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APPENDIX F  
DESCRIPTION OF WASTE TREATMENT OPERATIONS  
AT ROCKY FLATS DURING THE 1967 TO 1969  
TIME PERIOD IMPACTING INEEL PIT 9

**DESCRIPTION OF WASTE TREATMENT  
OPERATIONS AT ROCKY FLATS  
DURING THE 1967 TO 1969 TIME PERIOD  
IMPACTING INEEL PIT 9**

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## 1.0 Introduction

Manufacturing and chemical processing operations at the Rocky Flats Plant (RFP) generated both liquid and solid radioactive waste materials. It is expected that both solid radioactive waste materials and the radioactive sludge and salts resulting from treatment of the RFP liquid wastes, were disposed of in the Idaho National Environmental Engineering Laboratory (INEEL) Pit 9 in the 40' x 40' area proposed for Stage I/II activities. This expectation is confirmed in LMITCO Interdepartmental Communication RWT-01-99, dated April 16, 1999 from Thomas to Wilkins. Table I of the communication delineates the expected RFP waste contents in the subject 40' x 40' section of Pit 9.

This paper provides both a summary waste generation description and a more detailed description of the RFP waste generation processes. The emphasis of this waste process description is to generally describe the final waste form of the Building 774 sludge, nitrate salts and organic sludge and the form of the solid wastes that may be of particular interest in evaluating the Pit 9 safety concerns.

## 2.0 Summary Waste Generation Overview

Radioactive materials waste streams at RFP were generated in both solid and liquid forms. The determination of whether these materials were to be processed for the recovery of contained plutonium or discarded as wastes was made based upon an Economic Discard Limit (EDL). This EDL was based upon a comparison of the cost to recover the plutonium to a weapons specification metal form with the cost of virgin plutonium from the material production sites. If the cost to recover the contained plutonium was equal to or less than the cost to produce new plutonium, the material was classed as a residue and processed to recover the plutonium. If the cost to recover was greater than the cost of newly produced plutonium, the material was disposed of as waste.

Solid radioactive wastes were generated in various manufacturing and plutonium recovery glovebox operations. This solid waste consisted of a wide variety of materials. These materials included leaded glovebox gloves; paper, plastics, rags and other combustible wastes; various tools and other light metal or steel wastes; heavy metal wastes such as tantalum molds and funnels; graphite mold materials; glass and other items used in day-to-day glovebox operations.

Radioactive liquid wastes consisted of aqueous liquid waste from various plutonium recovery, analytical laboratory and R&D operations and organic wastes primarily from machining operations in the manufacturing process. The liquid wastes were transferred to Building 774 (then called Building 74, hence the sludge types 74-1, 74-2, etc.) via pipeline or from small generators by individual containers.

## 2.1 Solid Waste Generation Overview

The various solid wastes described above were segregated by Item Description Code (IDC) and then removed from the glovebox system using standard bagout procedures. In general the materials were placed into 55-gallon drums corresponding to the IDC codes. The separation of the materials by IDC was desirable for two reasons. First, to provide a material matrix in the drum that corresponded to the matrix in standards for newly developed Non Destructive Assay (NDA) equipment. Secondly, to simplify plutonium recovery if the material was to be processed as a residue rather than discarded as waste.

The separation of materials by IDC was less than desired. Improper segregation was frequently discovered in residue drums that were opened for processing. In these cases the drums were returned to the generator, operators re-trained or directed to be more conscientious, or merely separated at the recovery glovebox. The last scenario was the rule during the middle and late 1960's. After the middle 1970's significant progress was made in both NDA technology and in controlling the contents of drums, but this was not the case during the time period of interest for INEEL Pit 9.

## 2.2 Liquid Waste Processing Overview

The liquid waste processing operations in Building 774 treated all liquid wastes from the plutonium recovery areas and all other liquid process wastes. The purpose of the liquid waste treatment processes was four-fold:

- To remove radioactive contaminants from aqueous process waste and convert the solids generated to a shippable form for off-site disposal.
- To convert aqueous process wastes to water with purity suitable for atmospheric disposal through the waste treatment evaporator
- To solidify non-compatible aqueous wastes for disposal off-site.
- To solidify organic wastes for disposal off-site.

