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CHEMISTRY RESEARCH AND DEVELOPMENT
PROGRESS REPORT FOR
DECEMBER 1978 THROUGH MAY 1979

Frend John Miner

MASTER

Rockwell International

Energy Systems Group
Rocky Flats Plant
P.O. Box 464
Golden, Colorado 80401

U. S. DEPARTMENT OF ENERGY
CONTRACT DE-AC04-76DPO3533

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PROGRESS REPORT FOR
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Frend John Miner

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ROCKWELL INTERNATIONAL
ENERGY SYSTEMS GROUP
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P.O. BOX 464
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Prepared under Contract DE-AC04-76DPO3533
for the
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U.S. Department of Energy

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SUBJECT DESCRIPTORS

Absorbents	Minicomputers
Advanced Size Reduction	Niobium Alloy
Aluminum	Nondestructive Analysis
Air Pollution	Organic Polymer
Aqueous Cleaning	Packaging
Batching	Paint
Binary Alloy	Performance Testing
Calcium Compounds	Physical Security
Calibration Standards	Pipe
Calorimeters	Plutonium Residues
Carbon Steel	Polyethylenes
Caustic Contamination	Process Control
Cleaning	Radioactive Waste
Combustion	Radioactive Waste Processing
Compatibility	Reverse Osmosis
Computer Program	Safeguards Instrumentation
Contamination	Shipping Container
Control Systems	Sodium Chloride
Cracking	Soil
Container	Solidification
Corrosion	Solvent
Data Acquisition	Solvent Cleaning
Decontamination	Statistics
Depleted Uranium	Stress Corrosion
Detergent	Stress Corrosion Cracking
Environment	Surface Water
Equipment	Transport
Fluidized Bed Incineration	Transuranic Waste
Glove-Box Gloves	Uranium Base Alloy
Gloves	Uranium Incineration
Homogenization	Vitrification
Iron	Waste Disposal
Land Pollution	Waste Drums
Lead-Lined Gloves	Waste Immobilization
Liquid Waste	Waste Management
Measuring Instruments	Waste Processing
Microprocessors	Waste Transportation
Mild Steel	Water Pollution

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The advantages and disadvantages of three fixing agents for the solar evaporation pond sludge were studied. Flyash was selected and recommended as the primary fixing agent. (PPS)*

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*All Development and Development Support (DDS) endeavor numbers refer to FY-1981 Proposed Program for Process Development (JM-01-03-04) and Related Work, CD 79-192, February 13, 1979. Production and Production Support programs are identified as PPS.

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the presence of corrosion products indicate that stress corrosion cracking has occurred. A postulate based on corrosion product wedging theories is presented. (PPS)*

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The computer glove change files are being updated to improve accuracy and accessibility. (PPS)

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A basic solution was selected for potential use as an electrolyte in an electro-decontamination procedure. (PPS)

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*All Development and Development Support (DDS) endeavor numbers refer to FY-1981 Proposed Program for Process Development (JM-01-03-04) and Related Work, CD 79-192, February 13, 1979. Production and Production Support programs are identified as PPS.

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*All Development and Development Support (DDS) endeavor numbers refer to FY-1981 Proposed Program for Process Development (JM-01-03-04) and Related Work, CD 79-192, February 13, 1979. Production and Production Support programs are identified as PPS.

CAUSTIC WATER DECONTAMINATION 26

The reverse osmosis (RO) pilot plant has been used to demonstrate the decontamination capabilities of RO. A caustic solution at a pH > 10 and contaminated with uranium, plutonium, and americium was used. (PPS)*

SOIL DECONTAMINATION 27

A study was initiated to develop a dustless method for removing and processing plutonium-contaminated soil at the Rocky Flats Plant. Work is proceeding to locate suitable equipment, formulate equipment specifications, and produce an economic analysis for the entire soil removal and processing operation. (PPS)

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*All Development and Development Support (DDS) endeavor numbers refer to FY-1981 Proposed Program for Process Development (JM-01-03-04) and Related Work, CD 79-192, February 13, 1979. Production and Production Support programs are identified as PPS.

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SAMPLE AND INVENTORY FILE 32

A program has been written to determine total number of records on an inventory file and generate a new file containing a random sample record from the total inventory file. (PPS)

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A Monte Carlo study is underway to determine the effects of varying certain parameters in a production assembly problem. (PPS)

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An experimental design was set up to compare two methods of determining the percent of niobium in binary turnings. (PPS)

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A recently implemented disc operating system has been utilized to reduce a large amount of data generated by the air quality mobile van for the Brown Haze Study. (PPS)

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<p>A mechanical scanner has been designed to fit into a glove box to handle molten salt residues in the new plutonium recovery facility. (PPS)</p>	
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New end-point algorithms show that 3 percent accuracy is attainable with 75 percent time savings for low-wattage plutonium residue samples. (PPS)*

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New intrusion detection systems have been installed in plant vaults. Other systems are under evaluation. (PPS)

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Tests show the sodium iodide parts monitor, boron poison monitor, and X-ray fluorescence solution monitor will meet design criteria for the facility. (PPS)

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Problem areas have been identified in process control equipment for the new plutonium recovery building, and solutions have been obtained that will aid in start-up and operation of the processes. (PPS)

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A refractometer has been installed on the incinerator caustic recirculation line to control caustic molarity. (PPS)

WATER CONTROL AND RECYCLE PROJECT 45

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PROGRAMMABLE CONTROLLER STUDY 45

Efforts are underway to select a programmable controller standard for the Plant. Controllers from eight companies are currently under evaluation, and a final report expected in June 1979. (PPS)

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PROGRAMMABLE CONTROLLER - COMPUTER TRANSLATOR	46
/A minicomputer has been used to print programmable controller ladder diagrams. The result is a large savings in drafting time. (PPS)*	
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*All Development and Development Support (DDS) endeavor numbers refer to FY-1981 Proposed Program for Process Development (JM-01-03-04) and Related Work, CD 79-192, February 13, 1979. Production and Production Support programs are identified as PPS.

CHEMISTRY RESEARCH AND DEVELOPMENT PROGRESS REPORT FOR DECEMBER 1978 THROUGH MAY 1979

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COMPONENT DEVELOPMENT

Jean R. Gaines

SELECTION OF A FIXING AGENT FOR LOW-SPECIFIC-ACTIVITY, NON-RETRIEVABLE, SOLAR EVAPORATION POND SLUDGE

Dick M. Saiki and Paul G. Aguilar

Objective

The objective of this work was to compare advantages and disadvantages of three fixing agents and to select the best agent for fixing low-specific-activity (LSA), non-retrievable, solar pond sludge. The purpose of the fixing agent is to fix any excess water in the sludge prior to the sludge being shipped to the Nevada Test Site for burial.

Prior Work

Cement and a proprietary organic material from the Chemfix Corporation were briefly discussed and considered as fixing agents in a previous semiannual report (RFP-2818, March 30, 1979). Cement was selected as the best option for fixing the sludge.

Achievements and Discussion

Two additional fixing agents have been evaluated and compared to cement. These additional fixing agents are

- (1) A precipitator flyash generated by the Public Service Company of Colorado at the Comanche Plant, Pueblo, Colorado. This flyash contains a relatively high quantity of calcium oxide (~26 wt %), which reacts with water in the sludge. The flyash is primarily composed

of SiO_2 , Fe_2O_3 , Al_2O_3 , MgO , SO_3 , and TiO_2 . The final flyash-sludge mixture, when solidified, is similar to a crumbly clay.

- (2) A water-absorbent organic polymer called SGP Polymer, which is manufactured by General Mills Chemical Inc. This material is a starch-polyacrylonitrile graft polymer that absorbs moisture and forms a gel when mixed with the sludge.

Preliminary tests indicated that 0.4 kg of cement or flyash mixed with a liter of sludge would result in the desired final mixture—free of excess moisture. Approximately 0.02 kg of the SGP Polymer would be required for each liter of sludge.

In comparing the advantages and disadvantages of the three fixing agents, the following factors were considered: (1) availability, (2) costs (including the fixing agent, packaging, transportation, and processing facility), (3) hazards (including flammability, dust explosion, health and toxicity, and reactivity), (4) mechanics of handling (e.g., mixing and cleanup), and (5) data concerning the fixing agent/sludge mixture characteristics (e.g., setup time, temperature effects, and parameters affecting fixing efficiency).

Upon reviewing the advantages and disadvantages of the fixing agents, it was concluded and recommended that flyash should be used as the primary fixing agent. The flyash was preferred for the following reasons:

1. Compared to cement and SGP Polymer, flyash is the most economical.
2. The fixing efficiency and characteristics of flyash are not significantly affected by fluctuations in the salt content, pH, or temperature of the sludge.

3. Flyash has no significant problems concerning flammability, dust explosion, or reactivity.
4. The processing facility and bulk-handling equipment could be readily used with cement (a proven fixing agent) in the event difficulties are experienced with flyash.
5. The final flyash-sludge mix forms a crumbly clay solid that should minimize or eliminate migration of low level contamination. Also, because of the inorganic nature of the flyash, no long term effects or changes should occur during long term storage.

One disadvantage of the flyash is that a controversy does exist concerning the long term potential health hazards associated with trace metals (e.g., mercury, zinc, uranium, and thallium) in certain types of flyash. Also, there exists a potential hazard to personnel from respirable crystalline silica in the flyash. Therefore, a proper dust control system and proper protective clothing for personnel in processing and handling the flyash will be required. Similar precautionary measures would have been required for cement or the SGP polymer. A second disadvantage is that the calcium oxide content of the flyash must be maintained at a level greater than 25 wt % to obtain proper solidification. A quality control program therefore will be required for the flyash.

The SGP polymer was considered a viable alternative to flyash and cement. The primary advantages of the polymer included ease of handling, ease of mixing with the sludge, minimal health hazards, and the smaller quantity required to fix the sludge. However, a primary disadvantage, which makes the SGP polymer less acceptable than flyash, is that the absorption capacity and rates of reaction of the polymer are affected by pH, salt content, and temperature. Because the absorption capacity of the polymer is relatively sensitive to possible fluctuations in the pH and salt content of the sludge, problems could be encountered in maintaining a proper proportion of the polymer to sludge mix. Any changes, especially an increase, in the quantity of polymer required to fix the sludge will have a significant impact upon the total cost of the program because of the high cost per unit of the polymer.

In summary, flyash was recommended as the primary fixing agent, with cement as a backup. There appeared to be too many variables that could affect the performance and total cost of the SGP polymer.

Future Work

No future work is planned.

PERFORMANCE EVALUATION OF MIXING DEVICES FOR PROCESSING SOLAR POND SLUDGE

Paul G. Aguilar and Dick M. Saiki

Objective

The purpose for evaluating the performance of pug-mill mixers was to determine the effectiveness of the mixing devices in continuously mixing flyash and a synthetic sludge. The synthetic sludge is representative of evaporation pond sludge.

Prior Work

Tests were conducted on a Roto-Feed continuous mixer manufactured by Baker Perkins, Inc. Test results indicate that mixtures of more than 4 lbs of cement to 1 gal of slurry resulted in plugging at the extruder port. The consistency of the discharged slurry reached the thixotropic limitation. The sludge-flyash mixture plugged the extruder screw section, forcing the mixture back through the cement intake port. Also, the extruder screw section flute depth was approximately 0.25 in., thereby not allowing particles of larger size to be processed.

The Roto-Feed continuous mixer was eliminated as a possible method for mixing sludge and flyash. Tests have been conducted and reported in the previous semiannual report.*

Achievements and Discussion

Results from tests conducted on the single-screw pug-mill mixer manufactured by Plymouth Locomotive Works, Inc. indicated there were wet lumps in

*RFP-2877, May 28, 1979.

the mixture that possibly were caused by insufficient shear force. With mixing ratios of more than 4.2 lbs of flyash per gallon of sludge, mixture flow problems developed at the discharge because of the increased size and number of wet lumps. The wet lumps could not pass through the cone shape screw discharge with its restrictive flute clearance. This resulted in plugging of the extruder port. The manufacturer indicated that a higher shaft speed of 60 to 80 rpm, over the 45 rpm used for the test, would result in less residence time and would increase throughput with better dispersions. There was not adequate time, however, to make the extensive modifications required.

Results from tests conducted on the double-screw pug-mill mixer manufactured by Feeco International, Inc. indicated that the double-screw mixer improved the mixing action, thereby limiting the amount of wet lumps formed in the mixture. The minor wet lump that did appear in the mixture did not create any significant problems at the discharge. The discharge port consisted of an 8- X 10-in. opening at the end of the pug-mill trough. Results from the test indicated that of the three mixing devices tested, the double-screw pug-mill mixer performed best; consequently, this mixer will be recommended for use in the solar pond sludge removal program.

Samples of the mixtures taken from each test indicated that the time required for solidification was less than 12 hours. This would not pose a problem with the waste processing schedule.

Future Work

A report will be written summarizing results and recommendations.

SHIPPING CONTAINER FOR INTRAPLANT SHIPPING OF CONTAMINATED LIQUIDS

Paul G. Aguilar and Dick M. Saiki

Objective

The objective is to determine the type of protective housing required to protect a filter and relief valve

mounted on the lid of a DOT-17C 55-gallon steel drum from impact damage. The filter and relief valve would act to relieve excess internal pressures of the liquid waste drum.

Prior Work

Waste Management requested that Component Development assist in the testing and development of a liquid waste container. No experimental development tests have been performed previously on a liquid waste container.

Achievements and Discussion

An impact test was conducted on a prototype DOT-17C 55-gallon steel drum. Impact test parameters were selected from the test conditions required for a Type A Package. (See the Code of Federal Regulations, Title 49.) The purpose for the impact test was to determine if a two-inch-diameter pipe welded to the lid of the drum could withstand a four-foot drop with minimal damage to the pipe while maintaining a leak-tight drum. The pipe will act as a protective housing for the filter and relief valve.

A DOT-17H 30-gallon steel drum was filled to 98% of its capacity with sand and water to a gross weight of 382 pounds. The 30-gallon steel drum was then sealed and placed inside the prototype DOT-17C 55-gallon steel drum. Wood strips were placed around the outside, and a wood block was placed on top of the 30-gallon drum to keep it stationary. The prototype DOT-17C 55-gallon steel drum was then sealed and subjected to a four-foot drop onto a steel plate, exposing the pipe to the full force of the impact.

Upon impact, the lid of the DOT-17C 55-gallon steel drum suffered extensive damage; the steel pipe penetrated the lid. Adequate force was applied at impact to shear the lid, allowing the drum to be breached. From the test results, it was observed that the pipe suffered minimal damage and could have protected the filter and relief valve from damage. The lid of the drum will have to be reinforced, however, to withstand the impact test.

Future Work

Modifications to the previously tested design will be made and the good test characteristics will be incorporated into the future design.

IMPACT TEST OF A ROCKY FLATS MODEL RA-2 CONTAINER FOR TRANSPORTING LOW-SPECIFIC-ACTIVITY URANIUM

Paul G. Aguilar and Dick M. Saiki

Objective

The objective of this work was to determine if a Rocky Flats Model RA-2 container, when loaded to 630 pounds, would meet DOT drop test or impact requirements.

Prior Work

The Rocky Flats Model RA-2 Container was previously qualified in accordance with DOT requirements for transporting low-specific-activity (LSA) uranium billets.* The maximum gross weight limit allowed for the container was 500 pounds.

Achievements and Discussion

The Rocky Flats Model RA-2 container consists of a 30-gallon DOT-17H drum and celotex rings and disks for cushioning LSA uranium billets. Normally, two cylindrical billets are packaged per container. For this drop test, a test container was loaded with two billets, each weighing approximately 263 pounds. The gross weight of the test container was 630 pounds.

