

## 8.0 OPERATIONAL HISTORY OF BUILDING 771

### 8.1 INTRODUCTION

Building 771 was one of the first four major buildings to be constructed and placed in operation at the Rocky Flats Plant (RFP). For the first few years of RFP operation, Building 771 was the primary facility for plutonium operations. These operations included production of plutonium parts and recovery of plutonium from recycled materials and residues. The final product of this recovery operation was high-purity plutonium metal for use in casting and manufacturing operations (Crisler, 1992).

By the mid-1950s, it was clear that the space within Building 771 was inadequate to support all plutonium operations needed at RFP. This decision was partly prompted by the need to support a new weapons design which required more plutonium than the original weapons. Further, the plutonium shapes in this new weapon required more machining to achieve the necessary specifications. In addition, increased plutonium recovery operations were expected, partly due to the new weapons design. This expansion was known as the Part IV expansion, and included two major production buildings to support plutonium casting and fabrication operations. These buildings are now known as Buildings 776 and 777.

Plutonium part production in Buildings 776/777 began in 1957 when these buildings became operative although much of the production and fabrication equipment for plutonium remained in Building 771. In this manner "backup" plutonium production capabilities existed at RFP (Strangfeld, 1993). From 1957 onward the mission of Building 771 focused primarily on plutonium recovery.

The operations occurring within Building 771 prior to the 1989 shut-down included: (1) the chemical and physical operations for recovering and refining plutonium metal; (2) plutonium chemistry and metallurgical research operations; and (3) radiochemical analytical laboratory operations (EG&G, 1991). The storage of plutonium within the building has also been an essential feature of the building's activities since operations began.

This facility was placed in a curtailed mode of operation in November 1989 due to operational safety concerns. This curtailed mode of operation includes maintenance of the safety envelope

but no processing operations. Since January 1992, Building 771 has been in preparation for decontamination and decommissioning activities (Chew and Associates, 1992).

## 8.2 HISTORICAL TIMELINE

- 1951 Construction began on Building 771 in November (Buffer, 1993).
- 1952 Building 771 was occupied (Buffer, 1993).
- 1953 The first operations began in Building 771 in July (Epp, et al., 1957a)
- 1957 On September 11, a major industrial fire involving plutonium occurred within Building 771 (ChemRisk, 1992).
- 1958 A plutonium recovery incinerator began operation within Building 771 (Buffer, 1993).
- 1959 The solvent extraction process for plutonium recovery was replaced with the anion exchange process for plutonium recovery (Crisler, 1992).
- 1963/64 During this time period, the building was expanded to increase production. This expansion included the construction of new offices on the eastern portion of the northern wall of the building to accommodate a conference room, a new cafeteria, expansion of process operations into Room 114, and conversion of the original laundry area in Room 124 into additional locker room space (Chew and Associates, 1992). The addition containing the new offices, conference room and new cafeteria is known as Building 771A (EG&G, 1991).

Most of the expanded process operations in Room 114 had previously occurred in Room 149 of Building 771, although at a reduced scale. The new lines in Room 114 consisted of an americium recovery line, dissolution lines, filtrate recovery operations, batching operations, calcination operations, and fluorination operations (Weaver, 1994b).

- 1967 An office expansion was added to the western portion of the northern wall of the building (Chew and Associates, 1992). This addition is known as Building 771B (EG&G, 1991).
- 1970 Construction of a concrete block addition to the west side of the building for consolidation of all the maintenance, pipe, sheet metal and painting activities was completed (Chew and Associates, 1992).
- 1971 Construction of a drum-handling facility joining Building 771 to Building 774 was completed (Chew and Associates, 1992). This drum-handling facility is known as Building 771C or the Building 771 Annex (EG&G, 1991).
- 1974 The plenum system was upgraded (Crisler, 1992).
- 1979 Building 771 was shut down (Crisler, 1992) because Building 371 operations were expected to fulfill the plutonium recovery need of the RFP. Safety and cleanup operations for Building 771 began (Crisler, 1992).
- 1980 Operations restarted within Building 771 (Crisler, 1992) due to Building 371 material accountability problems.
- 1984 A plenum upgrade/replacement occurred (Crisler, 1992).
- 1986 Hydrofluorination upgrades began (Crisler, 1992), but these upgrades were never finished.
- 1989 Plutonium operations in Building 771 shut down in November as part of an overall plutonium operations shut down ordered by DOE.

### 8.3 PHYSICAL BUILDING DESCRIPTION

Building 771 is currently a one and two story structural-frame building of reinforced concrete construction and covers an area of approximately 300 feet by 260 feet. The second floor area dimensions are approximately 280 feet by 200 feet. The second floor area houses the ventilation equipment, chemical make-up operations, and miscellaneous storage facilities (Crisler, 1992).

The original Building 771 was a two-story, reinforced concrete structure partially buried in a hillside in the north-central part of the plant site (Figure 8-1). Except for the exposed north wall, the sides of the building are only slightly above the finished grade of the hill (EG&G, 1991).

Buildings 771A, 771B and 771C are major additions that were made to the original Building 771. Currently, Building 771A, located on the eastern portion of the north side of Building 771, contains a cafeteria and offices. Building 771B includes additional offices and is also located on the north side of Building 771. Production operations have never been conducted in Buildings 771A and 771B. Building 771C, commonly referred to as the Building 771 Annex, is on the east side of Building 771 and currently contains the shipping, receiving and drum radiological counting area. Other additions have been made to Building 771, but these additions have not been given building numbers. These additions include a loading dock and the maintenance shop (EG&G, 1991). For the purposes of this document, the term "Building 771" is considered to include references to Buildings 771A, 771B, 771C and other additions to the building.

