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**CHEMISTRY RESEARCH AND DEVELOPMENT
PROGRESS REPORT FOR
JULY 1977 THROUGH MAY 1978**

Frend John Miner

Component Development — — — — — Jean R. Gaines
Pilot Plant — — — — — Donald L. Ziegler
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**ROCKWELL INTERNATIONAL
ENERGY SYSTEMS GROUP
ROCKY FLATS PLANT
P.O. BOX 464
GOLDEN, COLORADO 80401**

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SUBJECT DESCRIPTORS

Air Pollution	Microprocessors
Aluminum	Minicomputers
Calibration Standards	Nondestructive Analysis
Calorimeters	Physical Security
Cleaning	Plutonium Residues
Control Systems	Radioactive Wastes
Corrosion	Safeguards Instrumentation
Corrosion Inhibitors	Shipping Containers
Decontamination	Soil
Environment	Stainless Steel
Evaporation	Statistics
Fiber Reinforced Plastics	Tributyl Phosphate
Fluidized Bed	Uranium
Glove-Box Gloves	Uranium Nitriding
Incineration	Vitrification
Incinerator	Waste Immobilization
Land Pollution	Waste Management
Leak Testing	Waste Processing
Materials Compatibility	Water Pollution
Measuring Instruments	Welded Joints

P R E F A C E

Previous Chemistry R&D Progress Reports have included work from all of the groups within Chemistry R&D. Beginning in January 1978, this format was changed. Progress Reports are now being published in three sections on the following time schedule:

<u>Chemistry R&D</u>	<u>Date</u>
Product Integrity and Surveillance (plus classified work from Process Chemistry and Engineering)	January and July
Chemical Research and Process Chemistry and Engineering	May and November
Component Development, Pilot Plant, and Instrumentation and Statistical Systems	June and December

The Product Integrity and Surveillance report will be the only one classified.

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

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

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Incineration of tributyl phosphate-solvent mixtures in a mixed bed of sodium carbonate-catalyst is effective in preventing the release of corrosive phosphorous pentoxide. For solid waste, a variable compression screw is now being fabricated to handle all types of waste. (Department of Energy – Division of Waste Products)

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

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

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

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A radiometric scanner has been provided for estimating the plutonium content of small packages. (PPS)

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

CHEMISTRY RESEARCH AND DEVELOPMENT PROGRESS REPORT FOR JULY 1977 THROUGH MAY 1978

Frend John Miner

COMPONENT DEVELOPMENT

Jean R. Gaines

COMPATIBILITY OF NON-NUCLEAR WEAPONS METALS WITH PROCESS MATERIALS

John L. Briggs and Marston R. Dringman

Objective

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

Prior Work

The results of other compatibility studies have not been reported in the open literature.

Achievements and Discussion

The compatibility of all non-nuclear weapons metals with processing materials currently in use is determined by corrosion and residue removal tests. These tests are part of a continuing Quality Control Program.

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

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 (in the case of non-fluid materials) placed in contact with the various corrosive materials and are 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 being visually examined, the coupons are reweighed for corrosion rate determinations. Following the corrosion tests, coupons that have been exposed to the corrosive 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. Rinse in distilled-demineralized water for 5 minutes.
3. Rinse in distilled-demineralized water for 5 minutes.
4. Air dry.

The coupons are then analyzed for residue by infrared, atomic absorption, and by 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 Trim Tap Heavy Cutting Fluid. This material could not be cleaned by the present aqueous detergent. In addition, a series of tests were performed with U-2.3 wt % Nb and Cu-1.87 wt % Be and 13 currently used processing materials.

*National Association of Corrosion Engineers

TABLE 1. Compatibility Test Materials and Metals

New Process Materials	Weapons Metals Tested
Alconox Detergent	Be
Chlorothene® V. G.	*
CIM-TAP® Milacron	*
CX34Y Lathe Coolant Milacron	303 and 304 stainless steel, 1100 A1, 7075 A1
Hefty Mark Felt Pen	5083 A1, U-2.3 wt % Nb, U-0.75 wt % Ti
Micro-Surf® Cleaner	* (less 303 stainless steel)
Polygel	*
Spindura® 10 Spindle Oil	*
Stencil Paper, 9003	* (less 303 stainless steel)
Stencil Paper, Type R	* (less 303 stainless steel)
Tergitol® 15-S-9 Detergent	1100 A1, 316 stainless steel, U-2.3 wt % Nb
Texaco Regal 32 R & O	*
Trim Tap® Heavy Cutting Fluid	*
Zyglo ZC7 Cleaner	*
Zyglo ZE3 Emulsifier	*
Zyglo ZL22A Penetrant	*
Zyglo ZL30A Penetrant	*
Zyglo ZP4A Developer	*
Zyglo ZP5 Developer	*
Zyglo ZP9 Developer	*

*Weapons metals include 1100, 5083, 6061, and 7075 aluminums; beryllium; monel; JBK-75, 21-6-9, 303, 304, 316, and 321 stainless steels; tuballoy; U-2.3 wt % Nb; and U-0.75 wt % Ti.

Future Work

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

CORROSION RESISTANCE OF 304L STAINLESS STEEL IN NITRIC- HYDROCHLORIC ACID ENVIRONMENTS

John L. Briggs and Marston R. Dringman

Objective

Corrosion tests have been performed to determine the feasibility of increasing the concentration levels

TABLE 2. Corrosion Resistance of 304L Stainless Steel in Nitric Acid-Hydrochloric Acid Mixtures

HCl Addition To 7N HNO ₃ (wt %)	Test Duration (days)	Corrosion Rate (mpy) ^a	Visual Correction
None	10	None	None
0.1	10	None	Light etch
0.3	10	None	Light etch
0.7	10	None	Light etch, shallow pitting
1.0	10	0.2	Light etch, shallow pitting
2.0	10	0.7	Light etch, shallow pitting
4.0	10	17.0	Heavy etch, deep pitting
6.0	10	415	Heavy etch, deep pitting
8.0	1	Complete dissolution after 24 hours	-----

$$\text{a. mpy} = \frac{534 \times \text{weight loss}}{\text{density} \times \text{area} \times \text{time}}$$

of hydrochloric acid in ion column tanks within the Plutonium Recovery Facility.

Prior Work

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

Achievements and Discussion

Sample coupons of 304L stainless steel (2 sq. in. surface area) were obtained for immersion tests performed to NACE* Standard Method TM-01-69 (as described in the preceeding report on compatibility testing of process materials). The samples were ultrasonic detergent cleaned, passivated in 50 vol % HNO₃ and weighed to 0.1 mg for gravimetric corrosion rate measurements. Duplicate sample coupons were immersed for 10 days at 50 ± 1 °C in various nitric acid-hydrochloric acid mixtures. Results of the corrosion test are shown in Table 2.

*National Association of Corrosion Engineers

No excessive general corrosion was measured for the 304L stainless steel coupons until the concentration level of HCl was increased to 6 wt %; however, corrosion pits were detected on coupons subjected to as low as 0.7 wt % HCl. Since pitting is not time related, chloride pitting could occur at the lower concentration levels with extended test duration.

In addition to localized chloride pitting, failure of austenitic stainless steel by chloride-induced stress corrosion cracking (SCC) has been thoroughly established. Because of pitting observed during immersion corrosion tests and because of possible chloride-induced stress corrosion cracking, it is not feasible to increase HCl in the ion column tanks to greater than the present level. The current HCl level varies with time but usually is below detection limits.

Future Work

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

EXFOLIATION CORROSION AND NITRIC ACID EXPOSURE TESTING OF 5083 ALUMINUM ALLOY

John L. Briggs

Objective

The objective of these studies was to determine whether two corrosion test methods could be correlated to the Alternate Immersion Stress Corrosion Cracking Test for 5083 aluminum. The two methods, both requiring only 24 to 48 hours, were the Ammonium-Salt Solution Exfoliation Test (ASSET) and the Nitric Acid Weight Loss Test (NAWLT).

Prior Work

Previous studies have shown that the Alternate Immersion Test, ASTM G44-75, is suitable for determining the susceptibility of 5083 Al to sensitization.

Achievements and Discussion

Six sample specimens of 5083 Al were obtained for the ASSET test by sectioning Hatch parts with varying heat treatments as shown in Table 3.

The samples were precleaned to Aluminum Association specifications and then immersed for 24 hours at 66 ± 1 °C in the corrodent solution, which consisted of

1.0M ammonium chloride
0.25M ammonium nitrate
0.01M ammonium tartrate
3 g/l hydrogen peroxide (10 ml of 30% stock solution per liter)

The sample specimens were immersed in a sufficient quantity of corrodent solution to provide a minimum volume-to-surface area ratio of 50 ml/in².

After 24 hours immersion, the sample coupons were removed from the corrodent solution, rinsed in distilled water, and soaked in concentrated nitric acid at 23 ± 2 °C until clean (45-60 seconds).

TABLE 3. Asset Test Sample Description

Part Number	Specimen Surface Area (in ²)	Fabrication Heat Treatment	
		Solutionizing Temperature (°C)	Forming Temperature (°C)
E8	11.53	3 hr at 400-WQ ^a	1 hr at 170
E10	10.57	2 hr at 260-WQ	1 hr at 170
E12	14.25	2 hr at 260-WQ	10 min at 210
E14	12.15	3 hr at 400-WQ	10 min at 210
E16	11.44	2 hr at 260-WQ	10 min at 228
E18	13.18	3 hr at 400-WQ	10 min at 228

a. Water quench

TABLE 4. NAWLT Test Results

Part Number ^a	Specimen Surface Area (in ²)	Specimen Weight Loss (mg)	Weight Loss Per Unit Area (mg/in ²)
E8	12.86	184.7	14.4
E10	12.61	219.7	17.4
E12	13.24	167.2	12.6
E14	12.95	149.3	11.5
E16	12.74	167.7	13.2
E18	13.20	160.0	12.1

a. Heat treatments previously described in Table 3.

The specimens were then rinsed in distilled water, air dried, and visually inspected for exfoliation.

Pit blistering was observed on Samples E10 and E16. Minimal pitting was exhibited by all of the samples. No evidence of exfoliation was detected for any of the samples tested. Sufficient data to distinguish between heat treatments were not collected from the ASSET test.

Six sample specimens (duplicates of the ASSET test specimens) were next examined by the NAWLT test. The precleaned specimens were weighed to the nearest 0.1 mg and immersed in 70 wt % nitric acid (125 ml/in² volume-to-surface area ratio) at 30 ± 1 °C for 24 hours. After 24 hours storage, the specimens were removed, rinsed in distilled water, air dried and reweighed to the nearest 0.1 mg. Results of the test are summarized in Table 4.

Results show some indication that E8 and E10 (the sensitized samples) had greater weight loss; however, none of the samples exhibited excessive weight loss as defined by the Aluminum Association criteria, i.e. >100 mg/in². Neither the surface appearance nor weight loss values provided sufficient difference to distinguish the degrees of sensitization.

