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RESEARCH AND ECOLOGY
SEMI-ANNUAL PROGRESS REPORT
JANUARY-JUNE 1972

CHEMISTRY RESEARCH AND DEVELOPMENT

M. A. Thompson

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DOW CHEMICAL U.S.A.
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401

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KILN INCINERATION

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Laboratory scale tests of a fluid bed waste incinerator concept with in situ neutralization of HCl have been performed. (PPS)

FILTER MEDIA PROCESSING

Studies were performed on the processing of HEPA filters for plutonium recovery. (PPS)

PLASTIC DRUM LINERS

A polyethylene waste drum liner with a screw-on lid has been developed and tested. (PPS)

UNCLASSIFIED CHEMISTRY RESEARCH AND DEVELOPMENT JOURNAL ARTICLES AND RFP’s FOR THE FIRST HALF 1972
RESEARCH AND ECOLOGY SEMI-ANNUAL PROGRESS REPORT
JANUARY-JUNE 1972

CHEMISTRY RESEARCH AND DEVELOPMENT
M. A. Thompson

PLUTONIUM INFORMATION
J. M. Cleveland

A 6-hour course in plutonium chemistry was prepared and presented to a total of 165 plant-site personnel during the first half of 1972. Two types of courses were presented: one for technically trained nonchemists; and one for non-technical personnel, such as foremen and Health Physics monitors, involved in plutonium handling. The course has been recorded on video tape for future presentations.

A "Plutonium Chemist's Periodic Table" has been compiled and published in collaboration with the Rocky Flats Technical Information Office. This chart contains all the elements in the conventional arrangement, and listed under each element, along with available physical data, are all the binary compounds the element forms with plutonium. The chart is color-coded to indicate those elements that do not react with plutonium, those that are slightly miscible, and those that alloy with plutonium but do not form discrete intermetallic compounds. Approximately 175 of these charts have been distributed in the 2 months since they became available.

PLUTONIUM SOURCE

There are many possible differences in the reactions of "soluble" forms of plutonium \([\text{Pu(OH)}_{x}^{-}\text{n}]\) and of insoluble forms of plutonium \([\text{PuO}_2]\) in the soil. Such differences include the physical reactions of the plutonium with soil particles, and differences in reactions with complexing agents present such as \(\text{CO}_3^{2-}\). Trying to identify these differences and determine their relative significance was futile. Therefore, both forms of plutonium will be used to determine distribution coefficients and elution characteristics on one or two soils. If there is an insignificant difference in the numerical values obtained, only \(\text{PuO}_2\) will be used as the plutonium source in subsequent work. If, however, there is a significant difference in the results, both sources of plutonium will be used in subsequent work.

Based on available information, it was determined that the mean particle diameter of the \(\text{PuO}_2\) used in this investigation should be in the range of 0.01 to 10\(\mu\). Particles of \(\text{PuO}_2\) this size were prepared by hydriding plutonium metal (to form \(\text{PuH}_2\)), then oxidizing the hydride to the oxide. The as-prepared oxide had a particle size distribution with 86% less than 10\(\mu\) in diameter. It was sieved using a 10-\(\mu\) "screen." The resulting material had a particle size distribution with 95% less than 10\(\mu\). This material will be used for the experimental work.

SOILS: MEASUREMENT OF PLUTONIUM TRANSPORT

Parameters available for measuring the movement of plutonium in soils include distribution coefficients, diffusion
coefficients, and elution characteristics. Methods for measuring these parameters are available in the literature. Most of these methods, however, were developed for measuring the movement of "free" ions and not relatively insoluble plutonium compounds in soil. As a result, the methods to be used will have to be selected with care.

The method being evaluated currently for determining distribution coefficients for the plutonium in the soil involves equilibrating 2 g of soil in 10 ml of water containing the required amount of plutonium. After equilibration, the soil and water are separated by centrifugation under conditions that will remove all particles 0.02μ or greater. The water will then be removed and analyzed for plutonium. Distribution coefficients will be calculated from these results.

SOILS: SELECTING, OBTAINING, AND CHARACTERIZING

The behavior of plutonium is being determined on many different types of soils. These soils have been carefully characterized, and a relationship will be obtained between these characteristics and the behavior of plutonium. The following criteria were established for the selection of the soils to be studied:

- A soil profile will consist of two parts — top soil (the top 8 to 10 inches) and underlying soil (soil about 2 feet down).

- Samples from 10 to 12 locations will be studied.

Based on these criteria, samples were obtained from the following sites: Los Alamos, New Mexico; Hanford, Washington, (two samples); Barnwell, South Carolina, (Allied-Gulf Nuclear Services); West Valley, New York, (Nuclear Fuel Services); Oak Ridge, Tennessee; Idaho Falls, Idaho, (Nuclear Reactor Test Site, four samples); Rocky Flats, Colorado (two samples). Top soil samples of approximately 10 pounds were obtained from each site. In addition, underlying samples of approximately 30 pounds were obtained from Idaho Falls and Hanford where plutonium-containing material is buried, or is known to be in the soil. A mineralogical analysis has been completed on all the soils. Rock fragments and liberated minerals were identified.

All the soils have been characterized physically and chemically by the Soils Testing Laboratory at Colorado State University. The results are summarized in Table 1.

ANALYTICAL METHODS: PLUTONIUM IN SOIL AND WATER

A number of methods have been evaluated for determining plutonium in the soil. A leach method was selected in which the plutonium is removed from the soil for analysis. Duplicate samples of five soils containing plutonium ranging from 0.59 to 193 d/m/g were leached by the usual

<table>
<thead>
<tr>
<th>Source</th>
<th>Location</th>
<th>Organic Matter (%)</th>
<th>CECb (meq/100 g)</th>
<th>pH</th>
<th>Saltsb (mmhos/cm)</th>
<th>Carbonate (meq/l)</th>
<th>Sand (%)</th>
<th>Silt (%)</th>
<th>Clay (%)</th>
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</thead>
<tbody>
<tr>
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<td>6.4</td>
<td>8.0</td>
<td>0.9</td>
<td>2.62</td>
<td>74</td>
<td>12</td>
<td>14</td>
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<tr>
<td></td>
<td>Underlying</td>
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<td>5.8</td>
<td>8.2</td>
<td>0.4</td>
<td>2.30</td>
<td>74</td>
<td>12</td>
<td>14</td>
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<tr>
<td>Rocky Flats A</td>
<td>Top</td>
<td>7.9</td>
<td>20.0</td>
<td>5.7</td>
<td>3.8</td>
<td>5.97</td>
<td>44</td>
<td>20</td>
<td>36</td>
</tr>
<tr>
<td>Rocky Flats B</td>
<td>Top</td>
<td>7.7</td>
<td>17.5</td>
<td>5.6</td>
<td>0.4</td>
<td>0.97</td>
<td>64</td>
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<td>22</td>
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<tr>
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<td>7.4</td>
<td>6.4</td>
<td>1.7</td>
<td>2.81</td>
<td>36</td>
<td>42</td>
<td>18</td>
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<tr>
<td>Barnwell</td>
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<td>2.9</td>
<td>5.4</td>
<td>0.4</td>
<td>0.53</td>
<td>78</td>
<td>2</td>
<td>20</td>
</tr>
<tr>
<td>West Valley</td>
<td>Top</td>
<td>8.1</td>
<td>16.0</td>
<td>5.4</td>
<td>1.2</td>
<td>1.36</td>
<td>32</td>
<td>38</td>
<td>30</td>
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<tr>
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<td>0.4</td>
<td>0.29</td>
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<td>32</td>
<td>36</td>
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<tr>
<td>Idaho Falls A</td>
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<td>15.5</td>
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<td>2.72</td>
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<td>24</td>
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<td>1.2</td>
<td>4.85</td>
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</table>

a CEC = Cation exchange capacity.

b Measured as conductivity.
method and the plutonium determined in the leachate. The soil residues from the leachings were then totally dissolved and their plutonium content determined. The mean recovery of plutonium by leaching was 83% with a standard deviation of 4%. However, the precision of the total plutonium content, as determined by duplicate samples, varied widely. On the five samples, the relative standard deviation ranged from 12 to 81%. This large variation can probably be explained by a nonhomogeneous distribution of the very small amount of plutonium in the soil. On a “standard” plutonium-containing soil from Brookhaven, New York, the relative standard deviation on six aliquants analyzed was 27%. The mean value of these six aliquants was 0.046 d/m/g; the accepted value is 0.042 d/m/g.

THERMODYNAMIC INVESTIGATIONS
F. L. Oetting and J. D. Navratil

DROP CALORIMETRY

The high temperature enthalpy of uranium monocarbide, uranium sesquicarbide, and uranium dicarbide has been determined by a copper-block calorimeter of the isoperibol type. The experimental data cover the temperature range to 1700°K relative to 298°K for the monocarbide and sesquicarbide, and to 1500°K relative to 298°K for the dicarbide.

The experimental data are expressed by the following equations:

\[
\begin{align*}
\text{UC:} & \quad H^0_T - H^0_{298} = -4975.5 + 14.390 \, T - 1.0695 \\
& \quad \times 10^{-4} \, T^2 + 4.0373 \times 10^{-7} \, T^3 \\
& \quad + 2.0391 \times 10^3 \, T^{-1} \, \text{cal/mol} \\
\text{C_p}^0 & = 14.390 - 2.139 \times 10^{-4} \, T + 12.119 \\
& \quad \times 10^{-7} \, T^2 - 2.0491 \\
& \quad \times 10^2 \, T^{-2} \, \text{cal/(°K mol)} \\
\text{U}_2\text{C}_3: & \quad H^0_T - H^0_{298} = 12642 + 36.003T - 5.7208 \\
& \quad \times 10^{-3} \, T^2 + 3.2950 \times 10^{-6} \, T^3 \\
& \quad + 6.9432 \times 10^3 \, T^{-1} \, \text{cal/mol} \\
\text{C_p}^0 & = 36.003 - 11.4416 \times 10^{-3} \, T \\
& \quad + 9.8850 \times 10^{-6} \, T^2 - 6.9432 \\
& \quad \times 10^2 \, T^{-2} \, \text{cal/(°K mol)} \\
\text{UC:} & \quad H^0_T - H^0_{298} = 5346.7 + 14.641T + 3.8060 \\
& \quad \times 10^{-3} \, T^2 - 6.6070 \times 10^{-7} \, T^4 \\
& \quad + 1.9690 \times 10^5 \, T^{-1} \, \text{cal/mol} \\
\end{align*}
\]

\[
\begin{align*}
\text{C_p}^0 & = 14.641 + 7.612 \times 10^{-3} \, T - 19.821 \\
& \quad \times 10^{-7} \, T^2 - 1.9690 \\
& \quad \times 10^5 \, T^{-2} \, \text{cal/(°K mol)}
\end{align*}
\]

If the heat capacity equations for \( \text{U}_2\text{C}_3 \) and UC are plotted (Figures 1 and 2, respectively), it is noted that there is a significant increase in the heat capacity as the upper decomposition temperature is approached. Such behavior is also found in many other compounds and elements at high temperature; however, the magnitude of the effect, especially in \( \text{U}_2\text{C}_3 \), is unusual.

An explanation for this effect is the creation of vacancies by the simultaneous removal of metal and carbon atoms. The energy, \( E \), for the formation of vacancies, can be found by applying the equation:

\[
\Delta H = E A e - \frac{E}{RT}
\]

where \( T \) is the temperature, \( A \) is a constant, and \( \Delta H \) is the excess enthalpy. An approximation for \( \Delta H \) was made by fitting the low temperature heat capacity data by a smooth curve, to give the solid heat capacity curves in Figures 1 and 2. After converting this to enthalpy and subtracting it from the least squares fit of the measured enthalpy, the excess enthalpy is obtained as a function of temperature.

According to the above equation, it was found that \( \text{U}_2\text{C}_3 \) and UC have energies of vacancy formation of 19.5 and
Figure 2. Heat Capacity Equations for UC.

23.4 kcal/mol, respectively. This corresponds to a vacancy concentration of 16% in each sublattice at the upper decomposition temperature for U$_2$C$_3$, and a vacancy concentration of 8% in each sublattice for UC at the melting point.