Plutonium is handled in most of the Building 771 operations. These operations are conducted within gloveboxes for primary containment of the radioactive materials. The design of the ventilation system further provides for air flow from areas where plutonium is not handled ("cold areas") to areas where plutonium is handled ("hot areas"). This is accomplished, in part, by supplying air to the cold areas and exhausting air from the hot areas (Crisler, 1991). Room air is drawn into gloveboxes through high efficiency particulate air (HEPA) filters (EG&G, 1991). Air exits the gloveboxes through the following: (1) a HEPA filter; (2) a booster exhaust plenum provided with a minimum of four stages of HEPA filtration; and (3) the main exhaust plenum which is provided with two additional stages of HEPA filtration (Crisler, 1992).

Building 771 does not have a separate process air vent system that specifically handles fumes from the chemical process area. However, individual fume scrubbers are typically used where acids or other noxious fumes require neutralization. Most of the operations involving heated acids are provided with a condenser unit near the operations in addition to offgas treatment with a fume scrubber. The condenser unit typically consisted of a heat exchanger chilled by a countercurrent flow of water. The water is chilled, providing for condensation of a majority of the acid entrained in the exhaust stream (EG&G, 1991).

A large fume scrubber is used to draw acid fumes from dissolver pots, evaporators, calciners, and vacuum systems. This large fume scrubber consisted of two packed media scrubbing towers in series, in which the acid-laden air is contacted by refrigerated potassium hydroxide passing counter to the air flow. Potassium hydroxide is collected in sumps at the bottom of the towers and recirculated through chillers and back to the top of the towers (EG&G, 1991).

#### **8.4 DESCRIPTION OF OPERATIONS**

The description of operations for Building 771 is divided into three sections: (1) historical operations (1952-1957), (2) recovery operations (1952 - 1989), and (3) support operations. Historical operations include the original mission of Building 771 as the sole plutonium processing facility at the RFP. Plutonium and americium recovery operations from the opening of the building to the 1989 production curtailment are discussed in detail in Section 8.4.2 of this report. These operations are discussed in detail because they were the major operations within Building 771 during most of its active production period. The discussion in the support operations section focuses on several laboratories which operated in the building. Additional information regarding many of the unit operations described in the following sections can be found in the "Plutonium Processing Materials Data Book" (Thompson, 1972).

##### **8.4.1 HISTORICAL OPERATIONS (1953 - 1957)**

Building 771 was originally constructed as a totally self-contained plutonium fabrication and reprocessing facility. For the period of May 1953 until 1957, when Building 776/777 entered operation, Building 771 was the plutonium handling facility for the RFP. During this time period the building housed the following plutonium part production-related activities: casting, fabrication (machining), coating, inspection, and testing, recovery operations, storage of plutonium metal, various laboratories, and other support operations (Chew and Associates, 1992; Strangfeld, 1993). The plutonium-related operations in Building 771 were arrayed along the southern hallway of the first floor of Building 771. Plutonium manufacturing operations were located on the south side of the south hallway while plutonium recovery operations were located on the north side of the south hallway. Only limited information is available on the original casting, fabrication, and other support activities that took place in Building 771. That information is presented below.

- Casting operations were conducted in the foundry in Room 182 (Chew and Associates, 1992).
- Plutonium machining facilities were located in the eastern portion of Room 182 (Chew and Associates, 1992; Strangfeld, 1993).
- Plutonium reprocessing operations were located in the east and west chemical glovebox lines located in Rooms 146 and 148, respectively. A process control room was located in Room 147, and a recovery process area in Room 149. Room 149 accommodated residue recovery using dissolution and a solvent extraction process (Chew and Associates, 1992). Details of the recovery process are discussed in detail in Section 8.4.2. These operations remained largely the same during active operations in Building 771.
- Radiography was located in Room 184.
- Plutonium metal was stored in Rooms 187 and 188 (Chew and Associates, 1992).
- Chemistry and Metallurgy Development Laboratories were located in rooms that are currently designated 172, 174, 175, 176, 178, 179, and 180 (Chew and Associates, 1992).
- Analytical Support Laboratories were located in the building in rooms that are now designated 151, 152, 153, and 157, as well as Rooms 154 through 156, and Rooms 158 through 169 (Chew and Associates, 1992).
- Laundry facilities were located in the northwest corner of the building in an area that is now part of Room 123 (the locker room). The laundry was located in roughly the northeast corner of the current locker room (Chew and Associates, 1992).
- Maintenance shops are known to have existed during this time, but their exact location is not currently known (Chew and Associates, 1992).

- Locker rooms were located in the general location of the current locker rooms for the building, but the original locker rooms were smaller and occupied only the northwestern one-third of the current area (Chew and Associates, 1992)

Offices, a cafeteria, a tool room, and a stock room were located in areas currently occupied by Rooms 101, 102, 106, 114, 120, 122, 127, 133, 135, and 141 through 144 (Chew and Associates, 1992).

Many of the plutonium operations were moved to Buildings 776/777 in 1957. On September 11 and 12, 1957, a fire occurred that caused considerable damage to Building 771 and considerable radiological contamination of areas inside and outside of the building. Although this was a serious fire, no injuries were reported (Epp, et al, 1957a), and the fire debris was cleaned up by 1958 (Barrick, 1989). Because of the damage caused to Building 771, and due to the impending completion of Buildings 776/777, some of the plutonium production operations started in Buildings 776/777 immediately following the Building 771 fire (Epp, et al, 1957a).

#### 8.4.2 RECOVERY (1953-1989)

Plutonium and americium recovery operations were conducted in Building 771. The following discussion primarily addresses plutonium recovery operations because the majority of the building was dedicated to this operation after 1957.

