uses do not add significantly to the water requirements at the WCF.

1.4 Electrical

Power failures or dips were the most frequent cause of upsets to calcining operation. Although the number of these upsets was quite high, the cumulative amount of downtime (that is with the feed shut off) was less than seven hours in 312 days. Except for loss of normal power, which was invariably caused by forces external to the WCF, the electrical system proved very reliable.

In most instances, a power dip or power failure was merely an inconvenience. Neither the NaK furnace nor the NaK pump is supplied with emergency power; thus, a loss of power for more than a few minutes requires that the feed be shut off. On March 12, however, a series of power failures occurred which finally culminated in a complete loss of power because the emergency generator failed to start. When this occurred, the WCF building was evacuated. Fortunately, normal power was restored in about seven minutes. When the calciner building was reentered, it was found that the process was already starting to recover from the extreme upset (switches were left in operating position) and that no lasting damage had resulted. The ability of the WCF to weather such things as complete loss of air or power (even though only for short times) proved to be one of the most pleasant surprises of the entire run.

The loss of all power pointed up a weakness of the emergency generator system. Compressed air is used to crank a diesel engine which drives the generator. The reservoir of air is sufficient for only a few successive start attempts before it is exhausted. On March 12, high winds caused certain street

lighting circuit power lines to blow together. Each time this happened a power failure resulted. The emergency generator responded to the first two failures, but on the third, the starting air pressure was not adequate, and the generator did not start. Three things were done in an attempt to prevent future instances of complete power loss.

- (1) When high winds came up, the fuses were pulled on the street lighting circuits. This was a temporary measure until the deficiency could be corrected.
- (2) When conditions were such that a series of power dips or failures might be expected (such as electrical storms), the emergency generator was started and operated on standby at no load until the danger was over.
- (3) The reservoir of air for starting the emergency generator was increased in capacity.

Although power dips and failures were not uncommon in the remaining part of the run, there were no other instances of complete power loss.

1.5 Fuel Oil

The NaK furnace is fired with No. 2 fuel oil, which is pumped to the furnace room from two outside storage tanks west of the calciner building. The oil is circulated through a closed loop, and most of it returns to the storage tanks through a pressure regulator valve set for about 30 psig. The fuel oil system operated very well during the calciner run. On December 9, shortly after the start of hot operation, the pressure regulator failed, and hand control with the bypass was required while the regulator was removed, repaired, and reinstalled. One of the fuel oil pumps became a little rough during the run, and operation was switched to the spare. Oil was consumed at an average rate of 0.44 gpm throughout the run, and a total of 201,000 gallons was used. Assuming a normal heating value for No. 2 fuel oil, the heat release from the oil averaged about 3.8 mm Btu/hr.

2. MECHANICAL EQUIPMENT

In the cold runs which preceded hot operation of the WCF, mechanical equipment breakdowns constituted one of the principal problems. To overcome these problems, major design changes were required for the off-gas blowers and the scrub system pumps, and the transport air blowers were eliminated altogether. Largely due to the improvements developed from the series of cold runs^[8], mechanical equipment performed extremely well during the hot run.

2.1 Fluidizing Air Blowers

Both fluidizing air blowers were overhauled during the final cold run, and there was no major mechanical trouble with either blower during the "hot" run. Blower WC-250A operated from the start of the run until January 14, 1964. Operation was then switched to WC-250B to permit a routine maintenance check of the A blower. WC-250B operated until March 2, when a slight oil leak developed. WC-250A was placed in service, and a faulty gasket was replaced

on WC-250B. For the remainder of the run, the blowers were switched about every 30 to 40 days to permit oil level checks which could not be made on an operating blower.

The first time the fluidizing air blowers were switched, a pulsation cycle was apparently set up which resulted in check valve hammering on the operating blower. This was eliminated by switching back and forth several times. It was discovered that an instantaneous switchover would not cause this problem, while a delayed switch frequently would. Subsequent switches were made smoothly and no check valve hammering occurred.

2.2 Main Off-Gas Blowers

The main off-gas blowers, WC-251A and B, were plagued by bearing failures during the early cold runs. The failures were due mainly to inadequate lubrication and to misalignment problems. These problems were solved by modifications prior to the final cold run.

2.21 Hot Run Performance. Blower WC-251A operated from start to finish of the hot run. Although there were occasions when one or more of the bearings showed a temperature rise, these were arrested before they became serious. Ambient temperature changes in the off-gas blower cell had a direct effect on bearing temperatures, and it is doubtful if the bearing temperatures could be kept low enough without adequate air flow through the off-gas blower cell. Several times during the run, the cell exhaust blower (WC-G-3302) had to be shut down for short periods. Each time, the blower cell temperature began to increase rapidly, and the blower bearing temperatures followed along. Early in the run, the cell temperature was about 128°F. By early March it had increased to 132°F with a corresponding increase in bearing temperatures, and in the middle of May the cell temperature reached 139°F. Bearing temperatures were getting close to the alarm point, and to lower the cell temperature, the air flow through the cell was increased by enlarging the louver area on the door. The cell temperature dropped to 135°F, and the bearing temperatures also dropped accordingly.

2.22 Lubrication. As originally installed, the blowers were grease lubricated. However, because of uncertain reliability of grease at the operating temperature and speed of the blowers (3600 rpm), a remote oil lubrication system was installed before the final cold run, which provided for circulation of the oil through the bearings and back to a reservoir. This system worked extremely well during the hot run. Oil flow was regulated independently to each blower bearing from a common gravity-feed tank. The oil flow to each bearing was checked regularly by the shift mechanic, while the feed tank level was maintained by plant operators.

2.23 Alignment. The alignment problems with the off-gas blowers were largely due to the effects of thermal expansion. Rather elaborate provisions for piping and blower expansion were required before satisfactory alignment could be maintained throughout the temperature range from startup to constant operation. After the hot run was over and decontamination of the WCF was completed, measurements indicated a radial misalignment of 17 mils between the motor and blower. Axial alignment of the blower sections and the motor remained "true". Both off-gas blowers were realigned even though maximum allowable deviations had not been exceeded.

2.3 Equipment Vent Blower

Blower WC-252, which maintains the vacuum on the equipment vent system, performed reliably through the cold phase of WCF operation. Before the start of hot operation, however, a connection was made between the suction of the equipment vent blower and the suction of the main off-gas blowers. A manual valve in the connecting line would normally be closed, thus isolating the two systems from each other, but in the event of failure of WC-252, the valve could be opened to provide vacuum for the equipment vent system. After seven months of hot operation, during which WC-252 ran satisfactorily, a bad motor bearing developed. The cross connection valve was opened and WC-252 was shut down. The bad motor bearing was replaced a few days later and WC-252 was returned to service. The blower performed satisfactorily for the remainder of the hot run and until decontamination of the facility was essentially complete. Later, all new bearings (for both motor and blower) were installed to put WC-252 in good condition for the next run.

2.4 Scrubbing System Pumps

Scrub pump WC-208A ran from November 30, at startup of the WCF on cold feed, until December 2, when loss of instrument air (see Section V-1.1) caused an automatic switch to WC-208B. The switch was not noticed until sometime after air pressure had returned, operations had been resumed, and routine data were being taken. Rather than switch back to WC-208A, the selector switch was changed to WC-208B, and it operated for all but a few minutes of the entire run.

The scrub pumps are located in an inaccessible area, and about the only indicators of pump condition are bearing temperatures, discharge pressure, and the current drawn by the motor. By all of these indications, WC-208B was still performing satisfactorily at the end of the run.

The remote lubrication system for the pump bearings worked satisfactorily, and bearing temperatures stayed below the danger level at all times. There was some leakage of oil from bearings onto the pump mounting flange, but the amount was slight. There was very little change in the discharge pressure of WC-208B during the run, and the current drawn by the motor also was quite steady.

After the run was over, the scrub pumps were used alternately during decontamination of the off-gas cell. After decontamination, both pumps developed operating troubles during maintenance checks. Both were pulled for inspection, and it was discovered that the impeller nut had dropped off the shaft of each pump (see Section VI-3.3).

After WC-208B pump had been pulled from the scrub tank and decontaminated further, it was inspected for wear. Comparison of dimensions taken before and after the run showed that very little wear had been experienced on the impeller or the impeller housing.