**SOLUTION CALORIMETRY**

An LKB reaction calorimeter used to measure the heats of solution of nuclear materials has been checked out by measuring the standard reaction of TRIS (NBS — Standard Reference Material 724) [TRIS = Tris (hydroxymethyl) aminomethane] with 0.1N HCl at 25°C. The calorimeter and mode of operation were altered in various ways to ensure reliability. For example, a separate electrical energy system was installed for comparison with the LKB system. The Regnault-Pfaundler and a modified Dickinson calculation method for the corrected temperature rise were also compared. The results from using a vented reaction vessel were compared to the results from using an unvented reaction vessel. The average enthalpy value for the TRIS reaction with 0.1N HCl was found to be 71.13 ± 0.5 cal/mol in good agreement with the certified value 71.16 ± 0.7 cal/mol, and also independent of the above-mentioned parameters.

Production operations currently use a chemical milling solution (750 ml/ℓ H$_3$PO$_4$, 31 ml/ℓ H$_2$SO$_4$ and 71 g/ℓ Cr O$_3$) to mill beryllium. Since temperature control of the milling operation is a necessary criterion, the enthalpy of solution of beryllium in milling solution was determined and found to be $-112.4 ± 2.8$ kcal/mol at 25°C. Concentration of beryllium was found to be independent (0.11 to 0.16 g/ℓ) of the heat of solution. Auxiliary heat capacity data indicated no significant change in the heat of solution at 70°C. This investigation on the chemical milling solution is being continued.

A comparable reaction of beryllium and H$_2$SO$_4$ solution was also investigated to determine the reliability of the calorimeter with very slow reactions, such as the milling solution. The following reaction,

$$\text{Be(c)} + 83.2 \text{H}_2\text{SO}_4 \cdot 3985 \text{H}_2\text{O} \rightarrow \text{BeSO}_4 \cdot 82.2 \text{H}_2\text{SO}_4 \cdot 3985 \text{H}_2\text{O}$$

has a measured heat of $-92.34 ± 0.22$ kcal/mol. This is in fair agreement with the reported value of $-92.83 ± 0.11$ kcal/mol. Combining our beryllium-sulfuric acid solution data with the heats of solution of the beryllium sulfates, we arrived at the following heats of formation at 25°C:

$$\begin{align*}
\Delta H_f &= -287.6 ± 0.2 \text{ kcal/mol} \\
\text{BeSO}_4 &- 435.3 ± 0.2 \text{ kcal/mol} \\
\text{BeSO}_4 \cdot 4\text{H}_2\text{O} &= -578.8 ± 0.2 \text{ kcal/mol}
\end{align*}$$

These values are in better agreement with the heats of formation of the beryllium sulfate as derived from hydrofluoric acid measurements than those obtained from other sulfuric acid studies.

**REFERENCES**

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CHEMISTRY INSTRUMENTATION

D. R. Cartwright

COMPUTER CONTROL FOR SAFEGUARDS INSTRUMENTATION

J. L. Lawless and H. N. Barton

Several radiometric counting systems are used in the plutonium chemistry recovery facility for safeguards and accountability purposes. These instruments are generally designed to measure the plutonium content in either 55-gallon drums of waste or 1-gallon packages of process residues by detecting gamma and neutron radiations. Complicated mathematical expressions, which are dependent upon the category of waste/residue, the particular counting system involved, net weight of the container, sample self-absorption, the gamma-ray and neutron data, and other variables, are used to arrive at a plutonium value. These calculations have been performed using the Rocky Flats IBM 360 computer facility, and usually 24 hours are required before the plutonium values are determined.

The need for immediate results, and the need to eliminate errors inherent in manually transcribing data, has led to the installation of a Hewlett Packard 2116-B computer in the chemistry recovery building. The computer has been designed to furnish on-line control for all the counting systems (see Figure 3). A mark card reader is located at each counting system where the operator, after inserting the sample in the counter, inputs the sample number, category, weight, etc. The computer then takes control of the counter and operates it through the counting cycle. After retrieving the counting data through appropriate interfacing equipment, the computer calculates the plutonium amount and prints the results on a teletype unit adjacent to the computer. Decisions can be immediately made concerning the sample disposition. The computer simultaneously punches a tape with all the sample information which then is sent to the IBM 360 computer for verification and the printing of the appropriate paperwork concerning the sample.

The first system to be brought under computer control has been the Helix Counter. The operation has been successful with only minor problems encountered. Plans now call for

Figure 3. Safeguards Computer Installation.
Can Counter II, the in-line Ash Leaching and Crusher Counters, and the Drum Counters to be interfaced to the computer.

REFERENCE


NEW WASTE-PACKAGING FACILITY
DRUM COUNTERS

R. N. Chanda, R. A. Deal, and J. L. Lawless

In the continuing program at Rocky Flats to utilize the best in radiometric safeguards instrumentation, two new drum counters are being constructed in the waste-packaging facility adjacent to the chemical operations area. When operational, these systems will replace the original drum counter, and the interim drum counting system built in 1971. Figure 4 depicts the physical layout of these new systems which will utilize three NaI(Tl) gamma-ray detectors and sixteen BF₃ neutron tubes.

Although similar in construction details, the two systems will be operated as two separate counters. One will be operated in a manner similar to systems presently in use; i.e., gamma-rays will be used for plutonium analysis and neutron/gamma ratios used for diagnostic information. However, only those drums with reasonably narrow n/γ ratios will be accepted by the system. It is estimated that greater than 90% of the waste drums can be analyzed successfully by this system. The remaining drums will be counted in the second drum counter which will be equipped with not only NaI(Tl) and BF₃ detectors, but also Ge(Li) detector(s), where a more detailed and lengthy analysis can be made. The operational philosophy of this second

Figure 4. Schematic View of Waste Packaging Drum Counters.
Can Counter II for gallon-sized packages of plutonium-bearing waste and scrap has been previously described and has been in production use approximately 6 months. This counter is standardized to assay materials in the following categories: ash, ash heel, graphite, graphite heel, insulation, soot, and glass. The accuracy is typically ±15% with a precision of 5%, which provides more accurate assays than were possible with the earlier system.

The computational program developed for this counter uses the gamma-ray data to calculate plutonium content, documents the raw and calculated data, and eventually prepares a universal transfer form for accountability records. Diagnostic tests and messages are included in the computer program to prevent errors caused by manual data handling and/or nontypical samples.

Future plans are to interface this counter to the Hewlett Packard 2116-B on-line computer, which will eliminate human errors in data handling, and provide instantaneous assay results.

REFERENCE

EXPERIMENTAL Ge(Li) BARREL COUNTER

H. R. Martin

Waste and scrap generated at Rocky Flats are segregated into various categories and assigned a code which identifies the matrix in the container. Because of differing matrix absorption effects in the various waste materials, encompassing approximately forty categories, a set of calibration standards representative of each unique type of waste is necessary. An investigation was conducted to determine the feasibility of obtaining matrix absorption corrections with a waste counting system equipped with a Ge(Li) gamma detector. Standards simulating combustibles, washables, scrap metals, and lead-glove categories were prepared with 0.5- and 1.0-gram PuO₂ samples sealed in small plastic vials. The vials were placed in the center of 1-gallon containers and surrounded by the matrix material. The gallon containers were placed on a turntable rotating at 4 rpm and a Ge(Li) detector was positioned 10 in. from the container edge. The maximum absorption of the 413 keV ²³⁹Pu line was about 60%.

The method used to obtain corrections was based on the relative attenuations of the 129- and 413-keV transitions from ²³⁹Pu. The absorption correction factor was obtained from an expression proposed by Cline. Results, with correction, were within ±10% of the 0.5-gram combustible, "no-absorption," mock standard. Additional improvement to ±5% can be obtained by using a varying empirical constant in the correction factor.

Because the results were obtained from a small number of samples, additional study is required. Present plans call for an in-depth investigation of 55-gallon drum standards from several categories using similar techniques. If the method continues to appear feasible, one of the new waste-packaging facility drum counters will be equipped to utilize it.

REFERENCE

PROGRAM FECS AS APPLIED TO COUNTER DESIGN FOR A NEW CHEMICAL PROCESSING BUILDING

R. A. Harlan, J. L. Lawless, and G. L. Kaes

A computer program names FECS (flux external to a cylindrical source) was written to aid design and evaluation of radiometric-assay counters. The program is quite flexible for the general problem of predicting the response of one or more detectors placed to the side of a cylindrical source. The response from lumps of course material in a cylindrical...
matrix, as well as the homogeneous-source problem, can be handled by the code.

Program FECS was applied to the design of in-line counters in a new chemical processing building which is being designed for Rocky Flats. Some of the counters in this building will be supported from the ceiling above approximately 1 ton of shielding per counter. Therefore, severe structural support problems associated with the production lines. Preliminary designs called for approximately 5 tons of shielding for each counter. Program FECS was applied to choose suitable compromises in the shielding design of the counters. The revised designs will require approximately 1 ton of shielding per counter. Therefore, severe structural support problems associated with the earlier designs have been minimized.

The counters under the present design will use two 2-in. by 2-in. NaI(1I) detectors. The detectors will be placed symmetrically along one side of the packages, which will rotate on an axis 40 centimeters from the face of the detectors. Lead collimators and shadow shielding will be used to conserve weight.

The system is now scheduled to be phased out and replaced by a more simple movable radiometric instrument which performs the same verification tests. This movable button verification counter has been constructed as shown in Figure 7.

In the new system, the 384-keV gamma-ray region will be detected with a 2-in. diameter by 1-in. thick NaI crystal. Four BF$_3$ neutron detectors are included in the design for further verification data since a given weight of plutonium will have a specific neutron emission. As with the Ge(Li) system, only 20 seconds of count are required. The new system has adequate shielding for counting in high background areas and can easily be moved to different locations where the canned plutonium is stored.

**REACTION GAMMA-RAY STUDY**

_H. R. Martin_

Generation of a catalog of reaction gamma rays, produced from alpha reactions on light elements, has been continued. These spectra allow identification of potential light element interferences in passive waste/scrap assay systems and can be useful in correcting results. In addition, they can sometimes be used to determine the chemical form or purity of plutonium compounds, or to follow chemical reactions involving plutonium and light elements.

Previously, spectra were obtained from plutonium compounds, alloys, and waste materials. Data have now been accumulated from PuO$_2$ mixtures with compounds containing fourteen light elements from $^3$Li to $^{19}$K. Neutron measurements were also carried out on each of the samples which contained 20 grams compound dry-mixed with 5 grams PuO$_2$. A summary based on preliminary analyses of these data is given in Table 2.

The spectra obtained in this study have proven useful in explaining anomalous neutron and gamma-ray counts observed in waste/scrap material. Information has also been provided which aids considerably in understanding potential pitfalls of present assay systems and in designing new systems.

**REFERENCE**

Figure 5. Plutonium Button Containing Trace Fission Product.
Figure 6. Gamma Ray Spectrum—2.3 KG Oralloy.
Figure 7. Movable Button Verification Counter.
RFP-1921-A

Table 2. Summary of Reaction Data.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Neutron Ratio ((20 \text{ grams})) (\text{mixture/PuO}_2)</th>
<th>Major Reaction Gamma Ray ((\text{keV}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\text{Li}_2\text{CO}_3)</td>
<td>1.58</td>
<td>479</td>
</tr>
<tr>
<td>(\text{BeO})</td>
<td>71.5</td>
<td>4430</td>
</tr>
<tr>
<td>(\text{H}_3\text{BO}_3)</td>
<td>9.43</td>
<td>3854</td>
</tr>
<tr>
<td>Graphite</td>
<td>1.14</td>
<td>None detected</td>
</tr>
<tr>
<td>Urea</td>
<td>1.01</td>
<td>971</td>
</tr>
<tr>
<td>((\text{NH}_4))_2\text{HPO}_4</td>
<td>1.03</td>
<td>871</td>
</tr>
<tr>
<td>(\text{CaF}_2)</td>
<td>10.81</td>
<td>1275</td>
</tr>
<tr>
<td>(\text{Na}_2\text{CO}_3)</td>
<td>2.36</td>
<td>1809</td>
</tr>
<tr>
<td>(\text{MgSO}_4)</td>
<td>1.43</td>
<td>1779</td>
</tr>
<tr>
<td>(\text{Al}_2\text{O}_3)</td>
<td>1.48</td>
<td>2235</td>
</tr>
<tr>
<td>(\text{SiO}_2)</td>
<td>1.08</td>
<td>None detected</td>
</tr>
<tr>
<td>((\text{NH}_4))_2\text{HPO}_4</td>
<td>1.03</td>
<td>2127</td>
</tr>
<tr>
<td>(\text{Sulfur Pu Powder})</td>
<td>0.94</td>
<td>1220</td>
</tr>
<tr>
<td>(\text{CaCl}_2)</td>
<td>0.98</td>
<td>2167</td>
</tr>
<tr>
<td>(\text{K}_2\text{CO}_3)</td>
<td>1.03</td>
<td>1547</td>
</tr>
</tbody>
</table>

*Contains 133 ppm \(^{241}\text{Am}\).