It was concluded that neither the ASSET or NAWLT test can be substituted for the Alternate Immersion Stress Corrosion Cracking Test to determine the susceptibility of 5083 Al parts to stress corrosion cracking.

Future Work

No further work will be performed to correlate the ASSET or NAWLT tests with stress corrosion cracking of 5083 aluminum. Further ASSET tests will be evaluated for other applications.

INTERGRANULAR CORROSION TESTING OF WELDED 21-6-9 AND 304L STAINLESS STEEL

John L. Briggs and Marston R. Dringman

Objective

The purpose of the corrosion tests was to determine the susceptibility of 304L and 21-6-9 stainless steel weld specimens to intergranular corrosion.

Prior Work

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

Achievements and Discussion

Austenitic stainless steels that have been heat treated in a temperature range of 350 to 800 °C become "sensitized." This means they become susceptible to intergranular corrosion. The susceptibility of such alloys to intergranular corrosion can be determined by either of two boiling acid tests. The first involves boiling nitric acid and is known as the Huey Test. The second utilizes boiling sulfuric acid-copper sulfate and is known as the modified Strauss Test. Welded samples of 304L and 21-6-9 stainless steel were examined by both test methods to determine the susceptibility of the alloys to intergranular corrosion following various joining processes.

The Huey Test consists of exposing specimens in fresh boiling nitric acid for five successive 48-hour

TABLE 5. Huey Test Results

Alloy and Type of Weld	Penetration (in./mo)*			Total Penetration (in./mo)
	1st Period	2nd Period	3rd Period	
21-6-9 and 304L Electron beam weld	0.0006	0.0013	0.0014	0.0009
21-6-9 and 308 Filler weld	0.00054	0.00043	0.00034	0.00047
21-6-9 and 308 Filler weld	0.00054	0.00046	0.0005	0.0005

*Reduced exposure time (from five to three 48-hour periods) is permitted by ASTM specification.

periods under a reflux condenser. If no weight losses are observed after three successive periods, the test can be terminated. The specimens are cleaned and weighed after each period for gravimetric corrosion rate determinations.

Two types of stainless steel weld samples were examined by the Huey Test method. One type consisted of two small blocks of 304L stainless steel, which were electron beam welded to a 21-6-9 stainless steel block. The other samples tested were 21-6-9 stainless steel welded by 308 stainless steel filler wire with inert gas. Results of the Huey Test are shown in Table 5. The constancy of penetration (Table 5) shows no intergranular corrosion for the welded samples tested.

The modified Strauss Test is less severe than the Huey Test. Intergranular attack takes place with minimal grain dislodgment resulting in minimal weight loss. Determination of intergranular corrosion susceptibility of a test specimen is therefore assessed by bending the specimen through 180 degrees and inspecting the bend surface for cracks. Cracks were used to define intergranular corrosion for this test.

Two sample specimens of 21-6-9 stainless steel were joined with 308 stainless steel filler wire by a gas tungsten arc weld method. The duplicate test specimens were then sectioned and pretreated for the modified Strauss Test as previously described.

No cracks were detected on either 21-6-9 alloy weld sample although some ductile flow (orange-peel effect) was apparent at the bend surfaces. The absence of cracks meant that no intergranular corrosion was detected for either sample specimen as defined by ASTM 262-Practice E for the modified Strauss Test.

It was concluded that both intergranular corrosion tests showed no indication of intergranular attack for any of the weld specimens tested.

Future Work

No future work on this objective is anticipated.

INTERGRANULAR CORROSION TESTING OF 304L STAINLESS STEEL BALL VALVE WELDS

John L. Briggs and Marston R. Dringman

Objective

The purpose of these corrosion tests was to determine the susceptibility to intergranular corrosion of welded 304L pipe to 304L valve bodies. These assemblies will be used for nitric acid service in the new Plutonium Recovery Facility.

Prior Work

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

Achievements and Discussion

A shipment of approximately 1000 ball valves welded to pipes were received at Rocky Flats for nitric acid service in the new Plutonium Recovery Facility (Building 371). Visual examination of these assemblies revealed a substantial weld underbead on the pipe interior. The underbeads were

TABLE 6. Results of Huey Test*

Sample Specimen	Mils Penetration Per Year					Average Penetration (mpy)
	1st 48-hr. Period	2nd 48-hr. Period	3rd 48-hr. Period	4th 48-hr. Period	5th 48-hr. Period	
1st Weld Sample	19.95	12.28	12.27	12.74	12.39	13.93
Control (no weld)	11.39	8.35	9.22	8.27	8.59	9.16
2nd Weld Sample	33.35	20.47	20.97	(**)	(**)	24.93

* An intergranular corrosion test involving specimens being exposed to boiling nitric acid for five successive 48-hour periods under a reflux condenser.

** Based on previous results and on criteria for Huey Test, only three test periods were needed.

severely oxidized and projected into the interior surface up to 0.125 inch. Since the underbead was obviously the result of excessive weld heat, it was necessary to determine whether the excessive weld heat had reduced material corrosion resistance.

Samples of the welded pipe were tested for intergranular corrosion using a boiling nitric acid test (ASTM designation A226-75) known as the Huey Test. The Huey Test consists of exposing specimens in boiling nitric acid for five successive 48-hour periods under a reflux condenser. The specimens are cleaned and weighed after each period for gravimetric corrosion rate determination.

Two specimens were subjected to the Huey Test. For one specimen, a region of the socket weld that exhibited weld underbead was removed from the pipe-valve assembly. This weld specimen weighed 63.5116 grams with a surface area of 6.0 square inches. An unwelded area of the pipe (52.6320 grams, 5.6 square inches) was also removed and tested for control purposes.

The Huey Test was repeated on another weld specimen (46.3415 grams, 4.8 square inches) to confirm results of the first test. Confirmation of the second test was obtained after three 48-hour test periods. The reduced exposure time (from five to three 48-hour periods) is permitted by ASTM specification. The Huey Test results are summarized in Table 6.

Huey Test criteria state that intergranular corrosion is not occurring if the corrosion rate does not increase for successive exposure periods. Corrosion rates for all of the 304L samples were highest after the initial 48-hour boiling period; however, the rates stabilized to normal corrosion rates for 304L stainless steel during subsequent exposure periods. Metallographic analyses of the "socket" welds also confirmed the absence of intergranular corrosion. Both weld specimens exhibited slightly higher corrosion rates than the unwelded (control) specimen; however, none of the specimens tested exhibited excessive corrosion rates.

It was concluded that no intergranular corrosion will occur for the welded 304L ball valve assemblies intended for nitric acid service in Building 371.

Future Work

No future work on this objective is anticipated.

CORROSION TESTS OF GLOVE-BOX AND CANYON WATER WINDOWS FOR THE NEW PLUTONIUM RECOVERY FACILITY

John L. Briggs and Marston R. Dringman

Objective

Corrosion tests were performed to determine whether corrosion or other adverse effects would result if Rocky Flats tap water or distilled water was used for filling water windows in the new Plutonium Recovery Facility (Building 371). Water treatments for corrosion inhibition and algae control were also examined in this study.

Prior Work

No prior work in this objective has been performed at Rocky Flats.

Achievements and Discussion

Water windows fabricated of double glass panes with 304L stainless steel frames have been designed for glove-box and canyon window use in Building 371. During service, space between the double glass windows will be filled with water for neutron radiation protection. Corrosion tests have been performed to determine whether corrosion would result from the use of distilled or Rocky Flats tap water for filling the glove-box windows.

Sample coupons of 304L stainless steel were obtained for the shield-water corrosion tests. The

corrosion tests were performed by total immersion of the sample coupons in various water solutions (NACE Standard Method TM-01-69) at 23 ± 2 and 50 ± 2 °C. Sample coupons with a surface area of 2 in² were used for the ambient temperature tests; larger 304L stainless steel coupons (8 in²) were employed for the accelerated (50 ± 2 °C) tests.

Prior to immersion, all sample coupons were abraded with 120 grit silicon carbide paper, rinsed in distilled water, air dried, and weighed to the nearest 0.1 mg. Duplicate sample coupons were then immersed in various test-water solutions, as shown in Table 7.

Cloudiness was observed for the Rocky Flats tap water treated with Roccal algicide within the first 24 hours of test. The white cloudiness remained throughout the 7-week test, which indicated the Roccal had reacted with dissolved minerals in the Rocky Flats tap water.

No apparent cloudiness was observed for untreated Rocky Flats tap water; however, visual clarity was slightly impaired after 7 weeks of storage. No weight changes were measured and no visual corrosion was detected for any of the 304L sample coupons immersed in the Rocky Flats tap water. Impaired visual clarity would reject the use of the tap water, however, for filling the water windows.

No weight changes were measured and no visual corrosion was detected for any of the 304L sample coupons exposed to the inhibited and uninhibited distilled water solutions. This indicates corrosion inhibition is probably not necessary. The visual clarity of all distilled water solutions was unchanged throughout the test and remained clear at termination of the tests.

In addition to the above tests, a glove-box window (filled with treated distilled water) has been placed in extended storage. After four months, the visual clarity is still unimpaired.

TABLE 7. Corrosion Test Results

Water Type	Water Treatment	Test Temp. (°C)	Sample Size (in ²)	Sample Weight Change	Test Storage Time (week)	Water, Visual Appearance
Rocky Flats tap water	None	23 ±2	2	None	7	Decreased clarity
Rocky Flats tap water	None	23 ±2	2	None	7	Decreased clarity
Rocky Flats tap water	500 ppm Roccal ^a	23 ±2	2	None	7	Cloudy
Rocky Flats tap water	500 ppm Roccal	23 ±2	2	None	7	Cloudy
Distilled water	None	23 ±2	2	None	7	Clear, no change
Distilled water	None	23 ±2	2	None	7	Clear, no change
Distilled water	500 ppm Roccal, 500 ppm NaNO ₂ ^b	23 ±2	2	None	7	Clear, no change
Distilled water	500 ppm Roccal, 500 ppm NaNO ₂	23 ±2	2	None	7	Clear, no change
Distilled water	500 ppm NaNO ₂	50 ±2	8	None	5	Clear, no change
Distilled water	500 ppm NaNO ₂	50 ±2	8	None	5	Clear, no change
Distilled water	500 ppm NaNO ₂ , 500 ppm Roccal	50 ±2	8	None	5	Clear, no change
Distilled water	500 ppm NaNO ₂ , 500 ppm Roccal	50 ±2	8	None	5	Clear, no change

a. Roccal is a proprietary algicide used to prevent possible algae growth.

b. NaNO₂ added for corrosion (if any) inhibition.

A recommendation was made to use distilled water treated with algicides and corrosion inhibitors.

Future Work

No future work on this objective is anticipated.

OPTION FOR PROCESSING AND PACKAGING SLUDGE FROM TRANSURANIUM-CONTAMINATED SOLAR EVAPORATION POND

Kazuji Terada

Objective

The objective of this work was to establish a method for fixing and packaging sludge from the solar evaporation pond.