2.5 Miscellaneous Equipment

The main ventilation blower, WC G-3301, and the cell exhaust blower, WC G-3302, performed virtually continuously during the WCF run. Their importance was clearly shown by the immediate effect on the off-gas blower

bearing temperatures whenever air flow through the off-gas blower cell stopped. It is certain that the bearing temperatures would have exceeded the maximum recommended operating temperature if air circulation through the cell had stopped for more than a short time. Fortunately, this never happened.

The cell exhaust blower began running a little rough early in July, but careful attention to lubrication and belt tension kept the blower running reasonably well until the run was over. On October 21, shortly after decontamination of the WCF was started, the blower had to be removed and completely overhauled. It was reinstalled October 23. A spare blower has been obtained. The main ventilation blower gave no trouble during the hot run.

The NaK furnace blower ran continuously during the WCF run except for the two occasions when the NaK loop was drained to repair leaks. The blower was inspected after the run and was found to be still in excellent condition. The furnace burner and the interior of the furnace were also inspected. Other than cleaning, the burner required no work; the refractory lining on the inside of the furnace was in excellent condition.

3. INSTRUMENTATION

The WCF performed very smoothly most of the time, and a minimum of operator surveillance was required. This would not have been possible without good instrumentation. Control of process conditions was extremely good for the most part, and recovery from severe upsets was usually fairly rapid. The problems which were experienced with instruments were mostly minor and were usually handled easily by a shift instrument man.

3.1 Measurement of Liquid Level, Density, Pressure, and Flow Rate

Most liquid levels, pressures, fluid densities, and flow rates at the WCF are measured by differential pressure transmitters, and transmitter signals are converted to readings on indicators, recorders, or recorder controllers. The hardware for this type of instrumentation held up quite well, although some makes proved more rugged and reliable than others. The most frequent cause of error in measurement of liquid level, density, pressure, or flow rate was restricted or plugged purge lines rather than faulty instrument components. The presence of air in liquid purged systems was another cause of error. These problems occurred most frequently in the scrubbing system instrument lines and the adsorber liquid level lines. Two particularly chronic offenders, however, were FR WC-251AB-1 (total off-gas flow to the stack) and the common line of PI WC-105-1 and LIA WC-105-1 (feed tank pressure and level instruments). In the first case the trouble was caused by freezing of moisture in the air supply line to the transmitter and purge meters, while in the other it was crystallization of solids from feed entrained by air from the air lifts. Perhaps the trouble with the feed tank instrument line plugging should have served as a warning, as late in the run the vent line from the tank became plugged. Surprisingly perhaps, there was virtually no plugging of instrument lines to the calciner vessel or cyclone.

The fluidizing air blowers were switched several times during the run. When the first switch was made from WC-250A to WC-250B, the fluidizing air

flow meter showed a drop from 61 to 42 percent of chart. From other instruments it appeared that no significant change in air flow had taken place. Additional testing confirmed that the same air flow rate gave considerably different flow meter readings for the two blowers. Presumably, pressure surges from the blower closest to the orifice meter caused an error in flow measurement. This problem persisted throughout the run but it was corrected after the run.

3.2 Feed Rate Measurement and Control

Control of feed flow to the calciner vessel was generally satisfactory, as previously indicated (Section III-1.3). However, problems with the feed system were frequent and often difficult to resolve. Failure of an instrument component was readily corrected by replacing the faulty item with a spare, but sometimes it was difficult to determine whether a particular control problem was due to component failure or to inability of the feed system to respond to a control signal. To check certain instrument components in place required that the feed be shut off. Rather than do this, the suspect item would be replaced by a spare unit which had been bench-tested in the shop. Often it turned out that nothing was wrong with the instrument. In those instances the cause of erratic feed rate was probably due to such things as:

- (1) Sticking valve and/or valve operator mechanism
- (2) Accumulation of solids at the valve opening
- (3) Accumulation of gelatinous material on the electrodes of the electromagnetic flow meter
- (4) Accumulation of solids at the spray nozzle tip.

There were two occasions when difficulties with feed rate control could definitely be attributed to plugging of the feed nozzles, control valves, or flow meters. In each case the start of feed control trouble coincided with high solids content in a batch of feed. The high solids content was caused each time by inadvertent agitation of the contents of the underground waste tank just prior to a waste transfer. The solids were apparently picked up off the bottom of the tank.

Erratic feed rate control caused by high solids content of the feed continued long after a return to normal feed. Gradually, the solids worked through the feed system, and control improved. Techniques used to cope with plugging caused by solids were (a) operation of the nozzle plunger, (b) flushing the nozzle and valve with water, and (c) manipulation of the valve. The last was generally the most successful.

3.3 Temperature Measurement and Control

Temperature measurement and control problems were infrequent during the WCF run, but a few worthy of mention are listed below:

- (1) The NaK furnace outlet temperature recorder drifted out of calibration at least twice during the run. The first time it was found to be reading about 35°C high, while another time it was reading 30°C low.

- (2) Off-gas temperature controller, TRC WC-908-1, showed a slight tendency toward a zero shift and was found to be reading high several times, once as much as 10°C.
- (3) The transport air temperature controller had very little influence on the transport air return temperature. Because of excessive heat loss in underground piping to and from solids storage, it was not possible to supply enough heat with heater WC-352, and the steam to the heater was at maximum flow for the entire run.
- (4) The electrically operated control valves in steam lines to the off-gas superheaters, WC-354 and -356, did not operate reliably as supplied, and the valves had to be modified to provide for better lubrication. After modification, the valves worked satisfactorily.
- (5) At low fluidizing air flow rates, there is so much heat loss between the furnace and the calciner vessel that it is impossible to raise the temperature to the desired point. At the air flow selected for the WCF run, the desired temperature was finally reached but there was virtually no margin left for control.
- (6) The off-gas humidity and superheater controllers, TRC WC-908 and -305, were interconnected so that the superheater set-point would automatically be maintained 10°C higher than the temperature of the humidity controller. This setup worked well except that the temperature difference had a tendency to drift away from the desired 10° spread.
- (7) Two thermocouples located in the calciner cell and one in the off-gas cell failed during the run. Inspection of the other thermocouples in the calciner cell after decontamination indicated severe deterioration of the thermoplastic thermocouple lead insulation due to high cell temperature. All thermocouples and lead wires in that cell have been replaced with asbestos covered wire (good to 1100°F).

3.4 Miscellaneous Instrument Problems

A few instrumentation problems occurred which do not fit in any particular category and are listed as follows:

- (1) Power dips and failures caused the NaK furnace to shut down many times during the course of the run. Restarting the furnace was sometimes very difficult because the flame detector did not "see" the pilot when it ignited. This problem has been remedied by increasing the propane pressure to the pilot.
- (2) The oxygen analyzer for the NaK furnace flue gas originally discharged condensate from an atmospheric steam trap to a concrete pad outside the NaK furnace room. During the coldest part of the winter, this created a large slab of ice outside the

building, and eventually this became a serious problem. A vacuum pump was substituted for the steam jet. This eliminated the problem.

- (3) Each time the fluidizing air blowers were switched, a large change in air flow was indicated by the flow recorder although it was obvious from other instrumentation that little, if any, actual change in air flow had taken place. Tests after shutdown showed that if pressure surges are damped out, this air flow measurement problem can be eliminated.
- (4) The radiation-type NaK expansion tank level indicator was improved before the hot run, but it still did not perform reliably. A different type of level measurement device has been installed.
- (5) Most of the run the NaK flow was not varied. However, two or three times when the controller output was changed, there was no response. The problem was traced to a sticking shaft in the powerstat control mechanism.
- (6) The transmitting rotamotor providing flow measurement for the calciner pressure control air quit operating properly near the end of the run. Repairs could not be made until after shutdown of the WCF.

4. VALVES AND PIPING

In contrast to expectations, the problems which caused the most concern were material failures in valves and piping rather than failure of rotating mechanical equipment such as the off-gas blowers and the scrub system pumps. In addition, a number of lines became plugged with solids during the run, but most of these were for use in decontamination and did not affect operations.

4.1 Valve Bellows Failures

After approximately two months of operation during which there were no serious problems, the seal bellows of one or more valves failed and scrub solution began leaking to the floor of the off-gas cell. Tests were conducted to try to determine which valve had failed, but at the time they were inconclusive. Later in the run, scrubbing solution control valve, FRC WC-208-1A, began sticking and could no longer be opened or closed by normal changes in air pressure. It was later confirmed that this valve had failed.