##### 8.4.2.1 Plutonium Recovery Operations

The overall process and chemistry of plutonium recovery at RFP remained largely unchanged since plutonium recovery operations began in Building 771 (Crisler, 1992). However, a number of the individual unit operations were significantly changed. Also, the floor space and equipment devoted to plutonium recovery in Building 771 was considerably increased.

Plutonium recovery operations in Building 771 were originally conducted in a batch fashion which consisted of simple manually operated equipment. At that time, batch operations were sufficient because little scrap was generated by the limited plutonium casting and machining operations. Similarly, site returns of retired or out-of-specification nuclear weapons or nuclear weapons components were minimal. Continuously operating and automatic control systems were

later introduced to increase the recovery capacity of the facility and to decrease radiation exposure of operating personnel.

The following discussion begins with the residue preparation process which prepares feed for the dissolution operations and flows through dissolution and leaching, ion exchange and conversion to metal. Other recovery-related operations including plutonium oxidation and incineration are then discussed.

#### **8.4.2.1.1 Residue Preparation**

Plutonium residues that underwent dissolution typically consisted of internally generated plutonium including scrap metal, plutonium residues associated with process-generated materials, and skull oxide from foundry operations (Crisler, 1992). Plutonium scrap metal was typically oxidized prior to dissolution operations (See Section 8.4.2.15). Some processing of plutonium-bearing liquids that originated at other DOE facilities was also performed in the 1950s. A brief discussion of these plutonium-bearing liquid feeds is provided below.

The first plutonium-bearing solutions were received from the Hanford Plant in Richland, Washington in 1953 (Crisler, 1992). Shipments of liquid plutonium solutions from Hanford were discontinued in 1959 (Crisler, 1992). After this time, other DOE facilities shipped solid plutonium materials, typically plutonium oxide, to RFP.

Until 1962, one of the feeds for dissolution operations was plutonium metal from foundry operations. This metal, which was called casting skull, had varying amounts of surface oxidation. Its reaction with the nitric acid was erratic, varying from slow to violent. In 1962, the operation was modified to eliminate this unpredictable behavior. Starting at this time the metal was completely oxidized prior to dissolution. This change also eliminated problems related with the storage of pyrophoric metal since the metal was oxidized as quickly as possible (Crisler, 1992).

**Crushing and Grinding.** Sand, slag, and crucible residues generated from the plutonium reduction process at RFP were reprocessed for plutonium recovery. These materials were primarily composed of calcium fluoride and calcium, along with traces of magnesium oxide, plutonium tetrafluoride, plutonium dioxide, and varying amounts of plutonium metal beads. The materials were broken into small pieces in a jaw crusher and then ground to a fine powder in a hammer mill. A disc-type grinder was also sometimes used for size reduction operations.

Following these operations, the sand, slag and crucible was in a form suitable for feed to the dissolution process.

**Glovebox HEPA Filter Processing.** Prior to discharge to the Building 771 plenum system, glovebox exhausts passed through HEPA filters installed at the glovebox. During active production when HEPA filters were changed out, the plutonium particulates on the used filter were knocked off by tapping the filter on the floor of the glovebox. The plutonium was then recovered and reprocessed. The used filter was disassembled to separate the wooden frame from the filter medium. The wood was discarded and the filter medium was held for further processing (EG&G, 1991).

#### **8.4.2.1.2 Residue Dissolution and Leaching**

**Dissolution.** Prior to 1965, dissolution was a batch-type operation performed in Room 149 in beakers of 2- to 4-liter capacity, which were heated by electric immersion heaters. The mixture of nitric acid and plutonium residues was agitated by the boiling action caused by the heater, an air sparge, mechanical agitation or a combination of these methods (Crisler, 1992). Typical residue feeds to dissolution consisted of sand, slag and crucible, graphite, plutonium fluoride, and feed heels.

In addition to being very labor intensive, the beaker method had other significant problems. The fumes from the many dissolution beakers corroded the electric heater connections and presented problems for the glovebox air-handing and filtration systems. The efficiency of dissolution was poor, primarily because of incomplete suspension of the solids in the acid solution. This type of dissolution was later used only for special materials or for systems with a low throughput (Crisler, 1992).

To improve dissolution, the batch pots were replaced in 1965 with a system of continuous cascade dissolvers (1965-1971). Additional space was also devoted to dissolution at about this time with the expansion of dissolution operations into Room 114 as well as Room 149. The continuous cascade dissolver system consisted of air-sparged dissolver vessels in series, overflowing by gravity from one to the other. The feed mechanism was a laboratory-sized vibratory feeder. Serious problems existed with this cascade system. The suspension of solids was incomplete. A buildup of solids, termed "heel," would slowly fill the vessels, rendering them inoperable. In order to achieve even minimal solid suspension, high air flow rates were required.

This caused the glovebox ventilation system to be overwhelmed. In addition, the vibratory feeders were subjected to transient equipment vibrations and fed erratically. This required that they be continuously monitored. These deficiencies led to the design of the air-lift with screw feeder dissolvers which were in use from 1971 to the end of process operations in 1989 (Crisler, 1992).

The first set of air-lift dissolvers was installed in 1971 and continued in use until 1989. High-level feeds consisted of various types of plutonium oxide and oxide heels. Incinerator ash and ash heel feeds were processed through the late 1970s, but because of off-gas silicon dioxide plugging problems, process-scale ash dissolution operations were discontinued and ash stockpiled since that time (Crisler, 1992). However, pilot-scale work was conducted in the 1980s to try to find a solution to the off-gas silica dioxide plugging problem (Weaver, 1994a).