The drop test was conducted in accordance with DOT requirements for a Type A package as described in the Code of Federal Regulations, Title 49. For this test, the test container was inverted and lifted to a

*Griffin, J. R. and Adling, D. A., "Certification of ERDA Contractors Packaging with Respect to Compliance with DOT Spec. 7A Performance Requirements," MLM-2228, Mound Labs, Monsanto Research Corp., June 1975, p. 43.

height of four feet with a special sling that was attached to a manual release hook. The container was oriented such that the edge of the drum lid and the clamping ring, at a location 90° from the nut and bolt, would be exposed to the full force of the impact. The container was dropped onto a 2-inch-thick steel plate.

Deformation to the lid, clamping ring, and the upper portion of the drum body was minimal. The maximum indentation from the impact was about 1.5 inches. The clamping ring conformed to the distortion and securely held the lid of the drum in position. Upon opening the drum, the billets were examined and found to be undamaged. Other than impressions made by the impact of the billets on the upper celotex disk, no significant changes in the physical condition of the celotex rings or disks were evident.

The drop test did show that the RA-2 container, when loaded to 630 pounds, is capable of withstanding the impact of a four-foot drop as per DOT regulations. No detrimental effect upon the integrity of the container and its contents should occur. Therefore, it was recommended that the container be used to transport LSA uranium billets to a maximum gross weight limit of 630 pounds.

Future Work

No further work is planned.

PRELIMINARY TESTS ON A PROTOTYPE MODEL RA-3 DOT-7A PLYWOOD BOX

Paul G. Aguilar and Dick M. Saiki

Objective

The objective of this work was to determine the impact and compression resistance of a prototype Model RA-3, DOT-7A plywood box.

Prior Work

No prior work has been conducted at Rocky Flats on testing a box of this configuration.

Achievements and Discussion

The prototype plywood box, designated Model RA-3 DOT-7A and designed by the Product Definition Group at Rocky Flats, was tested for impact and compression resistance. The box frame was constructed with 2- X 4-inch wood stringers. The sides, bottom, ends, and lid consisted of 0.75-inch-thick plywood panels. The various components were nailed together and all joints were sealed with elastomeric construction adhesive (B. F. Goodrich PL-200).

The approximate inside dimensions of the box were 43.5 inches long by 12 inches wide by 10.2 inches deep. If accepted, the container could be used to package and transport depleted uranium metal or alloy billets. The maximum weight capacity of the container would be 1500 pounds.

Test conditions were based on DOT requirements for a Type A package as given in the Code of Federal Regulations, Title 49. Prior to testing, the test container was loaded to 1500 pounds with lead bricks.

The compression test involved placing 7500 pounds (5 times the maximum weight of the box) onto the top of the test container for a period of 24 hours. During this test, no visible damage or compression occurred to the container.

The impact test was conducted by allowing the container to free-fall from a height of four feet onto one of the lid corners. The container was dropped onto a four-inch-thick steel plate. Upon impact, the end of the box suffered extensive damage and was breached. Adequate force was applied to the end of the container to split in half the 0.75-inch plywood panel and to separate the 2- X 4-inch wood stringers. In addition, lengthwise separation between the lid and body had occurred.

Future Work

Methods are being considered by Product Definition to strengthen the end of the box. Metallic banding will be required to prevent the lid and body from separating upon impact. Once these modifications are completed, a new series of tests will be conducted.

PRELIMINARY TEST RESULTS FOR A NEW OPEN-HEAD POLYETHYLENE LINER FOR TRANSURANIUM WASTE DRUMS

Paul G. Aguilar and Dick M. Saiki

Objective

The objective of this work is to determine the feasibility of using an open-head, rigid, polyethylene liner for packaging transuranium (TRU) waste. This liner would be used in place of the Type II 90 mil polyethylene liner presently used with a DOT-17C 55-gallon drum.

Prior Work

No prior work has been conducted on the new proposed liner.

Achievements and Discussion

Preliminary impact tests were completed on the open-head polyethylene liner (blue rigid liner), which is closed with a clamping ring configuration. The liner used for these tests is actually a 55-gallon polyethylene drum (Model 0555 DOT-E-7.768.55) manufactured by Plasti-Drum Inc. The lid of the liner is manufactured by injection molding of low density polyethylene while the body is fabricated from high density polyethylene by rotational molding. A 65-gallon, steel, recovery drum was used as the overpack since the blue rigid liners are not presently available in a size to fit into a 55-gallon DOT-17C drum.

The impact test parameters were selected from test conditions required for a Type A package, as given in the Code of Federal Regulations, Title 49. Prior to the liners being filled to the desired test weight, a Hedwin 10-mil, round-bottom liner was placed into each of the four rigid liners tested. The liners were filled to 98% capacity with a ethylene-glycol solution and sand, and the inner Hedwin liner was sealed with a pressure-sensitive adhesive tape. A closed, sponge-rubber gasket was placed into the groove of the rigid liner lid, and the lid was placed in position onto the body of the liner. A square back bolted ring was used to close and secure the

lid and body of the rigid liner together. The sealed liners were then overpacked with the 65-gallon recovery drums. The final gross weight of each package was 738 ± 2 pounds (335 ± 1 kilograms).

Two of the four test containers were impact-tested at -5 to 0 °F (-21 to -18 °C). The remaining two containers were tested at ambient temperature (70 ± 2 °F) (21 ± 1 °C). The impact tests consisted of dropping each container from a height of four feet onto a non-yielding surface. Each container was inverted and oriented such that the edge of the drum lid would be exposed to the full force of the impact.

Upon impact, leakage was observed for both containers tested at the low temperature. When the recovery drum lids were removed, the lids of both liners were found to have suffered extensive damage; each had a crack approximately 25 centimeters in length.

The rigid liners tested at ambient temperature had no visible damage; however, some moisture was noted on the outside of both liners in the vicinity of the clamping ring. It appears that the impact caused temporary deformation and leakage in the gasket area. For all four tests, the clamping ring securely held the lids and body of the liners together.

The impact tests showed that the lid of the rigid liner became embrittled and susceptible to cracking at a low temperature. To correct this condition, a high-density polyethylene will be used in the fabrication of the lid.

For the liners impact-tested at ambient temperature, it was found that the closed-cell sponge-rubber gaskets conformed to the shape to which they were compressed. This lack of resiliency in the gasket material and the roughness of the sealing surfaces may have contributed to the temporary leakage noted during impact testing. Presently a tubular neoprene gasket and a better finish on the sealing surfaces are being considered as means of eliminating this leakage.

Future Work

The required modifications will be made to the lid, gasket, and sealing surfaces of the liner. Once these modifications are completed, impact tests will be repeated. If the modified liner passes the impact tests, vibration and environmental stress cracking resistance tests will be conducted.

EVALUATION OF A TRI-WALL CORRUGATED FIBERBOARD PACKAGE FOR PACKAGING AND TRANSPORTING LOW-SPECIFIC-ACTIVITY NON-RETRIEVABLE WASTE

Dick M. Saiki and Paul G. Aguilar

Objective

The objective of this evaluation was to determine if an economical, disposable, Tri-Wall Corrugated Fiberboard (TWCF) package would meet present DOT regulations for transporting low specific activity (LSA) non-retrievable waste (activity levels $< 10\text{ nCi/g}$) to the Nevada Test Site for burial. Also, tests were conducted to determine performance characteristics of the package under certain adverse or hypothetical accident conditions.

Prior Work

No prior work on this objective has been conducted at Rocky Flats.

Achievements and Discussion

The TWCF package consists of a semioctagonal, tri-wall, corrugated box with single-wall lid and bottom caps, a 102-cm X 102-cm wooden pallet, a polyethylene (1.3-mm thick) liner, and non-metallic strapping. The final loaded configuration of the TWCF package is shown in Figure 1. The total height of the package is approximately 114 cm. Once filled, the liner is sealed with a pressure-sensitive vinyl tape. The non-metallic strapping is used to secure the single-wall lid and bottom caps to the

FIGURE 1. Final Packaging Configuration for the Loaded Tri-Wall Corrugated Fiberboard Package

tri-wall body and to hold the assembled box and pallet together. The total cost of the package is \$23, which is 75% less than the cost of the plywood box, with comparable volume, presently used to transport LSA non-retrievable wastes.

Currently there are no specific testing requirements in the DOT regulations for packaging LSA waste. However, the DOT regulations do state that the "waste material must be packaged in a strong, tight package so that there will be no leakage of radioactive material under conditions normally incidental

to transportation" (Code of Federal Regulations, Title 49). To determine if the TWCF package would meet these requirements, vibration tests simulating transport conditions were conducted. At the end of each test, the test packages and components were examined visually for possible physical damage or changes that could cause leakage or loss of waste material.

In addition to the vibration tests, other tests were conducted to determine the performance of the TWCF package under adverse or hypothetical

accident conditions. These tests included drop tests onto the pallet, rotational or tip-over drop tests onto the edge of the lid, moisture exposure through wet spray tests, puncture tests, and compression-by-stacking tests. Also, operating limitations and precautions were derived from the performance test results.

The packages used for these tests were loaded with a sand-vermalite mixture to between 820 and 860 kg. These weight limits were based on density values for pelletized evaporator salts. Prior to testing, the test containers were not pre-conditioned in any specific humidity or temperature environment.

The results of these tests indicated that if the TWCF package is used, the following precautions must be exercised and/or limitations exist:

1. The package should not be exposed to moisture during storage or shipment.
2. During storage, packages loaded to approximately 820 kg should not be stacked more than 2 high. Although the stacking test indicated the possibility of stacking the package 3 high, a variety of uncontrollable factors (e.g., humidity, stacking alignment, and fatigue) could alter or decrease the stacking strength of the package.
3. During transport, packages loaded to approximately 820 kg should not be stacked in the transport carrier. Packages loaded to lower weights possibly could be double stacked.

The TWCF package was recommended to Rocky Flats Waste Management as a possible alternative for packaging and transporting certain forms of LSA non-retrievable waste (i.e., material that will not abrade through, puncture, or chemically attack the polyethylene liner).

Future Work

A trial shipment to the Nevada Test Site of TWCF packages loaded with non-contaminated material is planned. Approval from DOE for this trial shipment is presently being sought.

GRAPHITIC CORROSION OF BUILDING 881 WATER MAIN

Marston R. Dringman and Richard A. Patterson

Objective

The objective of this investigation was to determine the cause of a premature water main failure.

Prior Work

No prior work has occurred on this task.

Achievements and Discussion

A gray iron water main developed a leak after 26 years in service. The pipe was 20.3 cm in diameter with a 0.89-cm wall thickness, and was buried approximately 2 m deep in clay-type soil. The leak occurred adjacent to a roadbed. Normal life expectancy for buried gray iron pipe is 35 years under normal, undisturbed, environmental conditions.

Excavation of the suspected leak region revealed an extremely moist soil condition. Upon exposure of the pipe, a circumferential crack was detected. It was not immediately obvious whether the crack was caused by impact during excavation or if it was the original leak source.

Attempts to rejoin and repair the crack were unsuccessful; consequently the affected pipe section was replaced. The removed pipe section was submitted for evaluation to determine the cause for the water leak and crack.

A visual examination of the fracture surface indicated that about 90% of the circumference was cracked prior to excavation; a fresh crack surface affected only 10% of the circumference. The older fracture surface was identifiable by a red-rust and mud-covered appearance, while the newly fractured sections had the typical dark gray appearance of brittle cast iron failures.

Examination of the outer surface of the pipe revealed a large region (10 cm wide by 35 cm long) of localized corrosion attack. Sediment within the

pipe indicated that this corroded region was located on one side approximately 90° up from the bottom. Sheets of corrosion product 25- to 35-mm square and 6- to 8-mm thick were easily removed from the pipe surface adjacent to the primary corroded region. The corrosion product was a dark-gray and red-rust conglomerate without obvious mechanical integrity. The previously mentioned crack appeared to originate within this corroded region and extend circumferentially around the pipe.

Soil samples were taken from the pipe exterior for chemical analysis. Infrared spectroscopy revealed that the soil contained mixtures of sulfates and carbonates. The soil was a very moist clay.

Metallographic examination of samples removed from the corroded region revealed extensive attack on both the interior and exterior surfaces. Wall thinning was measured at points of maximum attack and revealed that unaffected wall thickness was as thin as 2 mm in several regions.

Corrosion of this type in gray irons has been designated as graphitization or graphitic corrosion.¹ Graphitic corrosion is the selective leaching of iron from gray cast irons leaving an unaffected graphite network. The graphite is cathodic to iron, and an excellent galvanic cell exists with the addition of moisture. Exposure of gray iron for 26 years in moist soil that is high in sulfates or chlorides would be expected to produce severe graphitic corrosion.² Graphitic corrosion causes gray iron to lose strength and metallic properties. Dimensional changes do not occur, and dangerous situations may develop without detection.

It was concluded that the pipe failure resulted from external graphitic corrosion caused by moist conditions in the sulfate-containing soils surrounding the pipe. Natural groundwater or pin hole leaks from internal corrosion are assumed to be possible sources for the abnormal moist soil conditions.

If similar moist soil conditions are present on the remaining plant water mains, identical corrosion failures may occur in the near future.

Recommendations for avoiding this problem with future water main installations were made.

Future Work

No future work is planned.

References

1. M. G. Fontana and N. D. Greene, *Corrosion Engineering*, McGraw Hill Book Co., 1967, pp. 70-71.
2. American Society for Metals, *Metals Handbook*, Volume 10, 1975, p. 191.

BURIAL SITE CORROSION TESTS OF PAINTED WASTE DRUMS

John L. Briggs and Marston R. Dringman

Objective

The objective of the corrosion tests was to determine the suitability of the paint used to coat waste drums for storage at the Arco, Idaho burial site.

Prior Work

This is a new project.

Achievements and Discussion

Corrosion tests of coated mild steel samples conducted at the Arco, Idaho waste burial site have been concluded after a three year in-place test. The tests were initiated in collaboration with the Idaho Environmental Surveillance Group to determine the corrosion resistance of painted steel waste drums in the burial storage environment.

Painted mild steel (2 X 3 inches) for the corrosion tests were cut from the lids of open-head, mild steel drums manufactured by the U.S. Steel Company. Prior to testing, the sample coupons were cleaned with an aqueous solution, weighed to the nearest 0.1 mg, photographed, and then forwarded

to the Idaho burial site for placement in the storage cells.

The sample coupons were placed in three cells (Nos. 5, 6, 7) of transuranic storage area pad No. 1 in 1975. Storage of Rocky Flats waste in those cells began in 1971; the cells were closed November 7, 1973. The cells were opened for the first time in 1978, and a trip to the burial site was made to evaluate corrosion on the exhumed waste drums.

Fourteen of the 48 sample coupons sent to the burial site for tests were not recovered after the three-year test. After the remaining thirty-four test coupons were received, a visual evaluation was performed on the sample surfaces. The coupons were then cleaned with an aqueous detergent solution, and loose corrosion products and dirt were removed. The coupons were then reweighed. Most of the painted samples had no significant weight changes. However, high weight losses were measured for those coupons that exhibited large areas of perforated blistering. These coupons exhibited extensive rusting of the exposed bare steel surfaces.

A visual evaluation was performed on the sample coupons as specified by ASTM Method D610, "Standard Methods of Evaluating Degree of Rusting on Painted Steel Surfaces" and ASTM Method D714, "Evaluating Degree of Blistering of Paints." All of the sample coupons showed blistering of the paint surfaces to some degree ranging from large (0.25-inch diameter) to small blisters, visible only with a magnification of 8X. Although blistering can result from several causes, the blistering on the samples was probably caused by the high (~80%) relative humidity that existed in the storage cells.