From February 8 to February 24, solution leaking to the floor of the off-gas cell was drained to the hot sump tank, WC-119, and periodically recycled to the waste hold tank through RVC WC-119-3, which is located in the calciner cell. However, on February 24, a routine floor drain check disclosed liquid on the floor of the calciner cell. Tests showed that RVC WC-119-3 was leaking during recycle transfers from WC-119. The valve was closed for the rest of the run, and solution which leaked to WC-119 from the scrub system was transferred at intervals to WL-102, the ICPP waste evaporator feed tank. This arrangement

resulted in very little leakage from RVC WC-119-3 until about the end of July. By this time the operator for RVC WC-119-3 had corroded so badly that the valve was no longer held closed. Consequently, during transfers from WC-119 to WL-102, more solution would leak through RVC WC-119-3 to the waste hold tanks than would reach WL-102. Although operation of the calciner was possible for another 2-1/2 months, eventually the leakage from RVC WC-119-3 would probably have become so bad that it would have been necessary to shut down for decontamination and repairs.

Two other instances of block valve bellows failure occurred after the WCF had been shut down and was being decontaminated. RVC WC-119-2 seal bellows failed, and liquid leaked to the sump in the hot sump cell. RVC WC-208-4 failed during operation of the scrub system, and slightly contaminated liquid backed up the air line to the decontamination room.

4.2 Cause of Valve Failures

During and after decontamination of the WCF, eleven bellows-sealed valves were removed from the WCF for inspection and repairs. Ten were angle-type on-off valves constructed of 304 L stainless steel; the other was a throttling valve constructed of 347 stainless steel. Of these, two were known to have failed due to ruptured or corroded bellows during the campaign. Others were removed because of in-line leakage or because radiation levels from the valve bodies were abnormally high following decontamination.

Results of the inspection of the valves are presented in Table VI. In-line leakage was caused by corrosion of the Stellite facings (Haynes Alloy No. 6) on the seats and/or plugs of the valves. This alloy was originally specified (apparently with no regard to nitric acid corrosion) because a very hard material was considered necessary to resist erosion and abrasion of a two percent alumina slurry. Eight of the eleven bellows were corroded severely and perforations were visibly evident. The three remaining bellows were dented, but these dents may have been due to disassembly techniques (valve parts were contaminated and direct handling was necessarily restricted). Remote metallurgical examination indicated that failures in the on-off valve bellows were probably due to localized sensitization of the bellows material (Type 304 L stainless steel). Intergranular attack and stress corrosion were not prominent in the structure. About twelve leaves of the welded bellows in the throttling valve (FRC WC-208-1A) were Inconel instead of the prescribed Type 347 stainless steel. The Inconel corroded through after about 6000 hours and caused the first leak. Previously, the Stellite plug and seat of this valve were replaced (after 3500 hours) with 17-7 PH Condition 900 stainless steel for the plug and 17-4 PH Condition H-1100 stainless steel for the seat. These materials were not corroded nor was any appreciable erosion evident when the valve was disassembled.

Bonnets for the angle-type on-off valves were fabricated from carbon steel or cast iron. The yoke which supported the valve operator was attached to the valve body from the bonnet. In the event of bellows failure, leakage passed through the bonnet to a drain line to the cell floor. Severe corrosion of the bonnet can cause malfunction of the valve operator and uncontrolled leakage. Therefore, replacement of the carbon steel and cast iron with stainless steel for the bonnets is essential. This has been done for all block valves in liquid service.

TABLE VI

CONDITION OF BELLOWS-SEALED VALVES REMOVED FROM THE WCF

Valve	Failure Area			Service	Corrective Measure
	Plug	Seat	Bellows		
RVC WC-102-4	5-mil groove	Some burrs	Pin-point holes	12,500 hours -- transport air and decontamination solution	Stellite facings replaced with 308L weld deposit -- remachined -- new 7-mil bellows
RVC WC-107-1	Deeply pitted	Uncorroded	Warped, no pits	12,500 hours -- scrubbing solution: 5 M HNO ₃ , 1 M Al(NO ₃) ₃ , 50°C, trace solids -- decontamination solution -- 25 to 40 psig	Stellite facing on seat replaced with 17-7 PH overlay. Heat treated at 1100°F -- stellite on plug replaced with 17-7 PH overlay -- heat treated at 900°F -- new 7-mil bellows
RVC WC-107-2	Heavy general attack and pits	Burrs	Three dented convolutions	Installed in parallel with RVC WC-107-1	Same as for RVC WC-107-1
RVC WC-108-2	Severe corrosion of the stellite	Corrosion cracks	Perforations on an outside convolution	12,500 hours -- venturi scrubber drain. 5 M HNO ₃ , 1 M Al(NO ₃) ₃ , 65°C, trace solids, 8.0-in. Hg vacuum, decontamination solution	Stellite facings on plug and seat removed -- 308L weld deposit -- plug tapered to 45°, 1/32-in. land on the seat -- new 7-mil bellows
RVC WC-108-3	Severe corrosion of the stellite	Corrosion cracks	Perforations on two convolutions	12,500 hours -- same as RVC WC-107-1 except valve normally closed	Same as RVC WC-108-2
RVC WC-119-3	Severe corrosion of the stellite	Severe corrosion of the stellite	Separated from valve stem, severely corroded	Block valve in recycle system	Same as RVC WC-108-2
RVC WC-123AB-1	Uniform grooves in the stellite	Severe corrosion of the stellite	Numerous perforations	Directional valve in recycle system	Same as RVC WC-108-2
RVC WC-130-1	Groove in stellite facing	Burred	Severe corrosion with one hole	Drain valve in the equipment vent system	Same as RVC WC-108-2
RVC WC-208AB-3	Pitted stellite	Severe corrosion of the stellite	Dented	12,500 hours -- conditions similar to RVC WC-107-1	Same as RVC WC-107-1
RVC WC-208AB-4	General corrosion of the stellite	Severe corrosion of the stellite	Numerous perforations	Parallel valve to RVC WC-208AB-3	Same as RVC WC-107-1
RVC WC-208-1A	Uncorroded -- machine marks present on the 17-7 PH (900°F) stainless steel	Uncorroded -- machine marks present on the 17-7 PH (1100°F) stainless steel	No measurable attack on Type 317 bellows leaves. Inconel leaf section essentially corroded away	12,500 hours for bellows, 8,500 hours for plug and seat -- throttling valve for solution to the quench tower -- environment similar to RVC WC-107-1	Replace bellows with 15-mil welded type

4.3 Other Valve Problems

Although less serious, there were a number of other problems with valves during the WCF run. Some of these are listed below.

- (1) On May 5, during attempted operation of the NaK cold trap, throttling valve V-3 began to leak NaK. The leak was not serious and was stopped, but the cold trap was not operated for the rest of the run.
- (2) During tests of the scrub system valves in February, it was found that solution passed through RVC WC-208AB-3 almost as fast when closed as when open. There were also several other valves which did not give a good tight shutoff during the run or during the decontamination period that followed. These were RVC WC-105-2, -108-2, -108-3, -102-4, and -130-1. These and several other valves were removed for repairs.
- (3) The check valve in one-inch DCA-3030 to scrub system strainer, WC-123A, did not hold. During a recycle transfer, scrub solution backed up the line and some leaked to the floor of the decontamination room. The line was flushed out and the other recycle strainer was used for subsequent transfers.

- (4) Twice during the last three months of operation it was necessary to add NaK to the loop, indicating that the NaK dump valves and/or the manual valves were not holding completely tight.
- (5) Nearly all the steam block valves in the operating corridor were in poor condition by the end of the run. Several of them would not hold a tight shutoff, and several others had severe packing gland leaks.

4.4 Line Plugging

Lines and valves which were found to be plugged or badly restricted during the run or decontamination operations are listed below:

- (1) WC-105 vent line plugged in the latter part of May, and airlift air had to vent through the overflow line to the waste hold tanks along with excess liquid. During decontamination the plug was washed out without having to cut the line.
- (2) WC-119 sample pot drain line and valve plugged early in the run, and the plugged section of line had to be cut out after decontamination was completed.
- (3) Air line, one-half-inch HAA 3074, intended for particle size control was not properly purged and was found to be plugged at the end of the run. The plugged section of the line was cut out, and it will not be replaced. The opening to the calciner vessel has been sealed off.
- (4) The transport air return line drain, the silica gel transport line drain, and the drain from ten-inch PSA 3012 (downstream of WC-305 heater) were all found to be plugged at the end of the run when decontamination operations were started. The plugs could not be washed out, and the plugged sections had to be cut out.
- (5) Adsorbers WC-110, -111, and -113 were regenerated at the end of the run. None of the adsorbers would drain from the cone section drain line, indicating that either the valve, the line, or the very bottom section of the cone in each adsorber was plugged. This situation still exists, as the adsorber cells were not decontaminated. The adsorbers were drained from an alternate point higher on the bottom cone.
- (6) During decontamination, the sump jet in the hot sump cell became plugged while jetting decontamination solution from the floor of the cell. The jet discharge was cut and the plug was flushed out through the open section.
- (7) WC-105 drain line was badly restricted at the end of the run, and WC-105 would drain only very slowly. The line was cleared without being cut, but then it was found that the drain valve would not hold.