The Building 774 liquid waste treatment process was divided into four process groups. These were:

- First-Stage Processes
- Second-Stage Processes
- Evaporation Processes
- Solar Evaporation Ponds

These processes are shown as a simplified waste flow diagram in Figure 2. 1, Summary Waste Flow Diagram.

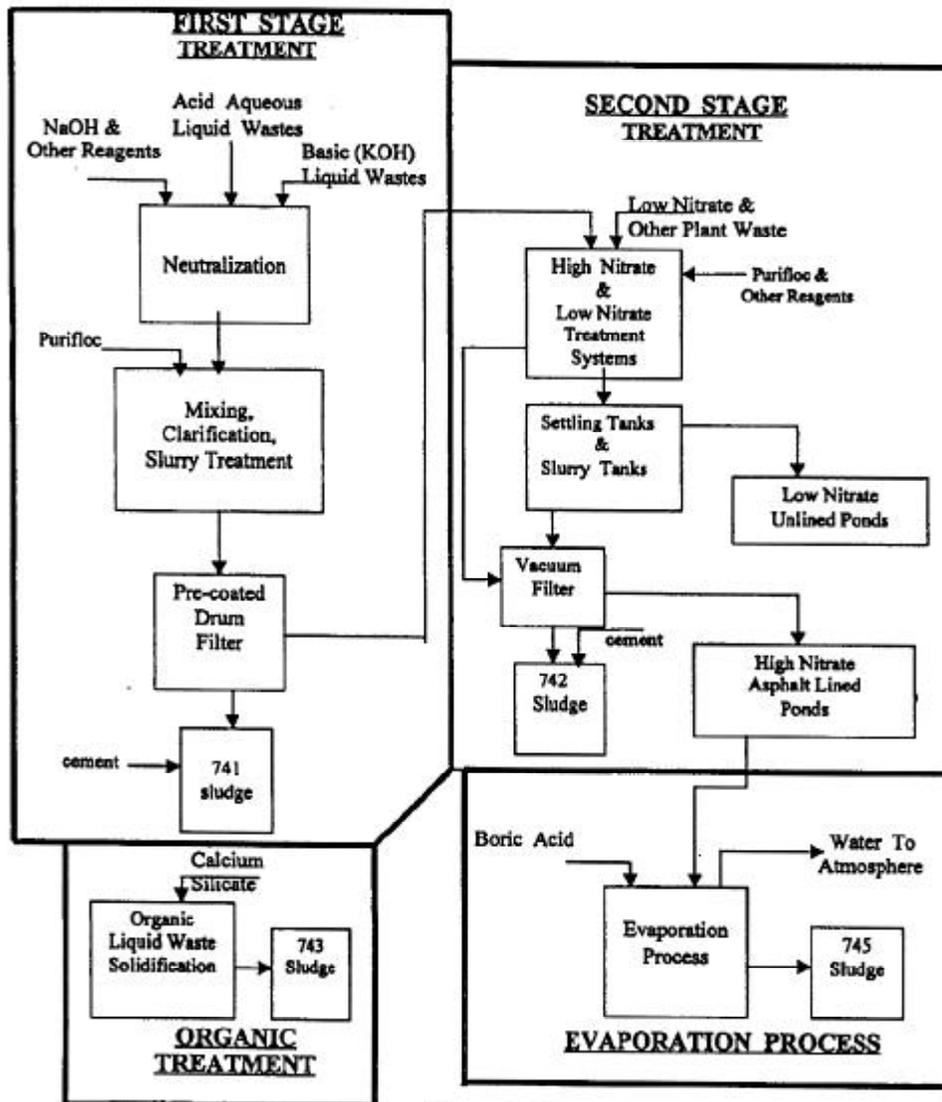


Figure 2.1 Summary Waste flow Diagram

### 3.0 Liquid Waste Treatment Detailed Description

Since the nitrate salts and sludge produced in the liquid waste treatment process are the focus of the Pit 9 independent technical review a more detailed description of the liquid waste treatment process is provided as follows. The following description is primarily based upon an informal document by Mr. Larry R. Crisler of the Los Alamos Technical Office/Rocky Flats. The document is titled, **A Comprehensive History of the Rocky Flats Plutonium/Actinide Recovery Operations, 1952 to 1991**, (dated 2/3/92). For a detailed description, including detailed waste treatment flow diagrams, the reader is referred to the document by Mr. Crisler