Prior Work

Several methods of processing sludge for packaging and shipping were examined. They were (1) fixing with cement, (2) fixing by a commercial process, and (3) dehydration by the following processes: solar evaporation, filtration, steam heat, and filter press. The use of filter press to dewater the sludge was recommended for further study.

Achievements and Discussion

Two pumps were obtained and used to demonstrate that the sludge in the solar evaporation pond can be pumped. One was a centrifugal pump with a capability of grinding stray solids; the other was a diaphragm pump. Pumping rates far exceeded the minimum rate necessary for processing and packaging the sludge within the scheduled period.

Laboratory-scale tests on filter press dewatering capabilities showed a reduction of water content from 75 to 55%. Although this is a significant reduction, it was decided that fixation by cement

was the better option. This decision was made on the basis of high capital expenditure and long process time for the filter press option.

The cost of cementing the sludge will be minimized by removing as much water as possible. This will be partially accomplished by transferring the sludge into 8,500-gallon tanks and allowing the sludge to settle for 24 hours. The clear supernatant liquid will be decanted and pumped to the new Waste Processing Facility (Building 374) for evaporation. The thin sludge below the supernatant liquid will be processed in a vacuum filter that is available. The thick sludge can be removed from the bottom of the tanks into a continuous cement mixer.

There are two 10,000-gallon tanks available for conversion to serve as settling tanks. After modifications to change the flat bottoms to conical ones, the tank volumes will be reduced to approximately 8,500 gallons. The two tanks will be alternated daily to allow a settling time of 24 hours. The capacity of each tank is more than the minimum volume of sludge that must be processed daily to complete the sludge removal objective within the scheduled period. Design criteria for removal, solidification, and packaging of the pond sludge has been submitted as part of an Engineering Job Order.

Future Work

An R&D report of selection and development of the sludge solidification plan will be prepared and issued. Input will be made to engineering and maintenance departments for designing the processing facility and installing the equipment.

QUALIFICATION OF A NEW RIGID LINER FOR WASTE DRUMS

Marston R. Dringman and Kazuji Terada

Objective

Approval must be granted by DOE for the use of rigid liners in 55-gallon drums shipped to the Idaho

burial site. The objective of this work is to perform and report on tests required to gain approval for the Plastidrum rigid liner.

Prior Work

Numerous attempts have been made to obtain alternate suppliers for rigid drum liners. For various reasons, potential vendors either lost interest or were disqualified. This work was a continuation of earlier efforts to qualify a new vendor and liner.

Achievements and Discussion

Qualification tests were conducted on a new rigid liner produced by the Plastidrum Corporation of Lockport, Illinois. This liner has a Rocky Flats designed closure system and is produced by blow molding instead of the rotational molding process used for the original liner. There is a substantial savings in the cost of the new liner (\$20) over the cost of the old (\$30).

The performance qualification tests were the same as those specified for quality acceptance. These tests involved a drop test using a loaded drum. Specified materials tests on the liner material also were included. The loaded packages were prepared by placing a liner in a drum, then filling it with gravel and an ethylene glycol-water solution to a gross weight of 700 pounds. Sealing and closure of the lid followed normal procedures for waste packages. The packages were then cooled overnight to 0 °F and subjected to vibrations and a 4-foot drop test. Material tests included a drop test of an empty liner, cooled to 0 °F, from a height of 6 feet, and an environmental stress crack resistance (ESCR) test. The ESCR tests were conducted according to ASTM Standard D568; a rating of 250 hours was required.

The Plastidrum liner passed all of these tests, and a report on the results was issued. This report was submitted to ALO by Waste Processing as the basis of a request for the use of this liner as the package for shipping transuranic wastes to Idaho for storage. This request was granted and the Plastidrum is currently used as the rigid liner in 55-gallon waste drums containing retrievable wastes.

Future Work

The manufacturer of the original rigid liner used at Rocky Flats filed for bankruptcy shortly before the Plastidrum liner became an approved liner. Therefore, the manufacturer of Plastidrum liners is now the only Rocky Flats supplier of rigid liners. Another source of rigid liners will be sought.

LEAK CHECK OF WASTE BOXES COATED WITH FIBERGLASS REINFORCED PLASTIC

Marston R. Dringman and Kazuji Terada

Objective

The objective of this work is to develop a method to leak-check fiberglass reinforced plastic (FRP) waste boxes. This test will be used as one of the receiving acceptance tests.

Prior Work

No prior work on this objective has occurred at Rocky Flats.

Achievements and Discussion

Several methods of leak-checking boxes were evaluated. They were (1) pressurization of the box and detection of escaping vapors, (2) immersion of the box in water, and (3) detection of holes in the FRP based on spark formation between a high voltage source and a conductive surface. The first method involved pressurization of the box with either helium, air and DOP (dioctylphthalate), or air. It was decided that the most promising test involved detection by spraying soap solution over an air pressurized box.

A leak test method was developed and demonstrated to Waste Processing, Quality Control, and Non-Destructive Testing representatives. They found it to be a suitable test. The test involves pressurizing the box to 0.25 psi and applying soap solution to

pinpoint leak sites by the appearance of bubbles. The equipment necessary to perform this test includes a temporary lid to provide an adequate seal for the pressurized box, belts to tighten the box against the lid so as to maintain the seal, an air pressure supply, and a soap sprayer. Accessory equipment includes devices necessary to move and position the 500-pound box in place on the test rig and a drip pan to collect the excess soapy solution.

A commercial grade vacuum cleaner is recommended as the source of air to pressurize the box. It has the advantage of being relatively inexpensive, readily available, and safe. It cannot generate pressures that could make the process hazardous.

The cost of a complete test unit is approximately \$5,500. This cost does not include a trailer if it should be decided that the test equipment must be portable.

It should be possible to test five boxes per hour as a two-man operation. An operational procedure was submitted to NDT.

Future Work

This project is considered complete.

COMPUTER STORED HISTORY FILE FOR GLOVE-BOX GLOVE CHANGES

Perry A. Patton

Objective

The objective of this work is to determine if a method can be developed to predict glove life through an accumulated history of glove failures and changes.

Prior Work

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

Achievements and Discussion

A computer storage file to record the history of glove-box glove failures and change information has been developed. To accomplish this, a number was assigned to each glove box and glove port. A history file was established containing all ports by line, room, and building. Reported glove changes list reasons for the change and how the need for change was determined.

Gloves are assigned an expiration time of 6, 12, 18, 24, or 42 months based on the work environment of the glove. Reports of change are entered on a continual basis. A computerized report is printed monthly listing gloves scheduled for change, according to expiration times, during the current month. A second monthly computerized report lists changes made in the previous month. A file data computer retrieval language, Mark IV, is used to retrieve historical information and manipulate the data to locate potential problem ports.

An additional computer program has been developed to analyze for useful glove life through statistical methods. This program is capable of analyzing glove expiration codes to determine if expiration times should be lengthened or shortened.

Future Work

A system will be developed to monitor error rates in the reporting system. Errors will increase glove costs by necessitating more frequent glove changes than are necessary. Errors will also affect the number of potential incidents by allowing gloves to remain in use beyond the recommended period.

CORROSION MONITORING - STEAM CONDENSATE LINES

John L. Briggs and Marston R. Dringman

Objective

The objective of the monitoring program for steam condensate corrosion is to identify the cause of

costly condensate line failures and to prevent such failures with suitable inhibitor treatment.

Prior Work

Previous monitoring studies of condensate corrosion have been described in earlier semiannual reports.

Achievements and Discussion

Sample stations, which provide sample coupon placement into the condensate lines and also allow for water sampling, were placed on the condensate return lines in Buildings 444, 439, 443, 334, 771, 881, 883, 865, 707, and 778 at Rocky Flats.

Sample stations were also located at areas of composite condensate returns from Buildings 331, 333, 334, 551, 442, 771, 774, 776, 777, and 779.

The mild steel sample coupons were rectangular ($2.5 \times 0.5 \times 0.0625$ inch) and weighed about 11 grams. The sample coupons were removed after a 12-month test period and new samples were placed in the stations for continued corrosion rate determination. After the samples were removed, loose corrosion products were removed and the specimens were rinsed first in distilled water and then in acetone before being air dried. The coupons were then reweighed to the nearest 0.1 mg for corrosion rate determination. Condensate water samples were taken at the termination of the test period, and chemical analysis were performed for pH, cyclohexylamine inhibitor level, and total dissolved solids. No significant levels of dissolved solids were detected in the condensate water samples. These samples showed generally a lower average pH (7.9) than those for previous tests, which averaged a pH of 9.3. A 9.0 pH level is necessary to neutralize any carbonic acid that may be formed.

No excessive corrosion rates were measured for any of the sample coupons. Severe pitting (indicative of dissolved oxygen attack) was visually detected for sample coupons from Stations 12 and 18, Buildings 778 and 707. These samples also exhibited higher weight losses than the other sample coupons but the corrosion rate was still not considered excessive.

Samples from Stations 12 and 18 showed pitting attack in the preceding test series. Termination of this test series coincided with a condensate line failure at Station 12 where about 15 feet of line was replaced because of pitting failure. An examination of the pipe showed pitting similar to the condition of sample coupons in that area.

Sources of dissolved oxygen in the condensate water are probably the large condensate holding tanks in proximity to Stations 12 and 18. Both holding tanks are vented to the atmosphere, which allows aeration of the condensate water and subsequent oxidation pitting of the condensate lines downstream from the holding tanks.

The possibility of auxiliary inhibitor treatment for the two problem areas (Stations 12 and 18) has been studied and when implemented, should reduce corrosion at those areas. Filming amines (e.g. octadecylamine) and film-forming inhibitors have been developed to supplement the use of neutralizing inhibitors such as cyclohexylamine. The filming amines will penetrate adherent corrosion products and form a protective film beneath, which will provide corrosion protection on already corroded systems.

Future Work

Corrosion monitoring of the steam condensate system will be performed continuously for this program.

PILOT PLANT DEVELOPMENT

Donald L. Ziegler

FAST FLUIDIZED BED INCINERATION PROCESS

*Louis J. Meile, Andrew L. Johnston, and
Seng F. Chung*

Objective

This work is being conducted to investigate the fast fluidization concept as a method for improved

incineration of radioactive waste materials. In addition, this concept is being evaluated to determine whether or not the technique offers significant advantages over the slow fluidized bed incineration process now being developed at the Rocky Flats Plant.

Prior Work

A literature search was made to provide information on fast fluidization techniques and their possible application in the incineration of radioactive wastes. A laboratory scale test system was designed, a process heat and material balance was calculated, and most of the test equipment was purchased.