Observations of the exhumed white painted waste drums at the burial site indicated red rust concentrated on lids and bottoms of the waste drums where water had collected and apparently remained for some length of time.

It was concluded that corrosion resistance of the white alkyd paint coating was not satisfactory for the humid storage cell environment. Results of the three-year burial-site coupon tests also indicate that the white alkyd paint is not suitably resistant to water corrosion.

Future Work

Based on this study, an upgrading of the paint has been requested by the burial site contractor, EG&G Company.

INVESTIGATION OF RUST CORROSION ON STEEL ACTIVATOR PISTONS

John L. Briggs and Marston R. Dringman

Objective

The objective of this investigation was to identify the source of rust corrosion on Category J steel pistons.

Prior Work

Previous studies have identified a dye penetrant inspection as one source of rust corrosion.

Achievements and Discussion

An investigation of the production processes for Category J S-6 steel activator pistons was performed to identify the sources of rust corrosion prevalent on those parts.

During production, the pistons are subjected to Zygo dye penetrant inspection, aqueous cleaning, aqueous rust removal, electroless nickel plating, and dimensional inspections.

A previous study concluded that aqueous Zygo inspection processes produce rust corrosion on the pistons. Unless properly performed, aqueous cleaning can also be a source of rust corrosion on the pistons. Four rejected S-6 steel pistons were obtained to determine if ultrasonic solvent vapor degreasing could replace aqueous cleaning for in-process cleaning of the pistons. The reject parts had been subjected to all processes of the normal production cycle.

Two pistons were cleaned by methyl chloroform ultrasonic vapor degreasing (ultrasonically cleaned

for 2 minutes, vapor-phase rinsed for 2 minutes). The other two pistons were cleaned with 2 vol % of Oakite NST detergent at 55 °C for 5 minutes, rinsed in warm (49 °C) deionized water, and rinsed in Formula 3A ethyl alcohol to accelerate drying.

Surface residue identifications were performed on the pistons by specific ion electrode, infrared spectroscopy, and emission spectroscopy.

Results of the cleaning residue studies indicated low levels of hydrocarbon residues (17 milligrams per piston) and chloride (30 milligrams per piston) on the pistons that were solvent cleaned. However, these trace residues would not preclude the solvent method for in-process cleaning.

The substitution of solvent cleaning for the current aqueous in-process cleaning method would reduce the occurrence of rust corrosion. However, final aqueous cleaning is required for chloride removal prior to assembly.

No residues were detected on the aqueous cleaned pistons and no rusting occurred. It is concluded that aqueous cleaning is optimum for final cleaning of the pistons.

As a result of investigating the piston production process, a revised process flow sheet was proposed that incorporated the solvent in-process and aqueous final-cleaning methods.

Future Work

No additional work is planned.

EXAMINATION OF THE CRACKING MECHANISM FOR THE LAKE ERIE EXTRUSION PRESS

Richard A. Patterson

Objective

The objective of this work was to examine representative cracks in the high-pressure water bottle (HPWB) of the Lake Erie Extrusion Press and to use

the resulting data to assess the reliability of the modified extrusion press system.

Prior Work

During the 20-year service of the HPWB, several incidences of pitting corrosion attack were reported. In 1969, corrosion attack was removed from the vessel interior by grinding until unaffected metal was reached. In 1976, localized corrosion activity again was found during inspection. Inspection data prior to 1976 indicated that the vessel had been subjected to corrosion attack in excess of the 1/16-inch design allowance, and that no crack-like defects had been detected.

In July 1978, during a routine inspection of the extrusion press accumulator system, numerous crack-like indications in the HPWB were detected. These defects were distributed randomly on both sides of the lower hemispherical cap-to-cylindrical laminate weld, presumably in the heat affected zone (HAZ). Measurements of defect dimensions indicated that they were hairline cracks ranging from 1/16 to 2 inches long and as much as 0.5 inch deep for the longest defect.

Representative samples of these cracks were removed from the HPWB. These cracks were then subjected to a detailed metallurgical examination.

Achievements and Discussion

Examination of representative cracks from the HPWB has revealed the following information:

1. Grind marks indicate that the cracks formed after 1969, and that little plastic deformation is associated with the origin of the cracks.
2. Inspection data indicate that these cracks were not present in 1976.
3. Metallographic examination revealed that these cracks nucleated at the lower cap-to-laminate weld toe and propagated into the weld metal to a depth of approximately 0.050 inches.

4. Scanning electron microscopy of the crack surfaces showed a highly corroded region followed by transgranular ductile overload, with crack propagation at an angle of approximately 45° to the primary tensile stress.
5. Scanning electron microscopy and metallographic results show multiple crack fronts and crack branching.

These major points indicate that the cracks are the result of a stress corrosion cracking mechanism that developed between 1976 and 1978.

A corrosion product wedging model has been proposed as the cause of the cracks. The model is based on one of the proposed theories of stress corrosion cracking in which the added force generated by volume expansion of metal transforming to corrosion product is sufficient to cause failure in stressed members.¹ This model is consistent with the physical appearance of cracks in the HPWB.

Specific environmental species responsible for the stress corrosion cracking (SCC) have not been identified. Suspected contaminants include chlorides, nitrates, and phosphates. However, absolute determinations of the responsible corrosion mechanism would require extensive SCC research with all suspected environments.

Future Work

A final report on this examination is being prepared for distribution.

Studies are in progress to compare the effectiveness of several corrosion inhibitors. This study should generate meaningful data for continued operation of the modified extrusion press system.

References

1. N. A. Nielsen, "The Role of Corrosion Products in Crack Propagation in Austenitic Stainless Steel. Electron Microscopic Studies," *Physical*

Metallurgy of Stress Corrosion Fracture, AIME Metallurgical Society Conferences, Vol. 4, 1959.

GLOVE CHANGE PROGRAM

Arlene F. Younger

Objective

The purpose of this work is to improve the accuracy and accessibility of the glove change program.

Prior Work

Work on the glove change program is described in RFP-2818, March 30, 1979.

Achievements and Discussion

Computer data in the glove change file are being examined and synchronized with physical conditions on the glove ports. Through the services of Product Information Systems, new capabilities have been developed regarding access to the files and manipulating data contained in the files. This will enable the user to have more accessibility to and responsibility for the files.

Use of glove change data presupposes the failure of the glove. User safety requires that this failure be estimated conservatively. Extensive cooperation through checks on submitted reports and checks on glove inspections is required in this initial phase of generating glove change data.

Future Work

Group presentations will be used to familiarize radiation monitors and users with new capabilities in the system.

As data in the glove change files expand, more data will be available to predict safe glove life expectancy and to develop more successful glove types.

ELECTRODECONTAMINATION WITH CAUSTIC SOLUTION

Everett L. Childs

Objective

The objective of this study was to develop an electrodecontamination method for the final decontamination of stainless steel and other metals processed in the Advanced Size Reduction Facility (ASRF).

Prior Work

R. W. Arrowsmith at Battelle Northwest Laboratories has reported considerable experimental work using phosphoric acid as an electrodecontamination medium. Problems associated with waste processing and plutonium recovery in this process made the phosphoric acid undesirable for use in the ASRF.

Achievements and Discussion

A test plan was implemented to compare phosphoric acid electropolish, nitric acid electroetch, and basic (sodium nitrate) electrodecontamination as the final decontamination process. The test plan is summarized in Table 1. Results of each of the 10 procedures are plotted in Figure 2. Procedures 3, 6, 7, 9, and 10 produced the most efficient decontamination (the most contamination removed for the least amount of metal removed).

The basic solution was picked as the most useful for the ASRF for the following reasons:

1. The basic solution has a long life.
2. The procedure does not dissolve plutonium. Plutonium is collected in the sludge and is readily removed with the sludge by filtration.
3. Plutonium can be recovered easily from the sludge with existing Rocky Flats technology.

TABLE 1. Test Parameters and Samples

Procedure Number	Test Solutions and Temperature	Current Density (A/in ²)	Sample Type and Number*		
			Standard	Welded	Line Generated
1	Basic Solution 25 °C	4.0	10	1	1
2	Basic Solution 25 °C	1.8	5	1	2
3	Basic Solution 90 °C	1.8	5	1	2
4	Nitric Acid 25 °C	4.0	5	1	2
5	Nitric Acid 25 °C	1.8	5	1	2
6	Phosphoric Acid 75%, 90 °C	4.0	5	1	2
7	Phosphoric Acid 75%, 90 °C	1.8	5	1	2
8	Phosphoric Acid 25%, 90 °C	4.0	5	1	2
9	Phosphoric Acid 25%, 90 °C	1.8	5	1	2
10	Basic Solution 90 °C	4.0	5	1	2

*All samples were stainless steel.

FIGURE 2. Average of Results for Each of 10 Procedures

Further testing of the basic solution was done in 1-liter baths. Additional tests were also run in a 21.5-liter bath to obtain information on solution life and required filter size.

Future Work

The basic solution procedure will be evaluated in a 400-gallon facility at Battelle Northwest Laboratory.

The following areas will be addressed at Rocky Flats:

1. Optimum pH for decontamination
2. Decontamination factors of aluminum, lead, copper, and iron
3. Decontamination at higher current densities
4. Decontamination at low temperatures

STEAM CLEANING TESTING FOR ADVANCED SIZE REDUCTION FACILITY

William R. Briggs

Objective

Steam cleaning testing was done as part of a program to design an Advanced Size Reduction Facility (ASRF). This testing was accomplished to determine the most desirable process for removing gross contamination and surface deposits (i.e., paint, grease, and adhesives) from metallic parts in preparation for electro-decontamination. A determination of the nature of the waste stream from the selected process was needed to establish ASRF design criteria.

Prior Work

No prior work has been reported.

Achievements and Discussion

Cleaning rate tests utilizing a wide variety of cleaning methods were performed on stainless steel test samples. The samples were first prepared with the projected worst case surface deposit, EA 901 Structural Adhesive. Steam cleaning exhibited the most promising cleaning rate and waste stream characteristics.

Cleaning rate tests were performed on various designs of steam cleaners. The Turco Steamerette Type D-1 steam cleaner cleaned as fast or faster than all other designs (approximately 2 minutes per square foot for EA 901 Adhesive) while generating only a 1.75 gallon per minute water waste stream.

The decontamination characteristics of the Turco steam cleaning gun were tested by cleaning both artificially contaminated stainless steel samples and samples cut from a decommissioned contaminated glove box. As shown in Figure 3, gross

FIGURE 3. Contamination Versus Steam Cleaning Time

contamination was removed quickly, but the non-retrievable burial limit was not reached.

The Turco Steamerette Type D-1 Steam Cleaner was incorporated into ASRF specifications for use in the initial decontamination area.

Future Work

There is no future work planned at this time.

REMOTE DISASSEMBLY FOR ADVANCED SIZE REDUCTION FACILITY

James M. Sherman

Objective

The object of this work is to develop and test methods of disassembling contaminated waste items for the Advanced Size Reduction Facility (ASRF). The output from this facility will be (1) size-reduced combustibles in drums and (2) decontaminated size-reduced metals. Work in the remote areas will be accomplished by using master/slave manipulators, bridge crane manipulators, and glove ports while handling standard small hand tools.

Prior Work

Design of the ASRF mock-up was reported in RFP-2877, May 28, 1979.

Achievements and Discussion

Complete disassembly operations were performed on sample glove boxes using the manipulators, glove ports, and disassembly tools. Tool changes, scrap material handling, minor maintenance, and material conveyance were performed and videotaped. Modifications to the disassembly tools have been completed. An example is the manipulator-held impact wrench shown in Figure 4. Modifications include the fingers on the manipulator and the adapter that is strapped to the wrench to facilitate

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FIGURE 4. Manipulator-Held Impact Wrench

holding by the manipulator. The modifications were tested and included in the recommendations for use in the facility. Specification sheets were completed documenting the entire range of disassembly operations covered by Remote Engineering in preparation for the next phase of the project.

The manipulators and positioning table for testing in the mock-up have been ordered.

Future Work

Assistance will be given Facilities Engineering to prepare final specifications so an architectural engineering firm can be hired. The scale model of the facility is to be completed for materials flow studies. The manipulator and positioning table will be installed when received for use in training.

THE COMPATIBILITY OF NON-NUCLEAR WEAPONS METALS WITH PROCESS MATERIALS

Marston R. Dringman and John L. Briggs

Objective

The objective is to perform tests that will determine the compatibility of process materials with non-nuclear metals.

Prior Work

The results of other compatibility studies have been reported in previous semiannual progress reports.

Achievements and Discussion

There is a continuing Quality Control program that requires compatibility testing of all non-nuclear weapons metals with processing materials currently in use. This is accomplished by corrosion and residue removal tests.

The compatibility tests for fluids are performed according to the total immersion corrosion test outlined in NACE Standard Method TM-01-69. Non-fluid materials are tested in a similar manner by contacting metal coupons with the materials.

The sample coupons are subjected to several steps: pretreated by surface finishing with 120 grit abrasive paper, rinsed first in distilled water and then in acetone, air dried, and then weighed to the nearest 0.1 mg. Next the metal coupons are immersed in or placed in contact with the various corrosive materials and stored for 10 days at room temperature.

At the end of the test period, the coupons are removed and loose corrosion products, if any, are removed from the surface. After visual examination, the coupons are reweighed for corrosion rate determinations. Following the corrosion tests, the coupons that have been exposed to the corrosive environments are cleaned with an aqueous detergent as follows:

TABLE 2. Compatibility Test Materials and Metals

Process Materials	Metals Tested	Metals Not Compatible
20 g/l $\text{Ca}(\text{NO}_3)_2$	JBK-75, 21-6-9	None
25 g/l NaNO_3	*	None
COE-flex injection (90-min exposure)	*	None
COE-flex injection (45-min exposure)	*	*
COE-flex injection (30-min exposure)	*	*
COE-flex regular (90-min exposure)	*	None
COE-flex regular (45-min exposure)	*	*
COE-flex regular (30-min exposure)	*	*
COE-flex heavy (90-min exposure)	*	None
COE-flex heavy (45-min exposure)	*	*
COE-flex heavy (30-min exposure)	*	*
Utilac 490-20 Red Spray Enamel	JBK-75, 21-6-9, 304L	JBK-75 21-6-9 304L
Utilac 490-00 Spray Enamel	JBK-75, 21-6-9, 304L	None

*Monel; Be; 1100, 5083, 6061, and 7075 Al; JBK-75, 21-6-9, 304L, 316, and 321 stainless steel; U - 2.3 wt % Nb; U - 6 wt % Nb; U - 0.75 wt % Ti; and U - 238.

1. Ultrasonically clean in detergent (Oakite NST Aluminum Cleaner, 20 ml/l at $55 \pm 5^\circ\text{C}$) - 5 minutes.
2. First rinse in distilled/demineralized water - 5 minutes.
3. Second rinse in distilled/demineralized water - 5 minutes.
4. Air dry.

The coupons are then analyzed for residue by infrared spectroscopy, atomic absorption, and emission spectrographic techniques. Table 2

summarizes the new process materials and non-nuclear weapons metals that were tested in this manner during this reporting period.

All materials listed in Table 2 proved to be compatible except where noted.

Future Work

Future compatibility studies will be performed as required for new weapons metals and production process materials.

CLEANING URANIUM - 6.2 WEIGHT PERCENT NIOBIUM ALLOY PARTS

John L. Briggs and Marston R. Dringman

Objective

The objective of the cleaning study was to develop a suitable final cleaning method for binary (U - 6.2 wt % Nb) alloy parts.

Prior Work

Previous studies have shown that aqueous detergent cleaning is effective for cleaning small binary alloy parts.

