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CHEMISTRY RESEARCH AND DEVELOPMENT
PROGRESS REPORT
JUNE THROUGH NOVEMBER 1978

Frend John Miner

Component Development	-----	Jean R. Gaines
Pilot Plant Development	-----	Donald L. Ziegler
Instrumentation and Statistical Systems	-----	Richard N. Chanda

Rockwell International

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

U. S. DEPARTMENT OF ENERGY
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**ROCKWELL INTERNATIONAL
ENERGY SYSTEMS GROUP
ROCKY FLATS PLANT
P.O. BOX 464
GOLDEN, COLORADO 80401**

**Prepared under Contract DE-AC04-76DPO3533
for the
Albuquerque Operations Office
U.S. Department of Energy**

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PUBLICATIONS

R. A. Harlan, "Experiences With a Counter for Plutonium in Crates," to be published in proceedings of American Nuclear Society topical meeting on Analytical Measurements in the Nuclear Fuel Cycle, Williamsburg, Virginia, May 15-17, 1978.

SUBJECT DESCRIPTORS

Advanced Size Reduction Facility	Manipulators
Air Pollution	Measuring Instruments
Aluminum	Microprocessors
Beryllium	Minicomputers
Brazing	Nickel
Calibration Standards	Nondestructive Analysis
Calorimeters	Physical Security
Carbon Steels	Pitting Corrosion
Chlorides	Plutonium Residues
Cleaning	Process Control
Compatibility	Radioactive Wastes
Contamination	Remote Handling
Control Systems	Residues
Cooling Water	Reverse Osmosis
Copper	Safeguards Instrumentation
Corrosion	Shipping Containers
Corrosion Inhibitors	Soil
Data Acquisition	Solar Pond Sludge
Decontamination	Soldering
Disassembly	Solvent Combustion
Environment	Stainless Steel 304
Evaporation	Statistics
Fast Fluidization	Stress Corrosion Cracking
Fluidized Bed	Surface Water
Fluidized Bed Incineration	Teleoperators
Fracture Properties	Transuranic Waste
Glove Box	Uranium Burning
Hydrofluoric Acid	Vitrification
Incineration	Waste Immobilization
Incinerator	Waste Management
Land Pollution	Waste Processing
	Water Pollution

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*All Development and Development Support (DDS) endeavor numbers refer to FY-1980 Proposed Program for Process Development (NM-01-04) and Related Work, CD 77-4282, December 30, 1977. Production and Production Support programs are identified as PPS.

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A remotely controlled vacuum cleaning system is being developed to clean the Building 371 storage vault floor. This is required to keep the background radiation level low and to aid in accounting for radioactive materials. (PPS)

*All Development and Development Support (DDS) endeavor numbers refer to FY-1980 Proposed Program for Process Development (NM-01-04) and Related Work, CD 77-4282, December 30, 1977. Production and Production Support programs are identified as PPS.

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*All Development and Development Support (DDS) endeavor numbers refer to FY-1980 Proposed Program for Process Development (NM-01-04) and Related Work, CD 77-4282, December 30, 1977. Production and Production Support programs are identified as PPS.

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FOR DEPARTMENT OF ENERGY DEFENSE WASTE 28**

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*All Development and Development Support (DDS) endeavor numbers refer to F.Y-1980 Proposed Program for Process Development (NM-01-04) and Related Work, CD 77-4282, December 30, 1977. Production and Production Support programs are identified as PPS.

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Two alpha scanning systems have been developed for monitoring oralloy parts. (PPS)

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*All Development and Development Support (DDS) endeavor numbers refer to FY-1980 Proposed Program for Process Development (NM-01-04) and Related Work, CD 77-4282, December 30, 1977. Production and Production Support programs are identified as PPS.

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A dual-chamber test stand has been constructed for leak-testing plastic air hoods. (PPS)	

*All Development and Development Support (DDS) endeavor numbers refer to FY-1980 Proposed Program for Process Development (NM-01-04) and Related Work, CD 77-4282, December 30, 1977. Production and Production Support programs are identified as PPS.

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CHEMISTRY RESEARCH AND DEVELOPMENT

PROGRESS REPORT

JUNE THROUGH NOVEMBER 1978

Frend John Miner

COMPONENT DEVELOPMENT

*Jean R. Gaines*COMPATIBILITY OF
NON-NUCLEAR WEAPONS METALS WITH
PROCESS MATERIALS*Marston R. Dringman and John L. Briggs*

Objective

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

Prior Work

Results of other compatibility studies have been reported in many previous progress reports.

Achievements and Discussion

There is a continuing Quality Control Program for which corrosion and residue removal tests are used to determine the compatibility of all non-nuclear weapons metals with processing materials currently in use.

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

Sample coupons are pretreated by surface finishing with 120-grit abrasive paper, rinsed in distilled water, rinsed in acetone, air dried, and weighed to the nearest 0.1 mg. The metal coupons then are immersed in or placed in contact with the various corrodent materials and stored for 10 days at room temperature, ($23 \pm 2^\circ\text{C}$).

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, coupons that have been exposed to the corrodent environments are cleaned with an aqueous detergent as follows:

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

The coupons are then analyzed for residue by infrared, atomic absorption, and emission spectrographic techniques. Table 1 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 1 proved to be compatible except where noted.

TABLE 1. Compatibility Test Metals and Materials

Process Materials	Metals Tested	Metals not Compatible
Polygel	*	
Alcoa No. 2 EJC	*	U - 0.75 Ti, U - 2.3 Nb, U-238, U - 6 Nb
Lubri-Bond A	JBK-75, 21-6-9 304L, 316	
Accu Pen P8F-3	*	
Bio Pen P7F-3	*	
Bio Pen P6F-3	*	
Sinoco Way Lube 1180	*	
Coe-Flex Heavy	*	
Coe-Flex Regular	*	
Coe-Flex Light	*	
Texaco Soluble Oil D	*	1100, 5083, 6061, and 7075 Al
Apiezon Grease T	*	

*Metals tested include 1100, 5083, 6061, and 7075 Al; Be; Monel; JBK-75, 21-6-9, 303, 304L, and 316 stainless steel; U-238; U - 2.3 Nb; U - 6 Nb; and U - 0.75 Ti.

Future Work

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

REMOVING SCALE FROM COPPER RINGS

Marston R. Dringman and John L. Briggs

Objective

The purpose of this study was to develop a method for removing production scale from copper compression rings before they are used as high pressure seals. The scale residues are present on the rings as received from the vendor.

Prior Work

This is a new effort.

Achievements and Discussion

Twenty copper compression rings were obtained for testing. The rings had a combined surface area of 1.47 in.² and weighed 1.6653 g. The average measurement was 0.168 in. outside diameter, 0.128 in. inside diameter, and 0.050 in. thick. They were ultrasonically cleaned in an aqueous detergent, rinsed, and air dried prior to weighing.

Battelle Chemical Polish No. 2 was selected as a potential cleaning agent based on previous copper cleaning agent studies. It is an acid solution containing 46 vol % phosphoric acid, 29 vol % acetic acid, 10 vol % nitric acid, and 15 vol % water. The chemical polishing mechanism of the mixture combines the dissolution of copper oxide with minimal copper metal removed by the inhibited nitric acid.

The rings were placed in the Battelle solution for 2.5 minutes at 23 ± 2 °C. The rings were then rinsed in distilled water and reweighed for a weight loss determination. The total initial weight

loss was 37.6 mg. Subsequent immersions gave weight losses of 12.8 mg, indicating these immersions removed only metal. This indicates that all of the manufacturer's scale is removed in the first 2.5-minute acid etch.

The rings were sent to the analytical laboratory for an acid residue analysis. No detectible nitrate or phosphate residues were detected.

The copper cleaning study concluded with a recommendation that all copper compression rings be cleaned with Battelle Chemical Polish No. 2.

Future Work

No further work on this objective is planned at this time.

PREBRAZE CLEANING OF 321 STAINLESS STEEL TUBES

John L. Briggs and Marston R. Dringman

Objective

Cleaning studies were performed to develop a suitable prebraze acid cleaning method for 321 stainless steel tubes used in the W-76 program. Another objective of the cleaning study was to establish a satisfactory range of upper and lower weight loss for tubes cleaned with the mixed acid etch process.

Prior Work

Previous studies resulted in a combined aqueous detergent - acid etch method. Excessive variation in tube weight loss necessitated revision of the method.

Achievement and Discussion

An evaluation of the previous acid etch (20 vol % hydrofluoric plus 30 vol % nitric acid) cleaning method indicated that excessive braze rejections

by radiography resulted when weight losses of <12 milligrams per tube were measured following the etch process. Since the braze rejects apparently occurred because of insufficient metal removal of the tubes, acid-etch exposure time was increased from 5 to 12 minutes to ensure adequate etching of the tube interiors.

One hundred 321 stainless steel tubes were preweighed in groups of five and then cleaned by M-Document specifications, including the 12-minute acid-etch exposure period. Measured weight losses for the 100 tubes averaged 28.5 mg with a high value of 42.0 mg and a low of 16.8 mg. No rejects were detected by radiography on any of the tubes after the brazing process. Further investigation of the cleaning process continued so as to narrow the range of weight loss during the acid etching process.

As a result of the investigation, the following revisions of the tube cleaning method were implemented: (1) the hydrofluoric acid concentration range (checked weekly) was increased from 20 ± 5 vol %, to 22.5 ± 2.5 vol %, (2) the tube-cleaning fixture was modified to eliminate excessive acid aeration, and (3) tube exposure time in the mixed acid was set at 8 ± 0.5 minutes.

After establishing optimum acid-cleaning parameters, 30 more sample tubes were obtained and cleaned by the revised method. The tubes were cleaned in sets of 5 at weekly intervals over a period of 27 days, with acid concentration determinations performed immediately prior to acid-etching the tubes. The results are shown in Table 2. One hundred production tubes were then cleaned with the revised acid-cleaning method. Weight losses averaged 27.0 milligrams per tube with a high weight-loss value of 33.7 milligrams per tube and a low weight loss of 17.5 milligrams per tube. The tubes were then brazed and submitted for radiographic inspection. No rejections were detected by that inspection process.

The results of these studies indicated that a satisfactory prebraze tube-cleaning method was developed. The method also incorporated a reduced range of tube weight-loss limits required for suitable braze operations. A meeting with

TABLE 2. Acid-Etch Weight-Loss Studies

Date	Weight Loss (mg/tube ^a)	Weight Loss Variation ^b (mg/tube)	Hydrofluoric Acid Concentration (vol %)	Nitric Acid Concentration (vol %)
11/9/78	25.4	2.7	19.3	29.0
11/11/78	31.5 ^c	2.3	24.8	28.3
11/18/78	32.4 ^c	1.8	23.9	27.9
11/21/78	22.2	1.5	23.3	27.4
12/1/78	19.0	1.5	23.0	26.2
12/6/78	31.4 ^c	1.9	24.8	31.3

a. Average weight loss per tube in sets of 5 tubes.

b. Variation between highest and lowest losses per set of 5 tubes.

c. Fresh acid makeup.

design agency personnel resulted in the conclusion that a weight-loss range of 25 ± 10 milligrams per tube would be an acceptable weight-loss limit. That range has been maintained in subsequent process cleaning operations.

Future Work

No future work is planned.

INVESTIGATION OF CLEANING METHODS FOR THE TOUCH-BRAZE PROCESS

John L. Briggs

Objective

The objective of this study was to identify sources of contaminant residues, if any, that could cause rejection of touch brazes on the W-79 program.

Prior Work

No prior work has been done on this objective.

Achievements and Discussion

The initial phase of the investigation included an evaluation of the U - 2.3 Nb alloy part cleaning

process. That process consisted of ultrasonic vapor degreasing in 1,1,1-trichloroethane solvent according to specified method F-0444-1012.

Two U - 2.3 Nb parts were designated for experimental surface residue studies. The parts had been previously completed through the production process and had been solvent-cleaned prior to a coating treatment. Immediately following solvent cleaning, the two parts were diverted from the production process for analytical rinses with carbon tetrachloride and deionized water. The rinses were analyzed for contaminant residues by infrared spectrometry and emission spectroscopy. Chloride analysis was performed by specific ion electrode.

After analytical rinsing, the parts were again solvent-cleaned and then hand-cleaned with an aqueous detergent plus distilled water rinses. This cleaning is routinely performed prior to coating the U - 2.3 Nb parts. After hand-cleaning the parts, excess water was flushed from the surfaces with reagent-grade methanol and the parts were allowed to air-dry.

Analytical rinses were again performed on the parts following the detergent cleaning and methanol rinse. The methanol used for drying the parts was analyzed by infrared spectrometry for possible container (polyethylene squeeze bottle) contamination. The residue analyses immediately

following solvent cleaning indicated no organic or inorganic contaminants on the parts. No residues were detected on the parts after the aqueous detergent hand cleaning, and no polyethylene contaminants were detected in the methanol used to dry the parts.

Two other possible contaminant sources (bacterial-infected lathe coolant and a metal tapping fluid) were identified during the production process investigation. Both materials were evaluated by cleaning and residue tests, and no residues were detected.

Although no sources of contaminant residues were detected by the investigation, the occurrence of stains and chloride pitting on solvent-cleaned production parts indicated that the solvent cleaning process was not satisfactory. Therefore, an aqueous detergent cleaning method was recommended to replace the solvent technique. The documented (M-21515-A) aqueous detergent method has been proven to be more suitable than solvent cleaning for removing production residues from nonfissile weapons metals at Rocky Flats* and for cleaning U - 2.3 Nb alloy. It has provided suitable cleaning since implementation for pretouch braze cleaning.

Future Work

No further work is planned.

STRESS CORROSION CRACKING OF 5083 ALUMINUM

R. Alan Patterson

Objective

Crack propagation rates as a function of applied stress intensity will be measured in an effort to define the plane strain stress intensity factor below which crack propagation is negligible. Slow strain

TABLE 3. Composition of 5083 Aluminum

Constituent	Commercial Range (wt %)	Plate Stock (wt %)
Mg	4.0 - 4.9	4.2
Mn	0.3 - 1.0	0.65
Cr	0.05 - 0.25	0.10
Fe	0.40 max.	0.20
Zn	0.25 max.	0.015
Cu	0.10 max.	0.035
Si	0.40 max.	0.06

rate tensile tests will be employed to monitor the susceptibility of 5083 Al to stress corrosion cracking (SCC) as a function of artificial aging treatments. Both of these experiments will then characterize the overall susceptibility of 5083 Al to SCC.