Achievements and Discussion

During this reporting period, a laboratory scale unit was installed, operational testing was started, and several equipment modifications were made. The 17-foot-long reactor vessel, the primary and secondary cyclones, and material transfer lines between the cyclones and the reactor were all fabricated from quartz components. The quartz equipment provides for visual observation of the process in addition to excellent thermal shock resistance and easy component modifications. Quartz can also withstand operating temperatures up to 1000 °C, which would be necessary to burn scrap graphite.

Early testing was directed toward achieving a state of fast fluidization with a single bed media of 50 to 80 mesh chromic oxide-alumina catalyst. Preliminary experiments were conducted at room temperature, without combustion, in an effort to duplicate conditions and results reported by other researchers. Fast fluidization has been investigated elsewhere at superficial gas velocities of 8 to 18 feet per second with suspended solids loadings ranging up to 20 percent of the total bed volume. These gas velocities transport solid particles to the top of the reactor where a cyclone separator is utilized to drop the fines back into the lower section of the bed. The slip velocities between gas and solids cause vigorous solids backmixing and exceptional solids-gas contact.

In testing the quartz unit thus far, suspended solids loadings of up to 9.6 percent have been achieved with fluidization gas velocities ranging up to approximately 6 feet per second. Under these conditions, the solids slip streams and backmixing patterns that are characteristic of fast fluidization were observed. A delicate balance between gas velocity, solids particle size, and suspended solids loadings appears to be necessary. This balance is needed to avoid material being transferred into the sintered metal particulate filters, and thus being lost from the system.

Several combustion tests were attempted after initial fast fluidization experiments. Prolonged, controlled burning was achieved in two of these tests. In each run, a lower, slow fluidized bed of sodium carbonate granules was utilized along with an upper, fast fluidized bed of chromic oxide-alumina catalyst. The system was preheated by electrical air heaters to between 200 and 300 °C; alcohol was then pumped into the beds to provide fuel for combustion for raising the system to an operating temperature of 450 to 550 °C. A finely shredded waste mixture consisting of equal parts of polyethylene, polyvinyl chloride, and paper was then screw-conveyed into the lower sodium carbonate bed. Combustion oxygen was limited in the lower bed by the addition of nitrogen gas to the fluidization air flow. Waste was decomposed in the lower bed where sodium carbonate was available for the neutralization of chlorides. Hydrocarbons produced during decomposition passed into the upper fast fluidized catalyst bed where additional air was added for combustion. The fast fluidized bed of oxidation catalyst apparently promoted complete combustion of the waste; no unburned waste or tars were present in the system after any run.

A variety of problems was encountered during combustion runs. Maintaining a level operating temperature of ~500 °C proved to be difficult in early runs. In two runs, however, control was achieved by regulating waste feeding rates and air-nitrogen gas ratios. Attrition of fine sodium carbonate particles from the lower bed into the upper bed of catalyst was also a problem. The apparent abrasion of sodium carbonate granules was attributed to excessively high gas velocities

within the lower bed. The reduction of gas velocities in subsequent runs appeared to curtail the amount of fines being generated. In addition, quartz breakage at certain points in the system also presented problems. Most breakage occurred where joints were clamped together or at points where quartz was attached to metal components. Improvements in component design, clamping techniques, and sealing compounds are being tested to overcome this problem.

Future Work

Additional work will be conducted to achieve greater suspended solids loadings with higher fluidizing gas velocities. Efforts will be directed towards eventually determining the optimum gas velocity, solids particle size, and suspended solids loading for the present laboratory system. Waste combustion experiments will be continued to determine burning rates and combustion efficiency. Off-gas will be analyzed to aid in the determination of combustion efficiency, and commercial catalysts will be purchased and tested both for fluidization properties and for burning efficiency.

DEVELOPMENT OF URANIUM CHIP BURNING PROCESS

*Donald J. Vickery, James R. Sheets,
Louis J. Meile, and Seng F. Chung*

Objective

The primary objective of this project is the development and pilot scale demonstration of a uranium chip incineration system. A secondary objective is to provide data to support the Engineering and Construction Department in designing a new off-gas system for the present chip burner.

Prior Work

This is a new program.

Achievements and Discussion

Uranium chip burning is a daily production operation being done at Rocky Flats. The chip burning incinerator is a multi-level, raked-hearth furnace with little process instrumentation or control. A process analysis revealed that dust and smoke released during feeding operations make it necessary for all personnel to wear respiratory protection during operation. Poor dust collection efficiency, occasional fire hazard while loading, and room contamination were also noted as problem areas. Development of a new chip burning process was initiated to resolve these problems.

Design

Formulation of a process design material balance has been completed using information and data collected to date. This conceptual design consists of a starved oxygen reactor that utilizes recycled off-gas, and air-locked batch feeder, a catalytic hydrocarbon afterburner, sintered metal filters, heat exchanger, high efficiency particulate air (HEPA) filters, recycle blower, exhaust blower, and automatic control instrumentation. An air-locked batch feeder is necessary because of the pyrophoric nature of uranium turnings. These turnings are sometimes coated with cutting oils containing hydrocarbons that, when pyrolyzed, could plug sintered metal filters, thus necessitating the use of a hydrocarbon afterburner. Recycled off-gas will be used to minimize the nitrogen requirements. Bottled nitrogen will be used since the process building has no Plant nitrogen supply.

Design Operation

Figure 1 is a simplified flow diagram of the proposed process. Uranium chips and turnings will be introduced into the reactor by an air-locked batch feeder. The proposed reactor will hold three 100-pound batches and will be designed to incinerate approximately 100 pounds per hour. An electric igniter will be used to initiate combustion. Flue gas will be used to carry the heat of combustion from the reactor to the off-gas heat exchanger. The rate of reaction of the uranium will be regulated by the amount of air blended with the recycle gas flow. Flue gases

FIGURE 1. Uranium Chip Burner

FIGURE 2. "Nitridr" - Coal Burner Reactor

containing pyrolyzed cutting oils will be diverted through the afterburner for complete combustion during start-up. Volatilization of cutting oils occurs between 450 and 500 °C. A cyclone and sintered metal filters will be used to collect incomplete combustion products. Flue gases will be cooled to 50 °C by a shell-type heat exchanger before passing either through the HEPA filters and exhaust blower or through the recycle blower. Exhaust and recycle gas flows will be regulated to control system pressure. The major portion of the U_3O_8 will be removed from the reactor at the completion of a cool-down cycle. Fly ash removed from the cyclone and sintered metal filters will be analyzed for suboxides and can be recycled to the reactor if necessary for complete combustion.

Design Testing

One process design was formulated when a computerized search of library information revealed that uranium chips can be rapidly converted at 400 °C into a nitride. Because uranium nitride has pyrochemical properties similar to coal dust, a coal burner design (Figure 2) was considered. Experimentation to determine rates of conversion proved that nitriding occurs too slowly to accommodate a 100-pound-per-hour system (Table 8). An experiment to control nitriding temperature by bleeding oxygen across uranium in a nitrogen atmosphere indicated that a direct conversion of uranium to U_3O_8 in a starved oxygen reactor was a feasible

TABLE 8. Uranium Nitriding

Reactor Type: Tube Furnace (Reactor Size - 2 in. dia × 2 in. long)
 Charge : Uranium Turnings (Charge Weight - 200 g)
 Flow : 100% Nitrogen (Flow Rate - 60 cfh)

Run	Temperature (°C)	Reaction Time (hr)	Unreacted Uranium (g)	Conversion Per Hour (%)
1	600	2	171	7.25
2	800	2	118.8	20.3
3	1000	2	128.3	17.9

TABLE 9. Uranium Oxidation

Reactor - Tube Furnace, 2 in. dia × 2 in. long
 Charge - Uranium Turnings, 200 g

Run	Temperature (°C)	Reaction Time (hr)	Total Flow (cfh)	Reaction Gas (% O ₂)	Conversion Per Hour (%)	Reaction Products (%)
1	500-700	1	159.0	2.2	100	U ₃ O ₈ = 87 UO ₂ = 8 U ₂ N ₃ = ~3
*2	500-700	0.6	64.0	1.4	83	U ₃ O ₈ = 55 UO ₂ = 30 U ₂ N ₃ = 10 U ₂ O ₅ = 1-5
3	400-660	0.33	195.0 230.0	1.8- 10 min 5.0- 10 min	300	U ₃ O ₈ = 90-95 UO ₂ = 5-8 U ₂ N ₃ = ~1.0

*Run 2 was not allowed to go to completion so incomplete reaction products could be identified.

alternative to the nitriding route. Further experiments were conducted and the results, as shown in Table 9, were used in the formulation of the present design.

Future Work

Future work will include design testing of the catalytic afterburner system, construction of a pilot scale system, testing and modification of the pilot scale system, and final recommendation for construction of the uranium chip burner processing plant.

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 to help formulate an economic evaluation for determining the best method for disposing of the soil.

Prior Work

Prior work in the Chemical Research Group of Chemistry R&D has involved laboratory scale development of a method for soil decontamination. This is the first work related to large-scale equipment specification and economic evaluations.

Achievements and Discussion

Preliminary studies have shown that contamination is attached to -35 mesh (-0.42 mm) soil particles. Based on this information, the contaminated soil can be concentrated to approximately 30% of the original volume by a wet screening, attrition scrubbing process. Further development work in the Chemical Research Group will be required to determine if an additional process can further decrease the volume of the contaminated soil to

1 to 10% of the original volume. The methods being considered are hydraulic, magnetic, extraction, centrifugal, and gravity concentration.

A conceptual design report (CDR) was prepared that included an estimate of the proposed facility cost. This CDR was presented to DOE on February 27, 1978. The program management plan and alternate economic studies have been completed and presented to Rocky Flats management. Approval was received to proceed with the development work, which will continue for approximately 12 months. The data generated will provide a much more accurate assessment of the technical and economic viability of the volume reduction process for this contaminated soils project.

Future Work

Test programs on uncontaminated soils from Rocky Flats are being initiated with mining equipment manufacturers. These tests will be done in the manufacturers' pilot plants to collect power requirements, sizing data, soil analysis information, and other pertinent data required to select equipment best suited for the soils decontamination project. A dustless method for removing the soil will also be developed. Any resuspension of contaminated soil will not be permitted.

Upon completion of the development work, the most economical methods and specifications can be generated. These data will then be supplied to the Rocky Flats Facility Engineering Department for input into the design criteria for an architectural and engineering contract.

FLUIDIZED BED INCINERATION SYSTEM FOR DEPARTMENT OF ENERGY DEFENSE WASTE

*David L. Anderson, Burke 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 two-fold: to demonstrate the adaptability of the fluidized bed process

for combustion of a wide variety of transuranic (TRU) contaminated waste materials and to prove scale-up information developed on pilot plant equipment. To accomplish the second objective, the pilot plant equipment was scaled up for a capacity of about 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 will play an important role in reaching the project objectives. It will perform real-time data acquisition, in-process calculations, documentation of incinerator equipment performance history, inventory control of nuclear materials, and off-line scientific data analysis.