RADIOMETRIC ASSAY OF MOLTEN SALT RESIDUES

R. N. Chanda, R. A. Deal, and J. I. Maes

Molten salt residues are the by-product of a production process for extracting americium from plutonium metal. Presently the plutonium content of these residues is determined by a weight difference of the plutonium button before and after extraction. A preliminary study using a Ge(Li) detector for the assay of these residues was undertaken.

Calibration data were obtained from cans containing 1400 to 1500 grams of molten salt material to which 20- to 50-grams \(\text{PuO}_2\) and 2-grams \(\text{Am}_2\text{O}_3\) in vials could be added. Samples were positioned on a turntable 18 in. from a 9% efficient Ge(Li) detector during counting. The very intense low energy \(^{241}\text{Am}\) gamma-ray emissions were reduced by a 0.25-in. cadmium absorber. Assay was based on the integrated response of the 413 keV gamma-ray emission of \(^{239}\text{Pu}\). This particular line was chosen because of lesser interferences from \(^{241}\text{Am}\). Counting times were 800 seconds.

Eight cans of molten salt residues containing 16- to 45-grams plutonium were assayed using a Ge(Li) gamma detector. The plutonium contents as determined by weight differences and Ge(Li) assay are shown in Table 3. Four of the eight cans were within \(\pm 10\%\) of the by-difference values.

Table 3. Results of Preliminary Molten Salt Ge(Li) Assay.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>By-Difference ((\text{grams}))</th>
<th>Ge(Li) Assay ((\text{grams}))</th>
<th>Difference ((%))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>33</td>
<td>32.5</td>
<td>-1.5</td>
</tr>
<tr>
<td>2</td>
<td>23</td>
<td>23.3</td>
<td>+1.3</td>
</tr>
<tr>
<td>5</td>
<td>45</td>
<td>44.3</td>
<td>-1.6</td>
</tr>
<tr>
<td>14</td>
<td>20</td>
<td>71.1</td>
<td>+8.6</td>
</tr>
<tr>
<td>9</td>
<td>16</td>
<td>32.6</td>
<td>+104</td>
</tr>
<tr>
<td>13</td>
<td>18</td>
<td>30.4</td>
<td>+69</td>
</tr>
<tr>
<td>15</td>
<td>23</td>
<td>28.8</td>
<td>+25</td>
</tr>
<tr>
<td>17</td>
<td>23</td>
<td>27.1</td>
<td>+18</td>
</tr>
</tbody>
</table>

The plutonium content of the remaining four cans will be assayed after chemical recovery.

Some explanations for the apparent large differences in four of the samples may be offered. During the extraction process, plutonium is known to build up on the stirrer of the molten salt extraction apparatus. In an individual extraction run, an answer may be high or low depending upon whether plutonium is adhering to the stirrer or whether plutonium is sluffing off from the stirrer. The same comments must necessarily apply to the extraction vessel, which is a 4\(\frac{5}{8}\) in. diameter by 10-in. high tantalum crucible.

This initial and preliminary study has not resulted in a definitive answer as to whether a radiometric assay is any better than the by-difference method presently used. Further work is needed and is under way.

SAFEGUARDS PERSONNEL MONITOR

J. L. Martinez and G. J. Cunningham

The safeguards personnel monitor described previously\(^1\) has been installed temporarily in a Rocky Flats production area guard post to gain experience with personnel traffic flow and to test the instrument reliability under actual operating conditions. It has been found that about 30 persons per minute can pass through the monitor which gives a problem of personnel delays only at shift change time. Where higher traffic conditions exist, multiple installations are now planned for Rocky Flats. Electronic design modifications, which will speed up the circuit response without sacrificing sensitivity, are in laboratory testing. If these modifications are successful, a personnel throughput of 60 or more per minute may result.
The monitor performance has been reliable, and no problems have been encountered. A slight de-sensitization was found necessary to prevent alarming on an occasional "hot" wrist watch. Efforts are being directed toward establishing the installation designs necessary for each of the guard posts at production areas. Hardware costs per installation have remained slightly below $5,000.

REFERENCE


IN-LINE IRON ANALYSIS

J. Benallo and C. M. Johnson

Investigation of in-line iron detection and analysis in a plutonium nitrate solution has been conducted. A vendor designed and built valve extracts a 1.5-ml sample of solution from the flowing stream. This sample is then passed through a small Cheminert ion exchange column and eluted with 0.35N HNO₃ solution flowing at a constant rate. Selected impurities such as iron are held up on the column and eluted at different rates. The eluate is passed through a Chromatronix MCC-75 microflow cell. The conductivity of the solution is measured as a function of time and read out on a Chromatronix CM-1A conductivity meter with an attached chart recorder. As each impurity is eluted at different times, the iron conductivity peak can be identified by proper calibration.

Sensitivity measurements in progress show excellent results both for the higher and lower concentrations of iron. The high concentration range is from 500 ppm to 2000 ppm, and the low from 10 ppm to 100 ppm. With the introduction of a newly designed temperature stabilization chamber, which houses the microflow cell, sensitivities are expected to reach the lower limits of 1- to 10-ppm iron concentration in a 0.35N HNO₃ solution.

MAGNETIC SCANNER FOR PLUTONIUM BRIQUETTES

J. Benallo, G. J. Cunningham, and J. L. Martinez

An instrument to detect the presence of para- or ferromagnetic materials in plutonium briquettes has been developed, and a unit suitable for glovebox installation has been constructed. The instrument achieves its sensitivity by using a Hall effect probe which detects aberrations in a magnetic field surrounding the briquette when para- or ferro-magnetic inclusion impurities are present. In laboratory experiments using a 1500-gram depleted uranium briquette as a stand-in for plutonium, 200-mg quantities of stainless steel could be detected at any location in the briquette. The sensitivity for iron is much greater. The entire scanning of a briquette can be accomplished in 30 seconds with an operator being required to push only one button. A logic circuit then controls the scanning. An audible signal indicates rejection of the briquette for impurities. Glovebox installation is scheduled in the near future for the magnetic scanner.

NASH PUMP CONTROL SYSTEM

D. E. Hausburg

Modifications have been made to the Nash pump control system briefly described earlier. Nash pumps are used in the plutonium processing areas to produce vacuum for transferring various solutions. A liquid level controller, with an associated valve to control the caustic level in the system mist tank, has been installed. The system has operated successfully for 3 months. The control panel for a second Nash pump system has been redesigned to accommodate the high level back-up alarm. Printed circuit boards with miniature plug-in relays, and a miniature plug-in time delay relay are used to facilitate maintenance problems.

Automatic shutdown of the pump will be caused by high outlet temperature of the caustic recirculation line, low cooling water pressure to the heat exchangers, and low bearing seal water pressure. Prevention of caustic from entering the vacuum system is accomplished with the time delay relay and a solenoid operated valve. Audible annunciators and warning lights are included in the control panel. A schematic diagram of the installation is shown in Figure 8.

REFERENCE

HYDROGEN ALARM SYSTEM

C. M. Johnson, D. E. Hausburg, and Y. Botello

Two systems for monitoring hydrogen gas concentrations have been assembled. One system is for chemistry milling operations where hydrogen gas evolves from the process, and the other system is for an acid spray process where hydrogen evolves from the cleaning of metal.

Hydrogen detection instruments with two (General Monitors, Inc.) sensors each enable the user to measure hydrogen in two locations simultaneously. The range of detection is 0 to 4%. An in-house-designed alarm system produces a warning light and audible warning at two separate and distinct levels. A condition of 1.2% hydrogen concentration is indicated by a yellow light and a steady audible warning signal. The higher alarm set-point of 2.4% hydrogen concentration is indicated by a red light and a pulsating audible warning signal. The Reset for each audible warning signal is provided by two pushbuttons.

REMOTE HANDLING

R. J. Nau, F. L. Crawford, and L. H. Faber

Four improved cannister-type filters designed to eliminate sealing problems while reducing change time have been installed in process streams in the plutonium recovery facility. Operation in acid streams is quite satisfactory, while the filters in the caustic streams are generally unsatisfactory for removing particles below 20 microns. Six more of these filter cannisters are being built for evaluation.
Fluidic devices have been utilized to develop a level sensor which warns of glovebox liquid levels prior to criticality drain overflows. The device will trigger on less than 0.1 in. of liquid level. It is less expensive than existing capacitance probes and has been successfully used in a production operation. A similar device has been used to control the high level on a caustic scrubber recirculation system. Another controller has been built using fluidic devices to shut off the caustic recirculation pumps in case of overflow to the glove box.

A manipulator arm being considered for use in the new plutonium recovery facility was tested and proved unsatisfactory in its designed configuration. Modifications were made to the cylinders and satisfactory operations were performed. However, an arm positioning device did not operate as expected because of erratic positioning. This was due to the varying load and the necessity to vary the supply air pressure as the load varied. Weight feedback would be necessary to approach satisfactory results but would not guarantee successful operation.

STACK EMISSIONS MONITORS

C. M. Johnson

In a continuing effort to control possible sources of pollution, originating within the plant, a program has been started to investigate all the stacks on the plant site. Eighty-eight buildings are being surveyed, and every roof penetration is being identified on roof prints. The identification of all roof penetrations has revealed some sources of pollution that were previously overlooked. When the survey is complete, a list of priorities will be established and major pollutants will be measured.

Instrumentation to quantitatively measure the pollutants is being investigated. It appears that an air quality chromatograph and an NO₂—SO₂ analyzer will measure the majority of the pollutants present. An NO₂, NOₓ analyzer is presently being utilized to check the efficiency of NO₂ scrubbers in several locations.

The successful completion of this program will give an accurate active control on possible pollutants leaving the plant site.

MOBILE AMBIENT AIR MONITOR

C. M. Johnson, D. E. Hausburg, and J. L. Lawless

As ambient air quality becomes increasingly important, it is essential that the quality of the air be known at each stationary emitter. A program has been started that will determine the quality of the ambient air within the Rocky Flats plant site and in the surrounding area. Plans have been completed to procure a van-type mobile air monitoring facility. Instrumentation will be provided to analyze all the pollutants specified in the proposed 1975 ambient air regulations. Space will also be provided in the van to perform wet chemical tests for the same compounds. Additional space is provided for radiometric type analyzers to be used in emergency situations. Equipment and instrumentation is being evaluated for placement in the van.

COMPUTER SPECTROCHEMICAL CALCULATIONS

H. N. Barton

FORTRAN programs for time share computer use were developed and are in use by the General Analytical and Plutonium Analytical Laboratories for the calculation of impurity concentrations in beryllium and plutonium determined by densitometric photographic emission spectroscopy. The computer calculation replaces, but is similar to, a graphical calculation method converting transmittance of photographically recorded emission lines to intensity by a Seidel Function emulsion calibration. Intensities are ratioed to an internal standard, and concentrations for each element are determined from an analytical working curve. Additionally, the computer method presents recovery factors, for each element, from an analysis of control-sample aliquots. Factors are recalculated following selective discard by element and aliquot, and optionally applied to sample calculations for selected elements. The averaging of aliquots and optional discard of sample aliquots is provided.

Alloyed and nonalloyed plutonium samples are calculated by the same program by providing separate analytical working curves, known values for control samples, and lower and upper reporting limits. Two internal standards and emulsion calibration curves are used for both alloyed and unalloyed plutonium.

Analytical working curves are represented by up to 14 linear segments of log concentration as a function of log intensity ratio. These curves are readily changed by replacement of concentration and intensity ratio values in a data file.