Prior Work

No prior work has taken place.

Achievements and Discussion

Table 3 compares the composition range for 5083 Al with the composition of the 2-inch plate stock procured for this experiment. Magnesium is the major alloying constituent and is primarily responsible for the good work-hardening characteristics of 5083 Al. Small quantities of manganese and chromium are added to the Al-Mg alloys to raise the recrystallization temperature and also for slight advantage in mechanical properties.

The resistance to general corrosion attack of the Al-Mg alloys is high and is particularly good in marine environments. However, because of the diminishing solubility of magnesium with decreasing temperature, there is a tendency for β -phase Mg_2Al_3 to precipitate in alloys with greater than 3 wt % Mg. The precipitation of β -phase at grain boundaries and sites of lattice disarray is generally considered to sensitize Al-Mg alloys to intercrystalline corrosion and stress corrosion cracking. This precipitation

*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.

is extremely slow at room temperature, but becomes increasingly rapid at temperatures above 65 °C. Typically the range of temperatures between 65 and 200 °C are quoted as the sensitization range for 5083 Al.¹

The two-inch-thick plate for these experiments has been subjected to the following thermomechanical treatment:

1. Solutionize at 260 °C for 1 hr.
2. Cross-roll at room temperature for a 20% reduction in thickness.
3. Heat at 230 °C for 10 min.
4. Uniaxially roll at a temperature between 205 and 230 °C for a single pass reduction in thickness of 20%.

This process was chosen in an effort to simulate the processing of parts at Rocky Flats. Yield strengths in excess of 35 ksi (240 MPa) without sensitizing the material to SCC should be obtained by this method. Metallographic examination of the resultant microstructure has indicated that a discontinuous precipitate (Mg_2Al_3) has formed in the test material, which should decrease its resistance to SCC.

Alternate immersion tests (ASTM G38-73) of 8.75-in.-o.d. "C" rings from the processed 5083 Al were performed to evaluate SCC susceptibility. Each "C" ring was stressed to 80% of the yield stress, 31 ksi (214 MPa). The samples are immersed in 3.5 wt % NaCl for a timed interval of 10 minutes and then allowed to dry for a second timed interval of 50 minutes. This cycle is continued for a 30-day test period when the test is terminated. Details of the test parameters and apparatus are explained by Briggs et al.² Three of eight processed 5083 Al "C" rings cracked during the alternate immersion test, thus confirming the sensitization of this material.

Future Work

Double cantilever beam specimens are being fabricated for use in the crack growth rate determinations. Samples will be stressed to con-

stant stress intensity increments ranging from the plane strain fracture toughness, K_{IC} , down to approximately 20% of K_{IC} , while immersed in a 3.5 wt % NaCl solution. Crack opening displacement measurements, as a function of time, will be used to monitor the extension rate of the stress corrosion cracks. A limiting crack extension rate, da/dt , of 10^{-8} cm/sec typically is used to define the lowest stress intensity below which significant crack growth stops. This stress intensity level, K_{ISCC} , is then assumed to be the critical stress intensity factor for 5083 Al stress corrosion and cracking in salt solutions. The value of K_{ISCC} can then be used as a design property for flawed pressure vessels subjected to marine atmospheres.

Cylindrical tensile specimens will be used to monitor the increase in SCC susceptibility of 5083 Al exposed to artificial aging treatments. In this manner, an assessment of room temperature aging can be extrapolated, and evaluations of long-term susceptibility can be made.

The tensile specimens will be tested by a slow strain rate technique. This technique consists of a slow extension rate (approximately 10^{-6} in./sec) tensile test in which the specimen is immersed in a corrosive environment (3.5 wt % NaCl) and also forced to corrode by an impressed potential approximately equal to the pitting potential for the metal. Increased susceptibility to SCC is measured by decreases in ultimate tensile strength or ductility, and changes in the fracture mode.

References

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SCALE REMOVAL FROM COOLING WATER LINES

John L. Briggs

Objective

The objective was to develop a method for cleaning diffusion pump cooling lines that had exhibited insufficient flow rate because of interior scaling.

Prior Work

No previous work was done on this objective.

Achievements and Discussion

A decrease in the cooling water flow rate was detected on a vapor deposition diffusion pump for the W-76 program. The reduced coolant flow rate resulted from interior constriction of the copper coolant lines. Scale deposition caused the constriction.

Approximately eight feet of the copper line was replaced in proximity to the diffusion pump, which is water-cooled. Since replacement of the copper cooling lines on the diffusion pump would require a lengthy shutdown of the vapor deposition process, a method to clean the diffusion pump cooling lines was required.

Analysis of the scale removed from the sectioned copper pipe was performed by infrared, emission spectroscopy, scanning electron microscopy, and X-ray diffraction techniques. The scale deposits were positively identified as $(\text{CaCO}_3)\text{Ca}_4(\text{PO}_4)^3$ (mineral name—carbapatite).

The literature has thoroughly described the reversion of polyphosphates to scale and sludge-forming orthophosphate in the presence of calcium carbonate. Higher temperatures such as those occurring at the diffusion pump surfaces, do accelerate orthophosphate scaling. No scaling was detected on the much cooler, mild steel connecting lines. In laboratory tests, a solution of 5 vol % hydrochloric acid com-

pletely dissolved the scale residue within 30 minutes at room temperature and with no adverse effects on the copper lines.

An acid-resistant chemical pump was obtained to flush the coolant lines around the diffusion pump. The coolant lines were fabricated exclusively of copper, with mechanical joints and no brazes.

An eight-liter reservoir of 5 vol % HCl was then circulated through the coolant lines for 45 minutes. Heavy accumulations of dissolved scale were observed in the acid reservoir after this first cleaning period.

The acid reservoir was recharged with fresh acid, which was circulated for a period of 30 minutes. Very little dissolved scale material was observed after this second flush. Next, the lines were flushed with distilled water for 10 minutes and then flushed with a neutralizing solution of sodium carbonate (8 oz/gal) for 15 minutes. The coolant lines were then reflushed with distilled water, drained, and reconnected to the closed cooling system.

Prior to cleaning the diffusion pump cooling lines, a decreasing flow rate of 0.3 gal/min was measured. After the lines were cleaned, the coolant flow rate nearly doubled, measuring 0.7 gal/min. This rate provides satisfactory heat transfer for diffusion pump operation.

Future Work

If scaling recurs in the diffusion pump coolant lines, periodic maintenance by acid-cleaning the lines may be necessary.

MONITORING GLOVE BOX CORROSION

John L. Briggs

Objective

The objective of the glove box corrosion monitoring program is to ensure that no structural defects occur

in glove-box lines because of corrosion and to provide a basis for preventive measures, if necessary.

Prior Work

Previous glove-box corrosion monitoring studies have been described in earlier progress reports.

Achievements and Discussion

Sample discs of 304 stainless steel (glove-box fabricating metal) were obtained for the corrosion tests. The disc dimensions were 5.72 cm in diameter, 0.16-cm thick with 0.79 cm centerholes and a total surface area of 8.1 in². The samples were abraded with 120-grit silicon carbide paper, aqueous cleaned, rinsed in distilled water, air dried, and weighed to the nearest 0.1 mg. They were then placed inside the glove-box lines in areas susceptible to corrosion. These areas were identified in previous investigations. Teflon strings were used to hang the samples to prevent possible galvanic contact.

After 12 months, the sample coupons were removed from the glove-box lines. Kim-wipes moistened with distilled water were used to wipe the surfaces of the samples and thus remove any loose corrosion products. The sample coupons were reweighed for gravimetric corrosion rate determinations and then were analyzed by microscopy for possible cracking or pitting corrosion. During the 12-month storage period, visual, in-place inspections of the corrosion samples were made in the acid environments of Lines 1 and 30. These lines are located in the plutonium recovery facility. Inspection results indicated no excessive corrosion on the sample surfaces throughout the test. The results of all the corrosion tests are included in Table 4.

A weight gain was measured for Sample 2, which indicated some passivation occurred during the storage period. The corrosion rates [<0.01 mils penetration per year (mpy)] measured for Samples 1 through 5 are not excessive for general corrosion attack. Some red rust was observed on sample discs located in the nitric acid and hydrochloric acid environment of Line 30. The corrosion rates (<1 mpy) measured for these sample coupons

TABLE 4. Glove Box Corrosion Test Results^a

Sample Identifications	Weight Loss (mg)	Corrosion Rate (mpy ^b)	Visual Appearance
1 - Line 1	2.9	0.003	No Change
2 - Line 1	+7.3 ^c	--	No Change
3 - Line 1	2.6	0.003	No Change
4 - Line 1	1.2	0.001	No Change
5 - Line 1	3.2	0.003	No Change
6 - Line 30	53.9	0.05	Light red rust ^d
7 - Line 30	485.5	0.47	Light red rust ^d
10 - Line 30	48.9	0.05	Light red rust ^d

a. Test duration - 12 months

b. mpy - mils penetration per year

c. Weight gain measured

d. Fe₂O₃

also indicated that no excessive, general corrosion was occurring in Line 30.

No localized (pitting, cracking) corrosion was detected for any of the sample discs examined by microscopic analyses. It was concluded that no excessive corrosion is occurring in the acid-vapor environments of either glove-box line.

Future Work

New sample disc coupons were located in Glove-Box Line 30 for a continuing program of monitoring. Since nitric acid is now the only acid employed in Line 1, no further corrosion of the 304 stainless steel line is anticipated; monitoring of that line has been discontinued. Line 30 still employs hydrochloric plus nitric acid for the chloride recovery process, and monitoring will be continued.

PITTING CORROSION OF BERYLLIUM PRODUCTION PARTS FOR THE W-76 PROGRAM

R. Alan Patterson and John L. Briggs

Objectives

The objectives of this work are to (1) evaluate certain beryllium parts to determine possible causes

for their speckled surface condition, (2) review the fabrication history to determine a possible operation responsible for the degradation, and (3) examine the possibility that this surface condition would affect a subsequent brazing operation.

Prior Work

This is a new effort.

Achievements and Discussion

Visual examination of a beryllium part revealed a speckled appearance on the inner and outer contour. Each speck is approximately 1 mm in diameter and is characterized by a brown annulus with a blue center. A low magnification (10X) inspection of the specks did not reveal the presence of surface contaminants or resolvable surface relief.

Attempts to absorb contaminants from these specks with distilled water-moistened cheese cloth did not yield any residue for chemical analysis. Therefore, 10 one-inch-diameter samples were cored out of the beryllium part. These samples were submitted for examination by the scanning electron microscope (SEM). The SEM examination of these specks revealed that they were actually small pits in the beryllium metal.

An energy dispersive analysis technique with the SEM revealed that a high concentration of chloride ions is associated with the pits. Beryllium has been shown¹ to be highly susceptible to pitting attack in solutions containing chloride ions. Thus, it is assumed that the speckled appearance of the beryllium surface is the result of localized pitting corrosion, with the active constituent being chloride-ion contamination.

A review of the operational sequence for these beryllium parts reveals that they are subjected to an ultrasonic cleaning operation in 1,1,1-trichloroethane, (Procedure M-38539) and are then transferred to the brazing area. The brazing procedure requires a surface preparation that involves etching the braze region with Ox-out 536 solution and rinsing in demineralized water. Two

possible sources for chloride ion contamination exist: the trichloroethane solvent or the demineralized water. No incidence of low purity water has been detected in the past year; therefore, the probable source for contamination is the chlorinated solvent cleaning process.

A review of Procedure M-38539 reveals that the beryllium parts are cleaned in an ultrasonic bath for a minimum of 3 minutes, but a maximum exposure time is not specified. Cleaning studies with chlorinated solvents² have revealed that significant quantities of chloride residues are retained on beryllium following 1,1,1-trichloroethane cleaning processes.

The relatively low incidence of pitted beryllium parts may be explained by the variable incubation period before visible pits appear,³ in conjunction with the loose time controls on the solvent cleaning procedures. The conditions to produce corrosion pits therefore are probably encountered on relatively few beryllium parts.

The Ox-out 536 cleaning procedure prior to brazing should eliminate most contaminants in the braze region. It is difficult to conclude that the solvent cleaning procedure or pitting corrosion is directly affecting the brazing procedure. Induction brazing generally involves many variables ranging from piece-part dimensional control to actual brazing parameter control. Therefore, the information obtained during this investigation is not sufficient to identify possible causes for braze rejects. The existence of surface contaminants, however, is generally detrimental to a brazing process.

Future Work

No further work is anticipated at this time.

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MONITORING CORROSION IN ROCKY FLATS OPEN COOLING WATER SYSTEMS

John L. Briggs and Marston R. Dringman

Objective

The objective of monitoring tests for corrosion in the open cooling water systems was to determine the effectiveness of polyphosphate inhibition and to provide preventive treatment if necessary.

Prior Work

Previous monitoring studies have been described in earlier progress reports.

Achievements and Discussion

Specimens of mild steel were obtained for the cooling tower corrosion tests. The specimens were rectangular coupons (3.0 × 0.5 × 0.06 in.) with a surface area of approximately 3.0 square inches. The sample coupons were cleaned with distilled water, rinsed with acetone, air dried, and weighed to the nearest 0.1 mg. The surface mill scale was not removed from the coupons to simulate steel equipment (heat exchangers, pipes) in the cooling tower water environments.

Duplicate sample coupons were immersed in the cooling tower sumps for a period of 26 months.

After the tests were terminated, loose corrosion products were removed and the coupons were

TABLE 5. Results of Cooling Tower Corrosion Tests

Cooling Tower	Test Duration (months)	Inhibitor Types	Phosphate Inhibitor Concentration (ppm)	Corrosion Rate (mpy ^a)	Sample Appearance
776	26	Sodium Pyrophosphate	8	1.9	Red rust plus gray sludge film
	26			2.2	
	4 ^b			1.4	
865	26	Sodium Tripolyphosphate	1	3.1	Red rust plus gray sludge film
	26			3.0	
	4 ^b			5.9	
559	26	Sodium Tripolyphosphate	7	0.9	Red rust plus gray sludge film
	26			1.1	
	4 ^b			2.7	
447	26	Sodium Tripolyphosphate	12	0.3	Protective gray film
	26			0.5	
	4 ^b			1.2	
881	26	Sodium Tripolyphosphate	4	0.7	Protective gray film
	26			0.6	
	4 ^b			1.0	
707	26 (lost)	Sodium Pyrophosphate	12	—	—
	26 (lost)			—	
	4 ^b			8.1	Red rust plus gray sludge film

a. mpy - mils penetration per year

b. Previous corrosion tests

rinsed in distilled water, rinsed in acetone, and air dried. The discs were then reweighed to the nearest 0.1 mg for corrosion rate determination.