Prior Work

Phase I, the construction of a containment area, began in June 1976. This work was completed in April 1977. A beneficial occupancy inspection of the containment area was accomplished on April 7, 1977.

Design of process equipment and associated process piping was completed in March 1977. After Title II review of all process equipment, a "control print" procedure was implemented. With this procedure, all equipment modifications, design changes, and corrections of possible safety hazards were channeled through a "control print" engineer to ensure proper documentation and overall project coordination. Phase II, the installation of process equipment, was started on April 11, 1977.

All hardware items for the data acquisition system and associated process sensors have been received. The data acquisition system consists of a Digital Equipment Corporation (DEC) programmable data processor (PDP-11/10), Foxboro Spec 200 and Interspec 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 delivered were thermocouples, pressure cells, flow meters, weight cells, and contact closures.

Achievements and Discussion

In April 1978, the fabrication, installation, and insulation of all process equipment was completed along with the placement of process sensors and wiring of the Foxboro Spec 200 and Interspec signal conversion system. A beneficial occupancy inspection was held April 27, 1978.

Work on the process monitoring and control system is continuing. The installation of system hardware is 90 percent complete. The remote weighing station and control panel have been built and installed. Hardware requirements for the graphics display of the fluidized bed incineration process are being specified and will be purchased during FY 1978.

The data acquisition system computer software package is 60 percent complete. This will enable the control room operator to examine any combination of 62 process variables and their associated alarm values.

An operational safety analysis (OSA) report has been written and circulated for review. Final approval of the OSA is expected in June 1978.

In preparation for experimental runs with the equipment, approximately a two-year supply of chromic oxide oxidation catalyst and sodium carbonate neutralizing agent have been obtained. Also, enough simulated waste has been collected to conduct the initial incineration test run.

Future Work

The process monitoring and control system should be completed by July 1978. This includes the computer software package, hardware diagnostic check, and final calibration of instrumentation. Concurrently, system operational (SO) tests of equipment will be made. Training of operating technicians will take place after SO testing has been completed. Cold testing with simulated waste should start during July 1978, and demonstrated operation with contaminated waste should be made in September 1978.

DEVELOPMENT OF A FLUIDIZED BED INCINERATION PROCESS

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

Objective

The objective of this program is to design and develop a fluidized bed incineration system (FBI) for processing combustible wastes contaminated with radioactive material. The system is to reduce both liquid and solid wastes to a dry, non-flammable residue suitable for actinide recovery, storage, or additional waste fixation operations, while accomplishing substantial volume and weight reduction. This system will have advantages over conventional incineration systems because it has a lower operating temperature, decreased equipment corrosion, and no need for an aqueous off-gas scrubbing system.

Prior Work

The FBI unit was installed in a canyon type enclosure for operation with plutonium-contaminated waste. Test runs with production-generated combustible waste revealed a high tramp metal content (8.5 wt %) that necessitated the design and installation of an air classifier. Test burns of high efficiency particulate air (HEPA) filters resulted in a weight reduction of ~2:1 and a volume reduction of ~4.5:1; the residue was then in an ideal form for further processing in the vitrification system.

Testing was initiated on a laboratory scale FBI on the burning of organic solvents containing tributyl phosphate (TBP). The tests were done in a 115-mm-diameter, quartz, fluidized bed incinerator. Test results showed that 99% of the phosphates were retained as thermally stable sodium phosphate. This led the way for further investigation in the larger scale pilot plant FBI.

Achievements and Discussion

Three, around-the-clock, week-long runs have been completed on the pilot scale FBI incinerator to

test its operability for TBP-solvent disposal. The first run was made using 15% TBP and 85% Texaco P-4 solvent by weight; the last run used 15% TBP and 85% Conoco L. P. A. solvent. No problems were encountered with either of the solvents. The TBP, however, did present a problem in the liquid pumping system since it attacks most elastomers that are used to seal pumps.

After testing several pumps, a positive displacement gear pump with a fluoroelastomer mechanical seal was found to work best for initial fluid handling. A fluoroelastomer diatube, positive displacement, injection pump worked best for pumping directly into the incinerator system.

Tests completed on the laboratory-scale quartz FBI indicated that a 50-50 mix of sodium carbonate and chromic oxide oxidation catalyst in the primary vessel and catalyst only in the afterburner vessel would be suitable for TBP-solvent runs. Approximately 1 kg of Na_2CO_3 should be added to the primary vessel each hour, and approximately 1 kg of bed material is elutriated to the cyclone separators to maintain a steady state condition in the primary vessel. Operation of the incinerator in this fashion maintains a 50% excess of sodium carbonate to neutralize acid gases formed in the process. At the completion of the runs, the incinerator off-gas system was dismantled and inspected for P_2O_5 formations. Results showed that nearly 100% of the available phosphate was retained as the stable sodium phosphate. No evidence was found of any P_2O_5 formation in the entire system. These preliminary results indicate that TBP-solvent incineration is feasible for fluidized bed type incinerators using a basic bed material.

Future Work

Several more runs are planned to complete data collection for TBP-solvent incineration. The off-gas system will be modified to smooth out several trouble spots. The off-gas analyzer system will be modified to achieve a more accurate assessment of the gas effluents. Plans are being made to purchase a reliable total hydrocarbon analyzer capable of operating on a process facility.

IMMOBILIZATION OF INCINERATOR ASH AND PLANT WASTE

*James A. Ledford, Paul M. Williams, Seng F. Chung,
Michael Del Hierro, and Roger A. Santeramo*

Objective

The objective of the project is to develop a method for processing several types of radioactive wastes into a form suitable for transportation and long-term storage. A suitable form would be one having sufficiently durable mechanical characteristics to resist breakage and dusting during handling and having sufficiently inert chemical characteristics to resist attack by the environment in which it is stored.

Prior Work

Wastes have been immobilized in dry and sludge forms at the Rocky Flats Plant through use of sintering, plastic bonding, plastic coating, cementing, and vitrification techniques. Vitrification is currently receiving the greatest emphasis since the product produced by this process appears to most closely fulfill the objectives of the project.

Simulated wastes currently being vitrified are diatomaceous earth, mineral resin, and three types of fluidized bed incinerator ash. The glass product contains approximately 67 wt % waste, 33 wt % glass-forming additives (boric acid and sodium carbonate), and occupies 16% of the volume of its dry powder constituents.

To study long-term physical stability, a mathematical model was developed. The model could calculate the effects of helium generated during alpha decay in glass containing 0.3% plutonium oxide. Data from the model indicate that more than 2000 years would elapse before internal helium generation could cause glass fracture.

An externally heated metal reactor was fabricated of Inconel 690 and was incorporated into a bench-scale, fully operational process. Free-formed glass pellets were produced that possessed good physical characteristics.

Achievements and Discussion

Resistance Heated Reactor

A larger metal vitrification reactor has been constructed in accordance with the same basic design as previous reactors. Made from 0.64-cm-thick Inconel 690, the reactor is square in cross-section, 23 cm on a side, and is 51 cm long. The reactor has been installed in a commercially available electric furnace and is presently producing approximately 4 kg of glass pellets per hour.

The furnace is heated by silicon carbide glowbars arranged in three independently controllable zones. It is capable of maintaining 1500 °C. The present furnace and reactor began operation in January 1978 and by the end of May 1978, had logged 900 hours of operation. Sections of the reactor have been opened to determine conditions within; however, no evidence of seriously destructive corrosion has been observed. Further studies of reactor corrosion will be undertaken at the time of the next major modification. Necessary maintenance operations have been infrequent and have required only unsophisticated efforts and little lost time. Data gathered during these operations indicate that complete reactor replacement could be accomplished in less than two hours of maintenance time.

Other components of the present bench-scale process are an Inconel 690 rotating forming table, vertical tube annealing furnace, and vibratory cooling conveyor.

Inconel 690 was selected over aluminum, stainless steel, and carbon steel for the forming table. The choice was based on the greater resistance of Inconel 690 to warping from thermal stresses imposed by molten glass during the forming operation. Serving as the annealing lehr is a vertically oriented tube furnace. This furnace contains a carbon steel pipe liner that is axially mounted, has a 2.6-cm inside diameter, and is about 56 cm long. Once the pipe is filled with glass pellets, travel time of the pellets through the furnace is controlled by the rate of withdrawal of pellets at the bottom. Pellets are withdrawn, cooled, and transported to a collection hopper by a variable-speed vibratory conveyor.

Joule Heated Reactor

A bench-scale, joule-heated, refractory melt reactor is in the process of being constructed. This reactor will be operated along with the resistance-heated reactor presently in operation. The two reactors will be compared to determine which appears to be more adaptable for operation in a pilot plant. The anticipated advantages of the joule-type furnace are

1. The container holding the molten glass can be of refractory brick; seams between the bricks or cracks are not critical since the melt will solidify as it seeps into the crack or seam.
2. The heat created by the furnace is generated in the melted product; therefore, the highest temperature will be in the product and the components of the furnace will experience a lower temperature.
3. Visual inspection of the reactor internals can be made by simply removing the top refractory.

The joule-type furnace has a melting chamber with a capacity of 2.3 liters and a fining chamber with a capacity of 1.2 liters. This furnace is expected to operate at a maximum temperature of 1050 to 1100 °C. The joule melter will be started with the use of a silicon carbide resistance element placed above the ash mixture. It is anticipated that the corrosive environment of the furnace chamber will cause a rapid deterioration of the resistance heating element. A method of starting up the joule furnace using electrically conductive compounds is being investigated. If a compound is found that can be used for starting a cold or frozen melter, the resistance heating element can be removed.

This furnace should provide data on conductivity changes in the glass mixture at various temperatures. The electrical power required to sustain the melt also will be monitored to provide input for the design of a larger second-generation furnace.

Annealing

Observations have confirmed that rapid, uncontrolled cooling of the glass introduces severe thermal

stresses that, depending in large part upon the size of the specimen, are often sufficient to cause spontaneous fracture. Two major types of fractures have been observed to occur: a completely destructive type in which the specimen immediately divides into two or more physically separated parts, and a "hidden" fracture that causes no immediate separation but that weakens the specimen, causing it to be less resistant to subsequent physical abuse. While the free-formed pellets are of such a size as to be relatively resistant to thermal fracture, breakage during cooling occasionally does occur. Both types of fracture are exhibited, with hidden fractures being most common.

Annealing was therefore explored to control cooling and relieve stresses in the glass. Evaluation of the effects of annealing is presently incomplete. Initial indications from tests are that annealed pellets are more resistant to thermal shock.

Future Work

Pellet annealing, off-gas cleaning, pellet chemical durability, quality control, and development of design criteria for a reduced-scope pilot plant facility will be investigated next in this program.