In recent operations, both plutonium oxide and plutonium-bearing residues were processed in dissolution lines containing a series of air-lift screw feeder dissolver vessels. Plutonium-bearing residues normally processed included impure oxide, impure fluoride, impure green cake, oxide heel, sand, slag, and crucible (EG&G, 1991). These materials were fed into the first dissolver at a controlled rate by a special screw feeder. Nitric acid, aluminum nitrate, calcium fluoride and water were also fed into the first dissolver at a controlled rate. The solution in the cascade dissolvers was heated to approximately 100° C by internal steam coils. The slurry overflowed (cascaded) from the first to the last dissolver in the set. From the last dissolver, the slurry overflowed to a filter, which separated the undissolved solids from the solution (EG&G, 1991). Agitation within the dissolvers was accomplished by internal air lifts which helped to keep the solids in suspension (Crisler, 1992).

**Laboratory Residue Processing.** Laboratory residue solutions were transferred to plutonium recovery in 4-liter bottles. These solutions contained plutonium from samples sent to analytical laboratories mixed with chemicals used in the analyses. These solutions contained complexing agents that adversely affected waste treatment processes and chloride ions that could be detrimental to process equipment. These residues were introduced directly into the recovery process by controlled blending of the laboratory residue with the feed to the cation exchange process. In the ion exchange process, chloride ions were separated from the solutions and the solutions go through normal plutonium recovery operations (EG&G, 1991).

**Oralloy Leaching.** This operation was transferred to Building 771 in approximately 1964 from Building 881 and took place in Room 174 area (Weaver, 1994). Oralloy materials consisted of enriched uranium. Oralloy residues were processed by selectively leaching pieces of scrap metal in a spray dissolver to allow oralloy metal to be recycled directly to the Oak Ridge, Tennessee DOE facilities without being subjected to chemical recovery. A mixture of sulfuric acid and nitric acid was used as the leaching solution to remove contamination present in the oralloy residues. Dissolved uranium and plutonium from the leaching process were ultimately precipitated from the solution and further processed in the special recovery area, Room 146 (EG&G, 1991). The uranium and plutonium present in the leaching solution were precipitated by sparging gaseous ammonia through the solution. The ammonia raised the pH of the solution, causing oxides of uranium and plutonium to precipitate.

**Part V Leach Process.** Part V Leach operations took place in Room 114. Special material residues, called Part V residues, were processed separately from the other plutonium residues. This process consisted of selectively leaching large pieces of scrap metal in a spray dissolver to remove impurities from the plutonium metal so that it could be recycled directly to MSE without going through chemical recovery (EG&G, 1991). A mixture of sulfuric and nitric acid was used as the leaching solution. The solution from the leaching operation was sent to ion exchange (EG&G, 1991).

#### 8.4.2.1.3 Ion Exchange

The ion exchange process purified and concentrated plutonium-bearing solutions to make them acceptable as feed for the conversion to metal process. The solutions resulted from (1) residue dissolution and leaching processes, (2) solution recycle from conversion to metal processes, and (3) solutions transferred from laboratories (EG&G, 1991).

Ion exchange equipment consisted of two separate gloveboxes and sets of tanks. These two systems were identified as the recycle recovery systems. Solutions were adjusted as batches to a set acid normality using nitric acid. Ferrous sulfamate and aluminum nitrate were added to adjust the valence of plutonium and to complex any fluoride present from the dissolution operation. The adjusted solution was pumped through sets of three columns in series containing nitrate-form resin. The plutonium was retained on the resin and the plutonium-depleted solution (ion column effluent) was collected for shipment to Liquid Waste Treatment Operations in another building. The columns were washed and eluted with nitric acid. The eluate was

transferred to the feed preparation process for conversion to metal. Ion columns were reconditioned for the next loading using a nitric acid wash. It was necessary to recycle some solution from the initial part of the elution and reconditioning steps. A separate set of cation exchange columns was used to remove chloride from laboratory residue solutions. The chloride waste solution was transferred to Liquid Waste Treatment Operations (EG&G, 1991).

This process replaced the historical solvent extraction process located in Room 149 which utilized tributyl phosphate (TBP) in a batch process (Crisler, 1992; Chew and Associates, 1992). This original batch solvent extraction process had been replaced by a small, continuous mixer-settler system in 1954, and the entire solvent extraction process was replaced in 1959 by the ion exchange system (Crisler, 1992).

#### **8.4.2.1.4 Conversion to Metal**

The operations associated with converting liquid feed solutions to plutonium metal included feed evaporation, batching, peroxide precipitation, calcination, hydrofluorination, reduction and button breakout. These processes are described below.

**Feed Evaporation.** This process was used to concentrate plutonium solutions originating from previous operations. Concentration of these solutions was necessary in order to have an acceptable plutonium concentration in the feed. Solutions selected to be concentrated were analyzed for plutonium concentration, normality, and iron concentration. From these data, the optimum degree of evaporation was determined. The solution was concentrated in a steam-heated, natural-convection evaporator. The concentrated solution, called "bottoms," was transferred as a batch to tanks prior to use in the batching operation (EG&G, 1991).

Feed evaporation was added to plutonium recovery operations in 1964. Prior to 1964, high-level plutonium solutions from dissolution were mixed with low-level solutions from anion exchange or diluted with nitric acid to obtain the desired precipitation feed concentration (Crisler, 1992; Weaver, 1994b).

In 1964, a feed evaporation unit began operations in Room 114 utilizing a single steam chest, thermosiphon-type evaporator in a semi-batch mode of operation. This system remained in operation until August 1988, when a new double-chest thermosiphon evaporator was installed along with a new controller unit. Prior to the installation of the new evaporator, the only changes

to the single-chest unit were upgrading of methods to calculate the amount of liquid to be boiled down (Crisler, 1992).