Achievements and Discussion

Previous and current cleaning studies have shown that single aqueous detergent cleaning (no solvent pre-cleaning) is sufficient for cleaning small binary alloy parts.¹ However, solvent pre-cleaning to remove the paraffin lathe oil from larger parts will prevent excess oil buildup in the subsequent aqueous detergent cleaning solution. Comparative studies therefore were performed to develop optimum cleaning methods for the binary parts.

Sample coupons of U - 6.2 wt % Nb alloy (1-in. diameter and 0.06-in. thick) were used for the pre-cleaning tests. The sample coupons were surface-finished with 120 grit abrasive paper, rinsed in

TABLE 3. Diagnostic Solvent Pre-Cleaning Test

Sample Identification	Test No.	Cleaning Method
1, 3	I	methyl chloroform, three immersions, 5 minutes each, ambient temperature, aqueous detergent clean ^a
14, 19	II	methyl chloroform, ultrasonic clean for 3 minutes, ambient temperature, aqueous detergent clean ^a
21, 23	III	aqueous detergent clean ^a
29, 30	IV	current M-document cleaning method (Freon TF immersion plus aqueous clean)

- a. Aqueous detergent (2 vol % Oakite) clean at < 43 °C as per M-32506-H; no Freon TF pre-clean.
b. Solvent cleaning method M-38539, Section 1.

distilled water, and air dried. Duplicate sample coupons were subjected to a simulated lathe oil treatment (wiped with paraffin oil) and then cleaned by the diagnostic cleaning methods listed in Table 3. After cleaning, the coupons were rinsed for residue identification by infrared and emission spectroscopy.

Following a pre-cleaning treatment, no detectable hydrocarbon residues (< 5 µg/in.²) were found by infrared spectroscopy for any of the four cleaning methods. Neither were any inorganic residues detected by emission spectroscopy for any of the cleaning methods.

Although single aqueous detergent cleaning (Test III) provides satisfactory cleaning, the use of solvent pre-cleaning is desirable to prevent excess lathe cutting oil buildup in the aqueous detergent solution.

The methyl chloroform (Test I) and Freon TF (Test IV) immersion dip-wipe methods provided suitable cleaning efficiency, but are inapplicable. The reason is the size and weight of the binary parts for triple immersion and wipe techniques.

The use of ultrasonic methyl chloroform solvent cleaning (Test II) provided suitable cleaning results

TABLE 4. Final Part Cleaning Test Results

Sample Identification	Hydrocarbon Residue C-H, ($\mu\text{g}/\text{in.}^2$)	Inorganic Residues (microgram per part)			
		Ca	Mg	Cu	Si
Control (No Cleaning)	1.1	20	5	10	10
5-Minute Aqueous Clean*	0.14	10	10	5	10
10-Minute Aqueous Clean*	< 0.1	5	5	< 1	10
15-Minute Aqueous Clean*	< 0.1	5	5	< 1	10

*Ultrasonic cleaning in 2 vol % Oakite, 40 °C, plus deionized water rinses. Ultrasonic solvent cleaning was performed prior to aqueous cleaning.

and is adaptable for the binary parts to be pre-cleaned. Fixtures have been designed and fabricated for all parts. The substitution of ultrasonic methyl chloroform cleaning for the current Freon immersion cleaning should result in reduced costs. This reduction will stem from decreasing cleaning time and solvent expense. A vapor degrease rinse phase was not included in the ultrasonic methyl chloroform cleaning because of the 75 °C boiling point of that solvent. Temperatures above 43 °C are restricted as a result of possible dimensional changes of the finished binary parts. The results of final aqueous cleaning tests performed on the binary parts are shown in Table 4.

The low level of hydrocarbons detected on the control part is typical of the amount of lathe oil retained on the parts after machining. The five-minute aqueous cleaning reduced the hydrocarbon level to 0.14 $\mu\text{g}/\text{in.}^2$. Extended aqueous cleaning times reduced residues to below minimum detection limits for infrared spectroscopy (<0.1 $\mu\text{g}/\text{in.}^2$). No significant inorganic residues were detected by emission spectroscopic techniques on the 10- and 15-minute aqueous cleaned parts.

It was concluded that a three-minute ultrasonic cleaning, using the room temperature solvent,

methyl chloroform, followed by a 10-minute aqueous detergent cleaning, provided adequate cleaning for the binary alloy parts.

Future Work

Studies will be performed to determine the effect of variable temperatures on aqueous cleaning efficiency.

References

1. J. L. Briggs and H. A. Goad, *A Comparative Study of Aqueous and Solvent Methods for Cleaning Metals*, RFP-2430, Rockwell International, Rocky Flats Plant, Golden, Colorado, April 19, 1976.

CLEANING METHODS FOR THE TOUCH BRAZE PROCESS

John L. Briggs and Marston R. Dringman

Objective

The objective of this study was to determine whether touch braze joint failures were caused by production metal working residues.

Prior Work

This was a new project.

Achievements and Discussion

An excessive number of rejected joints was occurring in the touch braze process. Because of this, an investigation was made to determine whether specified cleaning processes were adequately removing production processing materials from the aluminum- and silver-coated components prior to brazing.

Cleaning studies were performed to compare the effectiveness of a production-specified aqueous

detergent cleaning method with three cleaning processes specified by the design agency:

1. Pre-silver deposition component cleaning
2. Pre-braze silver-coated component cleaning
3. Pre-braze aluminum-coated component cleaning

Results of the pre-silver deposition cleaning studies indicated no significant residues remained after the components were cleaned with aqueous detergent or specified solvent. However, because of the occurrence of spots, stains, and chloride pitting on parts after solvent cleaning, the Rocky Flats aqueous method has been adopted for cleaning the components prior to silver coating.

Results of comparative cleaning studies for pre-braze silver-coated components revealed excessive hydrocarbon contamination on components cleaned by aqueous methods that were specified by the design agency. The source of the hydrocarbon residue was identified as a contaminated Freon 12 aerosol used to blow-dry the parts and clean the braze chamber. High purity nitrogen was substituted for the contaminated aerosol.

No residues were detected after using the Rocky Flats-documented (M10505-AZ, Section 2) cleaning method, which is being implemented for this cleaning process.

The results of residue analyses for pre-braze aluminum-coated components indicated no significant residues for either cleaning method on the sample coupons tested. However, the geometry of the aluminum-coated parts precludes complete cleaning of the part by the specified technique. The Rocky Flats aqueous cleaning method was recommended for this usage also.

During the evaluation of touch braze cleaning methods, it was observed that a pre-acid etch cleaning process (prior to aluminum coating) could be upgraded. Recommendations were offered to improve that cleaning method.

Future Work

No future work is planned.

STRESS CORROSION TESTING OF 5083 ALUMINUM

Richard A. Patterson

Objective

The objective of this work was to compare the experimental method and results of the alternate-immersion "C" ring test and the constant elongation rate tensile (CERT) test. Both methods reportedly are capable of quantifying susceptibility of aluminum specimens to stress corrosion cracking (SCC).

Prior Work

An extensive evaluation of SCC susceptibility of thermo-mechanically processed 5083 aluminum has been performed and reported by J. L. Briggs et al.¹

Achievements and Discussion

Table 5 summarizes the results of 8 C-rings tested by the alternate immersion technique. Specimen 5 was the only C-ring that did not require metallographic verification of cracks since this specimen was cracked across its entire width. Specimens 1, 2, and 4 exhibited only small cracks ranging from 1 to 2 mm in length. The remaining specimens exhibited extensive pitting corrosion attack.

Metallographic examination of Specimen 5 revealed intergranular cracking, which substantiates the existence of SCC in these specimens and thus verifies the sensitized nature of the "as processed" aluminum plate. The long times required for crack initiation and relatively slow crack propagation in Samples 1, 2, and 4 imply that the material is only slightly sensitized. No direct measurement can be made, however, of the degree of SCC susceptibility by this test.

Standard anodic polarization curves for 5083 aluminum in 3.5 wt % sodium chloride revealed that the characteristic pitting potential (E_p) was -740 mV versus the standard calomel electrode (SCE), and the protection potential (E_{pp}) was -770 mV versus SCE. The consensus is that pitting corrosion initiates and

TABLE 5. C-Rings Tested by Alternate Immersion Technique

Sample Number	Time for Visible Crack (days)	Crack Lengths After 30 days (mm)
1	8	1, 2
2	23	2
3	N. C.*	1
4	17	---
5	5	2.5, 2.5, 3, 7.5
6	N. C.	---
7	N. C.	---
8	N. C.	---

*N. C. - Did not crack within the 30-day test duration.

propagates freely at potentials more noble than E_p . In addition, existing pits will propagate, but no new pits will initiate at potentials between E_p and E_{pp} . Finally, no measurable corrosion occurs at potentials more negative than E_{pp} . Thus, if SCC is truly a combined electrochemical and mechanical phenomenon, this failure mechanism should be active only at potentials approximately equal to E_p or more noble.

Stress-strain curves for 5083 aluminum, as tested in air and in 3.5 wt % sodium chloride with several impressed potentials were obtained. Comparison of these curves revealed that significant reductions in total elongation to fracture and maximum load at fracture occur during SCC. Since samples tested in the sodium chloride solution failed before the air test yield point, the maximum load at fracture (P_{max}) was chosen as the best criterion for comparison, as recommended by deJong.²

Examination of the tensile test data led to the following observations:

1. If the potential is approximately equal to, or more noble than the pitting potential (E_p), the maximum load at fracture (P_{max}) is approximately 55% of the maximum load at fracture for the air test (P_{max}).
2. An intermediate value of P_{max}/P_{max}^0 was obtained for the sample with an impressed

potential between the protection potential (E_{pp}) and the pitting potential (E_p).

3. Finally, if the impressed potential is more negative than the protection potential, P_{max}/P_{max}^0 is approximately equal to 1.

A comparison of results from the C-ring and CERT tests indicates that both methods reveal that the 5083 aluminum specimens were susceptible to SCC. Differences in experimental methods and data analysis are thus the primary sources for choice of investigation technique.

The C-ring test realizes advantages because it more closely simulates static loading applications and is standardized by ASTM. However, the CERT test takes less time and allows the direct comparison of tensile properties rather than the pass/fail nature of C-ring tests. This final difference is the most important one because it provides the capability for a quantitative analysis of the degree of SCC susceptibility. Such analysis would require the generation of a data base that compares thermal aging experiences with SCC susceptibility; however, such analysis would be extremely valuable for quality acceptance or research programs.

Future Work

The constant elongation rate tensile test is a potentially valuable technique for investigations into the mechanism for stress corrosion cracking. Several extensions of this evaluation technique would generate data pertaining to the thermal dependence of SCC susceptibility, weld structure susceptibility, differentiation between hydrogen embrittlement and corrosion enhanced failure mechanism, and finally, baseline data for studies of crack growth rate versus plain strain stress intensity. These tasks will be accomplished through the following endeavors:

1. Determine the thermal dependence of SCC susceptibility by artificially aging 5083 aluminum specimens at several temperatures within the sensitization range of 65 to 200 °C.
2. Determine the susceptibility of simulated production welds made by electron beam cold

wire feed. Comparison of data from the artificially aged specimens and welded specimens would then generate a quantitative determination of weld structure susceptibility.

3. Perform CERT tests with the 5083 aluminum specimen polarized as the cathode rather than anode. The evolution of hydrogen at the cathode will thus expose the sample to external hydrogen atmospheres rather than the anodic dissolution experienced during SCC tests.
4. Use the data generated by the CERT tests to determine the most appropriate thermo-mechanical processing schedules for fracture mechanics specimens. Crack growth rate determinations would then be performed to determine the critical stress intensity factor for stress corrosion cracking.

References

1. J. L. Briggs, M. R. Dringman, D. E. Hausburg, and R. J. Jackson, *Alternate Immersion Stress Corrosion Testing of 5083 Aluminum*, RFP-2645, Rockwell International, Rocky Flats, Golden, Colorado, November 1978.
2. H. F. deJong, "Evaluation of the Constant Strain Rate Test Method for Testing Stress Corrosion Cracking in Aluminum Alloys," *Corrosion*, Vol. 34, pp. 32-36, January 1978.

PILOT PLANT DEVELOPMENT

Donald L. Ziegler

FLUIDIZED BED INCINERATOR ASH IMMOBILIZATION

*Paul M. Williams, James A. Ledford,
and Mike DelHierro*

Objective

The objective of this project is to develop a method for processing several types of radioactive residues

into a form suitable for transportation and long-term storage. A suitable form would be one having (1) sufficiently durable mechanical characteristics to maintain its integrity in the event of physical abuse during handling, and (2) sufficiently inert chemical characteristics to resist attack by the environment in which it is stored.

Prior Work

Of the several methods of immobilization initially tested and evaluated, vitrification, which is the conversion of residues into glass, was selected for further development. This choice stemmed from the product's inherent advantages of non-flammability, chemical stability, and mechanical durability.

The bench-scale vitrification facility has continued to operate in a satisfactory manner, producing free-formed glass pellets from the melt produced in a resistance-heated furnace. The product integrity has been increased by modification to the pellet cooling procedure. An alternate Joule-heated ceramic melter was designed, constructed, and operated.

Achievements and Discussion

Pilot Plant Progress

The purpose of the residue immobilization pilot plant is to obtain design and operational data for vitrification of fluidized bed incinerator (FBI) ash at Rocky Flats, and various simulated transuranic wastes from other nuclear processing sites. This experimental facility will furnish a processing capacity of 4.5 kg/hr.

Title I design for the facility was completed in March 1979. The Title II design phase is now in progress.

Pilot Plant Description

Nonradioactive incinerator ash and other waste material will be delivered to the pilot plant (see Figure 5) area in various containers. The various

FIGURE 5.. Residue Immobilization Pilot Plant

materials will be blended with glass-forming additives in predetermined proportions to produce feed for the process. Resultant feed will be packaged in standard 55-gallon shipping drums and manually transported, as needed, to the process area. The drums (Drum A) will be opened, and the feed conveyed via a pneumatic conveyor through the cyclone into a reactor feed tank. A screw feeder will then convey the feed at a controlled rate from the feed tank into the vitrification reactor. There, temperatures of about 1050 °C will form the feed into molten glass. Three remotely controlled plungers in the vitrification reactor will regulate the flow of molten glass from the reactor. The glass will drip onto a rotary table glass former, producing free-formed solid glass pellets. Pressurized cooling air, supplied by an air blower, will be

directed onto the pellets while they are on the rotary table glass former. This air will cool the pellets to about 50 °C. A vibratory separator will receive the buttons from the glass former. Pellets or fragments that are too large or too small will be removed from the product stream at this stage. The removed fraction will be manually conveyed, as required, to vitrification reactor for remelting.

A product elevator will receive acceptable discharge from the vibratory separator and will convey the pellets upward to allow free-fall discharge into Shipping Drum B. Data gathered from the weigh scales will provide information for process capacity and efficiency calculations. Filled drums will be sent to Waste Management for disposal.

Joule Reactor Development

The conductivity of solidified glass and the raw feed mixture was not expected to be sufficient to allow start-up by simply turning on the power. Therefore, a starting powder consisting of glass cullet (from previous Joule furnace melts) and graphite was prepared. An assembly of two electrodes was constructed to test the starting powder. The powder then was spread on top of the vitrification mix, and electrical power was applied. The carbon burned away in a very short time leaving the molten glass in an ideal state for continuous vitrification. By spiking the molten glass mixture just prior to shutdown, the reactor was able to be started by just passing current through the electrodes.

The design and construction of the second generation Joule reactor is underway. This reactor will be large enough to produce 4.5 kg/hr of waste glass. Thus, if the experimental runs prove it to be superior to the resistance-heated reactor designed for the pilot plant, this reactor will be installed in the pilot plant.