Results of the cooling tower corrosion tests are shown in Table 5. The results of previous short-term (4 months) tests are included for comparative purposes.

All samples became coated with film deposits of soft, gray material indentified as calcium orthophosphate sludge, which precipitated from the phosphate inhibitor. Some red rust was also apparent on all of the samples.

Analyses of water samples from cooling towers treated with polyphosphates indicated the inhibitor concentrations were too low for adequate inhibition. Analyses of all cooling-tower water samples showed phosphate inhibitor levels ranging from 1 to 12 ppm. The recommended treatment for effective inhibition with polyphosphates is 35 ± 5 ppm. Recommendations for proper inhibitor treatment were distributed to concerned utilities personnel.

Future Work

New sample coupons were placed in all cooling towers to continue the monitoring program.

HYDROFLUORIC ACID CORROSION OF SILVER-SOLDERED NICKEL

R. Alan Patterson and Marston R. Dringman

Objective

Cracks in a sintered-nickel filter had been repaired by silver soldering. Corrosion tests were performed on sample coupons to evaluate the performance of repaired filters when subjected to a hydrofluoric acid environment.

Prior Work

This was a new objective.

Achievements and Discussion

Sintered-nickel filters in the new Plutonium Recovery Facility's fluorination reactor are exposed to moist fluorine gas at approximately 250 °C. Fluorine gas could hydrolyze in the presence of moisture to form hydrofluoric acid (HF).^{1, 2} Boiling hydrofluoric acid represents the worst possible atmosphere for exposure by the nickel filters.

Sample coupons of nickel 200, Easy-flow 45 silver solder, and silver-soldered nickel 200 were obtained for total-immersion corrosion tests. Sample preparation and test procedures were performed in accordance with NACE Standard Method TM-01-69.

Duplicate sample coupons were immersed in 20 wt % hydrofluoric acid at the boiling temperature (115 ± 5 °C).³ The coupons were weighed and visually examined after 24-, 70-, and 120-hour test periods. Table 6 summarizes weight-loss results and visual observations for each coupon.

Corrosion attack was characterized by localized pitting of the nickel and by general uniform dissolution of the silver solder. Samples of silver-soldered nickel exhibited increased pitting of the nickel adjacent to the silver solder joint. The first sample actually fractured in the silver-soldered joint during the 120-hour inspection. After this sample failed, immersion tests were discontinued and an evaluation of the fracture was initiated.

Metallographic examination of the fractured coupon revealed that accelerated pitting attack had occurred in the nickel adjacent to the silver solder. Thus, pitting had reduced the effective area of bonded surface and subsequent load-bearing capability of the joint. A review of the electromotive force series and the galvanic series³ revealed that the major constituents of the silver solder (Ag, Cu) are more noble than nickel in supporting a galvanic coupon that could accelerate corrosion.

The observed pitting attack, which reduced overall bond integrity, was the primary cause for eliminating

TABLE 6. Weight Loss in Hydrofluoric Acid

Sample Type	Test Time (hours)	Weight Loss* (mg/cm ²)		Comments
		Sample No. 1	Sample No. 2	
Nickel 200	24	0.8	0.9	Extensive pitting attack noted
	70	2.0	2.2	
	120	5.3	4.8	
Silver Solder	24	2.2	2.3	General uniform corrosion attack (dissolution)
	70	5.0	5.3	
	120	8.7	8.4	
Silver-Soldered Nickel 200	24	0.8	0.8	Extensive pitting of Ni, increasing in intensity adjacent to the silver solder, general dissolution of silver solder. Sample No. 1 fractured at 120 hours during handling.
	70	2.3	2.5	
	120	5.1	5.0	

*Weight loss quoted for comparison purposes only. Since the major form of attack was extremely localized (pitting), no corrosion rates are reported.

silver-solder repairs of nickel filters. The recommendation to replace damaged filters instead of repairing them was based upon this observation.

Future Work

No further work is planned at this time.

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EXAMINATION OF CRACKING MECHANISMS FOR THE LAKE ERIE EXTRUSION PRESS

R. Alan Patterson

Objective

An evaluation of responsible cracking mechanisms will generate engineering data that will help ensure safe operation of the modified extrusion press system. Differentiation between corrosion-assisted and pure mechanical cracking will be required if similar events are to be avoided in future utilization of the extrusion press.

Prior Work

Rocky Flats purchased the used extrusion press system in 1969, after it had been in use for 12 years. An initial inspection of the accumulator bottles revealed several regions with corrosion pits. The high-pressure water bottle was approximately 3% affected by corrosion pits no greater than 0.080-in. deep in the cylindrical section, and a higher frequency of pits no greater than 0.100-in. deep in

TABLE 7. Extrusion Press Water Samples

Date of Report:	2-2-76 ^a	8-3-76 ^b	4-27-77 ^c	9-25-78 ^d
Constituent				
pH	5.0	7.1	4.8	7.5
Total Dissolved Solids	—	216 ppm	976 ppm	1529 ppm
Cl	490 ppm	8 ppm	25 ppm	27 ppm
NO ₃	<5 ppm	—	—	100 ppm
NH ₃	—	—	—	<20 ppm
Si	20 ppm	34 ppm	7.0 ppm	2 – 4 ppm
Ca	8.6 ppm	16 ppm	23.0 ppm	20 ppm
Mg	30 ppm	—	—	50 ppm
Na	330 ppm	—	—	100 ppm

a. Report number not known

b. LAB No. M76-1066

c. LAB No. M77-583

d. LAB No. M78-1146

the lower cap section. The pitted regions were manually ground to expose unaffected base metal and reinspected. The second inspection did not reveal any further anomolous conditions, thus indicating that the system was acceptable for use. A mixture of water, soluble oil, and sodium hypochlorite algicide was chosen for the working fluid.

In 1976, a pinhole leak occurred in the low pressure piping. Examination of the leaking pipe revealed that chloride-induced corrosion pitting had completely penetrated the pipe wall (0.218 in.). An analysis of the working fluid in the press system is shown in Table 7. The high chloride-ion concentration was assumed to come from the sodium hypochlorite algicide additive and was identified as the main corrodant. Dye penetrant and radiographic inspections of the system detected widespread shallow pitting with a maximum depth of 0.094 in.

J. L. Briggs from the Component Development group recommended that the chloride-contaminated water be replaced with an aqueous solution of soluble oil and sodium silicate without the sodium hypochlorite algicide. Sodium silicate (Na₂O:3.3

SiO₂) was added to the system in an effort to inhibit further corrosion activity. Six corrosion coupons were placed in the water sump to monitor corrosion activity within the system. Finally, a hydrostatic test was performed at 4,750 psi, which is 1.5 times the normal working pressure. The system passed the hydrostatic test and was accepted for further operation.

A routine dye-penetrant inspection in 1978 detected 27 cracks in the high-pressure water bottle. These cracks were located within the weld joining the lower cap hemishell to the laminated cylindrical section. The cracks ranged in sizes from 0.060 to 2.0 in. long. An ultrasonics inspection of the crack depth indicated that the deepest crack was deeper than 0.250 in. An extensive visual examination did not detect corrosion activity greater than that reported in the 1976 inspection.

Achievements and Discussion

Construction material for the high-pressure water bottle is assumed to be a carbon-manganese steel with nominal composition of ASTM Specification

TABLE 8. Composition Range of Pressure Vessel Steel

Constituent	Weight Percent
C	0.22 - 0.3
Mn	0.85 - 1.1
P	0.045 max
S	0.050 max
Si	0.1 max
Fe	Balance

A 212, as shown in Table 8. This steel should have an ultimate tensile strength of 70,000 psi and a fracture toughness of ≈ 50 ksi $\sqrt{\text{in.}}$ at room temperature.

An analysis of the stress state for the high-pressure water bottle has been performed. One analysis, assuming circular crack profiles with a depth equal to half the crack length and a static 3,150-psi internal pressure, revealed that the stress required to promote unstable crack growth is approximately 25 ksi $\sqrt{\text{in.}}$. Therefore the calculated critical stress has significantly lowered the load-bearing capacity of this vessel. A second analysis, which took into account residual stresses equal to the yield stress, indicated that the cracks within the vessel were close to a critical depth of one inch. Further use of this vessel therefore was not recommended.

A review of the available literature¹⁻⁵ indicated that corrosion-induced cracking for C-Mn steels has occurred in the following environments:

1. Chloride ion-induced pitting or stress corrosion cracking in low pH systems.
2. Nitrate ions in systems with low pH (3.0 - 5.0) at room temperature or higher.
3. Phosphate ions in systems with low pH (3.0 - 7.0).
4. Caustic solutions with the potential for OH^- concentration buildup approaching a high percent value (50 wt %) at elevated temperatures (above 40 °C) and high pH (above 8.0).

Table 7 summarizes all available chemical analyses of the water within the extrusion press. As can be seen in Table 7, the system had been exposed to high chloride ion environments prior to 1976. From 1976 to 1978, however, a relatively low chloride ion concentration has been maintained. Since the cracks were not detected in the 1976 inspection, direct correlation between high chloride ion concentration and cracking mechanism cannot be made at this time. Presently there is insufficient information to determine a possible mechanism for crack initiation within this vessel.

Six mild-steel coupons were placed in the extrusion press sump to allow monitoring of corrosion within the system. After two years of exposure, these coupons were removed from the sump and evaluated. The average corrosion rate was calculated to be 0.03 mpy with little evidence of localized corrosion pitting. This low corrosion rate verifies the observation of the inspection team that no obvious corrosion attack had occurred between the 1976 and 1978 inspections. However, the low corrosion rates do not preclude the possibility of a corrosion-assisted cracking mechanism being active within the high-pressure water bottle.

Future Work

An evaluation of cracks within the high-pressure water bottle will be required before a possible mechanism can be determined. After samples of the cracked metal are removed for evaluation, a determination of crack mode and analysis of available corrosion products will be performed.

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ADVANCED SIZE REDUCTION FACILITY

James M. Sherman

Objective

The objective of this work is to design the remote handling, disassembly, and size reduction portions

of the Advanced Size Reduction Facility (ASRF) for processing contaminated waste items from the Rocky Flats Plant. This facility will be used for reducing the size of all glove boxes and other contaminated material from decommissioned processing areas.

Prior Work

An outside consulting firm was hired to compile an initial conceptual design report covering methods of achieving disassembly and size reduction operations. A 25-to-1 scale model was constructed to allow visual reference to the proposed facility.

Achievements and Discussions

A full-scale mock-up (Figure 1) of a 40- X 20-foot bay has been constructed in the Remote Engineering Laboratory, and master/slave manipulators placed to simulate a remote bay of the facility.

FIGURE 1. Advanced Size Reduction Facility Mock-Up

24058-2

Full-scale testing of disassembly, cutting, and transfer operations are now within mock-up capability.

The manipulator hand tools have been selected and tested to provide baseline data for remote disassembly operations and specifications of the tools. A pneumatic impact wrench (Black & Decker model 778) has been used for bolt removal, a pneumatic chipper (Chicago-Pneumatic model 0456 LASAN) is effective for lead shielding removal and window removal, and a manipulator-held razor utility knife has been used successfully to slash plastic and tape coatings. Other tools for less frequent use are a ratchet wrench (Gardner-Denver model 54536 AAB), file saw (Chicago-Pneumatic model 3517 LEF), abrasive saw (Bosch model 1333), and a "super-nibbler" (Fenway). These tools, which are handled by a master/slave manipulator, will be interfaced with a tool holder integral to the tool and with manipulator finger attachments. (See Figures 2 and 3 for disassembly examples).

Contracts have been awarded to Atomics International* to provide specifications for remote cutting equipment and techniques, positioning tables, and high bay work for overhead bridge crane manipulators and hoists.

Future Work

Future efforts are expected to include the following: (1) Complete specifications for disassembly tools and manipulators in the remote sections of the ASRF, (2) Develop holding fixtures for all tooling and maintenance schedules for all remote equipment, (3) Develop and test material transfer equipment throughout the facility, (4) Work with Facilities Engineering on all utilities, (5) Interface Atomics International work with Rocky Flats' transfer system and perform full scale mock-up in

*Atomics International, now known as Energy Systems Group is headquartered in Canoga Park, California and is an organizational component of Rockwell International.

FIGURE 2. Window Removal Disassembly Operation

23764-12

FIGURE 3. Bolt Removal Disassembly Operation

23464-6

the Remote Laboratory, (6) Perform full-scale simulated operation—from receipt of a crated box through disassembly and cutting operations, and (7) Complete a working model of ASRF for materials studies.

VAULT CLEANING SYSTEM FOR THE NEW PLUTONIUM RECOVERY FACILITY

John D. Bert

Objective

The storage area in the new Plutonium Recovery Facility (Building 371) vault will require periodic cleaning to keep the background radiation level low and to aid in radioactive material accountability. Since it is undesirable to send personnel into the vault, a remotely controlled vacuum cleaning system must be developed.

Prior Work

This is a new endeavor.

Achievements and Discussion

A preliminary design has been completed for a system to clean the bins and the vertical walls. This system was to have been moved by the stacker retriever vehicle in the same manner as a storage pallet. However, this system has been shelved, and work on a system to clean the vault floor has been initiated since most of the dust accumulation will be on the floor.

A small vacuum cleaner has been partially designed. It will be used with the floor cleaning systems described in the "Future Work" section. The cleaner will have a critically safe canister plus filter material capable of retaining the small particle-sized dust expected in the vault.

Future Work

Two systems will be developed for cleaning the vault floor. The first system will be permanently

mounted on the stacker-retriever vehicle chassis. It will clean the floor between the rails and will have a simple on-off control to lower or raise the cleaning heads and to operate the vacuum cleaner. The other system will use a cleaning head on the end of an articulated boom and will be used to clean the floor under the bins. It will be remotely controlled and will require a television system for viewing the operation. The articulated boom, vacuum cleaner, and television camera will be mounted on a pallet, which in turn will be handled in the same manner as storage pallets are handled by the stacker-retriever vehicle.