#### 3.1 First Stage Liquid Waste Treatment

Most aqueous wastes from the Plutonium Recovery Building (771) entered the first stage of the Building 774 Liquid Waste Processing facility via pipeline. The first-stage receiver vessels were connected to the waste generator's tankage by pipelines. All transfers were made by vacuum. Packaged aqueous wastes were also introduced by vacuum into the appropriate vessels through dip tubes and hose connections attached to the receiver vessel.

In general, each waste received at the first stage was entered into one of four processes. Each process was tailored to meet certain characteristics of a particular waste. The waste passed through one or more of these processes before entry into the Second-Stage operation. The First Stage processes involved and the waste streams they treat are categorized as follows.

##### 3.1.1 Neutralization and Filtration Processes

Acid wastes containing large quantities of metal ions that were insoluble in basic solutions or chloride ions that were corrosive to the process equipment were neutralized with sodium hydroxide prior to vacuum filtration. The major cations found in the waste were aluminum, calcium, iron, potassium, magnesium, sodium, and silicon. The major anions found in the waste were nitrate, sulfate, fluoride, and chloride.

The main purpose of the process was to remove the large quantity of metal hydroxide solids produced from the waste stream since these solids would seriously hamper the decontamination ability of the succeeding flocculation and clarification processes. The metal ion acid wastes, such as ion column effluent and miscellaneous acid wastes, were received in the acid tank. This tank was drained by gravity into the neutralizer tank. Sodium hydroxide reagent was added until the waste reached a pH of approximately 11. The neutralized waste was then pumped from these tanks to a rotary-drum, vacuum filter, pre-coated with diatomaceous earth. The rotary drum filter separated the first stage liquid from the first stage solids.

The solids, labeled 741 Sludge, contained the bulk of the radioactive contaminants. The 741 Sludge was collected in specially prepared 55-gallon drums for disposal. Portland cement was layered into the drums to adsorb the high water content of the 741 Sludge.

The filled 55-gallon drums were separated from the rotary drum vacuum filter glovebox by the standard, bag cut procedure. The 55-gallon drums were closed, weighed, labeled, logged, and surveyed for internal and external radiation. They were then transferred to Building 664 for disposal. The liquid was transferred to the neutral or basic waste process feed tank for further treatment.

The chloride ion acid waste was received in a Kynar™ -lined HCl tank. Sodium hydroxide reagent was added to the tank until the waste reached a pH of approximately 11. The tank was then drained by gravity into the First Stage, slurry-holding tank. The chloride ion slurry was mixed with other slurries and was then pumped to the First Stage pre-coated rotary drum vacuum filter for the solid/liquid separation.

### **3.1.2 Batch Neutralization, Precipitation, and Filtration Processes**

Acid waste containing only small quantities of metal ions that are insoluble in basic solutions and basic waste that contains large quantities of undissolved solids were received in treatment and batch tanks.

Ferric sulfate and calcium chloride reagents were mixed into the waste, and the solution was adjusted to a pH of approximately 11 by the addition of sodium hydroxide. An adjustable dip tube was used to decant the solids-free liquid into the feed tank for the next precipitation and clarification process. The solids remaining in the bottom of the tank were gravity drained to the slurry holding tanks. The slurry from this process was combined with other slurries and processed through a pre-coated rotary drum vacuum filter for liquid/solids separation.

Caustic (KOH) scrubber solution from the Plutonium Recovery area was received in a batch tank and then drained by gravity to a slurry holding tank. The solids and liquids in the combined slurries were separated by vacuum filtration. This waste had to be filtered, because the solids would have affected the radioactive decontamination of the solution in the succeeding process.