Future Work

Title II design for the pilot plant facility is scheduled to be completed by the end of FY 1979. Construction should be under way during FY 1980. It is anticipated that the Joule reactor will be in operation by the beginning of FY 1980. During this year, experimental runs should provide enough data to make an objective comparison with the existing resistance-heated reactor destined for the pilot plant.

FLUIDIZED BED INCINERATION SYSTEM FOR DEPARTMENT OF ENERGY DEFENSE WASTE

*Andrew J. Johnson, Burke A. Bell,
Dean A. Burton, Scott C. Burkhardt,
Lori L. Luckett, Frank G. Meyer,
Ken E. Sedlmayr, and William F. Tosney*

Objective

The objective of the project is twofold: to demonstrate the adaptability of the fluidized bed incineration

(FBI) process for combustion of a wide variety of transuranium (TRU) contaminated waste materials and to prove scale-up information developed on the pilot plant FBI equipment. The process was scaled up for a capacity of 82 kg/hr (180 lb/hr), which corresponds to a heat release rate of 1,600,000 kJ/hr (1,500,000 Btu/hr).

A minicomputer-based process control and data acquisition system will play an important role in attaining the project objectives. The system will perform real-time data acquisition, in-process calculations, process control functions, inventory control of nuclear materials, in-process alarm monitoring, in-process graphics display, and storage of data for an array of off-line scientific data analysis and reduction.

Prior Work

Construction of the FBI system was completed in FY 1978, and a beneficial occupancy inspection was held on April 27, 1978. System operation tests were performed during May and June 1978, and minor equipment and piping modifications were made concurrently. Some operational problems were encountered during start-up runs, mainly with the sintered metal filters and the system blowers. The computer-based data acquisition network came on-line smoothly. It also proved capable of displaying any one of 61 process variables on the cathode ray tube (CRT), either individually or in prearranged groups. In addition, it can perform hardware diagnostic checks and assist in calibrating instruments. The acquired data is recorded periodically on magnetic media for record keeping and post-run analysis.

Final approval of the Operational Safety Analysis (OSA) was received in October 1978. An Emission Permit Request for operation of the FBI unit was submitted to the Colorado Department of Health Air Pollution Control Division. The license has been received.

Achievements and Discussion

An incineration run on non-contaminated solid waste was conducted in November. The run was

made to subject system components to operational conditions expected during contaminated waste disposal. Keeping the system uncontaminated simplified the maintenance that might otherwise have been required. Start-up proceeded as planned, and the system operated well during the first part of the run. The design feed rate of 82 kg/hr was exceeded by 10% and held for five continuous hours. At this point, the pressure drop across the sintered metal filters had increased to a level that was near the capacity of the exhaust blowers. The feed rate was reduced to allow a continuation of the equipment checkout. Two of the exhaust blowers appeared to operate less smoothly than the other two; impeller clearance later was increased by machining slightly to remove suspected high spots. No other major problems were encountered during the duration of the run.

The excessive pressure drop across the sintered metal filters was the major problem needing correction. A testing apparatus simulating the operating conditions the filters undergo was fabricated using a glass pipe for the filter chamber. This allowed visual observation of the loading and blow-back of the filters under varying conditions of flow and pressure drop. It was found that the 14 ft/min face velocity was too high for efficient blow-back; it was later found that others had also observed this. The old filters are now being replaced with a larger-diameter, new type filter. The increase surface area will permit operating with a total face velocity of less than 7 ft/min, which is within the accepted flow range for these filters.

Conceptual design has been completed on the liquid feed system and associated equipment. Pumps and tankage have been ordered, along with the associated transducers, to allow hookup to the computer data acquisition section and for remote control. This will increase the waste burning capabilities of the system to allow handling of another problem waste stream. The design of the tramp metal removal system for the primary fluidized bed reactor has been completed. The screw conveyor and the discharge enclosure are now being fabricated.

Data acquisition is being continually upgraded and extended by adding process variables to the data base and by extending computer software capabilities.

A Spec 200 Foxboro trend recorder and a twenty-point trend selector panel were added to the system in February 1979. A software addition allows for on-line process monitoring of the high-low alarm status of the process variables. An audible alarm will be tied in to the critical process variables for enhanced monitoring capabilities. Efforts are continuing in the areas of (1) automatic transfer of data from the remote weighing feed station to the computer, (2) automatic printout of data, (3) a graphics display panel, and (4) an on-line heat and material balance for the system. Detailed operating procedures for operation of the data acquisition system have been completed.

Future Work

An incineration run with non-contaminated waste was conducted in April 1979, to bring the process up to design capacity. The initial demonstration operation, with suspect-contaminated waste (< 10 nCi/g), is planned for June 1979. Process optimization will be accomplished by both on-line and off-line data correlation and reduction.

A liquid feed system has been designed and will be installed soon. This system will increase waste burning capabilities of the FBI demonstration-scale plant. Various liquids will be burned during test runs to determine combustion efficiencies, equipment operation, and maintenance histories.

SURFACE WATER DECONTAMINATION

Carl E. Plock and Terry N. Travis

Objective

The objective of this work is to demonstrate that the reverse osmosis (RO) process can be utilized to decontaminate Rocky Flats surface runoff water without damage to the RO membranes.

Prior Work

Prior work at Rocky Flats consisted of the development and evaluation of an RO system for purifying

and recycling effluent from the Sanitary Waste Treatment Plant. This work included laboratory studies and the construction of an RO pilot plant, which is housed in two 40-foot truck trailers. The pilot plant has a water recovery capability of 28,000 gallons per day (gpd).

Prior efforts also have involved the spiking of runoff pond water with plutonium in the RO laboratory. The spiked water was processed by a laboratory RO unit to determine the effectiveness of RO to remove plutonium. The resulting decontamination factors have been as high as 434 at pH values of less than 6. Additional details can be found in previous, classified, progress reports: RFP-2197 (February 13, 1974), RFP-2269 (March 21, 1975), RFP-2400 (June 20, 1975), RFP-2425 (September 16, 1975), RFP-2464 (March 5, 1976), and RFP-2546 (February 14, 1977).

Achievements and Discussion

Ten drums of pond water were spiked with plutonium and processed by the laboratory RO unit after the pH had been adjusted to a range of 6.5 to 8.5. The plutonium spikes ranged from 250 to 750 nanograms for each drum (170 liters) of water processed. The decontamination factor in this pH range was determined to be 2600; however, the plutonium recovery was poor—less than 5%.

Renovation of the RO pilot plant has been completed. However, the pilot plant has not yet been positioned in the drainage area for evaluation tests of the RO system for removing silt and colloids found in surface runoff water. Modification to the National Pollutant Discharge Elimination System (NPDES) discharge permit has not yet been received.

Future Work

The RO laboratory studies will continue. The RO pilot plant will be located in the creek drainage to evaluate the effectiveness of the RO pretreatment system to remove silt and colloids from Rocky Flats runoff water.

CAUSTIC WATER DECONTAMINATION

Carl E. Plock and Terry N. Travis

Objective

The objective of this work is to demonstrate that the reverse osmosis (RO) process can be utilized to decontaminate water containing low concentrations of uranium, plutonium, and americium.

Prior Work

A caustic solution (approximately 0.001M NaOH) became contaminated with very low concentrations of uranium, plutonium, and americium. The water amounted to approximately 400,000 gallons and was stored in one of the asphalt-lined ponds on the Rocky Flats Plant site until it could be processed. A decision was made to use the RO pilot plant, which had been renovated for another program, to process the caustic water. Through use of the pilot plant, data could be obtained to determine the effectiveness of RO for removing actinide elements on a large scale.

Prior work at Rocky Flats consisted of the development and evaluation of an RO system for the purification and recycling of effluent from the Sanitary Waste Treatment Plant. This work included laboratory studies and the construction of an RO plant, which is housed in two 40-foot truck trailers. The pilot plant has a recovery capacity of 28,000 gallons per day (gpd) and a programmable logic controller for control of the operations. Additional details can be found in previous, classified, progress reports: RFP-2197 (February 13, 1974), RFP-2269 (March 21, 1975), RFP-2400 (June 20, 1975), RFP-2425 (September 16, 1975), RFP-2464 (March 5, 1976), and RFP-2546 (February 14, 1977).

Current Achievements

The pilot plant was operated for two weeks. The only problem that developed was control of the pH. This was due to the large quantity of algae in the caustic water.

Analytical samples of the feed water and product water are being analyzed.

Future Work

No further pilot plant work is planned for this project. When the analytical data is received, it will be analyzed and a report will be written.

SOIL DECONTAMINATION

*Doyle L. Mitchell, Peter T. Faccini,
and John E. Garnett*

Objective

The objective of this work is to develop a dustless process for decontaminating soil at the Rocky Flats Plant. The project will include preparation of design criteria and equipment specifications for a production-scale facility at Rocky Flats. Data will also be generated to help formulate an economic evaluation for determining the best method for disposing of the soil.

Background

Drums of contaminated machining oil and solvent were stored onsite between 1958 and 1968. A 370- by 400-ft area of the Rocky Flats Plant was contaminated with plutonium, americium, and uranium when the drums deteriorated and leaked. Contamination levels were monitored at depths of 6 to 12 in. with readings of 20 to 60,000 dpm/g. The contamination is dispersed in 10,000 to 18,000 tons of soil. In 1969, the soil was covered by a barrier consisting of 3 in. of fill dirt, 6 in. of sub-base, and 3 in. of asphalt. The Atomic Energy Commission promised Colorado Governor John Love in 1971 that the contaminated soil would be removed and decontaminated as soon as an acceptable method was developed.

Prior Work

Laboratory studies have shown that plutonium contamination is attached to the -35 mesh (-0.42 mm) soil particles. A basic solution (pH 11) aids in dispersing the small particulate contamination from

soil particles. Using the basic solution in combination with scrubbing and wet screening, 65 to 70 vol % of the Rocky Flats soil can be decontaminated.

Achievements and Discussion

Pilot-scale equipment has been installed in the soil decontamination laboratory and is being operated on noncontaminated soils. Water recycle experiments are in progress to flocculate and remove colloids formed in the basic wash solution. Experiments have demonstrated that the chemical and physical makeup of the soil has a dramatic effect on flocculation of the colloid in the wash solution.

The Colorado School of Mines Research Institute (CSMRI) has been contracted to provide process information generated using noncontaminated soils. Tests at CSMRI are being conducted to demonstrate that the proposed process will disperse and separate size fractions of the soil as required. Equipment engineering data and utility requirements will be obtained from this study. The final report from CSMRI will be delivered to Rockwell at Rocky Flats by June 1, 1979.

Three tests were completed at the manufacturer's pilot plant. Additional tests that were planned were canceled, however, because of the difficulty in shipping Rocky Flats soil across the nation. One manufacturer, Telesmith, investigated scrubbing and obtained information as to retention time, scrubber size, and water usage. Another company, Dorr-Oliver, looked at thickening, phase separation via filtration, and centrifugation. Filtration was unsuccessful, but centrifugation looked promising. Bird, a third manufacturer, ran low-speed centrifugal dewatering tests, which also looked promising.

Future Work

Tests on contaminated soil will begin in the Rocky Flats soil laboratory on a process that includes washing/scrubbing, wet screening, flocculation, centrifuging, and ultrafiltration. Water recycle methods will be developed for treating the wash solutions used on soils having different physical and chemical properties. Process information will

be incorporated into a Conceptual Design Report for the production-scale facility.

LEAD-LINED GLOVE INCINERATION

Karin Y. Gallagher and Alfred L. Johnston

Objective

The objective is to develop and implement an experimental program to generate basic process information for combustion of used lead-lined rubber gloves.

Prior Work

Work on the incineration of lead-lined gloves was last performed in 1973.

Achievements and Discussion

At present, work is concentrated on fluidized bed incineration, with the intent of developing a process for demonstration on the pilot plant facility, Room 135, Building 776. Pyrolysis of the gloves in a fluidized bed is a new process.

Fluid bed incineration offers several advantages. It is both a simple means of agitating the ash, effecting exposure of lead oxide to the reducing atmosphere, and — facilitated by the drainage of molten lead through the bed — an efficient mechanism for separating the heavy metal product from the lighter carbon char. Moreover, with additional development effort, in situ chloride neutralization may be possible.

Several varied atmospheres were tested. They included nitrogen, one volume percent oxygen in nitrogen, nitrogen with 18.8 percent steam by weight, and nitrogen containing 0.506 percent carbon monoxide by weight. Incineration in both pure nitrogen and oxygen-enriched atmospheres proved unsuccessful even at bed temperatures in excess of 950 °C. Nitrogen pyrolysis failed to reduce the lead oxide to an acceptable, stable form. The addition of oxygen produced a predominant

yellow lead oxide (PbO) residue, unsuitable because of its corrosive properties and because of its tendency to form an eutectic melt with the ash or refractories that could be used in the reactor.

Steam-assisted reduction did produce large globules of metallic lead. Nevertheless, the process is made undesirable because of the slow rate of reaction (necessitating either a large reactor vessel or an extended operating period) together with the safety concerns inherent in the addition of water to a vessel processing plutonium-contaminated materials.

Preliminary tests of the carbon monoxide - spiked pyrolysis process indicate a rapid conversion of the red lead oxide, Pb_3O_4 , can be affected at approximately 700 °C. The resultant metal readily migrates to the bottom of the bed, amassing in large pools.

Three bed materials were evaluated: sodium carbonate, chromic oxide on alumina, and untreated alumina sand. The sodium carbonate was rejected because its porosity allowed absorption of the ash and metal residue. Both aluminas exhibited greater corrosion resistance.

Future Work

The fluidized bed incineration process involving a carbon monoxide - nitrogen atmosphere will be further refined. Development efforts will seek to devise a means of draining the metal from the bed and subsequently incinerating the carbon char by-product. In addition, a fourth potential bed medium, calcium carbonate granules, will be studied. It is hoped the carbonate sand bed will neutralize the chloride-rich offgas.

DEVELOPMENT OF URANIUM CHIP BURNING PROCESS

Donald J. Vickery and Patrick M. Arnold

Objective

The present Rocky Flats uranium chip burning system is an obsolete, inefficient operation and a

FIGURE 6. Proposed Uranium Chip Burner

potential health and safety hazard; consequently, a starved oxygen system for the safe incineration of depleted uranium is being developed. A current objective of this project is to develop and demonstrate a safe, efficient pilot-scale uranium chip incineration system. A second objective is to provide supporting data for a new offgas cleaning system being designed by Facilities Engineering and Construction (FE&C) for the present incinerator. Additionally, data from the pilot plant unit will provide FE&C with design criteria for the future construction of a new production process unit.

Prior Work

Laboratory tests were first run to determine if either nitriding or starved air combustion was a viable disposal process for depleted uranium chips. Data gathered indicated that nitriding reaction rates were too slow, but that the direct conversion of uranium turnings to U_3O_8 in a starved oxygen reactor was a feasible design alternative. A conceptual design, which was developed for chip disposal (Figure 6), incorporated the starved oxygen concept. Detailed design of a pilot unit featuring starved oxygen

combustion, 3.6 kg/hr capacity, recycled offgas, and an offgas cleaning system was then completed.

Achievements and Discussion

During this report period, assembly and installation of the pilot chip incinerator was completed. Initial operability testing indicated some exhaust modifications were required. These modifications have since been implemented. An electric igniter for initiating combustion within the reactor was found and ordered.

Upon completion of a detailed design, the required equipment was ordered. Off-the-shelf purchases included the exhaust/recycle blower, heat exchanger, instrumentation, and filters. Engineered equipment built at Rocky Flats consisted of the reactor vessel, cyclone separator, filter housings, and support stand. The air-locked batch feeder, catalytic afterburner, and exhaust blower (see Figure 6) are not necessary for pilot operation because both the exhaust and the recycle functions were combined into one blower.