Development of the small vacuum cleaner will continue toward selecting the filter type most suitable for small-particle retention.

RASCHIG RING REMOVAL PROJECT

Jeffrey K. Aldred

Objective

All chemical process tanks at Rocky Flats with greater than a 5-inch diameter are made critically safe by filling them with glass Raschig rings. These rings are subject to chemical and mechanical degradation and must be replaced periodically. Hand removal of these rings presents a hazard to employees working at this task. Therefore, a project was initiated with the objective being to provide devices that can mechanically remove the rings from tanks without exposing the working personnel.

Prior Work

A 6-inch auger was designed and fabricated, and was tested on a noncontaminated tank. Problems were noted, and work was undertaken to correct those shortcomings.

Achievements and Discussion

The Raschig-ring removal auger (Figure 4) has been redesigned and fabricated. All metal components

FIGURE 4. Auger Installed on Ring Tank. Scoop is shown in foreground.

24059-2

have been fabricated from stainless steel to better resist acid corrosion. The rear hopper has been enlarged to allow a freer path for the passage of the Raschig rings. The poker that was fitted to stir the rings was eliminated and a series of seals was added to prevent corrosion of the bearings.

The rate for ring removal is approximately 1 cubic foot of rings every 7 minutes.

A new scoop also was designed and fabricated. The scoop works through a ball joint and has a replaceable end to allow interchangeable work-ends to be installed.

Future Work

The auger scoop system is under review, and an applicability study will be done. Rates of ring

removal will be determined, and a contamination control study will be conducted.

REMOTE HANDLING OF HOT INGOTS

Jeffrey K. Aldred

Objective

A series of tasks at Rocky Flats involve the transport of metal ingots weighing over 150 lbs at a temperature of about 1000 °C. These ingots are now handled, with some difficulty, by men using tongs and grapples. There is a certain amount of manual contact by employees who wear heavy heat-resistant gloves for protection. This presents a hazard to the employees. The objective of this

FIGURE 5. Design for New Unimate Robot Hand

work is to provide a device or devices to remotely transfer the hot ingots.

Prior Work

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

Achievements and Discussion

A current study involves moving ingots of two different dimensions and weights from a furnace to a rolling mill. The first ingot under study weighs 170 lbs. A Unimate robot was modified so that it was capable of lifting the weight. A new lift cylinder was fitted, and the wrist joints were disabled

as they are not strong enough to bear the load. A special hand having a 7- to 12-inch handling capacity was fitted in place of the removed wrist (Figure 5). Opening and closure of the hand is controlled by the robot; these movements are driven by 100 psi of air. Using the modified robot, an ingot was successfully moved through a process while at room temperature.

An additional requirement for lifting a 270-lb ingot was generated as was a hand opening of 7 to 12 inches. A design for such a hand has been completed; opening and closure of the hand will be achieved by a small hydraulic pump. The robot was successfully tested to see if the weight could be borne.

Future Work

The new hand will be fabricated and tested on the robot. Demonstration of the system will follow.

QUALIFICATION OF A FLEXIBLE LINER FOR TRANSURANIC WASTE DRUMS

Marston R. Dringman and Paul G. Aguilar

Objective

The objective of this work was to perform a series of qualification tests on a flexible liner to determine if the liner (when overpacked with a 55-gallon drum) would meet DOT and Rocky Flats performance requirements. Final approval and use of this liner for certain categories of transuranic (TRU) wastes would result in a substantial cost savings.

Prior Work

Preliminary tests have been conducted and reported. Results of the previous tests have indicated that the flexible liner would have performance characteristics similar to the presently utilized Type II rigid liner.

FIGURE 6. Liner Configuration in Drum

Achievement and Discussion

The flexible liner or Hedliner® is fabricated by blow-molding a low-density polyethylene and is manufactured by the Hedwin Corporation of Baltimore, Maryland. The liner ranges in thickness from 0.020 inch around the midsection to 0.070 inch at the bottom to 0.060 inch near the open end. A special lid, which is 0.040-inch thick, will be used with the liner. The liner is sealed with an automated flame sealer, and the seal is exterior to the drum, as shown in Figure 6. The sealing operation is performed with the lid of the drum in position.

Specification 2U in Title 49 of the Code of Federal Regulations, in addition to Rocky Flats Procurement Specifications, state that the liner must have a minimum wall thickness of 0.015 inch. Liners of this exact thickness were not obtainable, thus, drop and vibration tests were conducted on a thicker liner (0.020 inch), which will be used at Rocky Flats, and a readily available thinner liner with a minimum wall thickness of 0.012 inch.

Qualification tests for the liner were selected and conducted in accordance with Specification 2U in Title 49 of the Code of Federal Regulations and with Rocky Flats Procurement Specifications. Included were free-drop and vibration tests on loaded liners overpacked with DOT 17C, 55-gallon drums. Also, the environmental stress cracking

resistance (ESCR) and the brittleness properties of the polyethylene were determined.

For the drop and vibration tests, the test packages were prepared by placing the liners into DOT 17C, 55-gallon drums and filling them with sand and water-ethylene glycol mixture to 98% of capacity. The final gross weights of the packages ranged between 575 and 600 pounds. The ethylene glycol was added to prevent freezing during the low-temperature tests. Both the drop and vibration tests were conducted on packages preconditioned to -18°C and at ambient temperature.

The drop tests were conducted from a height of 4 feet onto a steel plate. A special sling was used to lift the drums so that they could be dropped onto the edge of the drum lid. This edge is considered to be the most vulnerable area to impact. The vibration tests involved four complete sweeps from 5 to 540 cycles per second with a maximum amplitude of one inch and an acceleration of 1.5 G.

For both tests, the criteria used to judge whether a liner passed or failed were based on whether or not liquid escaped from the package. During the vibration tests, none of the liners failed. However, during one drop test at ambient temperature, the round-back bolted ring used to close the drum did come loose, which resulted in a failure. From this failure and from experience obtained while loading the package, it became apparent that the layer of plastic from the liner between the gasketed groove of the drum lid and the chime of the drum made it difficult to properly secure the normally used round-back bolted ring. A thinner lid gasket (0.26-inch o.d. instead of 0.375-inch o.d.) and a square-back bolted ring were therefore used to close the drums for a second series of drop tests. No failures were recorded during that series of tests. Visual inspection of the drums and liners revealed some deformation after the drop tests; however, none of the liners (except the one failure) suffered any physical damage.

The ESCR (Environmental Stress Cracking Resistance) tests were conducted in accordance with ASTM Standard D1693. A total of 10 specimens per test were cut to size, notched, stressed in a holder and immersed in a detergent

solution (Igepal®) at 50°C . The minimum-required stress crack rating for the polyethylene is 250 hours (i.e., 50% of the samples must not exhibit cracking for 24 hours). After three months of exposure, none of the samples exhibited any signs of cracking.

The brittleness of the polyethylene at -18°C was determined with a dart impact test. The samples tested had minimum thicknesses of 0.060 inch. The test involved dropping a 20-pound dart with a 1-inch-diameter hemispherical end from 4 feet onto the samples. Failure was judged by the formation of cracks, which extended more than 1.5 inches beyond the impact area. None of the four samples tested failed this test.

Results of the tests did show that the flexible liner, when overpacked with a DOT 17C 55-gallon drum, can be used to package, transport, and store TRU wastes that do not contain sharp points, edges, or corners. However, a 0.26-inch-o.d. head gasket and the square-back bolted ring must be used to close the drum. Test results have been submitted to the Albuquerque Operations Office by Rocky Flats Waste Management. With the results was a request for using the flexible liner for packaging and transporting TRU wastes to Idaho National Engineering Laboratory for storage.

Future Work

No further work on this objective is planned.

PERFORMANCE EVALUATION OF MIXING DEVICE FOR THE PROCESSING OF POND SLUDGE

Paul G. Aguilar

Objective

The objective of the performance evaluation of the Roto-Feed® continuous mixer was to

1. Determine the effectiveness of the mixing device for mixing cement and a sludge,

representative of evaporation pond sludge, into a homogeneous mixture.

2. Determining the time required for solidification of the mixture.

Prior Work

It was reported previously that cement would be used for sludge fixation. Cementing cost would be minimized by reducing the amount of water as much as possible. This would be accomplished by allowing the sludge to settle for 24 hours in holding tanks. The liquid would be decanted and pumped to Building 374 for evaporation, and the thick sludge would be pumped to a continuous cement mixer.

Achievements and Discussion

A series of tests were performed on a Roto-Feed® continuous mixer manufactured by Baker Perkins, Inc. The purpose of these tests was to evaluate the effectiveness of the mixing device in mixing cement and a sludge similar to the solar evaporation pond sludge.

The factors evaluated were homogeneity of the mixture, and time requirements for solidification using various extruder screw speeds and sludge-to-cement ratios.

The results indicated that mixtures of more than 4 lbs of cement to 1 gal of sludge developed problems at the extruder port. The consistency of the extruded sludge reached the thixotropic limitation. Cement and sludge plugged the mixing device, forcing the mixture back through the cement intake port.

The mixer was eliminated as a possible method for sludge and cement mixing.

Future Work

The Roto-Feed® continuous mixer did not operate to expectations; consequently, other types of mixing devices will be evaluated. Preliminary tests

will be performed on a pug mill manufactured by Plymouth Locomotive Works, Inc. Test results will be used to determine if further testing of the pug mill is justified.

PILOT PLANT DEVELOPMENT

DEVELOPMENT OF A FLUIDIZED BED INCINERATION PROCESS

*Andrew J. Johnson, Scott C. Burkhardt,
and Kenneth E. Sedlmayr*

Objective

The objective is the design and development of a fluidized bed incinerator (FBI) system for processing radioactively contaminated combustible wastes. The system will reduce both solid and liquid wastes to a dry, nonflammable residue that is suitable for actinide recovery or additional waste fixation operations. This system has advantages over conventional incinerator systems because it has a lower operating temperature, decreased equipment corrosion, and no need for an aqueous offgas scrubbing system.

Prior Work

Several one-hundred-hour runs were completed on the pilot-scale FBI unit using a tributyl phosphate (TBP)-solvent mixture as the waste-feed source. These preliminary runs made it possible to design a liquid feed handling system compatible with the various liquid waste streams. The earlier runs provided valuable data on the optimum ratio between oxidation catalyst and neutralizing agent (Na_2CO_3) for successful incinerator operation. Tests showed that for the TBP-solvent mixture, a 50-50 ratio was optimum. Initial analytical results confirmed that the phosphorous pentoxide (P_2O_5) formation normally inherent in TBP-solvent incineration was eliminated by the use of sodium carbonate in the primary reduction vessel.

The design and initial stages of procurement for the variable compression screw feeder were accomplished. This screw feeder will permit feeding materials of various densities into the incinerator

while maintaining an air-tight seal. Full-scale test burns of several relatively noncompressible wastes, such as ion exchange resin and high efficiency particulate air (HEPA) filters, could then be undertaken in the fluidized bed system. Previously such wastes could not be fed to the incinerator without being blended with highly compressible waste forms such as paper-plastic mixtures.

Achievements and Discussion

TBP-Solvent Incineration

The data collection portion of the TBP-solvent incineration program has been completed. The last series of runs was accomplished with a TBP-solvent mixture containing 27.7% TBP. This TBP ratio served as a severe test of the liquid-handling and acid-gas-neutralization qualities of the fluidized bed incinerator. Both of the runs went exceptionally well, with minimal downtime. Post-run inspection of the vessels and offgas piping revealed that the minor offgas modifications accomplished during the last reporting period were successful in eliminating potential plugging regions. Upon completion of the laboratory analytical sample results, an overall evaluation of the TBP-solvent incineration program can be accomplished.

Solid Waste Incineration

A number of 100-hour and one 225-hour runs have been successfully accomplished. The runs were made to determine the reliability of various mechanical apparatuses used in the solid waste feeding system. The system consists of a coarse blade shredder (15 mm), a fine blade shredder (7 mm), an air classifier (metal removal), a bridge-breaking apparatus in the feed hopper, and a compression feed screw. All components operated well with minimum downtime. Overall results proved the waste feeding system to be reliable, effective, and relatively trouble free.

During the reliability tests, a week-long run was conducted without the use of the fine shredder. This was done to determine whether or not fine shredding was a necessity for adequate waste feed preparation.

Results revealed that the advantages afforded by fine shredding far outweigh the disadvantages of periodic maintenance. Upon completion of this series of tests, the coarse shredder was disassembled and inspected for wear. Several manufacturer design deficiencies were encountered, and corrective maintenance was initiated. In cooperation with the Maintenance Department, various internal bearing and shaft assemblies were redesigned and modified to improve reliability. The shredder unit has been reinstalled and is operating satisfactorily.

Future Work

The fluidized-bed-incinerator offgas sampling system will include a nondispersive infrared (NDIR) total hydrocarbon (THC) analyzer as a replacement for the present instrument. This NDIR analyzer should be more compatible with a production process than the flame ionization detector unit now in use.

Delivery of remaining components for the variable compression feed screw should allow final assembly and initial testing in the near future. The unit will be constructed and tested using simulated waste feed in the laboratory before being installed in the actual process.

DEVELOPMENT OF URANIUM CHIP BURNING PROCESS

Donald J. Vickery and L. James Meile

Objective

One objective of this project is to develop and demonstrate a pilot-scale uranium-chip incineration system that is safe and efficient. A second objective is to provide data to support the Engineering and Construction Department in designing a new offgas cleaning system for the present incinerator. In addition, data from the pilot scale demonstration will provide Engineering and Construction with design information for the future construction of a new production process unit.

FIGURE 7. Proposed Uranium Chip Burner

Prior Work

A conceptual design and material balance was formulated for a uranium incineration system (Figure 7) that features starved oxygen combustion, 100-pounds-per-hour capacity, recycled offgas, and an offgas cleaning system. The conceptual design was based on laboratory experiments in which nitriding and starved air combustion were evaluated as a uranium chip disposal process. Laboratory efforts to improve nitriding reaction rates proved

unsuccessful; however, data acquired during these experiments, coupled with additional laboratory tests, indicated that the direct conversion of uranium turnings to U_3O_8 in a starved oxygen reaction was a feasible design alternative.