**Batching.** This process remained relatively unchanged after the batching line was moved from Room 149 to Room 114 in 1964 (Crisler, 1992; Weaver, 1994b). Relatively pure plutonium nitrate solutions received from oxide dissolution, ion exchange and feed evaporation were blended and adjusted to the proper pH and plutonium concentration before transfer to the peroxide precipitation process. Feed for this process was prepared in batches by blending the available solutions in the proper ratios in two tanks (EG&G, 1991). Sulfuric acid was also added to aid crystal growth in peroxide precipitation. Batches were mixed by circulation with a pump or by air sparging. The batch was sampled and analyzed for normality, impurities, and plutonium concentration. If the batched feed was within the prescribed concentration limits, it was transferred to the feed holding tank. If it was not, adjustments were calculated and solutions were added as needed (EG&G, 1991).

**Peroxide Precipitation.** The peroxide precipitation process converted the plutonium in solution to a solid form. The original precipitator for use in peroxide precipitation was made of a plastic or lucite material and had a volume of 5 to 8 liters, and was located in Room 149. The vessel contained both an agitator and a slinger plate used to distribute peroxide evenly into the vessel. Cooling coils along the vessel walls maintained a temperature of approximately 4°C during precipitation. A total of four precipitations were completed each day (Crisler, 1992).

For precipitation, hydrogen peroxide was added at a varying rate over the course of approximately one hour. The slurry was then allowed to digest. After digestion, the precipitate was pulled by vacuum through a dip tub in the precipitator vessel to a filter "G" boat. The filter boat was made of platinum-lined monel with a sintered-platinum fritter filter. The filtered precipitate was washed with water, then allowed to air dry. The precipitate was then transferred to a "G" furnace station where it was dried overnight. The dried precipitate cake was fluorinated the next day with anhydrous hydrogen fluoride gas. Peroxide remaining in the filtrate was destroyed with sodium hydroxide. During this period, the precipitate cake often decomposed while in the drying cycle (Crisler, 1992).

Starting in 1954, the precipitate cake was washed with denatured alcohol, eliminating plutonium peroxide decomposition. A major problem at this time was the reaction of hydrogen fluoride and

alcohol within the "G" furnace. An incident occurred in which the furnace pressurized and blew oxide out into the room. The use of alcohol was then terminated (Crisler, 1992).

On June 14, 1957, an explosion during the peroxide digestion cycle blew out the front panel of the glovebox. The primary cause of the incident was the catalytic decomposition of hydrogen peroxide. Shortly after, 35 weight percent hydrogen peroxide replaced the 50 weight percent hydrogen peroxide that had been used earlier. This method was used until the development of the continuous plutonium peroxide precipitation process in the 1960s (Crisler, 1992).

The entire continuous precipitation method was moved to Room 114 in 1964. The basic design remained similar to the methods used in Room 149. Major differences included the addition of a third digester. At about this same time, a calcination step, as opposed to the drying step identified above, was initiated (Crisler, 1992).

In recent operations, the feed solution and hydrogen peroxide were fed into a refrigerated, stirred reactor called a digester. Precipitation occurred and crystal growth began. The plutonium peroxide slurry cascaded through the digesters and into the rotary drum filter basin. A vacuum applied to the filter caused the plutonium peroxide to collect on the rotating filter drum surface. The plutonium peroxide cake which collected on the rotary drum filter, was automatically sliced off the filter wheel, collected in containers and transferred to the calciner (EG&G, 1991).

Filtrate was collected in a set of two receivers. The filtrate was fed to a steam-heated evaporator for conversion of the residual hydrogen peroxide into water and for concentration of the solution. This evaporation unit was referred to as the peroxide kill evaporator. The evaporator distillate was held for shipment to Liquid Waste Treatment Operations. Concentrated evaporator bottoms were transferred to tanks for sampling and analysis prior to transfer to anion exchange (Weaver, 1994b).

**Calcination.** The calcination process converted plutonium peroxide to plutonium oxide and drove out residual water and nitric acid, leaving a dry, powdered product. This process took place in an electrically heated rotary-tube furnace. The plutonium peroxide cake was fed into the heated tube by a feed auger drawing from a feed hopper. As material passed through the tube, it was dried by a counter-current flow of air that swept the acid fumes and some light particles into a solids scrubber (EG&G, 1991).

The solids scrubber used circulating nitric acid as the scrubbing solution. The calciner tube atmosphere was at a negative pressure compared to the glovebox. The solids scrubber solution dissolved most of the trapped solids. The solution was filtered and transferred to anion exchange. The dried plutonium oxide was collected, screened, weighed into batches, and transferred to hydrofluorination. The off-gas from the nitric acid scrubbing operation was passed through six HEPA filters in series (EG&G, 1991).

**Hydrofluorination.** Hydrofluorination was first conducted in Room 146 and 149 of 771. Originally, a batch of precipitated plutonium peroxide was filtered, calcined and hydrofluorinated using the same platinum-lined boat. Continuous peroxide precipitation and calcination were added in 1958, but the G-furnaces and platinum-lined boats continued to be used for hydrofluorination until a continuous hydrofluorinator was installed in 1964. A new continuous rotary hydrofluorinator, glovebox and support equipment were installed in 1980-1981 (Crisler, 1992).

In recent operations, plutonium oxide was converted to plutonium tetrafluoride in a continuous rotary-tube hydrofluorinator with a screwfeeder supply for the plutonium oxide feed. Plutonium tetrafluoride was formed by counter-current contact with hydrogen fluoride gas inside the rotating tube. The tube had three separate temperature zones for efficient production of plutonium tetrafluoride and for the removal of impurities such as sulfur from the feed. The plutonium tetrafluoride product was collected, weighed, and transferred in batches to the reduction process (EG&G, 1991).