INSTRUMENTATION AND STATISTICAL SYSTEMS

Richard N. Chanda

PLUTONIUM AND AMERICIUM SOIL ANALYSIS OF SELECTED LANDS ADJACENT TO ROCKY FLATS

Merril W. Hume

Objective

The objective of this work is to analyze data concerning soil samples from lands adjacent to Rocky

Flats to determine the levels of transuranic deposition attributable to Rocky Flats operations. These data are to be used in litigation proceedings in which the plaintiffs charge that activities of Rocky Flats have rendered their land contaminated.

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. This sampling plan, along with analytical procedures and some analytical results, were presented at pretrial hearings before the U. S. District Court for Colorado. As a result of those hearings, this testing program was modified, augmented, and will be referred to as the retesting program.

Achievements and Discussion

Plutonium and americium analytical results have been obtained on samples collected by three of the four collection techniques. Eighty-one sample sites were involved. The results were essentially as anticipated. 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 as previously noted. The major modifications over the original program consists 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.

The three participating laboratories received batched samples and began analysis in early February. In mid-March, when about half of the plutonium analyses were completed, it was discovered that the

field consensus standard had not been pretreated properly. Modifications were then made to the retesting program; however, this will not only be costly but also cause delays and some loss of quality control.

Future Work

A comprehensive statistical analysis will be performed when all of the data are available. The results will be presented at pretrial hearings this summer and play a major role in the trial next year.

LEADED GLOVE-LIFE STUDY

Elizabeth L. Baker

Objective

The objective of this effort is to determine the glove-life time for leaded gloves used in process-area glove boxes.

Prior Work

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

Achievements and Discussion

Component Development Research and Engineering is conducting a study on leaded gloves. One aspect of this study is glove-life time. The original expiration time for a glove was determined on a "best guess" basis. The Statistics Laboratory was asked to statistically determine glove-life time from current glove-life data.

Several computer programs have been written to massage and tabulate existing glove-life data and to indicate whether the expiration time associated with each glove box is appropriate. As a result of these programs, the expiration time on gloves has been extended or shortened on some 100 glove ports.

Future Work

With an increase in available glove-life data, a more comprehensive analysis will be performed to determine actual glove-life time for each glove port.

OPERATIONAL SAFETY ANALYSIS REVIEW PROGRAM

Elizabeth L. Baker

Objective

The objective of this program is to computerize the update and generation of an Operational Safety Analysis (OSA) review report.

Prior Work

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

Achievements and Discussion

Industrial Safety periodically reviews OSAs plant-wide. To reduce the man-effort required to update OSAs and to generate an OSA review report, the Statistics Laboratory has created a data file containing relevant OSA information and has written a computer program to regularly update this file and generate a current status report.

The program to generate the OSA review report has been completed and is now in use.

Future Work

After Industrial Safety has committed itself on final specifications, the computer program will be put into production. At this point, services of the Statistics Laboratory will only be required when changes are needed to the program; i.e., changing the report specifications and correcting errors.

STATISTICAL CONSULTING

Elizabeth L. Baker and Merril W. Hume

Objective

The objective is to provide statistical and mathematical guidance and technical support to all Rocky Flats operations upon request. 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 Discussion

Sampling plans have been developed for capital equipment items and various environmental monitoring programs, programming and statistical support to assess weather and air pollution over Denver and Rocky Flats, reliability determinations for wind energy systems and leaded gloves, error analysis for nuclear material control and for various production equipment and processes, monitoring and improving measurement control capabilities, and support for national ANSI and ASTM statistical subcommittees.

Future Work

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

CORRELATION STUDY ON INVENTORY DIFFERENCES

Elizabeth L. Baker

Objective

The objective of this project was to determine whether the declassification of Inventory Differences (ID) would divulge classified information.

Prior Work

This was a new objective.

Achievements and Discussion

The Department of Energy has been considering declassifying ID. As part of an overall study for Safeguards and Security to determine whether classified information, such as inventory and parts made, can be gleaned from ID data, the Statistics Laboratory has done a correlation study on ID versus relevant variables for plutonium, enriched uranium, and uranium-233. This study includes a review of a similar study done at Oak Ridge.

A rough draft on the correlation analysis has been completed and will be incorporated into the final report by Safeguards and Security for DOE.

Future Work

No future work is planned at this time.

AUTOMATIC SEQUENCER FOR WASTE DRUM LINER FLAME SEALER

Frederick L. Crawford

Objective

The objective of this project was to design, fabricate, and install an automatic sequencing system for sealing plastic waste containers.

Prior Work

A flame sealer to seal lids on plastic drum liners is currently undergoing testing by the Component Development Group, Research and Engineering. A system for automating and sequencing this

sealer has been designed and fabricated (see RFP-2680A, October 21, 1977, p. 36).

Achievements and Discussion

The automatic sequencing operation of the waste liner sealer has been installed with the necessary safety modifications (see Figure 3 shown on pages 26, 27, and 28). The added safety features include gas safety pilot burners with a bi-metal warp switch that will open if the pilot burner fails. The main gas supply valve outside of the work area and the burner supply valves automatically become inoperative if the pilot burner fails. If a pilot burner is extinguished, the main supply valve will be automatically shut off within 30 seconds, thus eliminating a buildup of explosive gases in the building and work area. An air cooler has been incorporated in the final operation to ensure greater tensile strength in the new weld.

The modified safety flame sealer has been tentatively accepted for sealing waste drums used to transport low-level radioactive sludge generated in waste treatment. Final acceptance will depend on waste-drum impact and integrity tests.

Future Work

Assistance will be provided as needed during acceptance testing, but no major modifications or additions are planned.

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.

FIGURE 3. Control System, Including Added Safety Features, of Flame Sealer for Waste Drum Liners

FIGURE 3. (continued)

FIGURE 3. (concluded)

FIGURE 4. Instrument Control Panel for Water Control and Recycle Plant

Prior Work

Support of this project has been on a continuing basis but the only results available through the open literature begin on page 31 of RFP-2417A, a progress report dated August 29, 1975.

Achievements and Discussion

Technical assistance during construction and installation of the instrument control panel for this project has been provided. Assistance included periodic inspections of the panel during construction and a final inspection before placement in the plant control room. (See Figure 4.)

An outline of a single control loop operation was prepared to be used as a guide in developing the plant operating manual. Loop variables, trip points, and plant start-up procedures were listed in addition to the loop operation. A first draft of the operating manual by the architect-engineers has been reviewed.

Future Work

Overseeing the installation of field instruments and the electrical tie-in of the control panel is in progress. A final review of the operating manual and two reviews of the System Operation (SO) Test Procedures are planned. Development of a program for the programmable logic controller to perform the SO tests and a program to operate the plant is in progress and will be continued.

HEAT REGULATION OF HYDROFLUORINATOR

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

Objective

The objective of this work was to provide an improved heating control system for a hydrofluorinator.

Prior Work

The original pneumatic and mechanical system failed to maintain proper heat on the rotary fluorinator tube. This resulted in an unacceptable product. Time lag from controllers and power rheostats, along with unreliable open thermocouples, allowed heat on the tube to fluctuate beyond set points.

Achievements and Discussion

There are three heat zones on the fluorinator tube, each operating at a different temperature. To protect and give a redundant thermocouple, two thermocouples have been installed in a sheath made of Inconel®*. Two of the dual thermocouples are located in each heater section and are connected to recorder-controllers and to digital readouts on the operators control panel. The redundant thermocouple assures the reliability of the controllers and thermocouples and enables the operator to observe the heat on all areas of the tube.

Three electronic recorder-controllers operate the solid state driving device (SCR) for the induction heaters. These are soft-heat SCR units to prolong heater coil life and to conserve electrical energy. All of the sensor points are alarmed for malfunction.

The project included specification of equipment, technical support, and preparation of adequate field drawings for Maintenance to work from. Obsolete equipment was removed and new equipment was relocated in a panel. The hostile environment of the fluorinator and its mating to existing usable equipment added a challenging dimension. The controls have provided better heat regulation on the tube and thereby aided in improving product consistency.

Future Work

Further improvements on other process heat and flow controls are planned.

*Inconel is a registered trademark of International Nickel Company.

PROCESS DATA ACQUISITION SYSTEM

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

Objective

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

Prior Work

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

Achievements and Discussion

At the request of Chemical Operations and in conjunction with Process Chemistry and Engineering of Chemistry R&D, the concepts of an ideal data acquisition and control system have been formulated. This system is to be capable of data acquisition reduction, record keeping, and in addition, have limited control on the process in the line. A general purpose microprocessor system will be designed and fabricated for each major wet chemistry production line. Each microprocessor will communicate with an available Hewlett-Packard minicomputer.

A prototype microprocessor system is presently being designed and fabricated. The general purpose data handling programs are currently being written for this microprocessor.

Future Work

The specific programs and interface electronics for each class of instruments to be connected to the system will be designed and written.

*CO-OP student

**Process Chemistry and Engineering

DEVELOPMENT OF OSCILLOMETRY FOR PROCESS CONTROL

Grover J. Cunningham and Charles M. Johnson

Objective

The objective of this work is to develop a method to indicate the presence or absence of solution in a process line or to detect when a significant change in solution composition occurs within the line.

Prior Work

No previous work has been done at Rocky Flats on this project.

Achievements and Discussion

An investigation has been initiated into the possible application of oscillometry in the chemical recovery operation. The technique, applicable mainly to solutions, involves measuring the resistance to conduction of an alternating current of radio frequency (10^5 to 10^7 Hz). As a result of various conduction mechanisms, the impedance of a solution varies in an intricate fashion with the dielectric constant, the electrolyte concentration, and the frequency. Interpretation of these relationships is difficult; however, the method is attractive since measurement does not require direct contact between electrodes and the solution. Possible applications include the presence or absence of solution in a line, breakthrough on ion columns, or other situations where a significant change in solution composition occurs. A circuit has been designed and built, and appropriate tests are being formulated.

Future Work

Future work will consist of evaluating and testing the designed circuit.

IN-LINE PROCESS CONTROL SENSOR FOR CHEMICAL PROCESSES

*Charles M. Johnson, L. Harry Faber,
Frederick L. Crawford, David E. Hausburg,
James L. Lawless, and Francis X. Haas*

Objective

The purpose of this work is to provide sensors for critical parameters in chemical processes used at the Rocky Flats Plant. The instrument under current investigation is a critical angle refractometer. The objective of this investigation is the adaptation of the principle of this instrument to nitric acid measurement in a plutonium nitrate stream.

Prior Work

Critical angle refractometers have been used in the past at the Hanford facility in Washington and at Rocky Flats for acid measurement in plutonium and uranium process streams. The use of refractometers has been limited to process streams having constant constituents with nitric acid as the only variable.

Achievements and Discussion

Critical angle refractometry is the physical measurement of reflected and absorbed light directed at the interface of two substances. In the case of a nitric acid-measuring instrument, the measurable change takes place at the interface of a sapphire prism and the nitric acid solution. Light directed at the interface at the critical angle of the compound of interest is 50 percent reflected and 50 percent absorbed. Changes in concentration of the substance of interest changes the percentages of reflected and absorbed light. The main disadvantage of this type instrument is that refractive index is an additive function; consequently, any change in the solution composition will change the angle of refraction to some degree.