REFERENCE

FLUIDIZED PARTICLE CONVERSION OF PLUTONIUM OXIDE TO PLUTONIUM TETRAFLUORIDE

R. L. Standifer, R. O. Wing, and J. R. Sheets

The plutonium recovery and purification plant processes various forms of plutonium scrap and metallurgical wastes to yield a purified metal product. After the plutonium is purified, usually as plutonium nitrate [Pu(NO₃)₄] solution, it is precipitated using hydrogen peroxide (H₂O₂). The precipitate is dried, and converted to plutonium oxide (PuO₂). The purified oxide is fluorinated to plutonium tetrafluoride (PuF₄) using hydrogen fluoride (HF) in a rotary tube reactor. The PuF₄ is subsequently reduced to plutonium metal by a calcium bomb reduction. Certain undesirable features of the rotary tube hydrofluorinator prompted an interest in a fluid-bed fluorine system as a possible substitute.

A process using fluorine and fluid-bed reactors to convert purified PuO₂ to PuF₄ has been developed and is included in the flow sheet for a new plutonium recovery facility. One of the advantages of the fluid-bed fluorine system relative to the system presently used is a potential reduction in the radiation exposure to operating personnel.

Based on an extended series of pilot-plant-demonstration runs, it was estimated that, with a well-designed system, exposure of the operators for this process can be reduced by 90% because of: (1) the lower PuF₄ inventory requirements of the fluid-bed fluorine process (2 kg/reactor compared to 10 to 16 kg in the hydrofluorination reactor), and (2) the relative ease of operating the fluid bed process remotely. The exposure levels for the production-scale fluid-bed fluorine system were calculated from time study and radiation determinations taken during the operation of the pilot plant. They were based on the following assumptions:

1. Three 2-kg batch reactors, requiring 4 hours/batch, will process 36 kg/day plutonium.
2. Individual reactors are isolated with sufficient shielding so that an operator would not receive exposure from adjacent reactors while working in a reactor cell.
3. No more than 1 kg residual plutonium (as PuF₄) will be present in each reactor cell.
4. Water shielding, 4-in. thick, will be installed on the operating side of each reactor glovebox.
5. The three reactors will be operated by two operators per shift. Each operator will spend no more than an average of 30 minutes per day working through gloves in the reactor cell area.
6. All routine operations will be performed from a well-shielded control room area, so that operators receive no significant radiation in normal operation.

All the above assumptions are considered realistic.

A suggested layout of a production fluid-bed fluorine system, which would satisfy these requirements, is shown in Figure 9.

REFERENCES


RECOVERY AND PURIFICATION OF PLUTONIUM BY A VOLATILE FLUORIDE PROCESS

R. L. Standifer, R. O. Wing, and J. R. Sheets

The development of a volatile fluoride process for the purification and recovery of plutonium has progressed from gram-scale laboratory experiments to the design and installation of a production-scale prototype system. The prototype system is scheduled to begin operation during the latter half of 1972. It is designed to provide a throughput capacity of 12 kg/day plutonium, using impure plutonium oxide (PuO₂) or plutonium tetrafluoride (PuF₄) as feed. The separation of most of the common
imperfections is attained with the volatile fluoride process by converting the nonvolatile plutonium compounds to volatile (b.p. -61°C) plutonium hexafluoride (PuF₆) using fluorine. Since most of the common impurities do not form fluorides which are volatile at moderate temperatures, they remain in the reaction vessel. The PuF₆ gas and unreacted fluorine are removed and the PuF₆ is condensed. The PuF₆ is subsequently reduced to PuF₄ by a thermal decomposition step.

Because PuF₆ and fluorine are hazardous materials, extensive safeguards to prevent their misuse have been incorporated in the prototype system design. Following is a description of some of the safeguards provided to prevent injury, and to minimize the possibility of damage to equipment or the release of contaminants.

**FLUORINE DETECTORS.** Low concentration (2 ppm) fluorine detectors are provided to continuously monitor the exhaust air from the gloveboxes and the fluorine supply building. Alarms, incorporated in the detectors, will be activated if significant fluorine leakage occurs.

**JACKETED TRANSFER LINES.** Fluorine supply lines, process piping, and pneumatic transfer lines, located outside of gloveboxes are jacketed and sealed where necessary. To provide added protection of some lines, the annular spacing between primary piping and jacketing is pressurized with inert gas.

**FLUORINE SUPPLY BUILDING SAFEGUARDS.** Fluorine is supplied to the process from two manifolds, each of which collects fluorine from 18 gas cylinders. The two supply manifolds are located in separate rooms of the supply building. Valve operation is arranged so that personnel need never enter the rooms while the manifolds are pressurized with fluorine. A slight negative pressure is
maintained in the manifold rooms and the exhaust air is passed through charcoal traps before exiting through a 40-foot-high stack.

WATER SCRUBBERS. The exhaust air from the five process gloveboxes will be contacted with water in recirculating scrubbers before passing to the main filter plenum. This should ensure hydrolysis of any fluorine or plutonium hexafluoride released if a rupture of the process equipment should occur.

OVERPRESSURE PROTECTION. Items of equipment, in which accidental overpressurization is possible through operator error or equipment malfunction, are protected by high pressure alarms and by rupture disks. The rupture disks vent through a trapping train should excessive pressures occur.

EXTERNAL SPRAY SYSTEM. The room sprinkler system, which is automatically activated by a rise in temperature, can be turned on manually in the event glovebox integrity is lost. This will minimize the spread of airborne contamination.

ALARMS, TRIPS, AND INTERLOCKS. Automatic alarms, safety trips, and interlocking control systems have been used extensively throughout the system to warn of impending unsafe conditions, and to prevent operating errors which could result in unsafe situations.

IGNITION TESTS. All materials used in the gloveboxes were tested to determine flammability in a fluorine atmosphere. All materials which ignited under test conditions (100% fluorine, 50°C) were removed or auxiliary protection was provided.

PERSONNEL TRAINING. A detailed description of the process and an operating procedure has been prepared as a guide for the training of operating personnel. The operating procedure includes itemized start-up, operating, and shut-down checklists; specific emergency procedures; and a list of important “do’s” and “don’ts.” A safety manual will be written, and a further safety analysis study will be performed prior to start-up. Because the volatile fluoride process utilizes techniques and materials which are not presently used in the Rocky Flats plutonium recovery process, the systematic and complete training of operating personnel is of utmost importance for the safe operation of the system.

REFERENCES


2. Ibid.


RECOVERY OF PLUTONIUM FROM HIGH SALT WASTE SOLUTIONS CONTAINING LOW LEVELS OF PLUTONIUM USING ION EXCHANGE METHODS.

G. H. Bryan and D. A. Burton

The plutonium recovery and purification facility at Rocky Flats generates large volumes of waste solutions containing 1 to $10^{-3}$ molar concentrations of such impurities as calcium, iron, silicon, and aluminum, as well as $10^{-4}$ to $10^{-6}$ molar ($10^{-2}$ to $10^{-4}$ g Pu/liter) concentrations of plutonium. At the present time these solutions are shipped from the chemical processing building to the waste processing building where a flocculating agent-carrier precipitation step is performed to lower the plutonium concentration to $10^{-8}$ to $10^{-10}$ molar ($10^{-6}$ to $10^{-8}$ g Pu/liter). This precipitate is filtered and sent to Idaho for burial.

These procedures have been adequate in the past; however, in anticipation of future requirements, development has begun on methods to recover the bulk of the plutonium presently sent to burial. Based on existing technology and equipment availability, ion exchange methods were chosen for the initial evaluation. Representative waste streams chosen for the initial investigations included ion column effluent, peroxide filtrate distillate, and caustic scrub solutions.

DOWEX® A-1

Dowex A-1 chelating ion exchange resin was found to remove plutonium quite effectively (from $10^{-6}$ to $10^{-9}$ g Pu/liter) on a laboratory scale after the waste stream was adjusted to
a pH of 4.0 to 4.1. The pH adjustment caused most of the iron, silicon, and aluminum present to precipitate and "carry" some of the plutonium. Filtration was therefore necessary prior to ion exchange treatment. Over 70% of the plutonium originally present was found to have been removed during the filtration procedure.

A small pilot plant was built to determine the feasibility of removing plutonium on a larger scale by Dowex A-1. The column used was 3 in. in diameter and 3 feet in length. A second column filled with activated charcoal was also used to act as a filter (see Figure 10). The results of the first four runs using this pilot plant were not as successful as the laboratory scale experiments. Resin shrinkage caused channeling within the resin column while using an upflow system during the first run. Uniform contact of the solution with the resin bed was, therefore, not achieved. A downflow system was used for the next three runs to alleviate the problem. Control of solution pH while passing through the resin column was also quite difficult. The results are shown in Table 4.

**DOWEX® 1**

In conjunction with and relating to the Dowex A-1 evaluation as a means of removing plutonium from waste streams, another project is also underway to evaluate the effectiveness of Dowex 1 for "waste polishing."

This system is being designed into the new plutonium recovery complex. Dowex 1 is used for this purpose because plutonium forms an anionic complex in strong nitric acid solutions, whereas most other metals do not. The use of a strong base resin (Dowex 1) therefore allows plutonium to be effectively concentrated and separated from most metallic impurities. A series of laboratory tests is presently being conducted to determine the ability of this resin to remove plutonium from high salt content streams with plutonium levels of $10^{-4}$ to $10^{-7}$ molar ($10^{-2}$ to $10^{-5}$ g Pu/liter). Preliminary results, shown in Table 5, indicate that effluent levels of $10^{-7}$ to $10^{-8}$ molar ($10^{-5}$ to $10^{-6}$ g Pu/liter) may be achieved. One run was made using the pilot plant described previously without a charcoal filter.
Table 4. Dowex® A-1 Ion Exchange Resin Pilot Plant Results.

<table>
<thead>
<tr>
<th>Volume in Feed (liters)</th>
<th>Pu Conc in Feed (g/l x 10^-7)</th>
<th>Avg Pu Conc in Resin Effluent (g/l x 10^-7)</th>
<th>Avg Pu Conc in Charcoal Effluent (g/l x 10^-7)</th>
<th>Decontamination Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Run I</td>
<td>72.0</td>
<td>1.7</td>
<td>1.8</td>
<td>7.7</td>
</tr>
<tr>
<td>Run II</td>
<td>72.0</td>
<td>1.4</td>
<td>0.94</td>
<td>1.0</td>
</tr>
<tr>
<td>Run III</td>
<td>96.6</td>
<td>62.7</td>
<td>12.3</td>
<td>4.9</td>
</tr>
<tr>
<td>Run IV</td>
<td>98.0</td>
<td>73.5</td>
<td>11.4</td>
<td>2.5</td>
</tr>
</tbody>
</table>

Note: Flow rates were varied from 0.5 to 1.5 gal/min/sq. ft. of resin; however, little difference was noted in the decontamination factors between any given flow rates.

Table 5. Laboratory Test Results Using Dowex 1® Ion Exchange Resin.

<table>
<thead>
<tr>
<th></th>
<th>Pu (g/liter)</th>
<th>Am (g/liter)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RUN I</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Feed</td>
<td>2.87 x 10^-4</td>
<td>3.24 x 10^-4</td>
</tr>
<tr>
<td>Effluent</td>
<td>5.5 x 10^-6</td>
<td>3.01 x 10^-6</td>
</tr>
<tr>
<td>Decontamination Factor</td>
<td>52.2</td>
<td>1.0</td>
</tr>
<tr>
<td>RUN II</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Feed</td>
<td>1.32 x 10^-3</td>
<td>3.23 x 10^-4</td>
</tr>
<tr>
<td>Effluent</td>
<td>2.2 x 10^-5</td>
<td>3.27 x 10^-4</td>
</tr>
<tr>
<td>Decontamination Factor</td>
<td>60.0</td>
<td>1.0</td>
</tr>
<tr>
<td>RUN III</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Feed</td>
<td>1.02 x 10^-3</td>
<td>7.93 x 10^-4</td>
</tr>
<tr>
<td>Effluent</td>
<td>2.6 x 10^-5</td>
<td>7.60 x 10^-4</td>
</tr>
<tr>
<td>Decontamination Factor</td>
<td>39.2</td>
<td>1.0</td>
</tr>
<tr>
<td>RUN IV</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Feed</td>
<td>2.45 x 10^-4</td>
<td>1.01 x 10^-3</td>
</tr>
<tr>
<td>Effluent</td>
<td>1.4 x 10^-6</td>
<td>9.80 x 10^-4</td>
</tr>
<tr>
<td>Decontamination Factor</td>
<td>175.0</td>
<td>1.0</td>
</tr>
</tbody>
</table>

This run indicates plutonium levels of 10^-9 molar (10^-7 g Pu/liter) may be achieved. These results are shown in Table 6.