(8) All calciner vessel sample lines stayed open during the run.

4.5 NaK System Leaks

NaK, when the oxygen content (as sodium oxide) is maintained below 0.005 percent, is not normally corrosive to Type 316 stainless steel, the material of construction for NaK loop components. However, the nipple connection between NaK expansion tank WC-103A and the NaK loop failed twice during the hot run. The nipple was repaired after the first leak was discovered, but after the second failure in the same piece, expansion tank WC-103A and the nipple were removed from the loop. The expansion tank instrumentation was transferred from WC-103A to -103B, and for the remainder of the run the loop was operated with only one expansion tank.

The calciner was not completely shut down while work was being done on the NaK loop. In each case, the feed was shut off, the bed was maintained in a partially fluidized condition, and the off-gas system continued operating more or less normally.

4.51 Discovery of the NaK Leaks. The first NaK leak was discovered March 6, when smoke was observed near the bottom of WC-103A. The leak did not appear to be serious and, to reduce thermal shock on the drain and charge tanks and the dump valves, the NaK was cooled considerably before it was dumped. Repairs were completed in rapid order, the loop was recharged, the bed was refluidized without difficulty, and the feed was restarted after the bed had been heated to 400°C. The total feed downtime was about 18 hours.

The second NaK leak occurred on March 18. This time the leak appeared to be more serious, and the NaK was dumped almost immediately. The work was much more extensive, and after repairs were completed there was some difficulty in refluidizing the bed in the calciner vessel. Despite these things, the feed was only off for a total of about 33 hours due to the second NaK leak.

4.52 Cause of NaK Leaks. After removal of the NaK expansion tank, specimens of the tank nozzle, pipe nipple, and a small section of the tee were examined metallurgically to determine the cause of the leaks. The results of these tests are as follows:

- (1) Visual examination of the specimens, in the "as received" condition, indicated that a circumferential crack had developed immediately above the nozzle-to-pipe connector transition weld. When NaK leaked from this crack, the reaction products and high temperatures cut a vertical groove in the pipe connector, which subsequently exposed a vertical crack in the pipe connector. This crack was probably the same crack through which the first NaK leak had occurred. (This had been repaired by "putty" welding and patching.) Additional vertical cracks found in both the pipe connector and tank nozzle had apparently formed as a result of excessive heat generation while welding the nozzle to the tank.

(2) Results of microscopic examination of the specimens were:

- (a) Numerous stress corrosion cracks, varying in depth and length, were found in the tank nozzle and pipe connector.
- (b) In general, cracks formed intergranularly at the inside surface, then propagated from there transgranularly.
- (c) Cracks were both circumferential and axial.
- (d) No evidence of cracking or corrosion of the tee was found.
- (e) There was no evidence of general corrosion of the inside surfaces of the nozzle, connector, or tee.
- (f) Chemical analyses of the specimens confirmed that all of the specimens were Type 316 stainless steel.
- (g) Acidified copper sulfate corrosion tests indicated that the tank nozzle had become highly sensitized as a result of carbide precipitation. The pipe connector and tee showed essentially no attack during the corrosion tests.
- (h) Microhardness surveys indicated that inside surfaces of the specimens were somewhat harder than the parent metal.

In addition to the above metallurgical examination, an analysis of the external stresses in the NaK piping system was conducted. A probable contributing cause of the stress corrosion cracking was that no provision was made for the expansion of the three-inch NaK line between the two expansion tanks. Thus, expansion forces may have resulted which exceeded the yield strength of the 1-1/2-inch pipe connector at the operating temperature of 1400°F. The review revealed that no other region of high external stress due to expansion forces should be present in the system.

4.53 Operation With a Single NaK Expansion Tank. Removal of one of the two NaK expansion tanks did not seriously affect operation of the NaK loop. The single remaining tank, however, was not large enough to take care of the change in volume resulting from a temperature change of over 700°C. In heating the NaK from ambient temperature to 740 to 760°C, therefore, NaK had to be drained from the loop at an intermediate temperature. Conversely, if the NaK were to cool much below its normal operating temperature, additional NaK would have to be added to keep a level in the expansion tank. With close surveillance of the NaK level in the remaining expansion tank, the main NaK loop was operated successfully for the rest of the hot run.

5. MISCELLANEOUS

5.1 NaK Cold Trap Operation

During the cold portion of the WCF run, the NaK cold trap was operated until the oxygen concentration dropped to less than 0.003 percent, as determined by plugging indicator tests. On December 31, after about one month of

operation, another plugging indicator test indicated an O₂ concentration of about 0.005 percent, and the cold trap was started up and operated for four days until the O₂ concentration again dropped below 0.003 percent. Checks of sodium oxide content were also made February 12, March 9, and March 23, and in each case it was not necessary to operate the cold trap. The last two checks were after NaK leaks, and indicated that very little oxygen had gotten into the NaK loop even while repairs were being made. On May 5, another oxide plugging test was being attempted when a trace of NaK was detected leaking from the cold trap throttling valve. The cold trap valves were closed to isolate the system and neither the cold trap nor the plugging indicator was operated for the rest of the run.

5.2 Tank Farm Operation

The number of different waste transfer routes used during the course of the WCF hot run required more valve operation in the tank farm than had previously been used. The tank farm valves were generally found to be very difficult to operate. The first instance of an inoperative valve was discovered December 6, 1963, when the waste transfer route from WM-185 was being checked prior to the start of hot operation. Valve PUV 61 would not open, and transfer of waste from WM-185 was not possible. On December 7 the valve box was excavated, and temporary repairs were made the same day to permit use of the transfer route. Many other instances of tank farm valves sticking open or closed were found in the course of the run, but there were no delays because of valve failures. An extensive program of inspection, repair, and modification of tank farm valves was started as a result of the numerous failures found during the calciner run.

5.3 Equipment Vent Filter Changes

The equipment vent filter was changed seven times during the run. On-stream times ranged from 30 days to 55 days with an average of 44 days. Generally, filter was changed because of the high-radiation level of the housing. However, in one instance the filter was changed early (after 33 days) while the radiation level was still relatively low because the efficiency of the filter was not as high as usual. The last filter change was made because of high-pressure drop (greater than ten inches H₂O) and was the only instance that pressure drop exceeded one to two inches H₂O.

Changing the equipment vent filter was a fairly simple operation. However, higher than normal radiation exposures were required to disconnect and change out the filter unit.

Although changing the equipment vent filter was not a big problem, a longer interval between changeouts was expected because of the installation of the demister vessel, WC-130, in the equipment vent system. However, the demister did not perform as expected, and at high air rates entrainment of moisture was excessive. It was only by reducing sparge air to a bare minimum that the filter life was increased to the above range. The demister vessel will be modified before the next run to achieve efficient operation at higher gas rates.

5.4 Off-Gas Filter Replacement

Final filtration of the main calciner off-gas is accomplished by three parallel connected filter units. Each unit consists of a removable housing which holds a roughing prefilter and a high-efficiency final filter. Each unit was in continuous

service during the WCF hot run except for brief periods when a filter was being replaced.

5.41 Filter Replacement Schedule. During the WCF hot run the east and west filter units were changed three times, and the center unit was changed four times. After decontamination of the WCF, all three filter units were removed.