The current work is directed towards minimizing the changes caused by compounds other than nitric

23064-2

FIGURE 5. Control Panel in Apparatus Used for Accelerated Stress Corrosion Cracking Tests

acid and bringing the associated error within an accepted level. In case of nitric acid measurement in plutonium nitrate solutions, the main variable, other than nitric acid, is plutonium nitrate. If the concentration of plutonium nitrate is known, an electronic correction is possible to give an accurate nitric acid measurement. Because of the accuracy of the radiometric determination of plutonium nitrate, this method has been chosen as the corrective function to use.

The instrument will consist of a critical angle refractometric measuring head, a radiometric germanium gamma-ray analyzer, and a micro-processor to process the data. These three components will be developed separately and then combined into one instrument.

The refractometric sensor is now being investigated. Serious consideration is being given to a fiber optic array at the sensor prism to produce a digital signal. Various configuration of fiber optics have been considered, and one has been chosen for further study.

Future Work

A laboratory refractometer will be purchased, then preliminary testing will be initiated.

ACCELERATED STRESS CORROSION CRACKING TEST APPARATUS

David E. Hausburg and William B. Schmidt

Objective

The objective of this work is to design and construct an apparatus to carry out accelerated stress corrosion cracking (SCC) testing of aluminum alloy specimens.

Prior Work

An apparatus to perform 30-day SCC testing on aluminum alloy specimens was constructed.

22930-4

FIGURE 6. Specimen Holder for Accelerated Stress Corrosion Cracking Test Apparatus

Achievements and Discussion

Replacement of the existing 30-day SCC test for aluminum alloy specimens with a 24-hour test would be advantageous. An apparatus to carry out a 24-hour specimen test has been designed and constructed. The control panel is shown in Figure 5. Components include a direct current power supply that applies voltage to the anode electrode (specimen) and to the cathode electrode, a digital readout to indicate current flow, a potentiometer to adjust current flow, and a cabinet to house the components.

A specimen holder was constructed that has an efficient means of measuring the physical deflection of the specimen. (See Figure 6.) In addition, the specimen holder provides unrestricted current flow between the two electrodes.

Future Work

No additional work is planned.

GRIT BLAST FEEDER

George P. Minges and Frederick L. Crawford

Objective

The objective of this work is to develop equipment for controlling and measuring the performance of the production grit blast machine.

Prior Work

This is a new objective.

Achievements and Discussion

The current grit blasting machine has no method for measurement of the sand-air ratio and therefore requires constant operator attention. The sand-air ratio is controlled by a crude gravity value set by the operator who judges the quality of the operation by the sound emitted from the machine.

A sand auger system has been designed and is currently being fabricated as the simplest and most cost effective solution to the control and measurement problem. Because of the nature of the operation, special consideration was given in the design to the elimination of foreign materials and oil from the sand.

Future Work

Upon completion of fabrication, the auger unit will be tested, evaluated, and installed.

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

Radiometric instruments have been developed in the past for measuring plutonium in process residues. These include the can, helix, and two drum counters. Improved measurement in certain categories will require an instrument of higher resolution capability.

Achievements and Discussion

The "Can-Scan" counter has been developed to improve the measurement on waste and scrap categories that exhibit difficult interfering characteristics such as lumpiness, high americium content, and segregation. The system is basically a commercial unit purchased from Canberra Industries, Meriden, Connecticut. It utilizes a high resolution germanium gamma-ray detector, a rotating table

that can be programmed to raise and lower in steps to provide segmented scanning, a transmission source to determine the amount of absorption contributed by the matrix media, and a correction source to determine the system dead time. These data are accumulated by the system computer, calculated, and the concentration of both plutonium and americium is reported for each segment on the system terminal. Diagnostic messages, processing information, and operator prompting messages also are provided.

The system has been installed in the chemical processing area and is undergoing checkout and calibration.

Future Work

System response to various category standards will be determined during the following months. Calculational factors based on these observations will be incorporated into the computer logic. Priority will be given to calibrating the system for the molten salt category.

CALORIMETER END POINT PREDICTION

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

Objective

The objective of this work is to develop equilibrium or end point prediction by computer analysis and thus reduce calorimeter analysis time by up to 60 percent.

Prior Work

This is a new objective.

Achievements and Discussion

A computer code based on a Mound Laboratory algorithm for equilibrium prediction was developed

*CO-OP student

and implemented on Instrumentation and Statistical Systems' Hewlett-Packard computer. Experimentally, the algorithm has proven to be too inaccurate for the large volume, low plutonium level samples required at Rocky Flats. A computer code that more nearly approximates the true multiple exponential curve of the calorimeter has been successfully developed.

Future Work

A data acquisition system has been ordered that will allow the collection of data necessary to evaluate and utilize the newly developed equilibrium prediction method. Results will be evaluated for possible implementation in all Rocky Flats calorimeters.

EFFLUENT MONITOR ANALYZER

*George P. Minges, George J. Cunningham,
and Mike A. Fox**

Objective

The objective of this work was to develop an analyzer for special nuclear materials (SNM) pipeline monitors.

Prior Work

This was a new objective.

Achievements and Discussion

A microprocessor-based electronic signal analyzer has been designed and fabricated. It analyzes signals coming from two radiometric detectors that are separated by a known distance along a pipeline. The instrument performs a statistical analysis on the signal from each detector and looks for a travel-time-related coincidence of a high level radiometric disturbance. When this

condition occurs, a relay contact changes, allowing for an external alarm or automatic valve action to isolate the disturbance. The instrument was patterned after the successful portal monitor analyzers.

Future Work

When this system is installed, Instrumentation and Statistical Systems will participate in the design fabrication and installation of detectors, alarms, and/or isolation valving.

CRATE RADIOASSAY COUNTER

*Ronald A. Harlan, Wayne E. Herren,
and E. Duane Frantz*

Objective

This objective is to develop a sensitive method to detect and estimate plutonium in 1.22 by 1.22 by 2.13 meter (4 by 4 by 7 feet) crates. The user desires a "go, no go" decision at 10 grams of plutonium. The counter is also designed to produce a rough assay to provide normal operating loss accountability.

Prior Work

The counter was constructed and a plutonium standards crate was built. For further details, see RFP-2680-A, October 21, 1977, page 23 and Figure 17, and *Nuclear Materials Management*, Vol. IV, No. III, 1977, p. 457.

Achievements and Discussion

The counter was operated under laboratory conditions for neutron counting. It was found capable of detecting 0.7 grams of War Reserve (WR) plutonium as PuO_2 in 6 hours or 10 grams in 20 minutes. The neutron detectors respond to spontaneous fission neutrons from ^{240}Pu and

*CO-OP student

neutrons generated from the α, n reaction in PuO_2 . The sensitivity varies, therefore, with the isotopic composition and chemical form of the plutonium. For reactor grade plutonium, with up to 30% ^{240}Pu , the counter would be approximately three times more sensitive than for WR material.

During our studies, we noted gamma-ray sensitivity to sources emitting gamma rays with energy greater than 100 keV. The low resolution detectors had been set to exclude response to plutonium gamma rays that are mostly below 500 keV in energy. New operating points have been chosen to determine the sensitivity with no response to gamma rays, plus or minus 4000 keV. This operating point excludes some neutron events, and the sensitivity is expected to be different.

Long term changes in the system's count of background was studied over 6 weeks. It was found that considerable drifting in the absolute counts did occur and that the ratio of counter background to an external background monitor changed about 5 percent. The ratio seldom changed more than 2 percent, however, between successive 30-minute counts. If background counts are taken between each sample measurement, the sensitivity figures previously quoted are valid should the old operating points be chosen.

Future Work

Data under the new operating points will be analyzed and a set of operating conditions will be chosen. Gamma detectors will be added to the counter and evaluated for assay purposes.

X-Y RETRIEVER VERIFICATION SYSTEM

*Grover J. Cunningham and Jorge L. Martinez**

Objective

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

*Safeguards and Security

Prior Work

No previous work has been done at Rocky Flats on this project.

Achievements and Discussion

Monthly inventories conducted in the X-Y retriever storage vault call for physically verifying the contents of each container using a remotely controlled manipulator arm. This operation takes approximately 6 minutes for each container or several hours for the entire vault. An effort is being made to provide a radiometric scanner that would verify the presence of SNM in a few seconds.

A battery operated instrument, Ludlum Model 300, has been purchased and modified to be carried by the manipulator arm. The instrument carrier will be similar to the SNM carrier presently used; consequently, the computer program in the manipulator will not require modification when scanning the canisters during inventory time.

The design of the lead shield to collimate radiation to the detector has been completed. Tests are being run to determine detector sensitivity and lead collimator design. Sensitivity should be sufficient to detect small SNM parts in addition to ingots with larger weights.

Future Work

Tests are to be run with different lead collimators and detectors to achieve adequate sensitivity.

INTRUSION DEVICES FOR THE PROTECTION OF SPECIAL NUCLEAR MATERIAL STORAGE AREAS

*Grover J. Cunningham and Jorge L. Martinez**

Objective

The objective is to lower the number of false alarms and upgrade the levels of response for special nuclear material (SNM) storage area intrusion systems at Rocky Flats.

*Safeguards and Security

Prior Work

No previous work has been done on this specific objective.

Achievements and Discussion

Data on the environmental and physical parameters of the two SNM storage vaults were gathered and used to design the physical layout of an intrusion detection system. These data were also used to select the type of intrusion system needed for these two SNM vaults.

The two SNM storage vaults have now been updated with new types of intrusion detection systems.

Future Work

Based upon the continued successful operation of these two intrusion systems, all Rocky Flats SNM storage areas will be updated in a comparable manner.

PORTABLE INSTRUMENTATION FOR THE DETECTION OF IODINE-131 IN VIVO

*Grover J. Cunningham and Jorge L. Martinez**

Objective

The objective of this work was to provide Plant Protection personnel with a means of checking employees who may have received *in vivo* radioactive tracers, such as iodine-131.

Prior Work

No previous work in this project has been done at Rocky Flats.

Achievements and Discussion

Employees entering or leaving special nuclear material (SNM) areas who receive *in vivo* radioactive tracer treatments trip the personnel monitors.

*Safeguards and Security

At present, Plant Protection personnel have no way other than a hands-on search to determine if the portal monitor alarm was tripped because of a radioactive tracer or because of the employee possibly having some SNM in his or her possession.

A portable instrument with a sodium-iodide probe was assembled that allows a security inspector to scan an employee and determine the location of the radioactive material. If the employee has received iodine-131 treatment, the source of radiation should be located in the neck or thyroid area.

A Ludlum Model 16 analyzer was fitted with a 1.5-inch-diameter by 0.25-inch-thick sodium-iodide crystal. An aluminum detector holder lined with a 0.125-inch lead sleeve was fabricated to house the crystal, tube, and plug-on divider, and provide a highly directional probe. Background count rate was further diminished by operating the single channel analyzer with a window range of 115 to 130 keV.