Achievements and Discussion

Design work was completed for construction of the pilot-scale, starved-oxygen-combustion system

for the incineration of depleted uranium. All equipment has been ordered and installation of the pilot plant is scheduled for completion by April 1, 1979. The pilot scale system will react eight pounds of uranium chips per hour. This is one-twelfth the capacity of the proposed production scale process. Uranium chips and turnings will be batch fed into the reactor and will be electrically ignited. Combustion will proceed down the reactor, countercurrent to the upward flow of gas. This concept was formulated to control operating temperatures and create a heat zone to volatilize and burn any cutting oil that may be present on the surface of the turnings.

Recycled flue gas will be used to carry the heat of combustion from the reactor to the offgas heat exchanger. The rate of reaction of the uranium will be regulated by the amount of air blended with the recycle gas flow. A cyclone separator and sintered metal filters will be used to collect solid combustion products from the offgas stream. Flue gases will be cooled to 50 °C by a shell and tube-type heat exchanger before being transferred. Gas transfer by the recycle-exhaust blower will be through HEPA filters or back to the reactor to remove combustion heat. Exhaust and recycle gas flows will be regulated to control system pressure.

Uranium oxide will be removed from the reactor at the completion of a cool-down cycle. Fly ash removed from the cyclone and from sintered metal filters will be analyzed for suboxides and will then be recycled to the reactor, if necessary, for complete combustion.

The pilot unit is designed to operate without the batch feeder, catalytic converter, and exhaust fan that are included in the production design. A batch feeder on the pilot unit is not a safety requirement and is not economically justifiable at this time. Laboratory investigations have indicated that the catalytic converter function might be replaced by countercurrent burning. This concept will be tested in the pilot unit. A single blower, combining recycle and exhaust functions, will be adequate for the pilot scale offgas volume.

The Safety Department has identified petroleum-base cutting oils (previously used at Rocky Flats

for machining uranium) to be carcinogens; consequently, water soluble, non-carcinogenic, synthetic oils were substituted. These synthetics, which contain 50% water, are further diluted 40 to 1 to yield a solution of 80 parts water to 1 part synthetic. Thermoanalysis of these synthetics indicated that water is released between 100 and 300 °C, an exotherm occurs around 500 °C, and a clean burn is accomplished by 700 °C. Laboratory investigation indicated that a heat zone created by burning countercurrent in the reactor would sufficiently pyrolyze or burn the small amounts of synthetic oil that coats the uranium turnings. Consequently, catalytic afterburning will not be employed on the pilot unit. Catalytic burning of cutting oils was tested during this investigation and was found to be a feasible alternative that can be added if countercurrent combustion proves inadequate.

A documentary film from Oak Ridge National Laboratory (ORNL) depicted a uranium fire, extinguished with nitrogen gas, bursting back into flame when an air atmosphere was re-established. A laboratory investigation was conducted to determine the exact nature of this phenomenon so that operational safeguards could be established if necessary. Every attempt to recreate a similar occurrence was unsuccessful. Uranium that was oxidized and nitrided to various stages of completion was tested under various air and nitrogen gas flows. Uranium turnings, UO_2 , U_2O_3 , and U_2N_3 were combined in varying proportions. Attempts to initiate spontaneous combustion of each of these combinations were unsuccessful. Sparking of pure uranium turnings to initiate combustion was successful only occasionally. Combustion inside the laboratory reactor could be extinguished immediately by terminating combustion air or by ceasing all inlet flows, even with the reactor top open to an air atmosphere. To ensure operational safety, however, incomplete combustion products will be handled in a sealed metal container or in an inerted atmosphere.

Future Work

Future work will include pilot plant construction and installation, operational testing, system modifications and the pilot scale demonstration of the

process. Specific areas of interest will be dust collection, reaction efficiency, process control, and cutting oil effects.

FAST FLUIDIZED BED INCINERATION PROCESS

L. James Meile and Alfred L. Johnston

Objective

This work is being conducted to investigate the fast fluidization concept as a method for improved incineration of radioactive waste materials. The primary objective of the investigation is to determine if fast fluidization offers significant advantages for use in the fluidized bed incineration process now being developed at Rocky Flats.

Prior Work

A laboratory unit, fabricated primarily of quartz, was installed for operational testing of the fast fluidized bed concept. The concept involves the suspension of solid media in a high velocity gas stream for vigorous solids backmixing and exceptional solids-gas contact. Fast fluidization has been investigated elsewhere at superficial gas velocities of 8 to 18 ft/sec with suspended solid loadings ranging up to 20 percent of the total bed volume. Early testing was directed toward achieving a state of fast fluidization with a chromic oxide-alumina catalyst bed media. Suspended solid loadings up to 10 percent of the bed volume at gas velocities up to 6 ft/sec were achieved. Several combustion tests were attempted; prolonged burning was achieved in two of the tests.

In combustion experiments, a heated, slow fluidized bed of sodium carbonate granules was utilized for partial burning of a simulated waste mixture. The mixture consisted of equal parts of paper, polyethylene, and polyvinyl chloride. An overhead bed of fast fluidized chromic oxide-alumina catalyst was used for hydrocarbon burnoff. Some of the variety of problems encountered during combustion tests included poor temperature control, excessive

particulate attrition, cyclone drain plugging, and quartz breakage.

Achievements and Discussion

During this reporting period, effort was directed toward increasing catalyst loadings and improving particulate removal from the offgas stream. Minor modifications were made to improve the performance of the air ejector, and a platform was installed to facilitate safe maintenance operations at the upper level of the 17-foot-high unit. Commercial fluidized bed catalyst samples were obtained for fluidization tests in the laboratory system. In addition, a pulverizer-screening unit was installed to provide the capability to produce various catalyst particle sizes for testing purposes. No combustion tests were attempted while these improvements were in progress.

Fluidization tests were conducted to optimize overall system performance. Catalyst loadings were increased during these tests to enhance conditions for subsequent combustion tests. Loadings ranging up to 15 percent of the bed volume with fluidizing gas velocities up to 10 ft/sec were achieved. Excessive losses of catalyst fines through the cyclones and into the sintered metal filters proved to be a problem, however. Since large amounts of catalyst must be removed from the offgas stream and returned to the fluidized bed by cyclone separation, the entire fast fluidization process is dependent upon efficient cyclone operation.

To improve catalyst removal and return, two new metal cyclone separators were fabricated and installed as replacements for the original quartz components. The original primary cyclone, which separates and returns the bulk of the suspended catalyst particles, was designed for high efficiency separation but was not efficient under the heavy solids loading conditions. The replacement, designed for a heavy flow of solids, was installed and has proved highly satisfactory for the particular application. A secondary cyclone, designed for high efficiency at low solids loadings, was fabricated and installed to further reduce particulate losses into the offgas stream. The increased efficiencies

of the two cyclones, coupled with improvements in the cyclone drains, have substantially improved catalyst separation and return into the fast fluidized system. With the addition of the two metal cyclones and drain lines, the entire offgas system has now been converted from quartz to metal construction.

Future Work

When gas velocities and catalyst loadings are optimized, a metal reactor will be fabricated to replace the present quartz unit. The quartz construction offered valuable visibility capabilities for early fluidization tests, but it has proven to be less desirable than metal for waste combustion experiments. Combustion tests are planned to determine waste burning rates, acid neutralization efficiency, ash characteristics, and offgas quality. Gas analyzers for CO, CO₂, O₂, and hydrocarbons will be installed to obtain emissions quality data. Commercial catalysts will be purchased and tested for fluidization properties and for burning efficiency.

FLUIDIZED BED INCINERATION SYSTEM FOR DEPARTMENT OF ENERGY DEFENSE WASTE

*Dave L. Anderson, Burt A. Bell, Dean A. Burton,
Jack N. Carlson, Pen K. Feng, Frank G. Meyer,
and Kenneth E. Sedlmayr*

Objective

The objective of the project is twofold: (1) to demonstrate the adaptability of the fluidized bed incineration (FBI) process for combustion of a wide variety of transuranic (TRU) contaminated waste materials and (2) to prove scale-up information developed on the pilot plant equipment. The process was scaled up for a capacity of 82 kg/hr (180 lb/hr), which will correspond 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 reaching the project objectives. The system will perform real-time data acquisition, in-process

calculations, process control functions, inventory control of nuclear materials, and storage of data for off-line analysis.

Prior Work

Construction of containment wall and process duct work (Phase I) began in June 1976 and was completed in April 1977. A beneficial occupancy inspection of the job was accomplished on April 7, 1977.

Design of process equipment and associated process piping was completed in March 1977. After Title II review, process equipment installation (Phase II) started on April 11, 1977 by Rockwell Maintenance. This job was completed in April 1978, and a beneficial occupancy inspection was held on April 27, 1978. As of May 1977, all hardware items for the data acquisition system and associated process sensors were received. The data acquisition system consists of a Digital Equipment Corporation (DEC) programmable data processor-11/10 (PDP-11/10), Foxboro Spec 200 and Interspec analog-digital signal conversion system, cathode ray tube (CRT) display and teletype console, and a remote weighing station microprocessor used for inventory control of feed material. Process sensors include thermocouples, pressure cells, flow meters, weight cells, and contact closures. Installation of the data acquisition system and associated instrumentation was completed during April 1978. Development of the software package for the system is 60% complete.

To perform the required number of experimental runs with the FBI demonstration scale plant, approximately a two-year supply of chromic oxide catalyst and sodium carbonate neutralizing agent has been purchased and delivered. Also, enough simulated waste has been collected to conduct initial incineration runs.

An operational safety analysis (OSA) report has been written and disseminated for review.

Achievements and Discussion

System operations tests were performed during May and June of 1978; minor equipment and piping

modifications were made concurrently. Approximately 200 kg of simulated waste were burned during start-up runs conducted in July and September 1978. Operational problems with the sintered metal filters and system blowers were encountered and are being corrected. The data acquisition system came on-line smoothly and is presently able to display any one of 61 process variables on the CRT. These variables can be displayed individually or in prearranged groups. The system also performs hardware diagnostic checks and assists in calibrating instruments. Acquired data are periodically recorded on magnetic storage media for the purpose of record keeping and post-run analysis.

The data acquisition system is being upgraded by extending computer software capabilities. Efforts are continuing in the areas of automatic transfer of data from the remote weighing feed station to the computer, high/low alarms, and automatic printout of data. Programming of these additional functions into the data acquisition system is 60 percent complete. A training program to familiarize operators with the equipment and the data acquisition system has been completed. Further training will be implemented should the need arise.

Preliminary approval of the OSA has been received and final approval is expected in the near future. An Emission Permit Request, requesting permission for the operation of the FBI demonstration plant, was submitted to the Colorado Department of Health's Air Pollution Control Division. A license permitting such an operation has been received.

Future Work

Several incineration runs with simulated waste will be conducted in early FY 1979 (October through December) to bring the process up to design capacity. Afterwards, equipment will be evaluated according to their performance and reliability; necessary modifications will be made. The initial demonstrated operation with contaminated waste is planned for January 1979. Work will be initiated to incorporate direct digital control (DDC) features into the data acquisition system. This will be done after more experience is obtained on the operation

of the FBI demonstration plant and the process dynamics are better understood. When implemented, the data acquisition system will acquire and analyze process data, and then transmit appropriate signals to control the process.

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

IMMOBILIZATION OF INCINERATOR ASH AND PLANT WASTE

*J. Andy Ledford, Paul M. Williams,
Douglas R. Hippler, Seng F. Chung,
Michael DelHierro, and Roger A. Santeramo*

Objective

The objective is the development of methods for processing several types of radioactive residues into a form acceptable 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 because of the product's inherent advantages of nonflammability, chemical stability, and mechanical durability.

A bench-scale vitrification facility was designed, constructed, and operated; it originally consisted of an externally resistance-heated metal vitrification reactor, vertical-tube-annealing furnace, and transport mechanisms handling input and output. Elements of the process were changed as operational

experience indicated that improvements could be made.

Achievements and Discussion

Furnace and Reactor

The present vitrification reactor is fabricated of Inconel® 690 and is wholly contained in a commercially available furnace heated by silicon carbide glow bars. Reactor corrosion was anticipated to be a problem; however, observations of the reactor internally and externally have revealed no evidence of serious corrosion after more than 1000 hours of operation. The commercial glow bar furnace has been able to maintain the continuous 1050 °C operating temperature without problems. A minor rewiring of the electrical leads to the individual glow bars has been the only maintenance required. The original copper wire was replaced with aluminum wire coated with high temperature insulation.

The glass buttons produced in the vitrification process were being annealed to improve product durability. Product tests, however, indicated improved product strength could be obtained by tempering. The annealing step has now been replaced by a tempering operation conducted directly on the rotating button-forming plate. Two rows of small air jets positioned on either side of the button path provide a cooling air flow that rapidly removes heat from the glass. The rapid cooling of the exterior of the button produces a large temperature gradient in the glass, resulting in a final product with high exterior compressive stress and high interior tensile stress.

Offgas from the reactor is now being handled by a low-efficiency ejector that adds a fine silica sand to the gas stream. Volatiles are condensed on the sand rather than on the pipe walls; the sand also serves to remove pipe wall deposits by abrasion. Solids are returned to the ejector for recycle while the cleaned gas is routed through sintered metal filters before final high efficiency particulate air (HEPA) filtration.

A number of physical tests are now being developed to determine product compressive strength, physical

appearance, temperature effects, and leach resistance. The tempered product has been found to qualify for the DOT's designation of "special form material." This demonstrates that the product presents minimum hazard of contaminating the environment in the event of a transportation accident.

Joule-Heated Ceramic Melter

As a possible alternative to the metal reactor and glow bar furnace, another type of glass melter is being developed in which heating is accomplished by passing an electric current through the molten glass. Instead of metal, the melter is constructed of ceramic refractory.

Several successful start-ups of the ceramic melter have been accomplished using a silicon carbide glow bar that spans the area between the main electrodes. Application of current to the glow bar results in surface heating of the material in the furnace, melting the material, and creating a conductive path between the main electrodes. Current applied to the electrodes then passes through the melt, further heating it and creating more melt until, ideally, all the material in the furnace becomes molten.

This ideal condition was not easily achieved in the present melter. As the electrical resistance of the glass decreases with higher temperatures, preferential paths of current are created in those areas of the melt that are the hottest. Current flows through these areas, almost to the exclusion of the colder zones. This current flow creates conditions that do not encourage spreading of the melt. This problem will be addressed with an arrangement of several electrodes that can be selectively switched to force the current to travel in various areas of the furnace as desired.