Results to date indicate Dowex 1 is a better ion exchange resin than Dowex A-1, for recovering plutonium from waste streams, because it does not require the close pH control or filtration. Future studies will concentrate on impurity variations in waste streams and their effect on the ability of Dowex 1 to remove plutonium.

**PRECIPITATION OF PLUTONIUM PEROXIDE**

_F. J. Miner, P. G. Hagan, and P. A. Glover_

Precipitation of plutonium from a nitric acid medium by the addition of hydrogen peroxide (H_2O_2) to form a non-stoichiometric plutonium peroxide is a basic unit operation in the plutonium recovery and purification process at Rocky Flats. The operation accomplishes two objectives: a liquid-to-solid conversion and concentration; and a purification from several impurity elements, notably americium.

The initial step of this unit operation is adjustment of the plutonium-nitric acid solution to a HNO_3 concentration of approximately 3.9M to 4.0M and a sulfate ion concentration of approximately 0.04M. The precipitation and subsequent filtration is presently accomplished using a H_2O_2 addition pot and a cascade of digestion pots, followed by a continuous rotary drum filter. The wet but solid product is scraped from the wheel in lots of approximately 2000 grams and subsequently transferred to a continuous rotary tube dryer where it is converted to a purified plutonium dioxide (PuO_2) form.

The flow sheet for the New Plutonium Recovery Facility (NPRF) calls for essentially the same chemistry of precipitation, except that no sulfate ion will be added in the initial batching operation. The objective here is to reduce equipment corrosion in subsequent operations. In addition, the equipment design criteria calls for batch rather than semi-continuous operation. An evaluation of certain equipment and operating parameters was therefore required.

Table 6. Pilot Plant Results Using Dowex 1® Ion Exchange Resin.

<table>
<thead>
<tr>
<th>Volume in Feed (liters)</th>
<th>Pu Conc in Feed (g/l x 10^-7)</th>
<th>Avg Pu Conc in Resin Effluent (g/l x 10^-7)</th>
<th>Decontamination Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Run I</td>
<td>7.0</td>
<td>411</td>
<td>1.7</td>
</tr>
</tbody>
</table>

Notes: 1. Flow rate was 0.5 gal/min sq. ft. of resin. 2. Feed solution was adjusted to 7.2N HNO_3.
FACTORS AFFECTING THE PRECIPITATION OF PLUTONIUM PEROXIDE

The plutonium peroxide precipitation process has been troubled intermittently by two problems. The first is filterability: at times the precipitate is slimy and difficult to filter. The second is incomplete precipitation of the plutonium: at times a significant fraction of the plutonium has to be recycled back through the precipitation process.

A number of variables are involved in the precipitation of plutonium peroxide. Six of these which were thought to be the most important were selected for investigation. The objective of this investigation was to identify those variables that did affect the precipitation of the plutonium peroxide, rank these variables in order of their effect, and then select levels for each of the variables that would give an optimum precipitation of plutonium peroxide.

The variables that were investigated, and the levels at which they were investigated, are shown in Table 7. An experimental design consisting of a fractional mixed factorial of five variables at three levels, and one variable at two levels, was used in the investigation. The experimental results were measured in terms of relative filtration time and the concentration of plutonium in the filtrate. The relative filtration time was the time in seconds required to filter a given quantity of plutonium peroxide slurry through a 1.5μ Millipore filter compared to the time required for a given volume of water.

The data obtained from the experiments were used to develop an equation that related either the relative filtration time or the plutonium concentration in the filtrate to the six variables investigated. This equation was used to calculate, via a computer, relative filtration times and plutonium concentrations in the filtrate for a number of combinations of the variables that were not determined experimentally. From these data, graphs were drawn, again via the computer, which showed the effect of changes of the six variables on either the relative filtration time or the plutonium concentration in the filtrate.

The variables that have a major effect on the plutonium peroxide precipitation were identified in two ways. The first involved a subjective evaluation of the relative importance of each of the variables based on a visual comparison of the computer-drawn graphs of the experimental and calculated data. The second involved the use of t-test values. The comparative magnitude of these values indicates the relative importance of the variables.

The results indicate that only the nitric acid concentration and the rate of hydrogen peroxide addition have a major effect on the relative filtration time. The other four variables influence the relative filtration time, but to a lesser extent. The nitric acid, hydrogen peroxide, and impurity concentrations, have the major effects on the plutonium concentration in the filtrate. The other three variables have lesser effects.

Unfortunately, a change in one variable can have a desired effect on the relative filtration time, but an undesirable effect on the plutonium content of the filtrate. This is the situation with nitric acid. Increasing the nitric acid concentration improves the filterability of the precipitate but at the same time increases the plutonium content of the filtrate. This same type of situation exists, but to a lesser degree, for all the other variables except the digestion time and the impurity concentration.

Based on the t-test data, the variables that are most important to the relative filtration time are the nitric acid concentration and the rate of hydrogen peroxide addition. The variables most important to the plutonium concentration in the filtrate are the nitric acid, hydrogen peroxide, and impurity concentrations. Therefore, the t-test data are in general agreement with the subjective evaluation and indicate that the variable that is most important to the relative filtration time is the nitric acid concentration. This is followed by the rate of hydrogen peroxide addition. The remaining four variables are of less importance, and, in fact, are not statistically significant.

Two of the variables have about equal importance as far as the plutonium concentration in the filtrate is concerned. They are the nitric acid concentration and the hydrogen peroxide concentration. These are followed in order of

Table 7. Variable and Levels Used in Plutonium Peroxide Precipitation Investigation.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>HNO₃ concentration</td>
<td>1.8, 2.5, and 3.1M</td>
</tr>
<tr>
<td>H₂O₂ concentration</td>
<td>2.9, 3.9, and 4.9M</td>
</tr>
<tr>
<td>Impurities</td>
<td>Present and absent</td>
</tr>
<tr>
<td>Digestion time</td>
<td>30, 60, and 90 minutes</td>
</tr>
<tr>
<td>Rate of H₂O₂ addition</td>
<td>1.8, 1.2, and 0.6 ml/minute</td>
</tr>
<tr>
<td>Temperature</td>
<td>22°, 14°, and 6°C</td>
</tr>
</tbody>
</table>

*When present, the individual metal ion impurities and their concentrations in the HNO₃-plutonium solution are as follows: Fe, 0.31 g/l; Cr, 0.04 g/l; Ni, 0.06 g/l; Cu, 0.03 g/l; Pb, 0.02 g/liter. These are typical stream averages as experienced in the existing process plant.*
importance by the impurity concentration and the rate of hydrogen peroxide addition. The remaining two variables are of lesser importance and are not, in fact, statistically significant.

The t-test data, as well as the experimental results, show that the plutonium concentration in the filtrate is more sensitive to the variables of the precipitation than is the relative filtration time.

Based on this investigation, the levels shown in Table 8 are recommended for the six major variables in the precipitation process. These levels were selected as the best compromise between the fastest filtration time for the plutonium peroxide precipitation and the lowest concentration of plutonium in the filtrate.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitric acid concentration</td>
<td>2.3 to 2.7M</td>
</tr>
<tr>
<td>Hydrogen peroxide/plutonium molar ratio</td>
<td>22 moles ( \text{H}_2\text{O}_2 ) per mole Pu, minimum</td>
</tr>
<tr>
<td>Impurities</td>
<td>Impurities present(^a)</td>
</tr>
<tr>
<td>Rate of hydrogen peroxide addition</td>
<td>2 moles ( \text{H}_2\text{O}_2 ) per min. per mole Pu in the feed</td>
</tr>
<tr>
<td>Temperature</td>
<td>( 14^\circ \text{C} )</td>
</tr>
<tr>
<td>Digestion time</td>
<td>30 minutes</td>
</tr>
</tbody>
</table>

\(^a\) Applicable only to the specific metallic impurities and their concentrations given in Table 7. However, it is felt that the impurities and their concentrations could vary somewhat from those listed without significant effect.

### STAINLESS STEEL FILTERS FOR THE FILTRATION OF PLUTONIUM PEROXIDE PRECIPITATE

Stainless steel fritted filters, of the type being considered for use in a new plutonium recovery facility, have been shown to be effective for the filtration of plutonium peroxide precipitates. Three different porosity size filters were tested with mean pore sizes of 5, 10, and 25 microns. All three filters showed neither appreciable loss of particulate matter nor significant change in filtration rates after repeated usage. As would be expected, however, the filtration rate of the 25-micron filter was faster than the 5- and 10-micron filters. For example, in seven separate filtrations for each filter, the 25-micron filter averaged a 16\% faster filtration time than the 10-micron filter and 69\% faster filtration time than the 5-micron filter. Other than tapping the filters free of material that had adhered to the surface, no special cleaning procedures were needed between filtrations.

### PLUTONIUM ELECTROREFINING DEVELOPMENT


An electrorefining capability of several hundred kilograms of plutonium per month is specified on the flow sheet for the new plutonium recovery facility. This ultra-pure material may be used directly, or it may be used to blend with slightly impure metal to yield an ingot of acceptable purity for fabrication purposes. To achieve this goal, with a minimum capital investment and a maximum confidence level, ways of increasing throughput and equipment reliability are under study.

In the electrorefining process at Rocky Flats, impure plutonium metal is melted in a ceramic cup under a transport salt cover, all of which is contained in a 4\% in. diameter cylindrical magnesium oxide (MgO) ceramic crucible.\(^1,2\) A tungsten anode electrode is immersed in the molten plutonium, making the molten plutonium metal the anode. The plutonium and salt are equilibrated with stirring to allow MgCl\(_2\) to oxidize plutonium to PuCl\(_3\), to provide plutonium ions in the salt phase. Plutonium ions may also be supplied by adding plutonium chloride (PuCl\(_3\)) or plutonium fluoride (PuF\(_3\)) and eliminating MgCl\(_2\) in the transport cover salt. The plutonium ions are electrochemically transferred to a tungsten cathode where the plutonium ions are reduced to form a purified metal product. This metal finds many uses because of its exceptionally high purity, \(<200 \text{ ppm total metallic impurities}\). Most of the metal produced at Rocky Flats is used for analytical standards, special projects, and research and development applications. Major potential uses include blending with off-specification plutonium to yield an acceptable product and general upgrading of the plutonium metal in the foundry to further minimize the ingot reject rate.

An increase in the electrochemical transport rate of an electrolysis cell may be obtained by increasing the electrode surface areas, if all other parameters are held constant. In this instance, an increase of the cell diameter from 4\% in. to 5\% in. will allow an approximate doubling of the effective electrode area. The design specifications for the
new facility call for such an increase in cell diameter. To ensure that appropriately sized ceramic components will be available for the larger cell, a development and testing program has begun. The larger ceramic crucibles performed satisfactorily when an equimolar NaCl-KCl salt was melted. A satisfactory performance was observed when 500 grams of an inert solid (Al₂O₃) was added to the anode cup and the NaCl-KCl salt was melted. When approximately 3000 grams (more typical of operational conditions) of inert material was placed in the anode cup and the NaCl-KCl salt melted, the outer crucible failed. An analysis of the failure suggested that the problem is due to stress induced by the inner cup pressing on a small area of the outer crucible. To minimize this stress, an attempt will be made to maximize the contact area by using flat, matched surfaces on both the inner and outer crucible at their points of contact.

Future work, and work in progress, includes development of equipment to allow slow, controlled lowering of components into the salt, development and testing of a large-scale (6 to 8-kg plutonium) critically safe slab configuration, testing of electrically insulated metal stirrers and metal outer crucibles, and testing of the electrorefining process in induction-heated, tilt-pour type furnaces.

REFERENCES


TIME DOMAIN REFLECTOMETRY (TDR) FOR LIQUID LEVEL MEASUREMENTS

G. D. Lehmkuhl

Large volumes of solutions containing plutonium are handled routinely in the plutonium recovery and purification plant at Rocky Flats. Because of the nature and value of plutonium, careful inventory control and measurement of these solutions is necessary. It is desirable to locate tanks holding these solutions behind shielding walls because of inherent radiation emanating from such solutions, and to furnish personnel protection in the event of an accidental nuclear excursion. The use of direct visual observations to determine liquid levels becomes increasingly difficult as the extent of shielding increases.