**Reduction and Button Breakout.** This process consisted of placing plutonium tetrafluoride in a magnesium oxide crucible, which was surrounded with magnesium oxide sand. A 30 percent excess of the stoichiometric equivalent of calcium was added to the crucible. Initiators were also placed in the crucible which was inerted with argon, sealed, and heated in a reduction vessel. This process was conducted in an inert, nitrogen-filled glovebox for fire protection. The initiator, which was made of potassium iodate, magnesium metal, and sodium peroxide, started an exothermic reaction at approximately 150° C. Temperatures in the crucible rose to approximately 2,000° C. During the reaction, the magnesium oxide sand acted as a heat sink, protecting the glovebox from temperature and pressure spikes (EG&G, 1991). In this reaction, pure plutonium metal buttons were formed.

After cooling, the crucible was opened and the plutonium button removed. The slag left in the crucible and the crucible were recycled through brushing/grinding and transferred to the dissolution process for plutonium recovery as sand, slag and crucible material (EG&G, 1991). The sand, slag, and crucible material included calcium fluoride and calcium, along with traces of magnesium oxide, plutonium tetrafluoride, plutonium dioxide, and varying amounts of plutonium metal beads.

The reduction operations remained largely unchanged since metal reduction operations began at RFP, with the exception of the introduction of initiators in 1972/1973 and increasing plutonium charges being introduced to the reduction vessel (Crisler, 1992).

#### **8.4.2.1.5 Plutonium Oxidation**

Plutonium oxidation converted pure plutonium metal, which was pyrophoric, to a more stable plutonium oxide. The plutonium oxide was then used as a feed to the dissolution operation. The source of plutonium for oxidation was solid pieces, such as buttons from recovery, flashing from button breakout, and drill shavings from button sampling. Oxidation of the metal was conducted in tantalum metal pans on electrically heated hot plates in a glovebox dedicated to this purpose (EG&G, 1991).

#### **8.4.2.1.6 Incineration**

Large quantities of combustible residues contaminated with small amounts of actinides, primarily plutonium and americium, were generated when RFP produced nuclear weapons components. One method to allow for recovery of plutonium from these materials was to reduce the amount of combustible bulk by incineration. Actinide contaminants remained in the ash, resulting in an ash having a plutonium concentration ranging from 5 to 10 weight percent. This ash was then sent to dissolution for subsequent recovery of the plutonium (Crisler, 1992). The incinerator was constructed in Room 149 (Chew and Associates, 1992) and became operational in 1957 or 1958 (Chew and Associates, 1992; Buffer, 1993).

Combustibles to be used as feed to the incineration process were stored in 55-gallon drums. The contents of one drum was emptied into the incinerator-sorting portion of the glovebox. Contents of the drum were searched for glass, metal and any other items which would not burn or would cause a hazard in the combustion chamber (Crisler, 1992).

The incinerator was comprised of three chambers: a firebox (with approximate dimensions of 2 feet by 2 feet by 3 feet) where combustibles were initially introduced to the system and burned, a main burner chamber where ashes which fell through the firebox grate continued to burn, and an afterburner section (Crisler, 1992). The offgases from the incinerator were treated through an extensive off-gas scrubbing and filtration system to minimize releases to the environment.

#### **8.4.2.2 Americium Recovery**

Plutonium-241, constituting less than one weight percent of the RFP plutonium stream, undergoes decay to americium-241. Equipment for removal and concentration of americium from plutonium solutions was installed in Room 149 in 1957 (ChemRisk, 1992; Chew and Associates, 1992).

Feed for the americium recovery process prior to 1967 originated from the plutonium recovery precipitation filtrate stream, which was stored in a set of tanks external to the gloveboxes. The filtrate from plutonium recovery operations was concentrated by evaporation, the bulk of the plutonium removed by anion exchange, and the americium recovered and purified by a thiocyanate ion-exchange process (Crisler, 1992). Pencil tanks for storage of americium solutions were also located in Room 149, with an americium nitrate solution tank located outside the building to the north (Chew and Associates, 1992).

In 1967, a molten salt extraction (MSE) process was developed to extract americium directly from aged plutonium metal. This MSE process took place in Building 776. Due to the new MSE process, the americium concentration in the plutonium-peroxide filtrate decreased. MSE residues were the only source of recoverable americium after 1968. After dissolution, actinides in the MSE residues were separated from the chloride salt matrix with a potassium hydroxide precipitation step. This precipitation step was later replaced by a cation exchange process (Crisler, 1992).

The americium purified at RFP was sold by DOE for use in commercial applications such as a medical diagnostic tracer and for smoke detector ionization sources. However, the demand for americium dropped off in the late 1970s, making purification and sale of the material uneconomical (ChemRisk, 1992). Americium was declared a waste by DOE in 1986 and no emphasis was placed on the recovery of americium after this date (Crisler, 1992).

### **8.4.3 SUPPORT OPERATIONS**

The major support operations conducted in Building 771 included several laboratories and shipping and counting operations which are described below.

#### **8.4.3.1 Chemical Technology**

Plutonium chemistry technology in Building 771 supported and developed improved methods for recovering, separating, and purifying actinides from acidic streams (EG&G, 1991).

Chemical research and development for actinide element separation and purification was conducted on laboratory-scale, pilot-scale, and production-scale equipment. Projects were conducted for the purpose of testing and gathering basic information. In addition to providing services for production at RFP, work was done in job lots for other DOE facilities, design agencies, and governmental departments (EG&G, 1991).

One of the primary operations studied by this laboratory was dissolution. Thus, nitric acid use by this laboratory was high, but use of other compounds was on an extremely limited basis.