Process and Equipment Description

The reactor vessel was sized and built for 3.6 kg/hr capacity. This was accomplished by charging the reactor with 10.9 kg of depleted uranium and controlling the reaction rate over a three-hour period. Rate control was achieved by recycling the oxygen-starved offgas and regulating the amount of combustion air introduced. The air-lock batch feeder is not required for charging the chips because of the small size of the pilot unit. Ignition is started with a flameless electric torch, and combustion proceeds down from the top of the reactor as countercurrent combustion air is introduced. By burning in this manner, a heat zone is created, which in theory will pyrolyze or burn cutting oil on the chips and therefore eliminate the need for a catalytic afterburner. The afterburner will not be included on the pilot chip incinerator because of the countercurrent burning.

Heat of combustion is carried from the reactor by recycled flue gas. The gas is first cooled by dilution with recycled exhaust gas and then passes through a

cyclone separator and sintered metal filters where any fly ash oxide that is present is removed. The gas then passes through a water-cooled shell and tube heat exchanger before (1) being exhausted through HEPA filtration to the building plenum or (2) being recycled to the reactor for heat removal. A single blower is capable of performing both the exhaust and the recycle functions because of the small size of the pilot facility.

The product, uranium oxide, is removed from the reactor at the end of the cycle, after cooling is complete. Some oxide, as fly ash, is also expected to be collected by the cyclone and sintered metal filters. The fly ash can be either recycled if combustion is not complete or included with the reactor ash for disposal.

Process Developments

The pilot incinerator system was designed and built as a single assembly on a support stand. It was constructed in noncontaminated zones as a unit to minimize the effort required by craftsmen in a contaminated area. The procedure was successful in that the assembly was transported and installed intact without the need for reassembly in the contaminated room containing the production incinerator.

Initial operability testing showed that exhaust piping modifications were required for better flow control. Because of higher dust loadings, flow through the system was too great, causing high pressure drop across the cyclone and the sintered metal filters. This was resolved by adding a blower recycle loop to reduce flow discharged to the reactor and cooling gas loops. Improved control was gained with the modifications.

A flameless electric torch was found for initiating combustion of uranium turnings in the reactor. The torch operates by heating an inert argon or nitrogen stream to approximately 800 °C to ignite the chips. The torch can be inserted through a nozzle in the sealed reactor. This eliminates the need of an open port for ignition as presently exists in the production reactor. The electric torch should bring about a safer operation.

Future Work

Planned work includes operational testing on actual depleted uranium, system modifications if necessary, and pilot-scale demonstration of the process. Specific areas of interest will be fly ash and particulate collection, reaction efficiency, process control, and effects of cutting oil combustion.

INSTRUMENTATION AND STATISTICAL SYSTEMS

Richard N. Chanda

PLUTONIUM DEPOSITION ON LANDS ADJACENT TO THE ROCKY FLATS PLANT

Merril W. Hume

Objective

The objective of this work is to develop soil sampling plans and controlled analytical procedures to determine levels of plutonium deposition on lands adjacent to the Rocky Flats plant. Data from initial sampling programs are to be used in litigation proceedings in which the plaintiffs charge that activities at the Rocky Flats plant have contaminated their land. In the process of meeting this objective, various soil sampling techniques, in addition to analytical laboratories and related procedures, are to be assessed.

Prior Work

Soil samples were collected from lands under litigation, according to a statistically designed sampling plan. At each sampling site, samples were taken using four different collection techniques. These samples, along with blanks and fabricated standards, were analyzed by Eberline Instrument Corporation of Santa Fe, New Mexico for plutonium and americium. This sampling plan, plus analytical procedures and some analytical results, were presented at pretrial hearings before the Colorado U.S. District Court.

At the suggestion of the court, an extensive retesting program was initiated. Major modifications over the original program consisted of additional sampling sites as selected by the plaintiffs, interlaboratory analyses, and increased quality control for the analytical laboratories. The last item required the use of field consensus standards and extensive blinding of samples. Rocky Flats personnel relinquished all monitoring of this retesting program. The results of this program were given to the court in January 1979.

In the fall of 1978, another soil sampling program was initiated to characterize plutonium deposition on unlitigated lands adjacent to the Rocky Flats plant. This program was statistically designed, as was the litigation program. Extensive quality controls, as employed in the retesting program, also were used. Only one soil sampling technique was used for this program. Results of the program were made available to participating land owners in April 1979.

Achievements and Discussion

Analytical results of all soil testing programs, totaling 168 sampling sites, have been completed. Analyses indicate excellent agreement with results of the various programs in addition to interlaboratory and intralaboratory replicate analyses. The data showed no surprises, and the goals of the sampling design were met. No analytical results exceeded the suggested EPA guidelines for transuranic deposition. A limited number of sites to the east of the Rocky Flats plant do exceed the Colorado guideline. A comprehensive health risk assessment employing results from lands under litigation has been performed by the Inhalation Toxicology Research Institute of Albuquerque, New Mexico. They conclude the health risk due to transuranic deposition is negligible.

Future Work

No additional sampling or analysis for plutonium in soil adjacent to Rocky Flats Plant is anticipated. Statistical analyses of data from this program will provide comparisons of sampling methods as well

as an assessment of laboratory capability. Results of this program will also play a major role in the upcoming land litigation trial.

STANDARDS FOR ATOMIC ABSORPTION ANALYSIS

Merril W. Hume

Objectives

Chemistry Standards Laboratory has developed a new technique for fabricating National Bureau of Standards (NBS) traceable standards for impurity analysis of plutonium metal by atomic absorption. The objective of this work is to perform statistical assessment of this technique for six elemental impurities. Several homogeneous plutonium metals are then to be characterized so that they may be used for quality control.

Prior Work

Production standards are currently used with each elemental atomic absorption run for calibration. No quality control metals are available.

Achievements and Discussion

Data have been collected using the new standards in addition to production standards. Preliminary statistical analysis indicates a large day-to-day variation in impurity analysis. There also appears to be a bias between the two sets of standards for some of the impurities.

Future Work

Statistical analysis of existing data is to be completed. Also, more NBS traceable metal is on order and will be used to fabricate a new set of standards. These standards will be useful for determining existing biases. Upon characterization of homogeneous metals, quality control programs will be developed.

MEASUREMENT ASSESSMENT PROPOSAL

Elizabeth L. Baker

Objective

The purpose of this proposal was to outline an action plan for a comprehensive measurement assessment program at Rocky Flats.

Prior Work

The Statistics Laboratory has on numerous occasions been involved in various aspects of the Rocky Flats measurement assessment programs; i.e., review, evaluation, modification, plus the use of these programs for such purposes as calculating limits of errors and reporting precision and bias.

Achievements and Discussion

A request was made by plant management for a plan of action on an integrated system for nuclear materials measurement control. A joint action plan was prepared with the Standards Laboratory to (1) determine the requirements for a measurement assessment program at Rocky Flats, (2) assess existing programs, (3) identify problem areas, and (4) design and implement a new measurement assessment system.

Future Work

The Statistics Laboratory will continue to provide assistance as needed. At present, a plant committee is preparing a comprehensive overview of Safeguards at Rocky Flats of which measurement assessment is a part.

SAMPLE AND INVENTORY FILE

Elizabeth L. Baker

Objective

The purpose of this project is to determine whether an inventory of a random sample of records from

a data file will give results equivalent to a total inventory. Cost savings may be appreciable.

Prior Work

No earlier work has been reported.

Achievements and Discussion

A computer program has been written for General Accounting to determine the total number of records on an inventory file and to generate a new file containing a random sample of records from the total inventory file. This program has the option of subsetting the inventory file before creating the sample file. It is now being used in a study by General Accounting to determine whether results from the sample are compatible with those from the entire inventory file.

Future Work

No further work is expected.

ACCIDENT SUMMARY PROGRAMS

Elizabeth L. Baker

Objective

The object of this project is to summarize accident data, using the computer, when the data becomes too unwieldy to manage by hand. The current work is necessitated by changes made in the data to yield more meaningful information.

Prior Work

Computer programs to summarize accident data were written by the Statistics Laboratory five years ago. Since then, the primary function of the Statistics Laboratory on this project has been to maintain the programs and correct errors in the data.

Achievements and Discussion

Since the beginning of the programs, use of the accident data has been increasing in new areas. These include use in accident prevention to determine causal factors as well as a working tool for supervision to pinpoint problem areas. As their usefulness expanded, further refinements in the data were required to contribute more meaningful information. These refinements include such things as additional variables, a breakdown of some existing variables into subsets, and secondary codes. To handle these changes, the computer programs have been rewritten, and the data file has been updated. The new system has been put into production.

Future Work

Program maintenance will be continued.

W-80 AND B-61 ASSEMBLY

Elizabeth L. Baker

Objective

The purpose of this study is to identify problems involved in meeting assembly requirements of the W-80 and B-61 programs as specified by Los Alamos Scientific Laboratory.

Prior Work

No previous work has been reported.

Achievements and Discussion

To identify problems inherent in the assembly of composite parts for the W-80 and B-61 programs, a Monte Carlo study is being made to determine the effects of varying certain parameters. Data on parts from other programs are being used where available. Orallo data from Y-12 have been incorporated into the study.

At present, a major problem area has been determined, and the percentage of assemblies that can be made has been estimated.

Future Work

Future work on this project will include merging additional data into the program as it becomes available, assisting in the definition (and possibly the implementation) of the selection algorithm used in production, and may include statistical analyses on the actual data once production begins.

AQUEOUS X RAY VERSUS DIRECT ANALYSIS

Elizabeth L. Baker

Objective

The purpose of this analysis was to compare a new method (direct analysis of compressed turnings) with the standard method (aqueous X ray) to determine the amount of niobium in binary turnings. If direct analysis shows no loss in precision over aqueous X ray, then direct analysis will replace the latter method, resulting in a significant time savings.

Prior Work

This was a new objective.

Achievements and Discussion

An experimental design was set up to compare the two methods of determining the percent of niobium in binary turnings. A statistical analysis of the resulting data showed that the two methods did not differ significantly with respect to measurement variability. Another analysis on production data showed that the variability for direct analysis was significantly smaller than that for aqueous X ray.

Future Work

No further work is expected.

CHEMISTRY STANDARDS LABORATORY CALORIMETRIC UNCERTAINTIES

Kenneth B. Gerald

Objective

The objective of this work is to provide a procedure for estimating the limit of error on the total grams of plutonium as determined calorimetrically by the Chemistry Standards Laboratory (CSL).

Prior Work

Since 1972, CSL calorimetry results for selected packages have been compared with calorimetry results from the Mound Facility through a sample exchange and verification program. During a meeting held December 7, 1978 with the safeguards and security auditors from DOE-ALO, it was agreed that for the 1979 verification program, calorimetry measurements made at Rocky Flats would not be exchanged with Mound. During this meeting, it was further agreed that Rocky Flats will publish a report comparing Rocky Flats calorimetry plutonium values from 1972 through 1978 with those from Mound. The report would include the uncertainties associated with Rocky Flats measurements.

Achievements and Discussion

The approximate variance of the total grams of plutonium in a sample was derived by using a general technique described by Jaech.* This technique was developed for estimating the variance of an arbitrary function of random variables. All meaningful sources of error given in American National Standard N15.22-1975 have been incorporated into the error propagation. A computer program has been written to calculate the limit of error ($2 \times$ standard deviation) on the grams of plutonium for any sample.

Future Work

Currently CSL is performing experiments to update the uncertainties of wattage measurements of the CSL calorimeters. This could greatly improve the

*Jaech, J. L., *Statistical Methods in Nuclear Material Control*, p. 152, TID 26-298, Office of Information Services, Department of Energy, Oak Ridge, Tennessee, 1973.

reported limit of error on CSL calorimeters since wattage measurement is the major contributor to overall uncertainty of the plutonium measurement.

STATISTICAL CONSULTING

*Elizabeth L. Baker, Kenneth B. Gerald,
and Merril W. Hume*

Objective

The objective of this effort is to provide, upon request, statistical and mathematical guidance and technical support to all Rocky Flats operations. Typical areas of assistance are experimental design, sampling plans, data analysis, technical programming, and mathematical analysis.

Prior Work

This function has been ongoing for a number of years.

Achievements and Discussion

Assistance was given with quality control programs for production, analytical laboratories, and environmental problems; modifications were made to the ID control charts; and limits of error were placed on shipper-receiver differences. Other areas receiving assistance included routine monitoring of control programs, part defective prediction modeling, and support to national ANSI and ASTM statistical subcommittees.

Future Work

Several of the above programs are on an ongoing basis. Requests for assistance are continual.

COMPUTER EXPANSION

James L. Lawless

Objective

The objective of this work is to expand the present computer system to provide a mass storage medium

with rapid access. Several pending projects require large data-base reduction or high-level language compilation.

Prior Work

The Hewlett Packard computer system has been in routine use for several years. Its primary use has been program development for a production computer used to control three radiometric counting systems.

Achievements and Discussion

The recently implemented disc operating system has been utilized to reduce a large amount of data generated by the air quality mobile van. These data will be used in the "Brown Haze" study. The system has also been used to place mnemonic coding on ladder diagrams for the Water Recycle Programmable Logic Controller. This will eliminate the need for hundreds of drafting hours. An INTEL 8080 Microprocessor cross assembler has been written to aid the developers of these process control functions.

Future Work

Digital plotting of spectral data, crate counter data, and general purpose routines are soon to be added. Interfacing to a Hewlett Packard calculator for calorimeter end point prediction will be implemented.

PART VERIFICATION COUNTER

James L. Lawless

Objective

The objective of this project is to provide instrumentation capable of determining or verifying that a specified part reported to be in a shipping container actually resides within the container. Also, where possible, the instrumentation should report any abnormalities that would give evidence of some

additional material that may inadvertently have been placed in the container. These measurements will be made by observing the penetrating gamma and neutron emissions.

Prior Work

The concept was defined and request for funding was specified about two years ago.

Achievements and Discussion

Meetings have been held with the ultimate user, the engineering staff, and instrument developers. Problem definitions and preliminary design criteria have been established. Specific instrumentation details and their related costs have been transmitted to the responsible project engineer for inclusion in the total project cost and layout.

Future Work

A considerable effort will be required to install the counter and to calibrate the system for the many types of parts anticipated. This will include determining typical gamma spectral data for each part. These data will then be stored on computer disc. An unknown will be compared to its associated stored spectra and/or gross gamma and neutron emissions. A window of acceptable limits will be imposed and the response of the unknown will be reported and documented.

DEVELOPMENT OF RADIOMETRIC MONITORS - BUILDING 371

*Francis X. Haas, James L. Lawless,
Wayne E. Herren, George P. Minges,
Michael A. Fox*, Charles H. Jacobson,
and Ronald L. Gornick*

Objective

The objective of this work is to develop design criteria and systems for radiometric assay

*Cooperative education (CO-OP) student.

instruments to be installed in the new plutonium recovery facility (Building 371).

Prior Work

Specifications for eight nondestructive assay instrumentation packages were written and the equipment was ordered. These systems include four NaI residue counters for 4-liter packages, a molten salt counter, a combination plutonium-acid normality analyzer for liquids, a calcined oxide counter, and a 20-cell calorimeter for oxide measurements. Building modifications and cable requirements were specified.

Achievements and Discussion

Molten Salt Counter

The computer system for the molten salt counter has been received and installed temporarily in Building 881. This system controls a stepping motor to drive the segmented elevator, and relay closures for controlling the turntable motor and operating the transmission source. The computer system has been inspected and accepted from the vendor. The turntable and segmenting elevator for the assay of material has been designed for installation in a glove box. Construction of the mechanism has started and is expected to be completed by July 30, 1979.