### **3.1.3 Continuous Radioactive Decontamination Process**

Caustic scrubber solutions (KOH) that were relatively free of solids, miscellaneous basic wastes, steam condensates, and cooling water were received in the decontamination process feed tanks. These neutral and basic wastes were mixed with the decanted solution from the previously described batch precipitation process and the filtrate from the rotary drum vacuum filter. These combined wastes, now basic from previous neutralization processes, were drained by gravity into a flash mixer tank where the decontaminating chemical reagents were introduced. The ferric ion of the ferric sulfate reagent combined with the hydroxide ions of the basic waste solution to form a ferric hydroxide floc. Residence time in the vessel provided the proper conditions for good precipitation and particle growth. The mixture, still in suspension, gravity flowed from the flocculator into the clarifier.

The clarifier was a non-agitated vessel where the previously formed floc was allowed to settle out, thus removing the radioactive contaminants from the solution. A slow-moving rake revolving on the clarifier floor gathered the precipitate to the slurry takeoff drain. The clarifier supernatant liquid overflowed, through a launder trough located near the top of the clarifier, to the clarifier effluent pump. This pump moved the effluent to the second-stage High Nitrate Receiving and Treatment tanks.

### 3.1.4 Aqueous Waste Solidification Process

Aqueous waste containing complexing agents known to be detrimental to the radioactive decontamination process and aqueous wastes containing certain radioactive isotopes or hazardous chemicals that were undesirable in the regular waste system were entered into the solidification process. The bulk of these wastes were received as package shipments. Specially prepared waste shipping drums, containing a mixture of Portland cement and an absorbent material, were attached by an o-ring drum liner to the solidification glovebox.

Packaged acid wastes were emptied by vacuum into the HCL receiver and neutralizer tank adjacent to the glovebox. Sodium hydroxide reagent was added until the color indicator indicated the contents of the tank to be basic. The neutralized waste was drained by gravity into the glovebox, where a connecting hose directed the waste into the prepared drum. An absorbent material in the drum helped to distribute the waste solution throughout the Portland cement. The cement chemically reacted with the waste solution to form a solid. Basic wastes were poured directly into the prepared drum with no pH adjustment.

## 3.2 Second-Stage Processes

The Second-Stage processes included two separate radioactive waste decontamination systems. A batch precipitation system was used to remove radioactive materials from wastes containing both radioactive and chemical contaminants in excess of DOR standards at the time. A continuous precipitation system was used to remove radioactive materials from wastes meeting the standard for chemical, but not radioactive, contaminants.

The benefit realized by segregation was better utilization of the waste-storage ponds. Wastes meeting the drinking water standards could be stored in unlined earthen ponds. Wastes that met the radioactive contaminant standards for on-site storage, but not the drinking water standards for the chemical contaminants, had to be stored in the asphalt-lined ponds.

Both processes utilized the ferric hydroxide carrier precipitation method of decontamination. The slurries generated by both processes were fed to the Second-Stage Rotary Drum Vacuum-Filter for liquid/solid separation. The filtrate from the filter was returned to the batch process system for further treatment. The solids were collected in specially prepared waste shipping drums. The solids taken off of the filter contain

approximately 70% water by weight. Dry Portland cement was added to the shipping drum, as it was filled, to absorb any free water.

### **3.2.1 Batch Radioactive Decontamination Process**

The treated effluent from the First-Stage processes, filtrate from the Second-Stage Vacuum Filter, liquids from the waste treatment process drains, and other plant wastes requiring radioactive decontamination by this process, were received and processed in the High Nitrate Receiving and Treatment tanks.

When a receiving tank became filled with the various wastes, the tank was isolated from the receiving system and placed in a mixing mode. The appropriate amounts of ferric sulfate and calcium chloride reagents were added and allowed to become intimately mixed with the waste solution. A flocculating agent was added near the end of the mixing cycle to increase the floc particle size. The mixing cycle was then ended and the treated waste remains static until the floc settles to the tank bottom. A rotary dip tube on the head of the tank was then adjusted to decant the clear supernatant liquid into the treated waste holding tanks. The treated waste was sampled, and the resulting plutonium analysis was used to determine its disposition. If the waste met the on-site storage guidelines for radioactivity, it was transferred to asphalt-lined evaporation ponds; otherwise it was reprocessed. The slurry remaining in the bottom of the batch tank was washed into slurry holding tanks. The slurry was processed by the Second-Stage Vacuum Filter.