In closed areas such as canyons, remote methods for liquid level measurements are particularly needed. Time domain reflectometry, using an electronic signal sent down a transmission line, was tested experimentally as a technique for such remote level measurements.¹ A ventilated transmission line probe was placed in a tank to make these measurements, and the measurements were compared with sight gage readings. When the time domain reflectometry probe was calibrated with the same liquid to be measured in the tank, level-measurement accuracy of 0.3 cm was possible. However, when different liquids were used for calibration and measurement, errors of 1 to 4 cm were observed. Experiments were successfully conducted to reduce this error (caused mainly by differences in ionic strengths). It is believed that the experimental equipment could be further developed into process equipment capable of making accurate, routine measurements of process tank liquid levels.

REFERENCE

A plutonium-metal plate was prepared for the Los Alamos Scientific Laboratory (LASL) using a tilt-pour casting procedure. The dimensions of the cast plate were 2.990 ±0.005 in. by 2.994 ±0.004 in. and 0.084 ±0.001-in. thick. The plutonium used for the fabrication was low NGS material having a $^{239}\text{Pu}$ content of 97.7 wt% and a $^{240}\text{Pu}$ content of 2.2 wt%. The plutonium had been previously alloyed with 1 wt% Ga to stabilize the plutonium in the delta phase. The dimensional specifications for the plate were very rigid as the plate had to fit into a prefabricated aluminum holder. The dimensions of the slot through which the plate had to slide were 3.000 in. by 0.087 inch. The cast plate was assembled into the holder without any dimensional problems.

The equipment used for the casting has been described. The plutonium feed metal was heated to 950°C in an oxidized tantalum crucible. The liquid metal was subsequently poured into a stackpole graphite mold which was heated to 700°C.

A photograph of the split mold configuration is shown in Figure 11. The interior surfaces of the mold were coated with a mold coating preparation of calcium fluoride. The finished plate is shown in Figure 12 after removing the casting sprue.

Figure 11. Split Mold Used for Casting Metal Plate Plutonium.

REFERENCE


CASTING OF MULTIPLE PLUTONIUM AND NEPTUNIUM METAL DISKS

W. V. Conner

Precision casting techniques have been developed at Rocky Flats for preparing small actinide metal targets for research and diagnostic purposes. These casting techniques were developed to eliminate the large amounts of scrap generated by the rolling procedure formerly used for preparing targets. A recent mold development program has resulted in a new mold design which makes it possible to cast multiple 0.006- to 0.010-in.-thick neptunium- and plutonium-metal disks. Using the new mold design and the proper casting parameters, it has been possible to prepare up to ten ½-in.-diameter disks in a single casting.

The basic techniques used for the multiple-disk castings are an extension of an injection casting procedure which was developed for preparing thin (<0.040-in. thick) actinide metal ingots and disks. The equipment and procedures used for injection castings have been described previously.

The primary difference between the previous injection castings and the multiple-disk castings is in the design of the
graphite mold. Several different mold designs were evaluated. The most successful design (see Figure 13) is for a split mold capable of producing up to ten \( \frac{1}{2} \)-in.-diameter disks per casting. The mold was designed with a 0.020-in. thick by \( \frac{3}{4} \)-in.-wide central sprue which extended from the bottom of the melting section to the bottom of the mold. The disk cavities were joined to the central sprue with tapered gates.

The results obtained using this technique can be illustrated by describing a typical plutonium casting.

The casting was made using 20 grams of plutonium metal alloyed with 1 wt% gallium. The ten \( \frac{1}{2} \)-in.-diameter disks are shown in Figure 14 after they were removed from the mold. The weights and thicknesses of the disks are given in Table 9. The average thickness of the individual disks ranged from 0.0105 to 0.0112 in. and the spread in thickness of the individual disks ranged from 0.0001 to 0.0005 inch.

This casting technique has also been used to prepare 0.007-in.-thick alpha plutonium and neptunium-metal disks. Twenty-two neptunium disks were prepared at the request of the Oak Ridge National Laboratory (ORNL) for the LMFBR neutron dosimetry program.

REFERENCES


Table 9. Weights and Dimensions of One-Half-Inch Diameter Delta-Plutonium-Metal Disks.

<table>
<thead>
<tr>
<th>Disk No.</th>
<th>Disk Wt (gram)</th>
<th>Disk Min. Thickness (in.)</th>
<th>Thickness Max. (in.)</th>
<th>Spread in Thickness (in.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.5586</td>
<td>0.0108</td>
<td>0.0111</td>
<td>0.0003</td>
</tr>
<tr>
<td>2</td>
<td>0.5538</td>
<td>0.0108</td>
<td>0.0110</td>
<td>0.0002</td>
</tr>
<tr>
<td>3</td>
<td>0.5672</td>
<td>0.0111</td>
<td>0.0112</td>
<td>0.0001</td>
</tr>
<tr>
<td>4</td>
<td>0.5648</td>
<td>0.0108</td>
<td>0.0112</td>
<td>0.0004</td>
</tr>
<tr>
<td>5</td>
<td>0.5632</td>
<td>0.0110</td>
<td>0.0111</td>
<td>0.0001</td>
</tr>
<tr>
<td>6</td>
<td>0.5597</td>
<td>0.0110</td>
<td>0.0107</td>
<td>0.0005</td>
</tr>
<tr>
<td>7</td>
<td>0.5293</td>
<td>0.0109</td>
<td>0.0113</td>
<td>0.0004</td>
</tr>
<tr>
<td>8</td>
<td>0.5651</td>
<td>0.0111</td>
<td>0.0111</td>
<td>0.0002</td>
</tr>
<tr>
<td>9</td>
<td>0.5698</td>
<td>0.0111</td>
<td>0.0113</td>
<td>0.0003</td>
</tr>
<tr>
<td>10</td>
<td>0.5665</td>
<td>0.0110</td>
<td>0.0113</td>
<td>0.0003</td>
</tr>
</tbody>
</table>

**PREPARATION OF HIGH PURITY NEPTUNIUM OXIDE**

*D. L. Baaso*

The Oak Ridge National Laboratory (ORNL) is involved in the preparation of diagnostic neutron detectors for LMFBR program. Neptunium-237 is one of the nuclides being used for neutron dosimeters. Rocky Flats was requested by ORNL to purify a 343-gram batch of neptunium oxide (300-g Np) for this program. The removal of plutonium-238 to a level that was as low as technically feasible was the primary purification objective. Other actinides and elemental impurities were also to be at very low levels in the final product. The neptunium feed material was supplied as oxide by ORNL, and contained 0.48 wt% thorium, 23-ppm plutonium (17-ppm 238Pu) and 20-ppm 235U.

The dissolution of the feed oxide was accomplished by three successive 1-hour contacts with 16M HNO3 – 0.1M HF at 100-110°C. A residual heel, which remained after the three acid contacts, was calcined and identified as ThO2 by X-ray diffraction. The nitrate feed solution was adjusted to 6.2M HNO3 along with the addition of ferrous sulfamate and aluminum nitrate in preparation for anion exchange. Reducing agents were used to hold the neptunium and plutonium in the Np(IV) and Pu(III) states, respectively. The neptunium was adsorbed onto a 6 in. by 24-in. column of Dowex 1-X2 nitrate form resin. The column was washed with nine column volumes (c.v.) of 6.2M HNO3, 0.05M N2H4. The first 3½ c.v. were also 0.05M in ferrous sulfamate.
The column was then converted to the chloride form by washing with 8 1/2 c.v. of 12M HCl-0.05M NH₂OH. This removed the thorium and plutonium. The fourth column-volume wash contained 0.1M HI and was left in a static condition with the column for 16 hours. After completing the HCl wash, the neptunium was eluted with 0.5M HCl and precipitated from the eluate by the addition of an excess of 0.1M oxalic acid. The oxalate precipitate was calcined for 17 hours at 600 to 700°C to form the neptunium oxide product.

Ninety-eight percent of the neptunium in the feed oxide was recovered in the purified product oxide. The plutonium, thorium, and uranium concentrations were reduced to <1 ppm. The total detectable impurity content of the final oxide product was 639 ppm (see Table 10).

### Table 10. Detectable Impurities in NpO₂ Product.

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (ppm)</th>
<th>Element</th>
<th>Concentration (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>16</td>
<td>Na</td>
<td>55</td>
</tr>
<tr>
<td>²⁴¹Am</td>
<td>&lt;0.001</td>
<td>P</td>
<td>3</td>
</tr>
<tr>
<td>C</td>
<td>30</td>
<td>²³⁸Pu</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Ca</td>
<td>38</td>
<td>Se</td>
<td>5</td>
</tr>
<tr>
<td>Cl</td>
<td>130</td>
<td>Si</td>
<td>25</td>
</tr>
<tr>
<td>Cr</td>
<td>1</td>
<td>Ta</td>
<td>15</td>
</tr>
<tr>
<td>Cu</td>
<td>4</td>
<td>Th</td>
<td>0.2</td>
</tr>
<tr>
<td>F</td>
<td>5</td>
<td>²³⁵U</td>
<td>&lt;0.2</td>
</tr>
<tr>
<td>Fe</td>
<td>4</td>
<td>W</td>
<td>4</td>
</tr>
<tr>
<td>In</td>
<td>13</td>
<td>Zn</td>
<td>1</td>
</tr>
<tr>
<td>K</td>
<td>12</td>
<td>Zr</td>
<td>3</td>
</tr>
<tr>
<td>Mg</td>
<td>5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Dowex 1-X2 resin was selected for the purification because of its greater capacity for neptunium and its favorable desorption and washing characteristics. Resin volume change problems were encountered at several stages in the processing cycle. A 7% shrinkage occurred during the 6.2M HNO₃ resin conditioning but the resin returned to its original volume when used with a hydrochloric acid medium. When eluted with dilute hydrochloric acid, the resin volume increased by 10%.

The major portion (89%) of the plutonium was separated from the neptunium during the initial nitrate anion exchange step. The remaining plutonium (11%) was separated during the chloride wash cycle used to remove the thorium from the column. The formation of the residual thorium dissolved heel was attributed to the low solubility of ThF₄ in the nitric-hydrofluoric acid dissolver solution.

### RECOVERY OF PLUTONIUM AND AMERICIUM-241 FROM CHLORIDE SALT RESIDUES

C. C. Perry and V. A. Casady

Americium-241 grows into plutonium from the beta decay of plutonium-241. The americium is removed from molten plutonium by extraction into a molten NaCl-KCl-6 wt% MgCl₂ solution. In general, the plutonium content in these extraction salts is about 2 wt% with an americium content of about 0.2 wt%. The spent chloride salts are processed for the recovery of the plutonium and americium, and to allow the discard of the chloride matrix. The present process consists of dissolving the salt residues in hot water and precipitating the plutonium and americium as hydroxides using 3M KOH. The resulting precipitate is filtered through a polypropylene pad which is coated with a one-inch-thick layer of filter aid. The major constituent of the filter aid is aluminum silicate. The resulting precipitate is dissolved in 8M HNO₃ in preparation for separating the americium and plutonium by anion exchange.

The following problems have been experienced with the existing process.

1. The salt residues are not completely soluble in hot water.
2. The separation of chloride into the hydroxide filtrate is not complete because the dissolved hydroxide precipitate often contains up to 4 g/l of chloride.
3. A heel remains after the dissolution of the hydroxide precipitate in 8M HNO₃. The major constituent of this heel is aluminum silicate from the filter aid entrained in the hydroxide precipitate.

In addition, the chloride salt residues generated from the two stage molten salt extraction process, which uses 8 wt% MgCl₂, cannot be dissolved effectively in hot water. This dissolution problem is related to the increased MgCl₂ content and oxidation products formed and entrained in the salt residue.

To reduce these process problems, a modified recovery procedure was developed. The modified process consists of dissolving the chloride salt residues in hot 1M HCl and precipitating the plutonium and americium as hydroxides using 3M KOH. The hydroxide precipitate is filtered through an organic (Solka-Floc)* filter aid and the precipitating liquor is decanted. A wash solution of 0.5M KOH is added.