Neutron Counters

A thermal neutron coincidence counter has been specified for assaying calcined oxide in the range of 800 to 2000 grams. Neutron coincidence counting electronics are on order with a delivery date of late June. The glove-box appendage is in place in Building 371. Construction of the ^3He detector assembly has started with an expected completion date of late May.

Residue Counters

The four residue counters are scheduled for installation to measure waste generated in the button

breakout area, metal and glass leach module, and high specific activity (HSA) ash collection area, and HSA combustibles from the combustible separation area. Each counter consists of two sodium iodide detectors, which are located one above the other. The shields around the detector and around glove-box appendages were modified to house a transmission source and shutter for each detector. This arrangement was chosen to eliminate the axial movement of the sample, yet retain the axial measurement of samples as presently employed in the Rocky Flats helix counter.* In addition, neutron coincidence electronics were ordered for each counter for adding at a later date.

Scaler electronics packages associated with each detector have been moved to the Stacker Retriever computer room in Building 371 to establish communications protocol for transmitting and receiving data.

Future Work

As equipment is received and/or built, it will be tested and calibration experiments will be undertaken to test the design criteria. Protocol will be established between each instrument and the Stacker Retriever computer for the transmission of information for nuclear material accountability and control.

SEGMENTED DRIVE UNIT

*Charles H. Jacobson,** John D. Bert,**
and James L. Lawless*

Objective

The objective of this work is to provide a mechanical device for handling cans of processed residues to be assayed in Building 371 for plutonium and americium content.

Prior Work

No prior work has been reported.

*J. L. Lawless and R. N. Chanda, "A Plutonium Waste Counter," *Trans. Am. Nucl. Soc.* 13, p. 748, (1970).

**Component Development personnel.

Achievements and Discussion

The existing Can Scan unit was used as a guide for the design of this device. Design modifications were necessary to make the unit small enough to fit into an existing well in a glove-box floor, and light enough to be easily removed for maintenance.

A satisfactory design has been drawn, approved, and is now under construction in the Instrumentation and Statistical Systems machine shop.

Future Work

This design will be turned over to Engineering for incorporation into a project in Building 776 that involves two of the scanning devices.

CRATE RADIOASSAY COUNTER

Ronald A. Harlan, Jack N. Carlson,
Wayne E. Herren*

Objective

The objective of this work is to develop a method to detect and estimate plutonium in $1.22 \times 1.22 \times 2.13$ m ($4 \times 4 \times 7$ ft) crates and to perform a safeguards check on crates being shipped to offsite burial.

Prior Work

The neutron-counting part of the crate counter was operational in June 1978. Two sodium iodide gamma ray detectors were installed on the top side of the counter to evaluate the detectors potential for estimating excess neutrons produced by (α ,n) reaction** on light elements.

Achievements and Discussion

The counter, which was calibrated with high-fired PuO_2 sources with relatively low (α ,n) production,

*Currently with the Wind Energy Systems group.

**Indicates nuclear reactions induced by alpha particles with emissions of neutrons and gamma rays.

responded to production crates by overestimating the contents. This was determined by comparing the results of crate counter assays to contents assayed separately in 55-gallon drums and subsequently emptied into crates. There was a mean overestimate of 5 with a range of 1 to 20.

For routine operations, the original calibration of the crate counter has been changed to lower estimates by a mean factor of 5. After 6 months of operation, detector response to calibration sources had changed enough that electronic resetting was required to restore original response.

A slow neutron coincidence counter has been borrowed from Los Alamos Scientific Laboratory (LASL). Discussions with LASL personnel indicate that this unit may be able to operate with the small signal-to-background ratio at the crate counter. If successful, the instrument can select only time-correlated neutrons from spontaneous fission of plutonium-240 in the crates and allow assays independent of variable (α, n) reaction neutrons.

A high-purity germanium (HPGe) gamma ray detector was placed on the side of the counter to augment the sodium iodide detectors on top. It was not possible, however, to detect enough (α, n) reaction gamma rays to correct neutron output for the highly variable (α, n) reaction neutron production. This was true even with 6- to 8-hour counting times except when PuF_4 was present in gram quantities. The HPGe detector does provide a second method for qualitative or even semiquantitative analysis of crate contents.

Combining gamma ray estimates with neutron estimates, the counter has identified several unusual situations; e.g., crates with tens of grams of plutonium that were thought by waste originators to have less than 1 gram, apparent mistakes in sequential labeling of crates, and the presence of uranium-235 in crates believed to have none.

It was recommended that the capability for high resolution gamma ray counting be installed when funds are available.

Future Work

The slow neutron coincidence counter will be adapted to this system. Attempts will be made to quantify errors in gamma ray assay to improve its present qualitative status.

HIGH RESOLUTION PACKAGE COUNTER "CAN SCAN"

*Francis X. Haas, James L. Lawless,
Wayne E. Herren, and Marvin E. Hughes**

Objective

The objective of this work is to provide an instrument capable of measuring the plutonium and americium content of process residues, with special emphasis on the molten salt category.

Prior Work

The system has been installed and operating as a production instrument since October 1978. System software had been modified since that time.

Achievements and Discussion

The credibility of the counting system has been improved. The initial calibration constants were obtained from a series of samples having a maximum of 80 grams of plutonium and less than 0.5 percent americium relative to plutonium. Routine samples contain up to 400 grams of plutonium and up to 10 percent americium relative to plutonium. Three plutonium samples containing 100, 200, and 400 grams of plutonium were made to increase the dynamic calibration range. Two americium samples containing 5 and 30 grams respectively were fabricated. These samples provided calibration constants to allow conversion of peak areas to grams of americium.

Interferences between the 414-keV plutonium peak and the 419-keV americium peak were investigated. This was done using three fabricated samples

*Chemical Operations group.

containing 4, 10, and 20 percent americium in the plutonium. Data taken from this study are still under investigation.

A working standard has been prepared for use in checking day-to-day operation of the electronic system. This standard contains 100 grams of plutonium, 0.43 gram of americium, and 38 grams of enriched uranium.

Future Work

A major effort will be placed on refining the plutonium and americium calibration and investigating peak overlap and interferences in the 410- to 425-keV energy region. System response to other plutonium waste categories and uranium bearing wastes also will be investigated.

GAMMA RAY ISOTOPIC MEASUREMENTS

Francis X. Haas

Objective

The objective of this work is to establish non-destructive techniques for measuring plutonium and americium isotopic ratios for safeguards assay and standards evaluation of nuclear materials.

Prior Work

Gamma-ray spectra were analyzed to help calibrate samples for the "CAN SCAN."

Achievements and Discussion

Fortran programs were obtained from Ray Gunnick of Lawrence Livermore Laboratories, and John Lemming of Mound Facility. The Fortran programs have been used to measure plutonium-americium ratios and plutonium isotopic measurements to help various groups meet quality assurance requirements for product materials. The isotopic values have been used in conjunction with calorimetrically determined sample heat to certify samples to be used in calibrating nondestructive assay instruments.

Future Work

Investigation of high resolution gamma ray spectroscopy will continue. The purpose will be to bring all applications to provide state-of-the-art analytical techniques for all appropriate applications at Rocky Flats.

UPDATE OF SALTS CRATE SURVEY METER

*Ronald A. Harlan and Jack N. Carlson, Jr. **

Objective

The objective of this project was to provide a simple, fast checkout of crates containing salts originating at the Waste Treatment Facility.

Prior Work

There was no prior work on this specific application; however, Instrumentation and Statistical Systems has provided development and user assistance for glove-box and tank surveys.

Achievements and Discussion

Crates of salts originate in the Waste Processing building when precipitations are carried out for cleanup of transuranic activity in chemical processing solutions. The activity is sorbed in the precipitates. In terms of radioactive species in the original solutions, concentrations of plutonium and americium are usually less than 10^{-3} grams per liter. The precipitates in the crates usually have less than 3×10^{-11} grams of plutonium or americium per gram of precipitate. Survey meters are used to check for "hot" spots or high concentrations that could be of safeguards or safety concern. In the meters being used, it was found that the sodium iodide detectors had been damaged. These were replaced and a simple lead collimator with a cadmium and copper end window was designed and installed over new detectors. The new configuration was tested with sources and calibrated for semi-quantitative use should a "hot" spot ever be found in these crates.

*Currently with the Wind Energy Systems group.

Future Work

No future effort is planned.

CALIBRATION AND INVENTORY VERIFICATION EXPERIMENTS

*Ronald A. Harlan, James L. Lawless,
and Kenneth B. Gerald*

Objective

This work provides a data base for nondestructive assay (NDA) calibrations for radiometric counters at Rocky Flats.

Prior Work

These studies have continued since 1964 when the first drum counter was installed. Instrumentation and Statistical Systems shares in this effort by designing sampling plans and calibration models, analyzing and interpreting data, implementing new or revised calibrations, and participating in the Measurement Control Task Force. Since 1974, counters have been compared, when feasible, to calorimetry results.* Since 1972, calorimetry results have also been obtained by shipping 4-liter packages of waste to the Mound Facility in Ohio.

Achievements and Discussion

Results of the 1978 exchange experience with Mound Facility were analyzed to compare Mound Facility calorimetry with that from Rocky Flats. Significant biases were found to exist in three categories: ash heels (IDC 421); pulverized sand, slag, and crucibles (IDC 398); and graphite (IDC 312). Based on these results, inventory adjustments are being made for ash heel residues in the Rocky Flats inventory. Programming assistance has been given to Production Control to adjust the ash heel values.

In cooperation with the Chemistry Standards Laboratory, a continuous sampling plan for inventory has been developed for 1979 and future years.

*See the previous progress report, RFP-2877, p. 35.

Under this plan, samples will be withdrawn over several months and counters will be "back-calibrated" against calorimetry values for the samples. This represents a change from past practice that tended to concentrate on statistical selections from production batches generated over long times but counted over short times of a few days. These data also will constitute a new base for the Measurement Control Program run by the Chemistry Standards Laboratory.

The Albuquerque Operations Office of DOE (DOE-ALO) has agreed to perform the 1979 verification using Rocky Flats calorimetry overseen by DOE/ALO or RFAO personnel. Some sample selections have been made. The LECO Crucible residues will be studied over the next year. Molten salt assays values from the newly operational CAN SCAN counter will also be verified.*

In the Waste Packaging Facility drum counters, a new check source was put into service, and operating limits were derived. Support was given to other R&D groups and Waste Management by data reduction on drums containing special materials not usually found in the Rocky Flats plutonium production stream.

Future Work

During the coming period, we expect to analyze data on the following residues: LECO crucibles, insulation, 8 X 8 filters, and cemented insulation by drum count; molten salts by CAN SCAN count; and by calorimetry, those 4-liter packaged residues selected in May 1979 by DOE/ALO auditors.

CALORIMETER END-POINT PREDICTION

*George P. Minges, Michael A. Fox,**
and James L. Lawless*

Objective

The objective of this effort is to develop techniques to predict the final value approached by a calorimeter

*See p. 33 of the previous progress report, RFP-2877.

**Cooperative education (CO-OP) student.

before that value is actually reached. The result would be a time savings in analysis and equipment use.

Prior Work

A minicomputer system has been demonstrated that collects and processes data to attain an end-point within 5 percent accuracy.

Achievements and Discussion

Further investigation into the algorithms generated to predict the end point yielded a new set of techniques, which include accuracy to within 3 percent at a 75 percent time savings. The algorithms are being completed and programming of these new algorithms is underway. Higher levels of accuracy are anticipated, and evaluation is currently underway with the assistance of the Chemistry Standards Laboratory calorimeter.

Future Work

Evaluation of the system and end-point prediction techniques will be completed. Maximizing throughput with respect to accuracy will be a consideration.

PHYSICAL SECURITY INTRUSION DETECTION SYSTEMS

Grover J. Cunningham and Joseph M. Milan

Objective

The objective of this work is to lower false alarm rates and to improve the levels of response for intrusion detection systems in various onsite storage vaults.

Prior Work

Work of a similar nature has been underway for some time; e.g., an infrared security system and a

vibration detection system have been installed and tested as reported in the previous progress report (RFP-2877, p. 37).

Achievements and Discussion

An outdoor microwave motion system has been received on loan from Sandia Laboratories and is being investigated for possible application in long hallways or tunnels at Rocky Flats. This system has been tested for outdoor use by Sandia Laboratory; however, they have no data on application for this system indoors. Another outdoor system received from Sandia Laboratories for indoor tests was an "E" field fence system. This fence system is being evaluated for possible use on or along shelving in Special Nuclear Materials (SNM) areas.

A combined system of infrared motion detection, vibration sensors, and a video motion system has been installed in a vault. The video motion system, which is new to Rocky Flats, consists of a video camera and alarm detection module.

A microwave detector was incorporated with the infrared and vibration systems reported earlier (RFP-2877, p. 38) to complete the detection system for another area.

Future Work

Sensitivity and false alarm information from installed systems will be used to improve the design of other intrusion detection systems. Data will be taken and analyzed for the outdoor microwave and E-field systems on loan from Sandia.

INSTRUMENTATION FOR A SOIL DECONTAMINATION PROJECT

*Ronald A. Harlan, Francis X. Haas,
and James L. Lawless*

Objective

This project will recommend, test, and specify equipment for continuously monitoring soil before and after it is decontaminated by a washing

process. A means is needed for continuous monitoring of transuranic alpha activity at 20 to 25 alpha counts per minute per gram (approximately 10 pCi/g) of soil.

Prior Work

No prior work has been reported, although some preliminary scoping and estimating have been performed.

Achievements and Discussion

Assay problems have been identified; however, solutions to those problems will require development work and experimental testing. The assay problems are summarized as follows:

1. In-situ analysis of soil before digging; thus facilitating digging decisions
2. Analysis of rocks and cobbles greater than 4 inches average diameter after processing and before return to landfill
3. Analysis of rocks and pebbles between 1/8 inch and 4 inches in diameter
4. Analysis of pebbles and sand between 17/10,000 inch (0.042 mm) and 1/8 inch in diameter
5. Analysis of fines for process decision on storage, further cleanup, or discard back to landfill
6. Analysis of process water for making a decision when to discard
7. Analysis of fines at another cleanup stage if attempted after item 5

This information has been reported to the Program Manager for factoring into engineering plans. Instrumentation requisitions have been issued for test systems except for a computerized multi-channel analyzer system. This system will monitor all detectors and will provide continuous process operations information for operations decisions.

Future Work

The requisitioned equipment will be purchased based on available funding. Specifications for the multichannel analyzer will be completed and issued. Following R&D experiments, final specifications will be issued.

ADVANCED SIZE REDUCTION FACILITY

Francis X. Haas and Wayne E. Herren

Objective

The objective is to provide design criteria for radiometric instrumentation that will be used in the Advanced Size Reduction Facility. Remote radiometric instruments will be built and tested to satisfy nuclear safety criteria and to measure contamination levels below 10 nCi/g on size-reduced items.

Prior Work

Instrumentation was specified for purchase that would measure plutonium contamination on items prior to their being electrodecontaminated, measure residual contamination after electrodecontamination, and measure plutonium concentration and nuclear poison concentration in the electrolyte baths.

Achievements and Discussion

Input Monitor

An array of nine sodium iodide detectors was assembled to measure plutonium contamination on size-reduced pieces prior to the pieces being electrodecontaminated. Detector response for the 350- to 420-keV gamma ray energy region from 1-, 3-, and 5-g encapsulated sources was mapped as the sources were moved to any one of 90 positions. These positions were located in a 3- by 3-ft plane located 10 in. from the detector faces. A linear fit to the data gave the following equation for count rate per 100 seconds (C) versus grams of plutonium (G):

$$C = 259470 G + 686000$$

Assuming that the minimum detectable amount of plutonium is six times the standard deviation in the intercept of this equation, the minimum detectable limit is 0.019 g with an uncertainty of ± 25 percent. Since this datum was taken with sources placed in the 90 positions in front of the detector array, the 0.019-g sensitivity corresponds to 1.71 g of plutonium evenly distributed over a 9-ft² area.