Two factors governed the operating life of the filter units. These were pressure drop increase and activity buildup. A radiation monitor was installed in each filter cell to provide a basis for estimating activity buildup on the filter. However, the radiation instruments proved unreliable, and an operating limit of 100 days was set for each filter unit on the basis of a calculated activity buildup rate. Table VII shows the date that each filter unit was replaced, the operating time

TABLE VII
FILTER REPLACEMENT DATA

	Center Unit F-2718-WC	East Unit F-2723-WC	West Unit F-2724-WC
Date of 1st Replacement	2-13-64	3-18-64	3-12-64
ΔP before (inches of H ₂ O)	3.9	3.8	4.1
ΔP after (inches of H ₂ O)	3.8	3.4	3.9
Time in service (days)	67	101	95
Date of 2nd Replacement	5-21-64	6-24-64	6-21-64
ΔP before (inches of H ₂ O)	7	4.5	5.8
ΔP after (inches of H ₂ O)	4	3.8	4.5
Time in service (days)	98	98	101
Date of 3rd Replacement	8-21-64	9-10-64	9-8-64
ΔP before (inches of H ₂ O)	5.8	6.6	7
ΔP after (inches of H ₂ O)	4.5	5.5	5.8
Time in service (days)	92	78	79
Date of 4th Replacement	9-28-64	After shutdown and decontamination	After shutdown and decontamination
ΔP before (inches of H ₂ O)	10.5		
ΔP after (inches of H ₂ O)	6		
Time in service (days)	38		

for each unit, and the change in total pressure drop as a result of each change. The first filter replacement was made after only 67 days service based on the radiation monitor in the cell. The next six filter changes were based on the arbitrary limit of 100 days of operation. The final three filter changes were made to reduce the overall pressure drop across the filters, and service for these three units varied from 38 to 79 days.

5.42 Filter Handling and Transport. Changing the final off-gas filter units proved to be a relatively simple operation. The equipment was designed so that all operations could be done remotely, but radiation levels were low enough to permit operation of the crane by direct visual operation after the filter cell hatch had been removed. The radiation level of a filter unit at the time of removal was about 10 to 20 R/hr gamma at 2 to 3 feet, but once the "hot" filter housing was disconnected and placed inside the shielding cask, the radiation field in the cell usually dropped to less than 100 mR/hr. The shielding cask, loaded with a hot filter housing, was transported by a low-boy trailer to a hot cell several miles distant from the WCF. At the hot cell, the filter housing was removed and the shielding cask was returned to the WCF. Usually the filter cask was transported to the hot cell the same day the filter unit was changed, but sometimes the loaded cask was left on the decontamination pad for one or two days. This did not cause any trouble.

The two sets of filter housings on hand at the start of the WCF run were easily installed and removed. However, a third set of housings, acquired during the run, did not fit properly. One of the three housings from this set was finally installed in the east filter cell after unsuccessful attempts to install it in the center cell and the west cell. The other two housings were not used during the run. The extent of misalignment of the third set of filter housings has been determined, and all housings have been altered to fit.

5.43 Remote Removal of Filter Elements. No provisions were made for reuse of the filter housings in the design of the WCF. It was assumed that the housings, along with the contaminated filters, would be transported in the shielding cask to the burial ground for disposal. However, because of the high cost of filter housings (about \$4500 each), there was considerable incentive to recover the housings for reuse. Before the first filter change, arrangements were made to attempt remote removal of the filters in the hot cell. Once the filter had been removed, the housing could be decontaminated to permit reuse. This technique proved successful, and none of the filter housings had to be discarded. During and following the WCF run, remote removal of the filters was accomplished thirteen times. The total cost of these operations was about \$5270 or about \$405 per housing. This is a saving of about \$4100 per housing over the cost of new ones or a total saving of about \$53,000.

5.44 Installation and Testing of New Filters. All of the prefilters and final filters are routinely tested for pressure drop and efficiency before they are accepted from the vendor. However, a test of each assembled housing was required to make certain that there was no way for gas to bypass the filter units. After installation of a roughing filter and a final filter in each housing, a DOP test^[12] was conducted. Several deficiencies were discovered and corrected as a result of the tests, and adequate filtering efficiency was thus assured. The equipment vent system filter housings were also DOP tested before installation.

5.5 Sample Room Contamination

Twice during the WCF run, contamination of the sample room became a problem. The cause in each case was different. In the first case, contamination took place several times during operation of the solids sampler, and was due to inadequate vacuum on the sample blister. By taping off most of the slit area

and taking special precautions during operation of the solids sampler, contamination from this source was contained. However, this was merely a temporary measure, and the sample blister vacuum has now been improved.

The second problem with sample room contamination began in July. At first the cause was not known, but contamination of the sample room was found to be coincident with transfers from the hot sump tank, WC-119, to the waste evaporator feed tank, WL-102. Eventually, it was discovered that even in the closed position, RVC WC-119-3 was leaking solution to the calciner cell floor when a transfer from WC-119 was made. A series of measurements showed that the sample room was at a slightly lower pressure than the calciner cell, and this caused air flow from the calciner cell to the sample room through the feed flow meter cubicle drain which was open in the calciner cell. When WC-119 was jetted, solution leaking from RVC WC-119-3 contaminated the air and, thus, also to some extent the sample room. After discovery of the cause and the mechanism, the contamination problem was eliminated by adjusting the pressure of the sample room until it was slightly higher than that of the calciner cell. With the driving force reversed, no more contamination of the sample room was experienced.

VI. SHUTDOWN, DECONTAMINATION, AND INSPECTION

1. SHUTDOWN AND PREPARATION FOR DECONTAMINATION

The routine shutdown procedure for the WCF was modified to take into account the condition of the calciner and the plans to start decontamination of the facility. Because of the poor condition of RVC WC-119-3, which made transfer of solution from WC-119 to WL-102 very difficult (Section V-4.1), waste transfers from the tank farm were discontinued considerably in advance of shutdown, and solutions from WC-108 and -119 were recycled and calcined. This left very little liquid in the various tanks at the time of shutdown, except for solution, held in WC-114, which had been used to wash the silica gel in adsorber WC-110. This solution had an extremely high activity and was held until it could be transferred out of the WCF facility without leakage through RVC WC-119-3 to the floor of the calciner cell.

1.1 Shutdown Sequence

Before the calciner was shut down, normal product transfer to solids storage was discontinued although the valves in the product takeoff lines were not closed.

Shutdown of the waste calciner began at 10:23 on October 15. At that time the waste hold tanks were empty and the feed was shut off. The remaining liquid in the feed tank, WC-105, was jetted to WC-119 (drain valve RVC WC-105-2 was restricted), and WC-105 was refilled with water. The water was fed to the calciner vessel to flush the feed lines. After the water flushes were completed, the feed system was secured with only the required air purges to prevent backup of solids into the feed lines. The NaK furnace was then shut down and transfer of the bed to solids storage was started. As mentioned previously (Section IV-1), this was completed without difficulty in about 4-1/2 hours. After the calciner vessel was essentially empty, a period of about 24 hours was spent in purging the calciner vessel to blow as much of the remaining solids as possible out the top of the calciner vessel. During this period, scrubbing solution was pumped to a waste hold tank and replaced with 4 M nitric acid to flush the scrubbing system. Also, all instrument lines to the calciner vessel and cyclone were blown out with air, and the decontamination lines to the NaK tube bundle recess were blown with air to clear solids from the recess as much as possible. After the "dusting out" period, the transport air system was shut down, the solids storage area was valved off from the transport air system, and the solids storage off-gas system was placed in service. Finally, on October 17, the fluidizing air blower and the off-gas blower were shut down and the cross connection to the equipment vent blower was opened. With only essential air purges on, there was no difficulty in maintaining adequate vacuum on the main off-gas system as well as the equipment vent system. At this point the shutdown was essentially complete.

1.2 Ruthenium Release During Shutdown

Drying of adsorber WC-110, which had been started October 13, was continued during the first part of the calciner shutdown. However, after the feed had been shut off and the bed had been started to storage, a very large increase in the ruthenium release rate took place. Drying of WC-110 was discontinued, but this did not stop the high release of ruthenium which for a short time reached a peak

rate estimated at 10 to 12 curies per hour. The humidity of the off-gas entering the other three adsorbers was then adjusted to about the same conditions in existence during routine operation. This did not help appreciably, and finally the adsorbers were bypassed. This brought the situation under control.

Later, while the calciner vessel was being purged, drying of WC-110 was resumed. At first it appeared that the drying operation could be completed without adverse effect. However, about 7 hours after drying was resumed, the ruthenium release rate increased abruptly. Drying was stopped and the release rate dropped to its former level. Drying of WC-110 was not attempted again until startup of the next processing campaign. At the time drying was discontinued, temperatures indicated that the top of the silica gel bed was dry (96°C), but that the middle (29°C) and bottom (24°C) of the bed were not dry.