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to the precipitate slurry which is filtered using a polypropylene pad coated with a \( \frac{3}{16} \)-in.-thick coating of the same organic filter aid. The collected hydroxide precipitate is dissolved in 3M \( \text{HNO}_3 \) which is filtered through a polypropylene pad to remove the organic filter aid.

A laboratory scale (200 g of salt) demonstration run has produced the following results.

1. Complete dissolution of the chloride salt residues from the two stage molten salt extraction process was accomplished.

2. The nitric acid solution from the dissolution of the hydroxide precipitate contained <1 g/l of chloride.

3. The remaining heel from the hydroxide dissolution represented only 0.13 wt% of the starting chloride salt and contained only 0.83% of the plutonium and 5.8% of the americium found in the starting chloride salt. The silacious nature of the heel was eliminated.

4. Plutonium and americium recoveries of 99% and 94%, respectively, were realized for the nitric acid solution.

Full scale demonstration runs with production equipment are planned.

REFERENCE


2. J. L. Long et al., Batch Two-stage Molten Salt Extraction of Americium, USAEC RFP-1875, in process.

PREPARATION OF URANIUM-235 AND URANIUM-238 METAL FOR OAK RIDGE NATIONAL LABORATORY

S. G. Proctor

Two, uranium-metal buttons (approximately 100 g each) were prepared from \( ^{235}\text{U}_3\text{O}_8 \) and \( ^{238}\text{U}_3\text{O}_8 \) received from Oak Ridge National Laboratory (ORNL). The metal buttons will be used to fabricate standard disks for the LMFBR diagnostic program. The high isotopic enrichment of the uranium involved (99.91% - 235\text{U} and 99.999% - 238\text{U}) required strict processing controls to maintain isotopic integrity, high purity, and to maximize the amount of metal produced.

A second request was received from ORNL to prepare a third uranium metal button from \( ^{236}\text{U}_3\text{O}_8 \) oxide (approximately 18 g) with a \( ^{236}\text{U} \) enrichment of 89.1%. The \( ^{236}\text{U} \) metal request was not part of the LMFBR diagnostic program. The \( ^{236}\text{U}_3\text{O}_8 \) was processed first, to serve as a pilot run for the other two batches of oxide which were prepared in a similar manner. A low metal yield (64.2%) resulted and was attributed to the poor reactivity of the \( ^{236}\text{U}_3\text{O}_8 \).

To produce a more reactive oxide, the as-received \( ^{235}\text{U}_3\text{O}_8 \) and \( ^{238}\text{U}_3\text{O}_8 \) were dissolved in nitric acid and were processed to a \( \text{UF}_4 \) form by peroxide precipitation and controlled calcination of the peroxide cake. During the hydrogen peroxide addition, close control was exercised to maintain the acidity at pH 2 and the solution temperature at \( 20^\circ\text{C} \). The \( \text{UF}_4 \) was subsequently converted to \( \text{UF}_6 \) by exposing the oxide to a \( \text{I}_2 - \text{II} \) mixture for a period of 6 hours at \( 600^\circ\text{C} \). The \( \text{UF}_4 \) formed was reduced to uranium metal using calcium metal with an iodine booster (1 mole \( \text{I}_2 \) per mole of uranium). Calcium chloride was also added to the reduction charge to improve metal coalescence by lowering the melting point of the slag. The reduction was carried out in a sealed pressure vessel which was placed in a preheated (1000°C) furnace.

Metal yields of 99.0% for the \( ^{238}\text{U} \) reduction and 99.2% for the \( ^{235}\text{U} \) reduction were realized. Isotopic analyses on the uranium metal products confirmed that isotopic integrity had been maintained. The elemental impurity level on a uranium basis increased from 646 to 698 ppm for the \( ^{238}\text{U} \) processing, whereas the impurity level for the \( ^{235}\text{U} \) processing decreased from 413 to 277 ppm. Minimal process losses of approximately 1% for both the \( ^{235}\text{U} \) and \( ^{238}\text{U} \) processing from oxide (\( \text{U}_3\text{O}_8 \)) to metal were experienced.

DECONTAMINATION DEVELOPMENT FOR BERYLLIUM AND DEPLETED URANIUM

F. E. Evans and R. L. Riegel

Present standards for classifying waste metal as noncontaminated with respect to depleted uranium are alpha smear analyses of \(<50 \text{ d/m ft}^2 \) and direct alpha counts of \(<250 \text{ c/m at any point on the surface of the material. Waste material is considered "noncontaminated" with beryllium at a smear analysis of } <25 \text{ µg/ft}^2 \). Two procedures have been developed for the decontamination of depleted uranium from tooling and of beryllium and uranium from stainless steel cladding used in the hot rolling of beryllium billets.
CONTAMINATED TOOLING

The fabrication of depleted uranium-based components generates contaminated tooling such as pot chucks, dies, punches, hold-down rings, and draw rings. These items are usually constructed from mild steels and have a weight range from 5 to 200 pounds. The spent tooling is packaged for storage at Idaho if the contamination limits for depleted uranium are not satisfied. If the limits are satisfied, the tooling can be disposed of as noncontaminated scrap metal. The gross surface contamination is initially removed by grit blasting. However, this technique does not remove contamination from holes and slots very effectively. The tooling is then immersed in warm (40 to 50°C) dilute (8 to 12 vol%) sulfuric acid which contains a small quantity (1 gal gal) of chromic trioxide. The immersion periods are varied from 30 to 60 minutes. The leached tooling is dip-rinsed followed by a warm water spray rinse. The tooling is dried and is subsequently smeared and counted directly to determine the residual uranium levels. The results for seven items processed is reported in Table 11. The smear and direct-count results, for the seven items processed, permitted the classification of the tooling as noncontaminated waste metal.

CONTAMINATED CLADDING

A second decontamination procedure has been developed for removing beryllium and depleted uranium from 304 stainless steel cladding used in the hot rolling of beryllium billets. During the rolling process, the inner surfaces of the cladding become contaminated with beryllium and the outside surfaces become contaminated with depleted uranium from the rolling mill rolls.

The stainless steel cladding sheets (15 in. by 27 in. by \(~1/16\) in.) are initially scrubbed with a mild abrasive and rinsed to remove any loose contamination. The bulk of the uranium contamination is removed during this step. The sheets are then immersed in a commercial descaling solution Ox-Out 536* or Derustit SS-3** for 40 minutes followed by a dip and spray rinse with water. The processed sheets are then surveyed for direct alpha count and smeared for uranium and beryllium removal.

The described decontamination procedure was developed using coupon specimens removed from the rolled sheet. These coupons were evaluated analytically before and after processing. The entire coupon (1 in. by 1 in. by \(1/16\) in.) was dissolved to provide information on the residual beryllium content because no smear was evident. The results are reported in Table 12. For the before and after results, two adjacent coupons were used. The uranium decontamination is completely removed during the descaling operation as approximately 0.001 in. of the metal surface is removed. Over 40 sheets have been decontaminated and classified as non-contaminated scrap metal.

*OX-OUT 536, Chemclean Corporation, 138-07 Eighteenth Avenue, College Point, New York, 11356.
**Derustit SS-3, Bradford Derustit Corporation, P.O. Box 51, Elmsford, New York 12065.

Table 11. Uranium Decontamination Results.

<table>
<thead>
<tr>
<th>Item No.</th>
<th>Description</th>
<th>(\text{H}_2\text{SO}_4) (vol%)</th>
<th>Immersion Period (min)</th>
<th>Smear Count (10^6) c/m Before</th>
<th>Smear Count (10^6) c/m After</th>
<th>Direct Count (10^6) c/m Before</th>
<th>Direct Count (10^6) c/m After</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8¼&quot; diameter hold down ring</td>
<td>0</td>
<td>45</td>
<td>136</td>
<td>0</td>
<td>60</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>15&quot;-diameter draw ring</td>
<td>8</td>
<td>45</td>
<td>136</td>
<td>45</td>
<td>200</td>
<td>150</td>
</tr>
<tr>
<td>3</td>
<td>18&quot;-diameter by 9&quot;-thick pot chuck</td>
<td>8</td>
<td>45</td>
<td>136</td>
<td>90</td>
<td>1000</td>
<td>500</td>
</tr>
<tr>
<td>4</td>
<td>Rerun of 18&quot;-diameter by 9&quot;-thick pot chuck</td>
<td>10</td>
<td>30</td>
<td>90</td>
<td>18</td>
<td>500</td>
<td>90</td>
</tr>
<tr>
<td>5</td>
<td>10&quot;-diameter by 8&quot;-thick pot chuck</td>
<td>10</td>
<td>50</td>
<td>1500</td>
<td>20</td>
<td>900</td>
<td>40</td>
</tr>
<tr>
<td>6</td>
<td>10&quot;-diameter by 8&quot;-thick pot chuck</td>
<td>12</td>
<td>60</td>
<td>300</td>
<td>18</td>
<td>400</td>
<td>70</td>
</tr>
<tr>
<td>7</td>
<td>18&quot;-diameter by 9&quot;-thick pot chuck</td>
<td>12</td>
<td>60</td>
<td>450</td>
<td>20</td>
<td>300</td>
<td>45</td>
</tr>
</tbody>
</table>
Table 12. Beryllium Decontamination Results.

<table>
<thead>
<tr>
<th>Run</th>
<th>Descaler</th>
<th>Immersion Period (min)</th>
<th>Metal Removed (in.)</th>
<th>Beryllium (ppm) Before</th>
<th>Beryllium (ppm) After</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SS-3</td>
<td>10</td>
<td>0.0010</td>
<td>800</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>SS-3</td>
<td>30</td>
<td>0.0010</td>
<td>800</td>
<td>10</td>
</tr>
<tr>
<td>3</td>
<td>OX-OUT-536</td>
<td>30</td>
<td>0.0017</td>
<td>700</td>
<td>6</td>
</tr>
<tr>
<td>4</td>
<td>OX-OUT-536</td>
<td>60</td>
<td>0.0030</td>
<td>700</td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>OX-OUT-536</td>
<td>60</td>
<td>0.0014</td>
<td>900</td>
<td>50</td>
</tr>
</tbody>
</table>

Note: ppm = µg beryllium per gram of steel sample.

NORMAL OPERATING PLUTONIUM LOSS STUDIES

R. E. Giebel

The routine practice at Rocky Flats for preparing spent equipment for discard is to thoroughly clean the accessible contaminated surfaces by using stripping agents and physical means. Individual equipment items that can be removed conveniently are placed in a 55-gallon drum and the residual plutonium is then determined by drum counting procedures.

Development studies have been initiated to evaluate and improve the procedures used to determine the amount of residual plutonium discarded with contaminated equipment. A glovebox, which was designated for discard, was selected for study. A gamma survey instrument fitted with a sodium iodide detector was used to locate and estimate the residual plutonium content.

The glovebox under study was subjected to three cycles of cleaning and gamma surveying in addition to the removal of items for drum counting. The experimental results indicated the following:

1. The surface cleaning procedures were inadequate.
2. The gamma survey instrumentation must be equipped to discriminate against the 60 keV $^{241}$Am gamma to obtain reliable plutonium values.
3. The gamma surveying practices need standardization and procedural improvements.
4. Significant variances were evident between gamma survey and drum counting values.
5. The data indicate that gamma survey values are low.

Continued studies are planned.

PLUTONIUM INVENTORY VERIFICATION

R. G. Leebl et al.

Plutonium metal, and other plutonium recovery process compounds, were selected from a recent Rocky Flats physical inventory for plutonium verification. The selected items will be assayed calorimetrically for their respective plutonium content by Mound Laboratory. The selected items were processed to a stable form and were packaged to comply with Safeguard and Department of Transportation specifications. A summary of the materials selected and associated quantities is presented in Table 13.

Table 13. Mound Laboratory Shipment.