### **3.2.2 Continuous Radioactive Decontamination Process**

The bulk of waste treated by this process originated from the laundry operation. Laundry waste (water and detergent), and similar low chemical content process wastes exceeding the radioactive standards for pond storage, were directed to the Low Nitrate Waste Storage Tank. The Second Stage Continuous Radioactive Decontamination process was identical in process chemistry to the First Stage. Waste stored in the Low Nitrate Storage Tank gravity flowed to the flash mixer tank. Here, the chemical reagents were added. This tank gravity flows to the flocculator tank, where the flocculating reagent was introduced. The gravity flow continued to the settling tank, where the floc particles were allowed to settle to the tank bottom. The settled solids were transferred through a manually-controlled drain line into the slurry holding tank. The clear liquor in the settling tank flowed into a launder trough around the top of the tank. This liquid pumped to a selected treated waste holding tank. The treated waste was sampled, and the resulting analytical lab data was used to determine its disposition. If the waste met all drinking water standards, it was pump-transferred to the storage pond; otherwise, it was reprocessed. The slurry held in the slurry tank was processed by the Second-Stage Vacuum Filter.

### 3.3 Evaporation Processes

Chemically contaminated wastes stored in the asphalt-lined evaporation ponds were processed through the evaporation system. This system consisted of two feed-storage tanks, a vertical long-tube natural circulation evaporator, a double drum dryer, a dust-scrubber system, and a steam condensate collection system. The total system was used to evaporate water from the liquid waste, leaving a dry solid (10- 15 % water) that was packaged for disposal. The waste was pumped by pipeline from the ponds to the evaporator feed storage tanks.

From these tanks concentrated salt water was continuously circulated through the heat exchanger, along with the waste feed stream. A portion of the concentrate was continuously removed from the evaporator and gravity fed through a pipeline to the steam-heated double-drum dryer. Here the remaining water was evaporated from the concentrate, leaving a film of dry solids baked on the rotating drum surfaces.

The dry solid consisted of approximately 60% sodium nitrate, 30% potassium nitrate and 10% miscellaneous. The miscellaneous is probably water since the salts contained around 9% water which was the limit of double drum dryer (per interview with Mr. Maury Maas a retired Rocky Flats waste treatment manager) when discharged from the evaporator. Small amounts of other cationic materials are also in the dried salts. Knife blades continuously scraped the solids from the dryer drums into catch containers (55-gallon drum). When a container was filled, it was weighed and shipped.

### 4.0 Liquid Organic Waste Treatment

Organic Liquid wastes were comprised of a variety of oils and solvents used in manufacturing operations. The organic waste treatment process consisted of blending the organic liquids with an absorbent powder (calcium silicate) to form a putty-like solid suitable for shipment. The blending operation took place in a blender inside a glovebox. The blender intimately blended the liquid organic waste with the calcium silicate and discharged the resulting solid into a drum attached to the glovebox by an o-ring bag.

During the time period around 1968 a large number of 55 gallon drums containing stored waste organic liquids were processed. The empty drums resulting from this processing were closed and disposed of as empty drums. Many of these contained drums solidified organic sludge that adhered to the drum and was not fed to the liquid waste treatment process. This resulted in "empty" drums that had a tare weight above that for a truly empty 55-gallon drum.

### 5.0 Disposal and Shipment

Waste drums and boxes were transferred from the Building 74 Liquid Waste Treatment Facility and solid wastes from other facilities to building 664 for loading either on trucks or railcars for shipment to the disposal site. A manifest sheet was generated for each shipment showing the number of drums, weight of the drums and a general description of contents. The detailed IDC was not included on the manifest, but instead the designation

as combustible, non-combustible, graphite, empty drum, or sludge type was usually listed. Waste was delivered to the Radiological Waste Management Facility within the Idaho National Engineering Laboratory and buried at Pit 9. Trucks were backed up to the edge of the pit and dumped. The dumping location was determined by either pacing off the distance from survey monuments or by estimating the distances from the monuments.

A caterpillar tractor pushed dirt over the drums while spreading them out. This probably compromised the integrity of the drums, but was not an issue since the wastes were not planned for retrieval and the drums would eventually fail. This was a typical landfill operation of the 1960's.