This sensitivity easily meets the 5-g sensitivity goal for which the system was to be designed. The 5-g total for the 9 ft² can be measured to ± 12 percent based on counting statistics and equation parameters. These limits are high by at least a factor of two based on measurements of gamma rays from a thin barium-133 source, gamma rays detected from encapsulated plutonium samples, and detector efficiency. These attenuation and encapsulation effects decrease the number of gamma rays seen by the detector by approximately 60 percent. Overestimates on the side of nuclear safety also exist because (1) americium contributions to the gamma ray signal used in the measurements are not discriminated against and (2) the limits were calculated assuming even distribution of contamination over the part without regard to any material voids that might exist on the parts to be electropolished.

Boron Poison Monitor

Nuclear Safety has requested a reliable method of measuring neutron poisons in the electrolyte solution. A neutron absorption technique outlined in ORNL/TM-6432¹ was used to measure boron in the sodium nitrate electrolyte solution. A 10^7 n/sec plutonium-beryllium source and $^{10}\text{BF}_3$ neutron detector were placed in an absorption cell measuring approximately $6 \times 6 \times 3$ in. The neutron absorption was measured over boron concentrations from 0 to 2.26 g/l. A linear least-squares fit to the data gave the following relationship between the count rate and the molar-barn per liter cross-section for boron:

$$[\sigma] = 1.321 + 154.47 R + 2.486 R^2$$

where R equals $(C_0/C) - 1$. (C_0 is the count rate with no boron in solution, and C is the count rate with the boron additive.)

The system can easily detect any change in boron concentration at the 1.1 g/l level requested by Nuclear Safety to ensure safe operation of the electrolyte bath. A 5-percent increase in the count rate is the result of a 15-percent decrease in the measured cross section.

Liquids Assay

An X-ray fluorescence system has been purchased to investigate the use of X-ray fluorescence for monitoring plutonium buildup in the electrolyte. The system was set up and accepted by measuring uranium concentration in liquids. Liquid samples containing 100, 200, 500, and 1000 ppm were prepared, and the system was calibrated. The lower limit of detectability is estimated to be 25 ppm ± 6 percent in counting times of 300 sec. Reproducibility on samples in the range of 100 to 1000 ppm is ± 3 percent.

Two systems have been proposed for analysis of plutonium buildup in the electrolyte solution. Both systems are to operate in the glove-box lines of the size reduction facility to avoid bagout, external contamination, and transportation problems. However, they are not to operate in the recirculating lines of the electrolyte. This decision was made because of the low energy region, 10 to 20 keV, to be used for assay. This would require thin foils or films to be used between the liquid and detector system. These foils would be fragile, difficult to maintain, and susceptible to material buildup, which would affect the measurement process. In addition, the electrolyte is operating at 80 °C, which can introduce thermal disturbances into the detecting system.

The X-ray fluorescence system will operate with the Si(Li) detector and cadmium-109 excitation source located beneath the glove box. The liquid sample and detector will be separated by a beryllium window in the glove-box bottom. The same geometry as the present test equipment can be maintained. Sensitivity for the measurement of plutonium will be the same or greater than that for uranium since the plutonium L-absorption edge lies closer to the excitation line than the uranium absorption edge. In addition to the plutonium, the system can provide analysis of the iron,

chromium, and other metal buildup in the electrolyte to ensure efficient operation of the electro-decontamination process.

The second system proposed is a thin, large-area, sodium iodide detector, such as the phoswich detector of FIDLER. Work done previously with the phoswich detector used in MONILLE gave a sensitivity of 1.06×10^{-4} mg/l in a 300-sec count.² Assays at the 200 mg/l level will be possible at ± 5 percent in count times of less than 10 sec.

Future Work

The three counting system described—the input monitor, the boron poison monitor, and the plutonium liquid monitor—will be integrated into an instrument control package to meet nuclear safety criteria. When each instrument reaches an alarm condition, interlocks will be activated and steps recommended by nuclear safety will be taken to bring the electropolish process back to safe operating procedures.

Work will be undertaken to specify an instrumentation package to measure the contamination level on electrodecontaminated parts at levels below 10 nCi/g.

References

1. J. E. Strain, "An In-Line Neutron Poison Monitor," ORNL/TM-6432, Oak Ridge National Laboratory, Oak Ridge, Tennessee, September 1978.
2. G. P. Minges, R. N. Chanda, and R. A. Harlan, "COMSAC and MONILLE: Safeguards and Environmental Systems," *Journal of Institute of Nuclear Materials Management*, Vol. IV, No. III, p. 480 (1975).

PROCESS CONTROLS FOR BUILDING 371

Ronald L. Gornick

Objective

The objective of this work is to provide technical support to Chemical Operations, Building 371, on problem areas, checkout, and start-up.

Prior Work

No prior work was done on this project.

Achievements and Discussion

Work has progressed on the sequence panel checkouts. Problems have been identified and solved that will aid in the start-up and operation of the processes. The major problems and the solutions found were as follows: (1) R-C time delay networks that were causing switching problems. The R-C networks have been removed on all panels checked to date without any loss of operating calibrations. (2) Power entering the test area was low and very unstable causing faulty operations and some card failure. A 45-kW transformer has been installed to replace a 15-kW transformer. A separate power circuit was run to the test area, and voltage suppression was put across the Sorensen regulator outputs. (3) Certain timers and lock-circuits were not latching into their assigned states when power was turned on, turned off, or when a power bump occurred. A circuit has been devised to allow initialization of the timers and lock-in circuits so that all upset conditions can be cleared without special jumping of signals by instrument technicians.

The tanks in Building 371 are being measured for level by differential pressure transmitters. This will cause problems since most tanks do not have a constant specific gravity (one of the basis for d/P cell level measurement). This problem has been brought to the attention of Chemical Operations personnel, and more accurate level and custody transfer measurement system is being designed.

Future Work

Technical support will continue to be given to Chemical Operations of Building 371.

CRITICAL ANGLE REFRACTOMETER

Charles M. Johnson and Robert C. Baker

Objective

The objective of this work is to provide an instrument capable of giving a constant readout of

caustic molarity in the recirculation line of the plutonium recovery facility. This instrument will enable process operators to eliminate foam in the system. The foam is caused by the caustic going acid.

Prior Work

The instrument has been installed in the plutonium recovery facility and wiring has been completed. Flow indicators were installed in the prisms wash and cooling water lines.

Achievements and Discussion

A critical angle refractometer measures the refractive index of solution at an interface of the liquid and the prism. The refractive index, such as molecular weight, density, and boiling point, is a widely accepted physical constant that can be used analytically as an identifying characteristic of a particular solution. The instrument has been constructed to identify KOH solutions in the zero to 6M range. Initial testing has shown that instrument meter readings vary no more than 0.2M from laboratory analysis of the caustic solutions. This instrument will be used during the next scheduled incinerator run.

Future Work

Installation of additional critical angle refractometers on different process lines will be done as requested by Chemical Operations.

WATER CONTROL AND RECYCLE PROJECT

*David E. Hausburg and Nancy D. Kirk**

Objective

The objective of this work is to provide technical support to the Water Control and Recycle Project until the recycle plant is operating and on-line.

*Electrical Design group.

Prior Work

Support of this project has been on a continuing basis since 1975 as reported on page 40 of the previous progress report, RFP-2877 (May 28, 1979).

Achievements and Discussion

Various problems have arisen during system operation (SO) testing of equipment and start-up of the water recycle plant. Technical assistance is being provided to resolve problems.

Installation errors by subcontractors caused damage to chemical injection equipment and instrumentation. Three contractors have assumed responsibility and the cost to place the systems in proper operation.

System operation testing of all pretreatment equipment is complete. A high silt density index of the feed water caused by algae growth delayed SO testing of the reverse osmosis trains. Copper sulphate was used to kill the algae, and SO testing of all three trains is now complete.

Future Work

Technical assistance will continue until all major technical difficulties are resolved and the plant is on-line. Work to complete the programmable controller documentation will continue.

PROGRAMMABLE CONTROLLER STUDY

*David E. Hausburg and L. R. Bailey**

Objective

The objective of this study is to evaluate programmable controller (PC) systems presently on the market and to develop a guide with recommendations to be used in setting a Rocky Flats Plant Standard.

*Control Systems Development group.

Prior Work

No prior work has been reported.

Achievements and Discussion

Both small-scale and large-scale PC systems are now controlling various processes at Rocky Flats, and additional installations of PCs are proposed. All indications point to the continual growth of PC use and greater development of systems by PC manufacturers. With these facts in mind, the Rocky Flats Mini/Micro Computers Users Committee initiated this study to set a Plant standard.

Brochures and engineering data from major PC manufacturers were reviewed, and eight companies were selected for further examination. A final report will include a table of salient features of each PC so ready comparison between systems can be made. Based on reviews and evaluations, three companies have PCs satisfactory for use at Rocky Flats and will be recommended to the Committee.

Future Work

The final report will be completed by June 1979. Review and evaluation of new PC systems will take place as manufacturers make new systems available.

PROGRAMMABLE CONTROLLER - COMPUTER TRANSLATOR

*David E. Hausburg, James L. Lawless,
and Joseph M. Milan*

Objective

The objective of this work is to use an existing Hewlett-Packard computer to print an electrical ladder diagram with mnemonics. Secondary to this work is the compiling and printing of important additional documentation to be used with the ladder diagram.

Prior Work

No prior work has been reported.

Achievements and Discussion

Programmable controllers (PCs) store soft-wired electrical ladder diagram elements in magnetic core memory or in semiconductor memory. A numerical address system generally is used to assign discrete elements a position within the memory structure. Hard-wired electrical inputs to the PC and hard-wired electrical outputs from the PC use the same address system. A reference table is required to correlate each address with discrete inputs and outputs. The use of a table is slow and unwieldy if the process has a large amount of inputs and outputs (I/O); e.g., the large-scale Allen-Bradley PC in service at the water recycle plant at Rocky Flats has greater than 550 I/Os. Adding alpha-numeric mnemonics to circuit elements reduces dependency on the reference table and speeds up insight to circuit operation.

A computer translator software program was purchased to add mnemonics to the water recycle plant ladder diagram. The program was developed for use with an Interdata computer, and modifications to program instructions were necessary to adapt the program to the Hewlett-Packard computer. A summary of mnemonics with addresses, I/O rack diagrams, and a symbolic table is printed in addition to the ladder diagram. Each of these three items increase the usefulness of the ladder diagram.

Computer printing of the ladder diagram is on 8- X 11-inch sheets that can be bound in book form; thus, blue-line electrical drawings are not required. With the use of computer printing, considerable cost savings are realized by eliminating a draft-person's time.

Future Work

Documentation for additional processes controlled by PCs will be generated as required.

DENVER 1978 BROWN HAZE STUDY

*Charles M. Johnson, Joseph M. Milan,
James L. Lawless, Frederick L. Crawford,
L. Harry Faber, and Robert C. Baker*

Objective

The purpose of this project was to install and man a background measuring station as part of the 1978 coordinated air pollution study of the Denver area. The background station was to furnish data on gaseous pollutants, meteorologic parameters, and particulate pollutants.

Prior Work

Early in 1978, several meetings were attended by interested organizations concerning the extent of the Denver-area air pollution problem. A review of past studies showed a lack of definitive characterization and source identification of the air pollution cloud. Because of the scope of the project, a large effort was mobilized.

George Wolfe of General Motors Research was project manager, and the Motor Vehicle Manufacturers Association (MVMA) sponsored the study. Selected organizations were invited to participate. Rockwell International at the Rocky Flats Plant was asked to furnish the background site for this study.

The 1978 Denver Haze Study was conducted from November 15 to December 20, 1978. Participating organizations, in addition to Rocky Flats, MVMA, and General Motors Research, were the National Oceanic and Atmospheric Administration, Environmental Protection Agency, the Colorado Department of Health, Ford Motors Research, Loren Crow (consulting meteorologist), Environmental Research and Technology, Inc., Meteorology Research Inc., and the University of Washington.

Achievements and Discussion

The equipment used to establish the background site was a Mobile Ambient Air Monitor (MAAM). (See

RFP-2417A, August 29, 1975, p. 29.) The MAAM was fully reconditioned from a shutdown state. Several pieces of equipment were added to align the site with the goals of the study. A nephelometer from the University of Washington, two low-volume particulate samplers from General Motors Research, and a high-volume particulate sampler were added to enhance our particulate sampling capability.

The background site was located at the Wind Energy Research facility on the northeast corner of the plant property. The MAAM was operated for a total of 37 days, 18 hours, and 45 minutes. During this time, there was equipment malfunction for 10 hours and 40 minutes; 98.8 percent of the available data is recorded on computer-compatible magnetic tape, the other 1.2 percent is available on strip chart records. A total of 215,060 data points were recorded. The magnetic tape will be furnished to the coordinating agency, Environmental Research and Technology, for inclusion in a data pool being established for this study.

Sixteen days were selected for intensive scrutiny by the brown haze study team. Fifteen days were designated pollution episode days and one day as a clean day. A schedule was adopted for submittal of the data to the study coordinator.

Preliminary examination of the overall data revealed two phenomena not previously known. The pollution cloud is not a homogeneous mass. It can occur in different areas at different times of the day. Also, the chemical composition of the mass is not constant but can vary from one day to another.

The raw data is being assembled into a usable form. The first issue, which is a tabulation of meteorological data, has been forwarded to the study meteorologist.

Future Work

The remaining data required for the project data bank will be assembled and forwarded to the coordinating agent. All of the data will be assembled into a usable report.

PRESENTATIONS, PUBLICATIONS, AND PATENTS

Presentations

P. M. Williams, "Vitrification of TRU Waste at Rocky Flats," at Waste Management and Fuel Cycles '79, Tucson, Arizona, February 25 through March 1, 1979.

J. A. Ledford, "Utilization of Borosilicate Glass for Transuranic Waste Immobilization," at American Ceramics Society Annual Meeting, Cincinnati, Ohio, April 29 through May 3, 1979.

C. E. Plock, "Use of Reverse Osmosis for Water Recycling at Rocky Flats," at The Role of Desalting Technology Workshop, Denver, Colorado, May 24-25, 1979.

R. A. Harlan, "Natural and Man Produced Radiation in Our Environment," at University of Denver, June 27, 1979.

Publications

D. L. Anderson, B. A. Bell, P. K. Feng, and F. G. Meyer, *Fluidized Bed Incineration System for U.S. Department of Energy Defense Waste*, January - June 1978, RFP-2811, December 8, 1978.

D. L. Ziegler, J. E. Garnett, and J. K. Fraser, *Recovery of Plutonium From Insulation, Scrap Glass, and Sand-Slag Residues*, RFP-2835, April 12, 1979.

A. J. Johnson, S. C. Burkhardt, J. A. Ledford, and P. M. Williams, *Status Report Waste Incineration and Immobilization for Nuclear Facilities*, October 1977 - March 1978, RFP-2863, May 17, 1979.

D. E. Hausburg, J. L. Briggs, Marston R. Dringman, and R. J. Jackson, "Alternate Immersion Stress Corrosion Testing of 5083 Aluminum," RFP-2645, November 13, 1978.

D. E. Hausburg, "Alternate Immersion Stress Corrosion Cracking Test Apparatus," *American Society of Certified Engineering Technicians*, January-February 1979.

Patents

C. E. Plock and J. A. Hayden, "Concentration and Purification of Plutonium or Thorium," U.S. Patent 3,948,735, Issued April 6, 1976.