Tests with persons having received iodine-131 treatments showed the probe to be sufficiently directional to locate not only the iodine-131 in the thyroid area but also to pinpoint small amounts of plutonium in close proximity. Two of these units have been assembled for Plant Protection use.

Future Work

No further development work is planned on this project.

RADIOMETRIC MONITORS AND ASSAY COUNTERS FOR THE NEW PLUTONIUM RECOVERY FACILITY

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

Objective

The objective is to develop design criteria for radiometric instruments to be installed in the new plutonium recovery facility.

Prior Work

Conceptual designs developed for these instrument systems fall into two categories: (1) monitoring systems used for safety and process control, and (2) systems used to assign a plutonium assay value to materials. This information will be for accountability and safeguards purposes. The instruments and their functions were identified. Cable runs for instruments and computer interfaces were identified and installed by the contractor.

Achievements and Discussion

Equipment specifications for the molten salt residues and liquids counters have been submitted to the contractor for quotation requests. The equipment will assay molten salt residue and analytical laboratory liquid samples by performance of Ge(Li) gamma-ray spectral data acquisition, data manipulation, and data transmission to the stacker retriever computer. In addition, controls are needed for a stepping motor drive to perform segmented gamma-ray scanning and for a refractometer interface. These controls, with relay closures and switch-closure sensors, are necessary for sample changing and transmission source activation. A third data acquisition and display unit was specified so that samples requiring special handling can be treated without interfering with routine work at either of the other two acquisition stations.

The spent resin ion column counters were dropped for the present because of cost overruns and the need to purchase and install "off-the-shelf" items that involves no development work for proper operation. Cable runs and a computer interface were provided, however, for future use with these counters.

Future Work

Work will continue on specifications for the calcined oxide assay counter and on modifications for the process residue counters to include gamma-ray attenuation measurements and neutron counting capabilities. Specifications for equipment will be reviewed and all counters will be purchased during this fiscal year.

CALIBRATION AND INVENTORY VERIFICATION EXPERIMENTS

Ronald A. Harlan

Objective

This effort seeks to provide a data base for non-destructive assay (NDA) calibrations of Rocky Flats radiometric counters.

Prior Work

Work of this type is a continuation of similar studies dating back to 1964. Instrumentation and Statistical Systems share in the effort by designing sampling plans and calibration models, analyzing and interpreting data, implementing revised or new calibrations, and participating in the Plant's Measurement Control Task Force. Nondestructive assay devices covered are the Can Counter, Helix Counter, Waste Packaging Facility (WPF) South Drum Counter, and WPF North Drum Counter. As a reference assay technique, calorimetry of material in 4-liter containers is widely used. Much of the work is done at Rocky Flats, but some calorimetry is performed at the Mound facility at DOE request.

Achievements and Discussion

Calibrations were updated for Material Description Codes (MDCs) 406, 407, 408, 409, 410, and 411, all of which are molten salts residues of various kinds. Also, in collaboration with the Chemistry Standards Laboratory, a calibration was developed for U-233 in waste. The effort on U-233 was preceded by several special drum assays that pointed out the need for this calibration.

The experimental portions of several calibrations were completed; however, it was not possible to complete the calculations nor adjust the calibration functions. Samples were selected for the 1978 exchange of samples with the Mound facility.

Future Work

Calculations will be completed and data from the 1978 exchange of samples with the Mound facility will be analyzed.

PACKAGE ESTIMATOR

*Ronald A. Harlan and Jill A. Irvin**

Objective

The objective of this work is to develop an improved counter, to estimate operating loss of plutonium in several small Research and Engineering nuclear material accounts.

Prior Work

A survey meter had been installed previously to estimate operating loss. Its design and calibration, however, were inadequate for assay purposes since its original intent was for nuclear safety purposes.

Achievements and Discussion

Instrumentation and Statistical Systems provided a collimator and shield to be used with the existing Ludlum survey instrument. The instrument was calibrated by Chemistry Standards Laboratory personnel according to a jointly designed experiment and was put into service following training of the user group by Chemistry Standards Laboratory personnel.

Future Work

No further work is planned at this time.

SOIL DECONTAMINATION

Francis X. Haas

Objective

The objective of this work is to establish design criteria for radiometric instrumentation to be used in the soils decontamination project.

Prior Work

No prior work has been performed on this project.

Achievements and Discussion

Radiometric instrumentation needed for the soils decontamination project has been delineated into three categories: (1) a water recycle monitor to measure radioactive contamination in the water used to wash the soil, (2) screened materials monitors to measure the contamination levels in various mesh sizes of screened material with a sensitivity of less than 10 nanocurie per gram, and (3) in-situ soil analysis monitor to locate contamination "hot-spots" in areas to be decontaminated. A manpower and expense matrix has been prepared for this instrumentation.

Future Work

When money for this project is approved, prototype instruments will be built and tested to perform the desired tasks.

PUBLICATIONS AND SPEECHES

Publications

Donald L. Ziegler, Jim White, Andrew J. Johnson, Leong Q. Fong, Alton R. Teter, and Seng F. Chung. "Status Report, Waste Incineration and Fixation for Waste Management, Production, and Reprocessing Division of the Department of Energy." RFP-2655. November 25, 1977.

Edward S. Goldberg, Charles R. Allen, James A. Bowman, Harry E. Hootman, Donald F. Luthy, Albert S. Neuls, and Donald L. Ziegler. "Transuranic Waste Technology Assessment Task Group Report." To be issued as a Department of Energy report.

Lewis L. Richey, Peter I. Faccini, and Pen K. Feng. "Status Report on the Fluidized Bed Incineration System for U. S. Department of Energy Defense Waste." RFP-2654. March 27, 1978.

*Chemistry Standards Laboratory

Off-Site Presentations

Donald L. Ziegler. "Radioactive Waste Incineration Technology." Adapted from RFP-2216, RFP-2540, RFP-2655, and RFP-2733. ASME-Chicago. October 3-6, 1977.

Donald L. Ziegler and Andrew J. Johnson. "Fluidized Bed Incineration of Transuranic Contaminated Waste." RFP-2693. NEA-IAEA Meeting in Paris. December 5, 1977.

Andrew J. Johnson. "Radioactive Waste Incineration and Fixation." Adapted from RFP-2540, RFP-2655, and RFP-2733. USA-U.K. Exchange Meeting in Los Alamos. June 29, 1977.

Raymond T. Heizer and Carl Plock. "Reverse Osmosis to Achieve Water Control and Recycle." RFP-2476. Industrial Pollution Conference, St. Louis, Missouri. April 11, 1978.

Donald L. Ziegler. "A Review of Incineration and Waste Immobilization Technology Development at Rocky Flats." Adapted from RFP-2216, RFP-2540, RFP-2655, and RFP-2733. EG&G TRU Waste Processing Task Force, Denver, Colorado. May 24, 1978.

Ronald A. Harlan. "Experiences With a Counter for Plutonium in Crates." RFP-2738. ANS Topical Meeting on Analytical Methods for Safeguards and Accountability Measurement of Special Nuclear Material, Williamsburg, Virginia. May 16, 1978.

Ronald A. Harlan. "A Crate Counter for Normal Operation Loss." RFP-2642. Institute of Nuclear Materials Management Annual Meeting, Washington, D. C. June 29-July 1, 1977.

On-Site Presentations

"Radioactive Waste Incineration and Fixation" by Donald L. Ziegler, Andrew J. Johnson, David Anderson, and Lewis L. Richey is a compilation of information from three reports: RFP-2540, RFP-2655, and RFP-2733. This technical presentation, along with tours of the operating pilot plant

fluidized bed incinerator, residue immobilization system, and demonstration fluidized bed incinerator, was held for a large number of visitors since June 1977. The following list is of the visitors and organizations represented.

James A. Bowman, INEL-EG&G, June 16, 1977

Al Blasewitz, HEDL, June 30, 1977

Hershel Phares, Dan R. Chapman, H. Zafer Dokuzoguz and Thomas D. Kendrick, Babcock & Wilcox, July 5, 1977

Gary R. Simmons, Atomic Energy of Canada, Whiteshell Nuclear Research Establishment, July 6, 1977

Chuck Allen, HEDL-Hanford, August 15, 1977

Harry Houtman, SRL-DuPont, August 18, 1977

F. S. Goldberg, SRO-DOE, August 18, 1977

George White, T. B. Cooper, Hanford-Rockwell, September 27, 1977

P. Dudley Josselyn, FMC Corporation, September 27, 1977

Roy Barker, West/Hanford FMEF Project Fuel Material Examination Facility, February 13, 1978

David M. Rohrer, NRC-High Level, March 1, 1978

Angelo Martello, DOE-ALO Waste Management, March 1, 1978

Robert Grayson, LLL, March 1, 1978

G. Ortel, DOE Waste Management, March 10, 1978

R. Lowrey, DOE-ALO Waste Management, March 10, 1978

Bengt Edwall, Claes Thegerstrom, South Swedish Power Company, March 13, 1978

Jim Knight, Ken Oma, Hene Kosiacic, Hanford-Rockwell, March 14, 1978

Herbert Koscheorke, Erich Schneider, KFK
(Kalrsrutte), March 17, 1978

Si Fred Kaufmann, DWK-Hanover, March 17, 1978

Roy L. Post, University of Arizona, March 30, 1978

Dennis Pence, Jerry Christian, Precision Scientific
Company, April 6, 1978

Mike McCormick, EG&G, April 13, 1978

John VanCleve, ORNL, April 21, 1978

Don Lorenze, LMFBR-Fuelcycle, April 21, 1978

Chester S. Meaders, Robert H. Ford, ORNL,
April 27, 1978

Dr. Robert L. Nelson, Harwell, England,
May 11, 1978

Texas Chee, DOE Washington, May 16, 1978

Robert Lowrey, DOE-ALO Waste Management,
May 16, 1978

"Radioactive Waste Incineration and Refractory
Development" (RFP-2216), by Donald L. Ziegler,
Peter T. Faccini, Carl E. Plock, and Lewis L. Richey.
This technical presentation, along with tours of the
pilot plant rotary kiln incinerator, laboratory refrac-
tory work, fluidized bed incineration facilities, and
the waste immobilization system was held for the
following visitors:

Ralph Erickson, William Shaffer, DOE-SRO,
November 7, 1977

H. S. Campbell, R. A. Clark, H. E. Hootman,
B. H. Kirk, B. D. Helton, E. L. Monroe,
J. Scafaria, C. D. Spenser, E. Clapp, SRL-
DuPont, December 7, 1977

D. Boersma, E. L. Albenesuis, SRL-DuPont,
April 19, 1977

William Shaffer, Ralph Erickson, DOE-SRO,
August 19, 1977

D. Boersma, SRL-DuPont, April 20, 1978

RFP-2818