Physical observation of the interior of the furnace, including electrodes, revealed no significant deterioration. Such results indicate that present materials used in the furnace construction will be suitable for use in future modifications.

Future Work

The test procedures that have been developed will be refined to evaluate variables in the process.

Another purpose will be to provide design data for the 4.5 kg/hr pilot plant facility to be installed in Building 777. Title I design for the facility is scheduled for completion in December 1978 to allow the start of construction in FY-1979. Design efforts for the project will be shared by the Pilot Plant group and Facilities Engineering. The group will provide design and document preparation for the process while depending upon Facilities Engineering and Construction to supply design efforts for building modifications and utilities and for over-all coordination of the project. Equipment development efforts will be directed primarily toward the Joule-heated ceramic melter to gather the data required for design and construction of an improved version of that unit.

SOIL DECONTAMINATION

*Doyle L. Mitchell, Leong Q. Fong,
and Loyal F. Richmond*

Objective

The objective of this work is to develop equipment specifications for use on the soils decontamination project at the Rocky Flats Plant. Data will also be generated that will help in the formulation of an economic evaluation to determine the best method for disposing of the soil.

Prior Work

Results of preliminary work have indicated that a wet screening and scrubbing process, with dispersants in the water, will concentrate the plutonium contamination into approximately 35 percent of the original soil weight. An estimate of the cost of a proposed facility was formulated and included in a conceptual design report (CDR).

Achievements and Discussion

Further studies have shown that contamination in the -35 mesh (-0.42 mm) soil fraction can be further concentrated by an additional process.

An intimate mixing of the fine fraction of the soil with water and a heavy organic liquid (nonpolar) separates out 99 percent of the activity. The mechanism appears to be a flotation of the contamination held by the <10- μ m soil particles and a partial dissolution of the plutonium into the aqueous media. The process has not proceeded beyond the bench-scale tests.

A mining consultant was contracted to provide expertise on concepts for digging and transporting the soil to the processing facility. Three concepts were generated, and a "screw excavator" was selected for further testing. The equipment is commercially available but would require some design and modification work. A negative pressure filtration system could be attached to the unit to help eliminate resuspension of particulates. The depth of cut could be adjusted from 5 to 60 cm (2 to 24 in.).

Scrubbing tests of non-contaminated Rocky Flats soil have been conducted by an equipment manufacturer. Test results will be compiled and included in a formal report. Filtration and centrifuge tests with Rocky Flats soil slurries are scheduled at two other equipment manufacturers.

Future Work

Rocky Flats personnel are scheduled to observe a screw excavator in operation at Baker, Montana. The digger will not be modified to meet Rocky Flats requirements; however, the digger can be observed in operation removing undisturbed soil.

Arrangements are in progress to engage outside laboratories and consulting firms to provide additional recommendations for soil removal and processing. Two development laboratories have expressed an interest in the testing of various manufacturers' equipment that may be applicable to the soils decontamination process. They will also provide recommended changes to the existing proposed flow diagram, operating parameters, and utilities requirements for the equipment. In addition, an engineering consulting firm will be contracted to make a paper study of the proposed flow diagram and recommend changes to improve

the process. The firm will be requested to provide a rough cost estimate for such things as engineering, equipment, mobilization, support equipment, and operation.

SURFACE WATER DECONTAMINATION

*Carl E. Plock, Terry N. Travis, John E. Garnett,
and Herbert N. Robinson*

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

A remote possibility exists that runoff water from the Rocky Flats plant could become contaminated with radionuclides and/or other inorganic substances such as nitrate. Therefore, as a part of the Surface Water Control Programs, new dams are being constructed in the creeks that drain the Rocky Flats plant. These dams will be large enough to impound the waters from a 100-year flood, which could be in excess of 100 million gallons of impounded water. If these waters, or the waters from a smaller flood, should become contaminated, the water will have to be returned to the Rocky Flats plant for decontamination. If the excess capacity of the Process Waste Treatment plant evaporators were to be used, it would require approximately nine years to process the 100 million gallons. However, by using the excess capacity of the Reverse Osmosis Recycling plant, the 100 million gallons of contaminated water could be processed in less than nine months. Before the surface runoff water can be processed through the Reverse Osmosis Recycling plant, data must be gathered to show that the processing of this water will not damage or adversely affect the membranes in the system.

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

and recycling of effluent from the Rocky Flats 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 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).

Achievements and Discussion

A new RO laboratory has been established in an area where small quantities of plutonium, uranium, thorium, and americium are permitted. Water is being transferred in 55-gallon drums from a pond on one of the creeks that cross the Rocky Flats reservation to the RO laboratory. The pH of the water is adjusted to 5.5 before it is processed in a small spiral-wound RO unit. The rejection rate of the total dissolved solids has averaged 93 percent at a water recovery of 90 percent.

Fifteen drums of the pond water were spiked with plutonium. The plutonium spikes ranged from 250 to 750 nanograms for each drum of water (170 liters) processed. The pH of the spiked pond waters varied from 2.5 to 5.4 prior to processing in the spiral-wound RO unit. The average plutonium decontamination factors ranged from 100 at pH 2.5 to 434 at pH 3.5.

The RO pilot plant, which was used in the previous RO project, is being renovated for future test use in a drainage area. Repairs of pilot unit equipment are now underway.

Future Work

Additional RO laboratory studies will be made to evaluate the decontamination of Rocky Flats runoff waters that have been spiked with plutonium, uranium, and/or thorium. The RO pilot plant will be located in a Rocky Flats creek drainage to

evaluate the effectiveness of the RO pre-treatment system for removing silt and colloids from runoff water at Rocky Flats. It will be necessary to obtain a modification to the National Pollutant Discharge Elimination System (NPDES) discharge permit for that creek drainage before placement of the pilot unit.

INSTRUMENTATION AND STATISTICAL SYSTEMS

RADIOMETRIC MONITORS - BUILDING 371

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

Objective

The objective of this work is to develop design criteria for radiometric assay instruments that will be installed in the new plutonium recovery facility.

Prior Work

Conceptual designs have been developed and in some cases construction begun on eight nondestructive assay systems. These 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. Cable runs for the instruments were identified and installed by the contractor. Specifications for the equipment were written and submitted to vendors with requests for quotation.

Achievements and Discussion

Gamma Ray Spectroscopy

A purchase order was placed to procure gamma ray spectroscopy equipment to assay molten salt residues and liquid samples. The system will control a stepping motor drive for the performance of segmented gamma ray scanning, relay closures, switch-closer sensors for sample changing and

transmission source activation, and a refractometer interface. The central processing system will be controlled by three terminals: one at the molten salt residue station, one at the liquids assay station, and the one located at the processor itself. Delivery of the system is expected in December 1978. The mechanical scanner and drive mechanism is being designed and will be fabricated in the Instrumentation and Statistical Systems machine shop.

Neutron Counters

A thermal neutron coincidence counter has been specified for assaying calcined oxide in the 800- to 2000-gram range. The electronics package to be used will be that developed by Los Alamos Scientific Laboratory, Group Q-1. Their construction drawings have been received and the bid package has been assembled and submitted to vendors through the building contractor. The glove-box appendage for the calcined oxide counter was designed and is being fabricated by the contractor. The ^3He detector assembly is being built in-house.

Future Work

Modifications will be made to the four residue counters as they are now designed. The counters will be built to include capabilities to perform gamma ray transmission measurements for sample matrix corrections. As equipment is received, 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 transmitting information concerning nuclear material accountability and control.

HIGH-RESOLUTION PACKAGE COUNTER "CAN SCAN"

*James L. Lawless, Francis X. Haas,
Wayne E. Herren, and William B. Schmidt*

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 in the plutonium recovery facility. The counting system, consisting of the sample cavity, turntable, and detector, is located in a counting room; associated computer and electronics are housed in an adjacent room. Air conditioning and regulated instrument power are supplied to minimize system failures.

Achievements and Discussions

The system hardware and software have been modified to simplify the operation. Diagnostic messages have been added to lead the operator through the various steps required for proper calibration and operation of the system. The instrument is presently calibrated for molten salts, packaged in Volrath cans, and is being routinely used for this purpose.

Future Work

A major effort will be placed on refining the americium calibration. Standards for this calibration are now being prepared by the molten salt facility. System response to other waste categories will be investigated.

ORALLOY DECONTAMINATION MONITOR

*James L. Lawless, Wayne E. Herren,
E. Duane Frantz, and Donald L. Baaso**

Objective

The objective of this work is to provide instrumentation capable of assuring that oralloy shipments to Oak Ridge are free of plutonium contamination.

*Baaso is associated with the Chemical Operations group.

Prior Work

Alpha scanning devices to detect plutonium hot spots have been used in the past. The first device was a zinc sulfide-phosphor detector that was held at a fixed critical distance from the surface to minimize U-235 alpha interference. This detector was later replaced by a more practical surface barrier alpha detector. These instruments were set to alarm if the count rate exceeded 300 counts per minute. Although this arbitrary limit was probably adequate, quantification of this limit in terms of grams of plutonium had never been determined.

Achievements and Discussion

Rocky Flats has built a new facility for decontaminating oralloy parts prior to shipment to Oak Ridge. Two new alpha counting systems have been developed to ensure shipments free of plutonium contamination.

The first is an alpha scanning system that involves surface barrier detectors held at a fixed distance from the surface. The response of the system has been determined by the use of standards. The system will confidently alarm and cause item rejection if any spot contains plutonium in excess of one microgram.

The second is a filter-paper-smear alpha counting system that uses energy-discriminating single channel analyzers. This is the final test in which the entire part is manually smeared with a filter paper to remove loose particles. The filter paper is then counted. A count rate exceeding 60 disintegrations per minute in the plutonium channel will cause item rejection. This satisfies Department of Transportation criteria.

Rocky Flats representatives met with Oak Ridge personnel at Oak Ridge. These tests and limits are acceptable to both shipper and receiver. Oak Ridge is planning to install a verification alpha scanning system, similar to the Rocky Flats design, in the near future.

Future Work

A multichannel analyzer is now being purchased to replace the single channel analyzer, alpha smear

system. The new equipment will simplify the operation and produce documented results.

CRATE RADIOASSAY COUNTER

*Ronald A. Harlan, Wayne E. Herren,
Kenneth B. Gerald, George P. Minges,
and E. Duane Frantz*

Objective

The objective of this work is to develop a method to detect and estimate plutonium in 1.22- X 1.22- X 2.13-m (4 X 4 X 7 ft) crates.

Prior Work

A counter was placed in operation in June 1978 and found to be sensitive to less than 10 grams of PuO_2 in a 20-minute count. The first calibration was found to be unacceptable because of gamma ray response in the neutron detectors. The counter was subsequently recalibrated. Other details are reported in the previous progress report RFP-2818, March 30, 1979.

Achievements and Discussion

Drifting of the background causes an uncertainty of 8 grams at the 0.95 confidence limit. Part of this drifting is related to variations in room temperature at the neutron detectors. The detectors reside in an open building with no temperature control. A temperature sensor and oscillator circuit were built and installed. Output of the oscillator circuit is read into a scaler to give a number related to the average temperature during a count. These data may be used to numerically correct the background and sample counts for drifting. Such corrections are not a part of routine operations at this time. The counter was declared operational for the user, Waste Management, to count samples. Estimates are being calculated from neutron data only. Experience to date shows that the counter gives generally higher plutonium assays than estimates given by those originating the crates. This matter is under further study.

It is well known that (α, η) reactions are produced by alpha emitters in close proximity to light elements. This situation occurs in most of the waste and was presumed to be accounted for in the calibration sources. Overestimates would be expected if the (α, η) reaction yields are increased for some reason. One reason expected for a small fraction of crates at Rocky Flats would be high specific alpha activity. This activity is due to americium-241, which is present in amounts larger than the stream average of approximately 150 ppm. This should not cause much problem until the americium-241 content exceeds 1000 ppm relative to plutonium. Another reasons for high (α, η) yields would be the presence of plutonium flouride or oxyfluoride. The cause of high (α, η) reaction yields can be detected by using gamma ray spectroscopy to seek gamma rays that serve as a signature for americium decay, or for the $^{19}\text{F}(\alpha, \eta)^{22}\text{Ne}$ reaction.

Sodium iodide detectors were installed at the crate counter to look for gamma rays from americium and fluorine in the waste. So far, no cases have been clearly identified. Thus, further study is needed to clarify the cause and meaning of differences between counter assay and originator estimates or assays.

Future Work

Plans are to examine some of the "hotter" crates with a high resolution germanium detector. Some basic research into (α, η) yields may also be indicated.

CALIBRATION AND INVENTORY VERIFICATION EXPERIMENTS

Ronald A. Harlan and Kenneth B. Gerald

Objective

The objective of this work is to provide a data base for nondestructive assay (NDA) calibrations of Rocky Flats radiometric counters.

Prior Work

Work under this endeavor is a continuation of similar studies dating back to 1964. 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 by participating on the Rocky Flats Measurement Control Task Force. Nondestructive assay devices covered are the Can Counter, Helix Counter, Waste Packaging Facility's (WPF) South Drum Counter, and WPF North Drum Counter. Since 1974, counter results frequently have been compared to calorimetry results, and the latter results used to back-calibrate the counters. Some samples are sent to the Mound Facility in addition to being calorimetered at Rocky Flats.

Achievements and Discussion

Samples were selected for the 1978 Mound exchanges, counted, and the shipment made. In addition, a number of samples were calorimetered at Rocky Flats. Recently received results on these samples are actively being studied; however, final results are not available at this time. A calibration for uranium-233 in the WPF North Drum Counter was developed with the cooperation of the Chemistry Standards Laboratory.

Future Work

Results from the 1978 Mound exchange calibration experiment will be used to implement any required changes in NDA calibrations at the can and helix counters.

CALORIMETER END POINT PREDICTION

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

Objective

The objective of this effort is to develop techniques for predicting the final value approached by a

*CO-OP Student

calorimeter before the instrument actually reaches this end point value. The result will be a savings in analysis time and equipment.

Prior Work

A minicomputer code that approximates the true multiple exponential curve of a calorimeter has been executed off-line. The success of this project justified further investigation.

Achievements and Discussion

A multiple calorimeter data acquisition, control, and computing system has been interfaced, programmed, and tested to allow further on-line calorimetry work. The system consists of a scanner, ratiometer, programmable calculator, and a mini-computer. This system is being used to improve the accuracy of the end-point prediction algorithms. Algorithm tuning and curve smoothing currently are being pursued. Because of the unavailability of a calorimeter in our area, off-line work is still being done.