1.3 Preparation for Decontamination

1.31 Blanking RVC WC-119-3 Inlet Line. Before starting full scale decontamination operations, it was necessary to cut and blank off the line to RVC WC-119-3 so that decontamination solutions could be transferred to WL-102 without leaking to the floor of the calciner cell and to the waste hold tanks. This was done on October 16 after the calciner cell had been sprayed several times with water to remove some of the gross surface contamination in the working area. Despite a general radiation level of about 5 R/hr gamma in the working area, the line was cut and blanked without anyone receiving more than authorized radiation dosage. After the line was blanked, solutions from waste hold tank, WC-100, and adsorber regeneration solution tank, WC-114, were transferred to WC-119 and from WC-119 to WL-102.

1.32 Regeneration of WC-111 and -113. For several reasons, it was decided to regenerate WC-111 and -113 ruthenium adsorbers before starting full scale decontamination efforts in the off-gas cell. Both adsorber beds were washed on October 19 without attempting to complete the saturation of the beds with water. Each bed was washed once with 1 M HNO₃. WC-111 was washed a second time with 1 M HNO₃ while H₂O was used for the second wash of WC-113. Each wash was drained separately to WC-114 and sampled. The results are tabulated in Table VIII. WC-110, which was regenerated before shutdown, had been washed

TABLE VIII

COMPOSITION OF ADSORBER REGENERATION WASH SOLUTIONS

	WC-110 Water Washes		WC-111		WC-113	
	First Time Through	Third Time Through	First Wash 1 M HNO ₃	Second Wash 1 M HNO ₃	First Wash 1 M HNO ₃	Second Wash H ₂ O
Aluminum (g/l)	1.15	1.38	2.99	< 1	2.71	< 1
Acid (N)	0.06	0.06	----	----	----	----
Undissolved solids (g/l)	0.27	0.27	0.23	0.19	0.45	0.31
Hg (g/l)	0.07	0.09	0.22	0.06	0.18	0.05
Ru-106 (d/m/ml)	4.8 x 10 ⁹	5.5 x 10 ⁹	2.0 x 10 ¹⁰	2.8 x 10 ⁹	5.9 x 10 ⁹	5.0 x 10 ⁷
Cs-137 (d/m/ml)	5.4 x 10 ⁷	7.0 x 10 ⁷	< 1.5 x 10 ⁸	< 3.6 x 10 ⁷	< 7.3 x 10 ⁷	7.0 x 10 ⁵
Sr-90 (d/m/ml)	----	----	1.6 x 10 ⁸	3.9 x 10 ⁷	1.4 x 10 ⁸	6.0 x 10 ⁵
Volume (liters)	----	0,700	3,000	3,280	2,720	4,070
Curies (Ru-106)	----	21,500	27,960	4,070	7,320	93

three times with water. The same wash water was used each time, and the first wash and the last wash were sampled in WC-114. These results are also tabulated in Table VIII. The amounts of aluminum, solids, and mercury removed by the regeneration washes were comparable for the three adsorbers, but the amount of ruthenium removed varied considerably. Comparing WC-110 and -111 only, it would seem that the 1 M HNO_3 was considerably more effective in removing ruthenium than was water. However, looking at only WC-110 and -113, the opposite conclusion could be drawn. Considering all three sets of data together, no conclusions can be drawn about the relative effectiveness of water and nitric acid as regeneration solution.

2. DECONTAMINATION OF THE WCF

From October 18 to November 2, decontamination of the WCF was pushed as hard and fast as possible, and during this period progress was rapid. On November 2, however, fuel processing in the ICPP was started, and decontamination work in the WCF had to be curtailed due to lack of manpower. Progress was sporadic after that, and there were periods of several days that no decontamination work was done at all. By January 7, however, decontamination of the calciner cell, the waste hold cell, and the off-gas cell was essentially complete. Later, the hot sump cell, the off-gas blower cell, and the off-gas filter cells were decontaminated to varying degrees over a period of about two months. In the off-gas cell and the calciner cell, removal of external contamination due to valve leaks required as much or more effort as decontamination of interior equipment surfaces. If it had not been for the external contamination problem, it is probable that the entire decontamination operation could have been completed in a month of concentrated effort.

2.1 Waste Hold Cell

Decontamination of equipment in the waste hold cell was started October 16, and alternate flushes with water and nitric acid were made through October 26. Caustic, water, and nitric acid were used in turn from October 27 through November 22, and caustic-tartaric acid was used starting November 24.

Equipment in the waste hold cell was decontaminated by the same methods used in the uranium processing plant, and there were no unique problems. There was no external contamination, and except for the bottom of tanks WC-100 and -101 and a few valves, the equipment decontaminated quite easily. The cell was entered October 19, at which time general background radiation levels varied from about 500 mR/hr at the floor level to about 1000 mR/hr near the top of the cell. The tanks and valves were considerably hotter, and some spots reading greater than 25 R/hr were detected. After about a month of work, the general background in the cell had been reduced to about 150 mR/hr with spots ranging up to about 5 R/hr. Very little further improvement resulted until use of caustic-tartaric acid was started. This proved very effective. Within a few days, the general background radiation had been reduced to about 25 mR/hr, and maximums were no greater than 150 to 200 mR/hr. This completed the waste hold cell decontamination.

2.2 Off-Gas Cell

2.21 Initial Radiation Levels. Daily radiation surveys were made in the calciner cell, the off-gas cell, and the transport air cell after hot feed was started. At the entrance to the off-gas cell, the radiation level reached equilibrium at about 8.5 R/hr after 11 days of hot operation. Very little change in radiation level took place with continuing operation until solution started leaking to the floor of the cell from the bellows of FRC WC-208-1A. At the time of shutdown, radiation levels were much higher in the off-gas cell, and much of it was due to external contamination of the equipment. The first survey of the cell was taken October 17, after the scrubbing system had been emptied and flushed with 4 M HNO₃. The general cell background in the south end of the cell was about 5 R/hr gamma and 7.5 R/hr gamma plus beta. WC-114 was 15 R/hr gamma. WC-108 was 12 R/hr and floor drain area was 15 R/hr gamma and greater than 25 R/hr beta plus gamma.

2.22 Interior Surfaces. Conventional methods of decontamination were used in decontaminating the interior surfaces of off-gas cell equipment, and the only real problems encountered were due to inability to flood certain equipment items. Most of the scrubbing system equipment and lines were easily decontaminated by alternate contact with nitric acid, water, and caustic. The regeneration solution tank, WC-114, was quite difficult to decontaminate, although it had been contacted for a relatively short time with radioactive solutions and then only with wash solution from the adsorbers. However, repeated contact with nitric acid at concentrations from 4 M to 13 M finally reduced radiation to satisfactory levels. Boiling 13 M HNO₃ appeared to be the most effective treatment. Off-gas heater, WC-305, was difficult to decontaminate due to inability to fill it with solution. However, once a method for filling WC-305 was devised, a series of contacts with hot nitric acid, water, and hot caustic reduced the radiation from greater than 25 R/hr to less than 150 mR/hr in a short time. Other problems which were encountered were: (a) A one-inch line which drains the ten-inch off-gas line was plugged. The plugged section was cut out and a temporary drain was installed. (b) Several valves would not hold in the closed position and solution leaked through them.

2.23 Exterior Surfaces. Most of the equipment in the off-gas cell was contaminated to some extent on the outside. The floor of the cell, scrub solution tank WC-108, and part of the wall opposite FRC WC-208-1A were highly contaminated and had to be scrubbed many times before external contamination was reduced to a satisfactory point. The problem was aggravated by the fact that the bad valve, FRC WC-208-1A, could not be removed in the early stages of decontamination, and leakage of solution from the valve during decontamination tended to recontaminate equipment to some extent. Finally, on December 23, the valve was cut out and the open lines were plugged. Intensive scrubbing over a period of seven days then reduced the external contamination to satisfactory levels except in a few difficult-to-clean places such as the pump flanges and housings. Part of the concrete wall was deeply etched and impossible to decontaminate completely. Where radiation was high from this source, lead was used to shield it out. For cleaning contamination from external surfaces, scrubbing with Turco 4502 followed by oxalic acid proved to be the most effective treatment.