#### **8.4.3.2 Plutonium Metallurgy Research**

The plutonium metallurgy group of Building 771 assisted in the development of processes that required metallurgical production of materials and related manufacturing techniques. Plutonium metallurgy operations consisted of casting, heat-treating, rolling, forming, forging, sizing, and swaging. Supporting operations included metallography, X-ray diffraction, tensile testing, density measurements, and powder metallurgy (EG&G, 1991).

All plutonium metallurgy operations were conducted in gloveboxes. In recent years, the primary emphasis was on plutonium casting operations. Castings were made in ceramic-lined metal molds or ceramic-lined graphite molds (EG&G, 1991).

#### **8.4.3.3 Plutonium Operations Support Laboratory**

Liquid and solid samples were received by or prepared in the Building 771 analytical laboratory. Samples were analyzed for plutonium, americium, uranium, neptunium, and other radioactive

isotopes (EG&G, 1991). Small liquid samples and sludges were prepared for analysis in "B" boxes that could be opened to the room. Sample analysis involved the use of hydrochloric acid, nitrous oxide and hexane in some cases (EG&G, 1991).

#### 8.4.3.4 Shipping and Counting

Shipping, receiving, and measurement of the amount of radioactivity (counting) of containerized materials entering or exiting Building 771 were performed in Building 771C. Building 771C was separated from the remainder of Building 771 by an airlock. Typical operations in Building 771C included temporary storage of materials in containers and counting of individual containers for activity prior to transfer into or out of Building 771. Containers remained closed while in temporary storage, and counting was a non-destructive, non-intrusive activity (EG&G, 1991).

### 8.5 CURRENT CONTAMINATION STATUS

Building 771 is considered hazardous due to the existence of quantities of Special Nuclear Material (SNM) in storage and due to plutonium contamination present in the building (EG&G, 1992a).

Sources of radiation exposure in Building 771 are plutonium and uranium solutions in tanks and bottles; plutonium metal, oxides, and fluorides in vaults; and other residues stored in drums. Exposure levels in the vaults are up to 15 millirem per hour. The average exposure level for chemical operators during curtailment has been 150 - 160 millirem per year (EG&G, 1992a).

Some Building 771 process areas are contaminated with plutonium, ranging from less than 200 disintegrations per minute per 100 square centimeters, to in excess of 1,000,000 disintegrations per minute per 100 square centimeters. The latter is the limit of the detection instruments used by the Radiation Protection Technologists (RPTs) (EG&G, 1992a).

Room 141, an old pump room, is the most heavily contaminated area. The pumps, which were not housed in gloveboxes, were used to transfer plutonium nitrate solutions. The solution leaked and the acidic solution contaminated, corroded, and diffused into the concrete floor (EG&G, 1992a).

Many contamination incidents have occurred in the process areas during the 40 years of processing and storage of plutonium solutions and solids. Decontamination of the working areas was accomplished to the extent possible at the time of the incidents. Contamination which could not be removed was painted over. Thus, there can be contamination within the layers of paint (EG&G, 1992a).

Hazardous wastes present within Building 771 include mixed low-level radioactive and hazardous waste, mixed transuranic and hazardous waste, as well as straight hazardous waste. There are also approximately 72 tanks containing hazardous or mixed radioactive and hazardous waste (EG&G, 1992a).

Building 771 also has appreciable amounts of asbestos insulation, but this material has been identified and wrapped in plastic to await decontamination and decommissioning efforts (EG&G, 1992a).

Because the 1989 curtailment status was initially expected to last for a short period of time, plutonium bearing residues and process operations were stabilized only for short term storage and long term actions were not taken. During the 1989 to 1992 curtailment period, multiple assessments of the building equipment and infrastructure resulted in a decision that the plutonium recovery operations would not resume within this building. At this time, the specific schedules and details concerning the type and magnitude of future operations within Building 771 are not well defined (EG&G, 1992).

## REFERENCES

- Barrick, C.W., 1989, Building 771 Accumulated Fire Ashes Story, Rockwell International Internal Letter to D.S. Hurt. November 9, 1989.
- Buffer, Pat, 1993, Highlights in Rocky Flats Plant History, EG&G Rocky Flats, Inc. February, 1993.
- M. H. Chew & Associates, Inc., 1992, Facility History for Building 771 at the Rocky Flats Plant, Compiled for EM-30 by M.H. Chew & Associates, Inc., Under Contract to BDM International, Inc., Contract Number 06S1004, Prime Contract Number DE-AC001-90EW48063. April, 1992.
- Crisler, L.R., 1992, A History of the Rocky-Flats Plutonium/Actinide Recovery Plant - 1952 to 1991, EG&G Rocky Flats, Production Technology Development Report, RT92-003. February 3, 1992 (February 11 also given as the date of publication).
- EG&G, 1991, Air Pollutant Emission Notice (APEN) Report, Building 771, EG&G Rocky Flats, Inc. February 28, 1991.
- EG&G, 1992, Revision of Building 771 Stabilization Plan, Phase One, May 1992, EG&G Rocky Flats, Environmental Management Department, Air Quality and Chemical Tracking Division, Air Emission Inventory. May, 1992.
- EG&G, 1992a. Draft Mission Transition Program Management Plan, Appendix A-1, Surplus Defense Nuclear Production Facilities Element. Revision 7. October 1, 1992.
- Epp, J.G., B.P. Shepherd, W.R. Cornelison, E.R. Turnquist, R.R. Harrison, H.A. Spavin, 1957a. Report of Investigation of Serious Incident in Building 71 on September 11, 1957, The Dow Chemical Company, Rocky Flats Plant, Denver, Colorado. October 7, 1957.
- Epp, J.G., 1957b, Supplementary Report on Fire in Building 71, September 11, 1957, The Dow Chemical Company, Rocky Flats Plant, Denver, Colorado. December 10, 1957.