Personnel from the Savannah River facility have seen and have shown significant interest in our system. They are currently sending some of their calorimeter data to be analyzed at Rocky Flats for possible end point application.

Future Work

Work will continue with the goal of a 60 percent reduction in analysis time and less than 5 percent error. The system is ready to be installed on a calorimeter for on-line use and evaluation. If downtime can be arranged, the system will be installed on an existing 2-cell Chemistry Standards Laboratory calorimeter for use and evaluation.

GAMMA RAY ISOTOPIC MEASUREMENTS

Francis X. Haas

Objective

The objective of this work is to establish nondestructive techniques for measuring plutonium and

americium isotopic ratios for nuclear materials safeguards assay and standards evaluation.

Prior Work

No prior work has been reported on this topic.

Achievements and Discussion

High-resolution gamma-ray spectroscopy has been used to measure the americium-241 and plutonium-239 ratios in 22 samples of pulverized, 8 percent molten salts. This information, together with calorimetry data on these samples, was used to provide process calibration standards for calibrating the segmented package counter—"Can-Scan." The measured amounts of plutonium ranged from 0 to 70 grams, and the material was packaged in 8801 Volrath cans contained inside 8802 Volrath cans.

An interlaboratory exchange of FORTRAN programs and procedures for gamma-ray data reduction has been initiated. A hard disk has been sent to the Battelle Pacific Northwest Laboratory for a copy of the gamma-ray reduction programs developed by Ray Gunnick from Lawrence Livermore Laboratory.

Future Work

Instrumentation and Statistical Systems will investigate gamma-ray spectroscopic techniques for measuring plutonium and americium ratios and for measuring other isotopes. The goal is to help various groups meet quality assurance requirements for product materials and to bring such nondestructive analytical techniques to the state-of-the-art. The first application will be in setting up procedures for determining plutonium isotopic concentrations for a pending incoming shipment of plutonium oxide.

PERIMETER SECURITY ZONE

Grover J. Cunningham

Objective

The objective of this project is to lower the cost of detection and lighting for the Perimeter Security Zone (PSZ).

Prior Work

No previous work has been done on this project by Instrumentation and Statistical Systems.

Achievements and Discussion

The new PSZ, which will envelop the entire production complex, has a double-fenced no-man's area that is to be under high-intensity lighting and closed circuit television (CCTV) surveillance. Installation and operational costs are high enough that alternate means are being sought to provide equal security at reduced cost.

A new type of infrared security system, Pyro Electric Vidicon Thermo Imaging System (PEV), that would possibly eliminate the need for the high-intensity lighting, CCTV, and one type of motion detection in the PSZ has been identified and proposed to the Security Department for evaluation. A demonstration at Rocky Flats by the manufacturer is forthcoming.

Future Work

Based upon a successful demonstration at Rocky Flats, the system may be purchased for further evaluation, testing, and possible installation in the PSZ.

INTRUSION DETECTION SYSTEMS

Grover J. Cunningham

Objective

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

Prior Work

No previous work has been done on this project by Instrumentation and Statistical Systems.

Achievements and Discussion

An infrared security system has been installed in one area, and testing is now complete. This system is ready for security use.

In addition, a vibration detection system has been installed and is undergoing tests for false alarms and sensitivity. A conceptual layout for other systems has been completed.

Future Work

Intrusion detection system will be installed in other areas on an as-needed basis.

DURESS ALARM

Grover J. Cunningham

Objective

The objective of this project is to provide the capability of remote duress alarms at Rocky Flats guard posts.

Prior Work

No previous work on this project has been done by Instrumentation and Statistical Systems.

Achievements and Discussion

Tests were conducted on a remote triggering device that would give an electrical contact closure at a considerable distance from an alarm point. Successful tests have resulted in the purchase of this instrument for all Rocky Flats guard posts.

Future Work

No further work is planned.

OPEN-SAFE WARNING DOOR SWITCH

Grover J. Cunningham

Objective

The objective of this "open safe" warning system is to alert safe custodians to an open safe condition and thereby decrease the possibility of security infractions related to unlocked safes.

Prior Work

There has been no prior work on this objective.

Achievements and Discussion

A warning alarm was installed to alert office personnel of an open safe situation and to decrease the possibility of an unattended safe. The alarm is installed above the office exit door and triggered by a magnetic switch attached to the door (Figure 8). When the office door is opened, the alarm is activated, thus emitting a one-time 2-second audible tone. The system can also be activated by a motion detector. An On-Off switch is mounted on the safe and is manually activated when the custodian opens

FIGURE 8. Door-Switch Warning Alarm for Open Safe

the safe. This device, or a modification thereof, may be useful to others on the plant site.

Future Work

No additional work is planned.

X-Y RETRIEVER VERIFICATION SYSTEM

Grover J. Cunningham

Objective

The objective of this project is to provide manufacturing personnel with a quick check system for verifying at a distance the presence or absence of special nuclear material (SNM) in each storage location within the X-Y retriever vault.

Prior Work

Instruments were purchased and a collimator design was completed. (See RFP-2818, March 30, 1979.)

Achievements and Discussion

A radiometric scanner has been installed in the X-Y retriever vault to provide special nuclear material (SNM) verification at inventory time. Normally an operation requiring up to two days, SNM verification has been reduced to a few hours. Tests show the scanner will verify the presence of 95 percent of the SNM without a sensitivity range change. To verify the other 5 percent, a range change must be made to the instrument or the SNM material moved to a lower radiation background area within the vault.

Future Work

Instruction on the care and use of the instrument will be given to manufacturing personnel.

ADVANCED SIZE REDUCTION FACILITY

Francis X. Haas

Objective

The objective of this work is to provide design criteria for radiometric instrumentation for use in the Advanced Size Reduction Facility. Remote radiometric instruments will be built and tested to satisfy nuclear safety criteria and to measure contamination levels on reduced-size items at levels below 10 nanocuries per gram.

Prior Work

No prior work has been reported.

Achievements and Discussion

A variety of radiometric devices will be specified to accomplish the dual purpose of providing measurements for nuclear criticality safety and process control. The major part of the size reduction operation involves removing actinide contamination by physical means, i.e., by such means as scraping and spraying. However, final decontamination will take place in aqueous electropolishing tanks sized to handle 3 ft (91.5 cm) by 3 ft (91.5 cm) by 3 in. (7.5 cm) pieces of hardware. These tanks present special nuclear safety problems, and attention to date has been focused on the electropolishing operation.

Nuclear safety devices will be built to ensure the plutonium limit established for the electropolishing tanks is not exceeded. This will be accomplished by (1) measuring the plutonium content on each item before it enters the electropolishing tank, (2) continuously measuring the plutonium and nuclear poison concentrations in the electrolyte itself, and (3) monitoring plutonium buildup in filters, pumps, and tank bottoms associated with the electropolishing system.

The electropolishing input monitor will be a shielded array of NaI(Tl) crystals placed on each side of the assay item. Americium-seeded detectors have been chosen for the array. The americium

will provide a built-in calibration source for monitoring the correct operation of the instrument in addition to providing a reference pulse for stabilizing the electronics in the working environment. Work has begun on mapping the detector response for the 350- to 420-keV gamma rays from a 1-g source as a function of distance and solid angle. This is being done to determine the optimum detector array for remotely measuring items that fit into a 3 ft X 3 ft X 3 in. envelope.

The buildup of plutonium in the electropolishing bath will be measured using X-ray fluorescence. Maximum allowable plutonium concentration will be about 200 mg/l. Specifications for the X-ray fluorescence unit have been written and the quotations reviewed. Delivery of the equipment is expected by February 1979.

In addition to monitoring the plutonium concentration buildup in the electrolyte, Nuclear Safety requires that a nuclear poison be placed in the solution and its presence be monitored continuously. If cadmium is used as the poison, its concentration will be measured by X-ray fluorescence at the same time the plutonium is measured. Boron is another poison suggested; its concentration will be measured using neutron transmission measurements through the solution and/or the measurement of the 478-keV gamma ray associated with the $^{10}\text{B}(\eta, \alpha)^7\text{Li}$ reaction. A moderator has been built to produce a thermal neutron beam for a plutonium-beryllium neutron source. Measurements are presently underway to measure the neutron attenuation in samples containing various concentrations of boron.

The remote retrievable-nonretrievable waste monitor will consist of an array of NaI(Tl) detectors similar to the input monitor. In this array, however, thin NaI(Tl) FIDLER detectors will be used to measure the 17-keV X rays emitted in the plutonium decay. Detectors are on order and will be calibrated by measuring their response to X rays emitted from a plutonium dish source as a function of solid angle and distance from the detector.

Future Work

Instrumentation will be purchased and experiments run to determine instrument design criteria. Scheduled completion date is April 1, 1979.

WATER CONTROL AND RECYCLE PROJECT

David E. Hausburg

Objective

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

Prior Work

Support of this work is on a continuing basis as reported in Progress Reports RFP-2417A, August 20, 1975, p. 31; RFP-2818, March 30, 1979, and in two classified progress reports.

Achievements and Discussion

Beneficial occupancy of the Water Recycle Plant has been obtained from the construction company so that shakedown of equipment, system operation testing, and instrument calibration can proceed. Installation of all process equipment, instrumentation, and the process control panel is complete. Also, the final editions of the Plant Operating Manual and the System Operation Test Procedures have been received.

A program to provide alarming functions and limited control functions has been entered into the programmable logic controller so that system operation testing can take place.

Programmable controller technology training for beginning engineers, technicians, and plant operators is being provided so that efficient operation of the plant can occur when on-line operations begin.

Future Work

Technical assistance to resolve process start-up problems normally associated with new construction will continue. Program development for the programmable logic controller will be a major task until the plant is operational. In addition, work to document the control program and the training of plant personnel will be required.

GRIT BLAST FEEDER

*George P. Minges, Frederick L. Crawford,
and E. Duane Frantz*

Objective

The objective of this work is to provide a means for measurement and possible control of the performance of the production grit blast machine.

Prior Work

The current grit blasting machine has no provision for measurement of the sand-to-air ratio and thus requires constant operator adjustment. The sand-to-air ratio is controlled by a crude gravity value set by the operator. The operator judges the quality of the operation by the "sights and sounds" being emitted by the machine.

Since the air is currently controllable, Instrumentation and Statistical Systems suggested a design for a sand auger delivery system that could be calibrated in flow in addition to being easily controllable by a microprocessor. This would then give a known sand-to-air ratio.

Achievements and Discussion

A sand auger delivery system has been designed, fabricated, and tested in our laboratory. This system will control, measure, and repeat the sand-flow requirements. Coupled with the known air flow, this system will provide for the objectives of sand-to-air ratio measurement and electrical control.

Future Work

The auger system will be installed when sand-blaster downtime can be scheduled. After installation and testing, an evaluation will be made; a second auger may be fabricated to provide a spare and to incorporate any indicated improvements.

PROCESS DATA ACQUISITION SYSTEM

George P. Minges, Mike A. Fox,
James L. Lawless, Charles M. Johnson,
and Charles R. Forrey***

Objective

The objective of this project is to provide Chemical Operations with a means of limited control, data acquisition and reduction, and central record keeping for the main chemistry lines.

Prior Work

At the request of Chemical Operations and in conjunction with Process Chemistry and Engineering, the concepts of an ideal data acquisition and control system were formulated. This system was to be compatible with each major wet-chemistry production line. (See previous progress report RFP-2818, March 30, 1979.)

Achievements and Discussion

A prototype general-purpose microprocessor system has been designed, fabricated, and programmed to accept data from, and interface to, all types of production line instrumentation. This device utilizes a plant standard 8080 microprocessor designed to accept data in analog, digital serial and parallel, and of a varying frequency.

A Hewlett-Packard minicomputer is used for data reduction and central record keeping. To provide a means of guaranteeing noise immunity in a production area, a 100-meter fiber optic cable was used in the 2400 bit per second mini-to-micro system interface.

Future Work

Documentation of this system is now being prepared. Upon completion of the documentation and system testing, the unit will be ready for installation. If

*CO-OP Student

**Process Chemistry and Engineering

the prototype system is satisfactory to Chemical Operations, future microprocessors can be fabricated by other groups or outside vendors.

GAS SAMPLING SYSTEM FOR FLUIDIZED BED INCINERATOR

*L. Harry Faber, Charles M. Johnson,
and William B. Schmidt*

Objective

The objective of this project is to design and fabricate a gas-sampling interface system for the fluidized bed incinerator that will extract, transport, condition, and distribute a representative effluent gas sample to the continuous analyzers.

Prior Work

No prior work was done on this project.

Achievements and Discussion

The stack-gas analyzers on the incinerator effluent require an extracted gas sample that is low in particulate matter and has a dew point low enough to prevent condensation in the sample lines and analyzer sensors. The removal of particulates, which in this application could be radioactive, and moisture from the combustion process must be accomplished with minimal effect on the concentration of components to be monitored. Further, this interface system must provide a constant flow of conditioned stack gas in sufficient volume and at a pressure sufficient to operate the on-line analyzer instruments.

An interface system has been designed and built to the above specifications just mentioned and to operate as follows. By operator demand, the interface system (Figure 9) will introduce zero or calibration gas to the analyzers. The same flow route will be used as that followed by the sample gas, thereby checking the integrity of the system for component dilution or degradation that would

24033-9

FIGURE 9. Fluid Bed Incinerator Gas Sampling System

affect the analytical results. Solenoid valves operating the calibration system will remain "ON" for a preset time period, then return to "NORMAL OPERATION." Lights on the front panel indicate when the function is in progress.

Individual flow meters with front-panel readout control the output to analyzer instruments and monitor the return flow. This return gas and excess sample gas is returned to the stack to prevent the possibility of a contamination release.

To keep a constant flow going to the analyzer, an automatic backflush is inaugurated periodically that purges the stack intake filter from particulate buildup. Excessive loading of this filter will "SHUTDOWN" sampling from the stack and introduce ambient air into the analyzers.

The gas sampler system is being installed in the instrument area of the fluidized bed incinerator.

Future Work

From tests conducted to date, it appears that a higher volume pump will have to be installed to overcome a higher-than-expected negative line pressure on the stack. Technical backup will be provided as needed; however, no major work is planned on this project.