2.24 Final Radiation Levels. From October 16 to November 2 during full time decontamination operations, progress was fairly rapid. Gamma radiation

levels were generally lowered by factors of 10 to 20 over beginning levels, and in many areas the beta to gamma ratio had been reduced to 2/1 or less. However, there were still a number of hot spots, and general cell backgrounds were still 500 to 1000 mR/hr depending on the location. By January 7, when decontamination of the off-gas cell was essentially complete, the radiation level in most parts of the cell ranged from 50 to 100 mR/hr gamma. The contribution from external contamination had been virtually eliminated.

2.3 Calciner Cell

2.31 Initial Radiation Levels. After radioactive feed was started to the WCF, the radiation field at the entrance to the calciner cell increased to an equilibrium value of about 8 R/hr. The field remained essentially constant until valve RVC WC-119-3 failed, allowing radioactive solution to leak to the cell. As the run progressed, the condition of RVC WC-119-3 got worse, and during transfers from WC-119 to WL-102, liquid would spray from the valve into the calciner cell contaminating all the equipment in the cell to varying degrees. The floor of the cell and the cell entry, the walls of the cell entry, and nearby piping were contaminated the worst. A survey taken after the calciner was shut down and the bed had been dumped (but before any decontamination work) revealed a general field of 10 R/hr gamma and greater than 25 R/hr beta plus gamma at the entrance of the cell. After the floor and walls at and near the entrance were washed several times with water, the general field at the cell entrance dropped to about 5 R/hr gamma and 8 R/hr beta plus gamma. It was at this stage that RVC WC-119-3 was isolated to prevent further leakage.

2.32 Interior Surfaces. Great care was taken in introducing decontamination solution for the first time to the calciner vessel, the calciner cyclone, and all other lines which had handled dry solids, to prevent formation of plugs. The calciner vessel was filled from the bottom in an attempt to dissolve solids as they were contacted and to establish a counter flow through lines where solids would tend to settle. Once the calciner vessel was filled, the NaK loop was used to heat the decontamination solution to boiling temperature and hold it there for the required period. This technique worked very well, and not a single major line to or from the calciner vessel became plugged. Because amorphous aluminum oxide is quite soluble in 4 M nitric acid, this was used as the initial decontamination solution for the calciner vessel and all other solids handling equipment and piping. Later, other solutions, including plain water, caustic, and caustic-tartaric acid, were used. Nitric acid (or water) was also used initially on all other equipment and piping to make certain all aluminum was rinsed out before any caustic solution was used.

During the course of decontaminating the equipment in the calciner cell, a few plugged lines were discovered. Some of these were known to be plugged before the calciner was shut down, but for others it was impossible to establish whether the plug occurred during operation or decontamination. Most of these plugged lines could not be cleared except by cutting out a section.

2.33 Exterior Surfaces. The external contamination problem in the calciner cell was similar to that in the off-gas cell. The floor, walls, and equipment surfaces close to the point of leakage were highly contaminated, but higher in the cell the contamination problem diminished rapidly. The same chemicals and methods of decontamination were used in the calciner cell as in the off-gas cell. Some of the concrete near the cell entrance and in the entry way itself could not be adequately decontaminated and had to be covered with lead sheet.

2.34 Final Radiation Levels. For about the first three weeks after the WCF was shut down, decontamination was pushed continuously. However, as two other cells were also being decontaminated, probably no more than 1/3 to 1/2 of that time was actually spent on calciner cell decontamination. Progress was quite rapid during the early period, and by November 8 gamma radiation fields in the calciner cell were generally less than 500 mR/hr. The top of the calciner vessel was 700 mR/hr, the middle of the vessel was generally about 1000 mR/hr, and the bottom cone was about 400 mR/hr. There were still numerous hot spots with much higher fields, and external contamination was still a problem in some areas, principally the cell entry and the floor. By January 7, decontamination of the calciner cell was finished although when the calciner vessel was opened for inspection, some work was done on specific hot spots through the open manway. General radiation fields in the cell ranged from 40 to 100 mR/hr gamma with practically no beta. The fields were generally highest in the lower half of the cell and lowest in the upper half.

2.4 Secondary Decontamination Work

2.41 Hot Sump Cell. Decontamination of the equipment in the hot sump cell was not originally planned while the WCF was down. However, late in December liquid buildup on the floor of the hot sump cell was detected while transferring solution to WL-102. All indications pointed to failure of the bellows seal for one of two valves in the cell. About a month later, after decontamination of the off-gas cell, the waste hold cell, and the calciner cell was finished, part-time decontamination of the hot sump cell was started. The cell hatch was removed January 27, and it was established that RVC WC-119-2 was the faulty valve. On March 3, the bad valve was removed and the lines were capped. The valve is not needed and will not be reinstalled.

2.42 Transport Air Cell. The transport air cell was entered many times during the WCF run to check for "hot spots" on the transport air line and valving. The radiation background in the cell never got above 100 to 200 mR/hr even close to the transport air line. However, at places on the bottom of the line and upstream of the slide valves, radiation levels reached 5 to 10 R/hr due to accumulation of solids. The transport air line and valves were flushed with nitric acid during decontamination of the calciner cell, and that was sufficient to eliminate hot spots. No additional decontamination was required.

2.43 Off-Gas Blower Cell. Radiation levels in the off-gas blower cell remained relatively low throughout the hot run, permitting entry to inspect equipment whenever desired. Actually, the high ambient temperature in the cell was a more serious deterrent to staying in the cell for extended periods than was radiation. However, the equipment vent blower, WC-252, and the off-gas blower, WC-251A, which operated for the entire run, were considerably above the background radiation level, and several days were spent decontaminating both. Efforts were only partially successful, and parts of the blower and associated piping remained considerably above general background levels. Work on the blowers was completed, however, without excessive radiation exposure to maintenance personnel.

2.44 Off-Gas Filter Cells. In the middle of February 1965, the off-gas filter housings were removed from the cells. Except for the open ends of the off-gas lines, the radiation fields in the cells were quite low, generally under 100 mR/hr. However, the floors were contaminated too highly to permit routine work. Covers were taped over the open off-gas lines, and the floors, walls,

and piping were washed and scrubbed. This was adequate to permit work in the cells to determine the extent of misalignment of the second set of spare filter housings and to modify the connecting rods to correct this problem.

3. FACILITY INSPECTION

3.1 Calciner

Subsequent to decontamination, the calciner was opened for visual inspection of the baffle plate, NaK tubes and tube support, feed nozzles, and distributor plate caps. Photographs of the baffle plates, gas outlet screen, and upper decontamination header are shown in Figures 11 and 12. The baffle plates were

Fig. 11. Opened calciner vessel after decontamination.

Fig. 12. Calciner decontamination header, baffle, and off-gas outlet screen after decontamination.

essentially free of dust, and the stainless steel angles used to hold down the plates had no buildup of solid material even in difficult to reach corners. The basket-type screen placed over the off-gas outlet was clean as was the decontamination header. All of the 1/8-inch holes, some of which can be seen in Figure 12, were clear.

A remote metallurgical examination of one baffle plate (Type 347 stainless steel) showed that carbide and sigma phase precipitation were absent. There was no evidence of intergranular attack, and micro-hardness measurements indicated that there was no significant change in the metal surface. The corrosion film on the plate was too thin to measure accurately, but the thickness was estimated to be less than one micron. This plate had been subjected to the calciner atmosphere at temperatures between 325 and 410°C for 10,000 hours.

After the baffle was removed from the calciner, a photograph of the interior of the vessel (Figure 13) was obtained. Essentially all of the solids had been removed from the vessel. Shiny surfaces were evident at various locations. Some of these were obvious reflections of the camera flash from flat surfaces. The reflective surfaces around the distributor plate caps may have been caused by abrasion of the oxide coating from the plate and/or accumulation of solids. This could not be resolved even with the aid of a telescope. It was apparent, however,

Fig. 13 Calciner interior after decontamination.

that no irregularities such as would be caused by severe abrasion were present. Because radiation readings in the immediate vicinity of the caps were quite low, accumulation of solids undoubtedly was very minor at most. A polished surface caused by the fluidizing action of the alumina is the most probable reason for the pronounced reflection.