Hornbacher, D.D., 1994, Interview with Mr. D.D. Hornbacher, Former RFP Employee. May 24, 1994.

Putzier, E.A., 1982, The Past 30 Years at Rocky Flats. November, 1982.

Strangfeld, D.K., 1993, Interview with D. K. Strangfeld, EG&G Rocky Flats Plant Employee. September 7, 1993.

Thompson, M.A., 1972, The Plutonium Processing Materials Data Book, Dow Chemical Company, Rocky Flats Plant, Golden, Colorado. June 30, 1972.

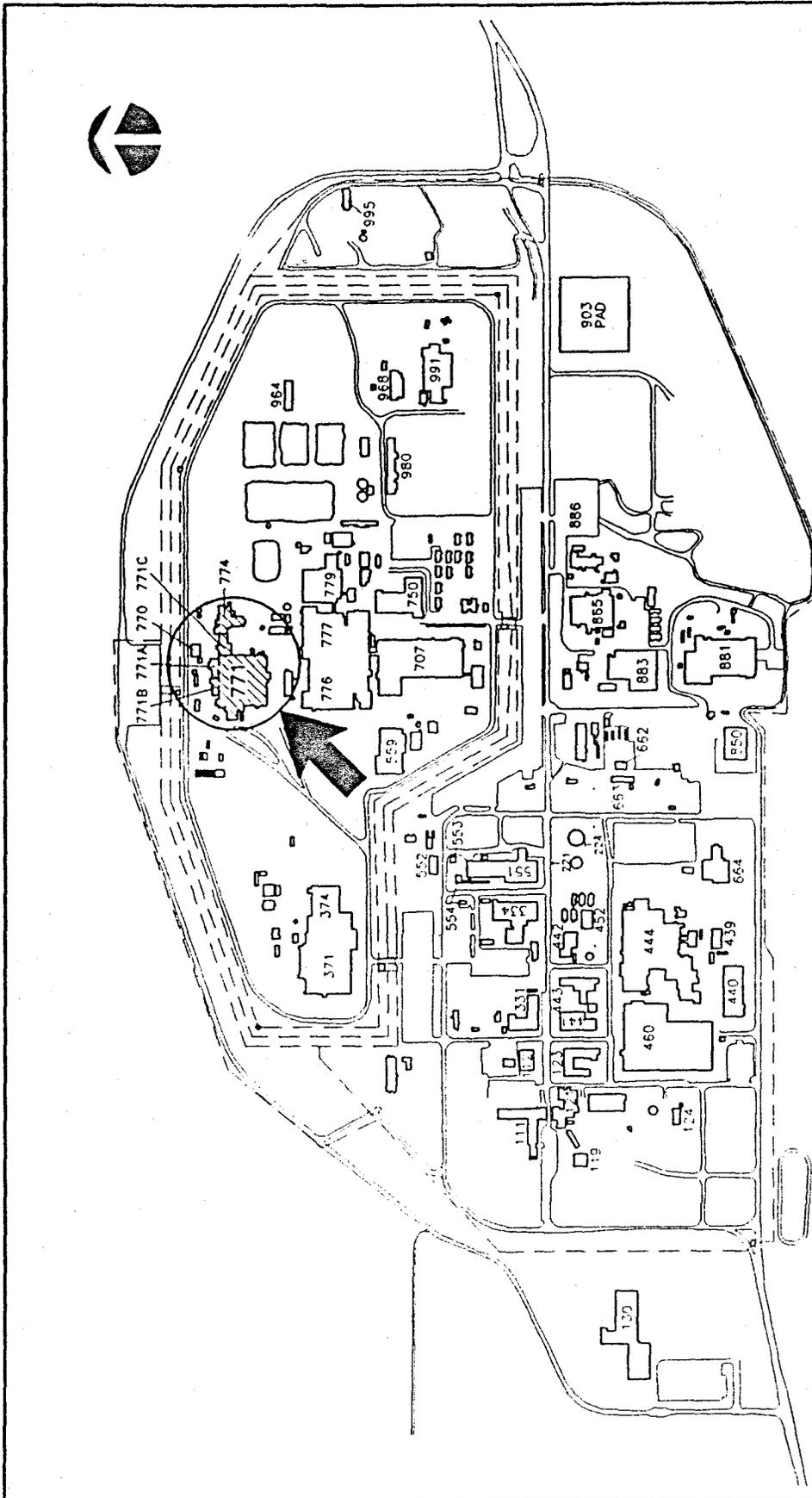
Weaver, J.D., 1994, Interview with J. D. Weaver, EG&G Rocky Flats Plant Employee. June 9, 1994.

Weaver, J.D., 1994a, Interview with J. D. Weaver, EG&G Rocky Flats Plant Employee. September 14, 1994.

Weaver, J.D., 1994b, Interview with J. D. Weaver, EG&G Rocky Flats Plant Employee. September 19, 1994.

931-082\000cb\771.hrr

**FIGURES**



		<b>FIGURE 8-1</b>	
		<b>BUILDING 771</b>	
PREPARED FOR <b>U.S. DEPARTMENT OF ENERGY</b> ROCKY FLATS PLANT GOLDEN, COLORADO			
BY <b>WRIGHT WATER ENGINEERS, INC.</b> 2400 W. 26TH AVE. SUITE 100A DENVER, CO 80211 (303)480-1700			
PROJ. NO.	931-082.000	DWG. NO.	-
DESIGN BY	JKC	CHECKED	JKC
DRAWN BY	KAL	APPROVED	-
DATE	NOV. 28, 1994	SCALE	1" = 900'

24  
24