BREATHING-AIR HOOD TEST APPARATUS

*David E. Hausburg, Frederick L. Crawford,
William B. Schmidt, and Ollie I. Buttedahl**

Objective

The objective of this work is to design and construct a semiautomated test apparatus for quality acceptance of breathing-air hoods.

Prior Work

No previous work has been reported.

Achievements and Discussion

Vendor-supplied, plastic, breathing-air hoods require testing for minuscule leaks before being

accepted by the Rocky Flats Warehouse. Non-destructive Testing is responsible for the quality acceptance program. Presently, a modified glove box is used as a chamber for testing plastic air hoods, plastic barrel liners, and plastic bags. The glove-box volume is greater than required for hood testing, and evacuation of this large volume to comply with procedures is time consuming. An apparatus designed specifically for testing hoods would speed up hood testing and thus would increase efficiency.

A prototype single-chamber test stand was constructed for evaluation (Figure 10). However, the final design of the test stand included an additional test chamber. Thus, testing of one hood in a chamber can take place while the second chamber is being loaded with another hood for testing.

Industrial Safety was consulted concerning the rest position of the chamber lid. Subsequently the lid hinge was relocated and a rest stop provided for

*from Component Development

FIGURE 10. Test Apparatus for Breathing-Air Hoods. Shown is one of two test chambers.

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operator safety. With proper instruction concerning pinch points, the apparatus will not present safety hazards for the operator.

Included in the electrical test sequence is an alarm function. When a leak in a hood is detected, an alarm occurs and testing cannot continue until the alarm is acknowledged. Testing of hoods to provide a reliability study of the apparatus is in progress.

Future Work

Modifications to the apparatus may be necessary to accommodate the users' requirements, e.g., counterweights to the chamber lids to reduce operator fatigue.

CORRELATION SPECTROPHOTOMETRIC ANALYSIS OF PROCESS MATERIALS

*Charles M. Johnson, L. Harry Faber,
Frederick L. Crawford, George P. Minges,
and Mike A. Fox**

Objective

The objective of this project is to develop an instrument for analyzing minute quantities of compounds in gaseous and liquid process streams. It is the further objective of this project to develop component parts of a correlation spectrometer that will be adaptable to the several configurations necessary to analyze plant process streams.

The development of these instruments will fill a void that now exists in process control instrumentation technology. First application will be in monitoring the fluidized bed incinerator offgas for hydrochloric acid.

Prior Work

Basic work in correlation spectrometry has been performed by several investigators at various

locations throughout the country. This work has been directed primarily to laboratory-type instrument and gas samples. In early 1974, a need developed for a process instrument to analyze low concentrations of HCl and HF in effluent stack gases. A contract was later awarded to Science Applications Inc., La Jolla, California, to develop two analyzers, one each for HCl and HF. Although two units were built, electronic drift and other stability problems prevented the analyzers from meeting specifications. These units were later purchased by Rockwell with the intent of modifying electronic and mechanical functions to meet specifications.

Work at this laboratory has been directed toward the development of reliable components and their incorporation into a process type instrument.

Achievements and Discussion

Filter correlation spectrometry has the capacity to enhance the compound of interest and to nullify interfering compounds. The correlation spectrometer has many components that are common to standard nondispersive infrared analyzers. For instance, the infrared source, detector, and chopper are the same as those found in all infrared instruments.

The most critical measuring element of the correlation spectrometer is the wave form produced by the detector. All elements of the instrument are designed to produce and analyze this wave form. In a process instrument it is necessary to design dependable, rugged components that will withstand process use over many months without maintenance. For this reason we have separated the measuring cell and the electronics.

Thus far our design efforts have produced a stabilized power supply to eliminate random noise and voltage fluctuations. An improved detector preamplifier has enhanced the wave form. This improved wave form has indicated a fault in the detector mounting and temperature regulation system, which is being redesigned. Improvement in the chopper wheel and chopper motor has produced a stable wave form that enhances the analysis. A stabilized source regulator has

*CO-OP Student

eliminated wave amplitude variations that confuse the analysis. Because of the complexity and interaction of the components, corrections of one element indicate faults in another element.

Future Work

Further modification of the present instrument would be nonproductive. With the experience gained so far, any additional work should begin with design of a new spectrometer.

Work on this project has been deferred because of the lack of funds and available manpower.

1978 DENVER BROWN HAZE STUDY

*Frederick L. Crawford, L. Harry Faber,
and Charles M. Johnson*

Objective

The purpose of this project is to furnish an accurate, reliable background measuring station for the 1978 coordinated pollution study of the Denver area. This background data station will be operated to furnish information on gaseous and particulate pollutants at ambient levels outside of the main pollution cloud. Validated information, on nine-track computer magnetic tape, will be furnished to the coordinating agency for inclusion and correlation with information gathered in this area-wide study.

Prior Work

As the air pollution problem in the Denver area worsens, the civic obligation of major area industries to seek solutions to this problem becomes more obvious. In 1973, the Environmental Protection Agency sponsored a month-long study of the "Denver Brown Cloud." Twenty-seven companies, organizations, and installations participated in the study. Instrumentation and Statistical Systems participated in a limited way in this 1973 study. A large amount of data and information was gathered, but the study failed to produce any definitive characterization of the major constituents of the Brown Cloud. Many participants felt the

study lacked strong central direction to accomplish specific goals. Early this year, a project was proposed by General Motors Research to characterize the Denver Brown Haze problem.

The Motor Vehicle Manufacturers Association (MVMA) was asked to sponsor this project. The MVMA accepted and will fund most of the project, which is entitled the MVMA - Denver Haze Study. Selected organizations were asked to participate on the basis of their expertise or their location and available equipment. Participating organizations include the National Oceanic and Atmospheric Administration (NOAA), the State Air Pollution Control Division, Environmental Research and Technology Inc., General Motors Research, and Rockwell International - Rocky Flats Plant.

Achievements and Discussion

We have been asked to furnish the site and equipment for a background measuring station during the months of November and December 1978. Our Mobile Ambient Air Monitor - MAAM (see RFP-2417A, August 29, 1975, p. 29) van will measure and record ambient levels of gaseous and particulate pollutants in an area outside the major Denver pollution cloud. The van has been partially reconditioned as of this writing.

Future Work

The MAAM will be fully reconditioned and brought up to the standards required for the study. A nephelometer furnished by the University of Washington will be installed together with additional air filter equipment furnished by MVMA. The measuring station will be maintained during the time required, and the data produced will be included in the overall study.

PLUTONIUM AND AMERICIUM SOIL ANALYSIS OF LANDS ADJACENT TO THE ROCKY FLATS PLANT

Merril W. Hume

Objective

The objective of this work is to develop soil-sampling plans and to analyze soil-sample data

concerning lands adjacent to Rocky Flats. The results are intended to help determine the levels of transuranic deposition attributable to Rocky Flats Plant operations. These data are to be used in litigation proceedings in which the plaintiffs charge that activities at Rocky Flats have contaminated their land. An addendum to this program is the characterization of all lands adjacent to Rocky Flats Plant. As an aside, various soil-sampling techniques, analytical laboratories, and related procedures will be assessed.

Prior Work

Soil samples were collected from the 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 to be analyzed by Eberline Instrument Company of Santa Fe, New Mexico for plutonium, americium, and a limited number of other elements. The sampling plan, analytical procedures, and some analytical results were presented at pretrial hearings before the Colorado U.S. District Court. Analytical results for both plutonium and americium by three sampling techniques taken at 81 sample sites were almost as anticipated. The goals of the sampling design were met. The data in no way indicate that the suggested EPA guidelines for transuranic deposition have been exceeded.

At the suggestion of the court, an extensive retesting program was developed. Major modifications over the original programs consist of additional sampling sites, interlaboratory analyses, and increased quality control for the analytical laboratories. The latter required the use of field consensus standards and extensive blinding of samples. Rocky Flats personnel relinquished all monitoring of the retesting program.

Achievements and Discussion

Analytical results of the retesting program have been completed. Preliminary analysis indicates excellent agreement with results of the original

testing program and with inter- and intra-laboratory replicate analyses. Results from the soil samples collected by the sampling method developed by Carl Johnson (Jefferson County Health Officer) appear to correlate well with results from the other sampling methods but are lower than anticipated. No results exceed the suggested EPA guidelines.

Sampling has been completed for the adjacent unlitigated lands. Extensive quality controls, as employed in the retesting program, will be used with these samples.

Future Work

A comprehensive statistical analysis will be performed with all of the data. The results will be presented at pretrial hearings and will play a major role in the ensuing trial.

TANK CALIBRATION EXPERIMENTS

Merril W. Hume

Objective

The objective of this work is to (1) perform volumetric tank calibration experiments to evaluate existing Rocky Flats Plant calibration procedures, (2) test the validity of American National Standards Institute (ANSI) Standard N15.19-1975, and (3) assess the detectability of volume perturbations that may exist in the tanks because of Raschig ring voids or sludge buildup.

Prior Work

Volumetric tank calibration is important not only from the viewpoints of nuclear material control accountability and safeguards but also the nuclear safety aspect. The Rocky Flats Plant has nearly 150 Raschig ring-filled tanks containing fissile solutions that are routinely calibrated. The calibration data are processed by computer codes that

also generate the tank calibration certificate from which decisions are made.

Achievements and Discussion

Numerous volumetric tank calibration experiments were conducted using two typical production tanks and routine Rocky Flats Plant calibration procedures. The calibration data were collected for ring-filled, ring-free, and volume-perturbed situations. Review of both Rocky Flats Plant and ANSI calibration procedures resulted in suggestions for improvement. Also the statistical treatment of calibration data, as advocated by both Rocky Flats Plant and ANSI, proved to be inadequate since the required assumptions were not met. Each tank calibration presents unique problems, so the balance between rigorous analysis and practicality must be resolved on an individual basis. Residual analysis is recommended for the detection of volume perturbations.

Future Work

Similar calibration experiments are being conducted with the PDPP (Positive Displacement Piston Prover) by the Chemistry Standards Laboratory. The knowledge gained from these programs should greatly enhance required tank calibrations for the new reprocessing facility at the Rocky Flats Plant. The revised ANSI Standard N15.19 will incorporate improvements resulting from these volumetric calibration experiments.

FACTOR STUDY

Elizabeth L. Baker

Objective

The objective of this study is to review factors used frequently in lieu of actual measurements to transfer accountable material. Errors in a factor can indicate abnormal losses or gains in material where in fact none exist; consequently, these factors are reviewed periodically to determine their validity.

Prior Work

The Statistics Laboratory has been involved regularly in reviewing and/or updating some of the factors used at Rocky Flats Plant.

Achievements and Discussion

At the request of the Nuclear Materials Control and Safeguards Committee, a general review was made of factors used on accountable material at Rocky Flats. This study showed the factors in use were generally up-to-date or were currently under study. The use of factors tends to cause large fluctuations in ID (inventory difference) in areas where large amounts of material are handled. The Statistics Laboratory strongly recommended that factors be eliminated and measurement capability be provided wherever possible.

Future Work

Factors will continue to be reviewed on an as-needed basis.

MEASUREMENT ASSURANCE PROGRAMS

Elizabeth L. Baker and Merrill W. Hume

Objective

The objective of this study is to evaluate the measurement capability at Rocky Flats through use of measurement assurance programs.

Prior Work

The Statistics Laboratory has been involved on several occasions with the review, evaluation, and/or update of various measurement assurance programs that exist at Rocky Flats. Previous studies have revealed that some of the statistics generated in these programs are, at best, inappropriate.

Achievements and Discussion

Major renovations of existing measurement assurance programs are needed to provide meaningful quantitative assessment of Rocky Flats measurement capabilities. To this end, a proposal has been made to perform an exhaustive review of all present programs and to begin a concerted effort to amend and coordinate these programs as required.

In the meantime, assistance has been provided to Nuclear Materials Control in calculating precision estimates when needed. Also, at the request of the Department of Energy's Albuquerque Operations Office, a computer program was written to simplify the monitoring of the helix, can, and drum counters.

Future Work

Interim measures will continue to be used until there is sufficient manpower to revise these programs.

BRIQUETTE OIL FACTOR

Elizabeth L. Baker

Objective

The objective of this study is to re-evaluate the factor used to estimate the amount of machining oil trapped in briquettes made from plutonium machine turnings. Since the briquettes are transferred on a weight basis, any oil not accounted for will be transferred as plutonium.

Prior Work

The Statistics Laboratory, on several occasions, initiated a review of this factor. Available data, however, has been insufficient to provide a precise factor estimate.

Achievements and Discussion

Larger-than-desirable variations in inventory differences (IDs) for areas handling plutonium-turnings briquettes prompted a re-study of the oil factor used to transfer these briquettes. This study was conducted by Industrial Engineering with data analysis by the Statistics Laboratory. Parts were weighed before and after machining, and the difference in weight was compared with the weight of briquettes made from the machine turnings. Results show the current factor to be too small; however, the uncertainty on the new factor is quite large.

Future Work

To update the current briquette oil factor, more work is needed. The R&D Joining Group is currently working on a project that, as a side result, should produce a current oil factor. In the long run, however, plans should be made to eliminate the factor altogether by providing direct briquette measurement capability or by eliminating the transfer of the briquettes.

INVENTORY DIFFERENCE CONTROL CHARTS

Elizabeth L. Baker

Objective

The objective of this study is to ensure, through the use of inventory difference (ID) charts, that Material Balance Areas (MBAs) handling radioactive material do not have losses in material over that which may be accounted for by normal operating procedures.

Prior Work

The current version of the ID control chart program was written in 1974. The Statistics Laboratory has maintained the program and data files since then. This includes adding, deleting, and changing MBAs

in addition to correcting errors that may occur in the data.

Achievements and Discussion

At the request of DOE and Nuclear Materials Control at Rocky Flats, a separate computer program was set up to produce control charts for uranium-233 and other special MBAs that are not in the regular ID program.

Future Work

The Statistics Laboratory will continue to maintain these programs and their respective data files.

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 reliability.

Prior Work

This function has been ongoing for a number of years.

Achievements and Discussions

Limits of error have been placed on shipments and receipts of Special Nuclear Materials and internal working standards. Quality control programs were developed for analytical laboratory and environmental monitoring problems, and a manuscript was reviewed for Nuclear Technology. Other achievements involved a sampling plan that was developed for waste salts, and support that was given for national ANSI and ASTM statistical subcommittees.

Future Work

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