A radiation survey of the entire vessel indicated that the highest readings were near the center of the NaK tube bundle. Analysis of a "smear" indicated a full range of fission products, the major source, however, being cerium-144. When water was added to submerge about two-thirds of the NaK tubes, the radiation level in the vessel dropped to about 30 mR/hr. The vessel was then entered for inspection. The NaK tubes showed no evidence of wear though a very thin layer of alumina coated the surfaces. The tube support was completely intact and had not loosened. Lack of any markings on the tubes indicated that all the tubes had expanded as an integral unit. Micrometer measurements of two of the tubes were obtained and these are shown in Table IX along with measurements obtained previously. The variations in readings are felt to be within measurement accuracy which resulted from the cramped and undesirable conditions associated with the wearing of radiation protection clothing and special breathing equipment.

TABLE IX

NaK TUBE DIAMETER MEASUREMENTS (in.)

Tube 1	Near Tube Sheet		Center		End of Tube	
	Vertical	Horizontal	Vertical	Horizontal	Vertical	Horizontal
November 26, 1963	1.551	1.551	1.550	1.552	1.545	1.545
January 28, 1965	1.545	1.545	1.543	1.548	1.548	1.552

Tube 2	Near Tube Sheet		Center		End of Tube	
	Vertical	Horizontal	Vertical	Horizontal	Vertical	Horizontal
November 26, 1964	1.551	1.551	1.550	1.552	1.549	1.549
January 28, 1965	1.553	1.557	1.550	1.550	1.548	1.555

All of the distributor plate caps were tested using a hooked rod and found to be intact and firmly in place. The feed nozzle caps which were fabricated from Type 440 C stainless steel showed very slight wear. Later the feed nozzles were removed and the nozzle air caps were found to contain severe corrosion cracks and pits. The austenitic portions of the nozzles, however, were not corroded. The air caps have been replaced with a precipitation-hardened austenitic steel for the next campaign.

3.2 Cyclone Fines Ejector

The cyclone fines ejector provides the motivating force for transfer of solids from the calciner cyclone to the product transport line. This ejector has a boron carbide liner and contains a motive air line with a tip fabricated from Type 440 C stainless steel. X-ray photographs of the ejector indicated that both the liner and the motive air line tip were intact. No evidence of abrasive wear was apparent from the photographs.

3.3 Quench Pumps

Several Type 304 stainless steel nuts installed on a flange below the solution depth in the scrubbing solution tank suffered severe corrosion and had loosened. The accompanying Type 304 L stainless steel bolts were unattacked. Metallographic examination indicated that localized attack occurred at the grain boundaries of the nuts. Chemical analyses indicated carbon concentrations in the nuts and bolts of 0.151 and 0.050 percent, respectively. Chromium carbide precipitation probably resulted during fabrication of the nuts. This carbide later was slowly dissolved out by the nitric acid in the tank. Stabilized stainless steel such as Type 347 or low carbon stainless steel such as Type 304 L will be used for future campaigns.

3.4 Venturi Scrubber

Pipe plugs in the piezometer ring of the venturi scrubber were removed for inspection of the holes to the throat of the venturi scrubber. Polishing of the metal immediately around the holes was evident but even moderate erosion was not observed. The piezometer ring was essentially free of solids.

3.5 Electromagnetic Flow Meters

Electromagnetic flow meters in the feed streams to the calciner nozzles were removed for inspection of the vitreous enamel liners. There was no apparent damage to the enamel except possibly in the vicinity of the platinum electrodes. Boroscopic observations were hindered by the need for plastic covering over the boroscope to prevent contamination of the instrument. However, lower resistance values from the platinum electrodes to ground than those observed with new meters suggest the possibility of deterioration of the enamel in the regions of the electrodes. A new meter registered 100,000 megohms while those from the WCF varied from 8000 to 30,000 megohms. Three new meters will be installed before operation of the WCF is resumed.

3.6 Filter Support Media

The high-efficiency filters originally specified for the WCF contained stainless steel spacers between the filter media. These spacers, though excellent from the corrosion standpoint, were costly and presented many problems because sharp edges of the metal frequently damaged the filter media during fabrication, handling, and shipping. During the last two months of the campaign, filters containing "asbestos-type" and "glass-type" spacers were used in the WCF. Rapid pressure-drop increase across the filters necessitated change-out of one of the "asbestos-type" filters. The spacers had lost their rigidity and the filter media was severely matted, thus reducing the free surface area of the filter. The major constituent of crystalline deposits removed from the filter case was magnesium nitrate. (The "asbestos-type" spacers contained approximately twenty percent magnesium by weight.) Laboratory tests of the asbestos-type and glass-type material later indicated weight losses of 38 and 3.7 percent, respectively, after eight days exposure at 65 to 70°C to air and vapor from boiling 4 M nitric acid. Thus the "asbestos-type" filters are not suitable for service in the WCF off-gas environment.

VII. CONCLUSIONS AND RECOMMENDATIONS

1. CONCLUSIONS

The first hot run of the Waste Calcining Facility was successful in every respect. In slightly over ten months of virtually continuous operation, fluidized bed waste calcination was proved to be a safe, reliable, and very attractive process for converting radioactive waste liquids to solids. The principal conclusions reached during operation, decontamination, and after inspection are:

- (1) The WCF is basically very stable and can withstand severe upsets and temporary changes in operating conditions without detriment to the product or to off-gas decontamination.
- (2) Mechanical equipment held up very well, and planning for runs of a year or more in duration appears quite reasonable.
- (3) Without modifications or changes in operating conditions which would increase the amount of heat transfer to the fluidized bed, the WCF is probably limited to a gross feed rate of about 100 gph or about 20 percent above design capacity. Gross feed rates 50 to 75 percent greater than design could probably be achieved with no changes except those necessary to supply more heat to the fluidized bed.
- (4) Undissolved solids of the type twice entrained from the bottom of the underground waste tanks cause considerable feed control problems. However, pilot plant tests indicate that finely divided solids would not be expected to cause a great deal of trouble.
- (5) The overall efficiency of the off-gas cleanup system for removal of particulate material was excellent as evidenced by an average decontamination factor of about 1.3×10^7 for strontium-90. However, the efficiency of the calciner cyclone, the first solids removal step, was only about 50 percent. If the cyclone efficiency could be increased to 75 percent or more, the solids carry-over rate to the scrubbing system would be reduced by a factor of two or more, and the rate of recycle of scrubbing solution could be reduced considerably.
- (6) The conditions which govern adsorption and desorption of ruthenium in silica gel beds need to be better defined by additional studies. During the run, ruthenium released to the stack averaged less than 1 curie per day, while in a four-hour period during shutdown about 55 curies of ruthenium were released to the atmosphere from the adsorbers.
- (7) As demonstrated in the existing solids storage facility, heat transfer from the storage vessels to the vault walls to the ground is an important factor in removing heat from stored solids and will be taken into account in the design of future storage facilities.

- (8) Successful decontamination of the WCF proved that the "direct maintenance philosophy" is feasible for the WCF.
- (9) Erosion and corrosion were negligible in the calciner vessel, the NaK tube bundle, the cyclone fines jet, the scrub system pumps, and those portions of the transport air system which were inspected.

2. RECOMMENDATIONS

To improve operation of the WCF and to further increase reliability of equipment, the following modifications are planned prior to the next campaign:

- (1) The bellows-drain manifold for valves in the scrub system should be routed to a collection pot instead of the floor drain. Provisions for detection of a leak and return of the solution to the scrub solution tank should be included.
- (2) The recycle strainers should be eliminated.
- (3) Carbon steel and cast iron bonnet plates for Fisher-Governor block valves in regular liquid service should be replaced by stainless steel plates.
- (4) The equipment vent system demister vessel should be revised to reduce liquid entrainment. A larger vent should be installed from the sample gallery to the equipment vent system.
- (5) The decontamination manifolds at the WCF should be revised to eliminate leaks and corrosion of carbon steel steam lines.
- (6) The transport air return line should be rerouted to eliminate low spots and reduce pressure drop.
- (7) Improved facilities should be installed for sampling the off-gas at intermediate points.
- (8) The fluidizing air metering system should be revised to eliminate the effect of pressure fluctuations from the blowers.
- (9) The adsorber drain lines should be rerouted directly to the drain header in the off-gas cell.
- (10) Piping should be modified so that the scrub tank pump-out jet will be independent of the scrub solution recirculation system.
- (11) New feed nozzles and new electromagnetic flow meters should be installed in the feed system.
- (12) Stainless steel liners should be installed on the floor and part way up the walls in the entry way of the calciner and off-gas cells.

- (13) The calciner vessel and transport air line drain should be separated from the waste hold tanks and feed system drains.
- (14) The off-gas filter connecting rods should be modified to permit installation of all the off-gas filter housings.

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