<table>
<thead>
<tr>
<th>Material Category</th>
<th>Number of Items</th>
<th>Net Weight (g)</th>
<th>Plutonium Weight (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Molten Salt Metal</td>
<td>10</td>
<td>19,313</td>
<td>19,239</td>
</tr>
<tr>
<td>2. Rocky Flats' Buttons</td>
<td>10</td>
<td>13,123</td>
<td>13,078</td>
</tr>
<tr>
<td>3. Reburned Skull Oxide</td>
<td>10</td>
<td>9,319</td>
<td>6,969</td>
</tr>
<tr>
<td>4. Dirty Green Cake</td>
<td>4</td>
<td>3,870</td>
<td>3,196</td>
</tr>
<tr>
<td>5. Dirty Fluoride</td>
<td>16</td>
<td>11,443</td>
<td>5,167</td>
</tr>
<tr>
<td>6. Incinerator Ash</td>
<td>15</td>
<td>73,181</td>
<td>3,678</td>
</tr>
<tr>
<td>Totals</td>
<td>65</td>
<td>130,249</td>
<td>51,327</td>
</tr>
</tbody>
</table>

PREPARATION OF PLUTONIUM AND URANIUM STANDARD MATERIALS

R. E. Giebel

Custom plutonium and depleted uranium materials were prepared for the Analytical Standards Program at Rocky Flats. Depleted uranium metal with low elemental impurities (Al – 8 ppm, C – 15 ppm, Fe – 14 ppm, Ni – 3 ppm, and Si – 4 ppm) was selected for the preparation of analytical metal standards. The metal was homogenized by casting and was rolled into a 100-mil-thick sheet.

A strip, $\frac{1}{2}$-in. wide, was removed from the center of the sheet. The ends were cut from the strip, and the remainder was cut into $\frac{1}{2}$-in. long coupons.

Plutonium oxide (2100 g) was prepared for source material in the preparation of chemical standards. The stable oxide was prepared from plutonium peroxide cake which was muffled under controlled conditions to remove nitrates, sulfates, and moisture.
PLUTONIUM STORAGE STUDIES

R. E. Giebel

Experimental studies have continued to establish practical guidelines for packaging and storing plutonium materials for extended periods of time. The packaging item of particular interest is the plastic (PVC) bag which is used as a vehicle to remove the plutonium material from the glovebox line and which also serves as the initial contamination barrier.

An alpha plutonium button (2.1 kg) recovered from the molten salt extraction process was wrapped directly in aluminum foil, removed from the glovebox line in a PVC cut-out bag, and sealed into a produce can. The sealed produce can was placed in a Vollrath can to complete the storage configuration. After 6 months of shelf storage, the packaging system and contents were examined. The PVC cut-out bag was slightly discolored but was structurally sound. The aluminum foil did not stick to the plutonium button but was contaminated with plutonium oxide. Approximately 17 grams of plutonium metal were lost as oxide during the storage period.

A batch of plutonium oxide was divided into two segments consisting of 3862 g and 1531 g of oxide, respectively. The thermal energies involved were 8.1 watts for the larger batch and 3.2 watts for the smaller batch. The two oxide batches were packaged separately using a No. 19 screw-cap-glue-can for the primary container, PVC cut-out bag for the alpha barrier, a sealed No. 901 can, and a 6M shipping container. Again, the longevity and structural integrity of the PVC cut-out bag was the main objective of the study. After a 3½ month storage period, the packaging system was examined. The PVC bag surrounding the 3.2 watt container was slightly discolored but was structurally sound.

The PVC bag surrounding the 8.1 watt container was a light rust color. An oily material assumed to be plasticizer had collected on the inside of the No. 901 can. No integrity failure of the PVC bag was evident. Additional storage studies are planned for longer periods of time and for material with higher thermal energies.

ELECTROPLATING AND ANODIZING SUPPORT

T. A. Blatter and K. R. Souply

NICKEL PLATING OF FIRE SPRINKLER HEADS

Fire sprinkler heads located in a decontamination facility which uses caustic solutions, were corroding badly. For corrosion protection, the brass sprinkler heads were plated with 0.0005 in. of nickel. Experimental tests demonstrated that the nickel plate did not interfere with sprinkler head performance and did not alter their activation temperature.

The area to be plated was scrubbed with a mild abrasive followed by additional ultrasonic cleaning using an alkaline solution at 60°C. The zinc solder detection areas were masked off prior to plating. The head was dipped into a 50 vol% HCl solution for 30 seconds followed by a water rinse. A Wood’s nickel strike was applied followed by a water rinse. The area was then plated from a Watt’s nickel bath at a current density of 40 A/ft² to a thickness of 0.0005 in. of nickel.

NICKEL PLATING OF BRASS RINGS

The Health Physics Group has been experiencing disassembly and deterioration problems with the brass rings used in the air sampling heads located in the beryllium fabrication areas. The rings become pitted with use and stick to the gasket which forces their discard to contaminated waste. In an attempt to increase ring life, the ring was electroplated with 0.0005 in. of nickel. Preliminary evaluations of the plated rings have been favorable.

The ring surface is cleaned cathodically using Oakite 162 followed by a water rinse, a 5-second dip in a 50 vol% HCl solution, and a water rinse. A Wood’s nickel strike is applied followed by a water rinse. The final 0.0005-in.-nickel plate was deposited from a Watts nickel bath at a current density of 40 A/ft². The rings are rack-plated at 20 rings per rack.

HARD ANODIZING OF ALUMINUM GEARS

The Rocky Flats Inspection Group was using an inspection gage fitted with a Teflon-fiber gear system. The Teflon gears had a tendency to strip the fiber gears. This gear
system also seemed to suffer from accelerated wear which adversely affected the accuracy of the gage. An aluminum gear system which was hard-anodized (0.002 in.) to increase wear resistance was substituted and has been performing satisfactorily. The aluminum gears were prepared for anodizing by a 5-minute immersion in a commercial aluminum cleaner followed by a water rinse and a 1-minute dip in a 20 vol% HNO₃ solution. The operating conditions for the sulfuric-oxalic acid anodizing were as follows:

- Sulfuric acid concentration: 12 vol%
- Oxalic acid concentration: 1 wt%
- Solution temperature: 8 - 11°C
- Current density: 40 A/ft²
- Potential: 10 - 50 volts
- Anodizing time: 2 hours

**ENVIRONMENTAL RESEARCH**

_W. C. Bright_

**HYDROCHLORIC ACID PLUTONIUM RECOVERY PROCESS**

_L. J. Meile and D. L. Ziegler_

Initial results obtained in the development of a chloride plutonium recovery process have been reported. Two additional 40-gram bomb reductions of dicesium plutonium hexachloride (Cs₂PuCl₆) have been made. These reductions were made in a tantalum crucible with a 30% excess of calcium metal. In the first reduction, coalescence of the metal was poor and only metal shot was obtained. The charge began to react at 110°C and was heated to 445°C. Iodine was added to the second charge to increase the heat of reaction. Reaction began at about 80°C and heating continued until the internal temperature reached 700°C. An intact button was obtained with a metal yield of 85%. Table 14 compares properties of the two reductions.

The tantalum reduction crucible and iodine booster are considered to be primarily responsible for the higher yield and increased purity of the metal in the second reduction. This crucible proved far superior to magnesium oxide for containing the corrosive molten chloride salts during the reduction process. The iodine booster may or may not have been responsible for the lower firing temperature, but it is believed to have improved coalescence by increasing the reaction temperature.

**REFERENCE**


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**PROPERTIES OF EVAPORATOR FEED SOLUTION IN THE NEW AQUEOUS WASTE TREATMENT FACILITY**

_T. C. Johnson and L. J. Meile_

Criteria for a new aqueous waste treatment facility have been developed. The major process in the facility will be multiple-effect evaporation of the combined waste streams and recovery of water for reuse. Process and equipment
design require data on the physical properties of the solution being evaporated and on changes in composition as the solution is evaporated. Laboratory scale evaporation tests were made on a simulated feed solution to obtain this information.

Table 15 gives the chemical analyses of the simulated feed solution and of the solution as it was concentrated in the laboratory evaporator. These analyses show two significant facts concerning the concentration of the solution: (1) the pH decreases as the concentration of the solution increases, and (2) ammonium ion is lost from the solution as evidenced by a lower concentration factor for HN₄⁺ than for solids and other ions. The apparently constant nitrate ion concentration may be due to an analytical method limitation.

Table 15. Analysis of Evaporator Solution at Various Stages of Concentration.

<table>
<thead>
<tr>
<th>Property or Species Analyzed</th>
<th>Simulated Feed Solution (ppm)</th>
<th>Concentrated Solutions (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>~3% Solids</td>
<td>~15% Solids</td>
</tr>
<tr>
<td>pH</td>
<td>8.2</td>
<td>6.5</td>
</tr>
<tr>
<td>Total Solids</td>
<td>29,930</td>
<td>138,200</td>
</tr>
<tr>
<td>Dissolved Solids</td>
<td>29,990</td>
<td>136,250</td>
</tr>
<tr>
<td>NH₄⁺</td>
<td>820</td>
<td>1,850</td>
</tr>
<tr>
<td>NO₃⁻</td>
<td>15,000</td>
<td>13,200</td>
</tr>
<tr>
<td>Cl⁻</td>
<td>4,700</td>
<td>21,370</td>
</tr>
<tr>
<td>F⁻</td>
<td>450</td>
<td>1,280</td>
</tr>
<tr>
<td>PO₄³⁻</td>
<td>610</td>
<td>2,710</td>
</tr>
<tr>
<td>HCO₃⁻</td>
<td>1,230</td>
<td>3,080</td>
</tr>
<tr>
<td>SCN⁻</td>
<td>49</td>
<td>240</td>
</tr>
</tbody>
</table>

Figures 15 and 16 are plots of specific gravity and boiling point of the solution as functions of the solids concentration. Boiling point rise is an important consideration in multiple-effect evaporator design; this solution exhibits a relatively small boiling point rise. Specific gravity is needed for mechanical design considerations and was determined for that reason.

Observations were made during the evaporation runs on foaming and precipitation of solids. Foaming was anticipated because of the presence of detergents in the laundry water. The foaming was very minor, and only occasionally rose more than one inch above the surface of the liquid. Some precipitation of solids occurred when the solution was initially prepared. The precipitate was present in a concentration of 0.1 grams/liter, was amorphous, and was not readily identified. As the solids concentration was increased by evaporation, precipitation of a small amount...
Differential thermal analyses of the reactions of delta-stabilized plutonium metal with carbon tetrachloride and Freon TF have been completed. Experimental runs were made with the reactants sealed in 1.5-ml borosilicate glass ampules at a heating rate of 15 deg. min⁻¹. Several runs were made with the ampoules evacuated to about 5 mm Hg. Additional runs were made in the presence of oxygen which was added by purging the ampoules before sealing. Metal used in the experiment was production grade delta material. Samples used consisted of fresh filings measuring approximately 0.2 by 0.2 by 0.2 mm. Approximately 15 mg of filings were used in each run.

Results of the tests are given in Table 16. All runs listed, with the exception of the one involving metal and oxygen, were characterized by an extremely sharp exothermic peak. Temperatures given represent the point of initial deviation of the DTA trace from its null position. In the case of the metal and oxygen run, the DTA trace rose at a moderate rate then leveled-off, signifying depletion of the available oxygen.

**Table 16. Differential Thermal Analysis of the Reactions Between Delta Stabilized Plutonium Filings, Carbon Tetrachloride and Freon TF.**

<table>
<thead>
<tr>
<th>Reactant</th>
<th>Onset Temperature (°C)</th>
<th>Reaction Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen</td>
<td>230</td>
<td>Ignition.</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>240</td>
<td>Violent.</td>
</tr>
<tr>
<td>Carbon tetrachloride and Oxygen</td>
<td>200</td>
<td>Violent.</td>
</tr>
<tr>
<td>Freon TF</td>
<td>565</td>
<td>Violent — more exothermic than CCl₄ reaction.</td>
</tr>
<tr>
<td>Freon TF and oxygen</td>
<td>582</td>
<td>Violent — more exothermic than CCl₄ reaction.</td>
</tr>
</tbody>
</table>

Data obtained during glovebox fire testing and previously reported,¹ have been used to predict the average air temperature expected during a fire within a radiochemical processing facility. A worst-case assumption of a fire involving methyl methacrylate glovebox windows and neutron shielding was made for the model. A bulk air temperature rise of about 200°F is predicted for a room containing 126,000 cubic feet of space. However, the air temperature leaving the glovebox would be about 1600°F and a localized temperature near the glovebox of 1000°F might occur. Therefore, ducting and structural members should be designed to withstand this type of exposure.

**REFERENCE**


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**WATER RECYCLE USING REVERSE OSMOSIS FOR PURIFICATION**

C. E. Plock

Each month approximately 10.5 million gallons of process, sanitary, cooling tower, and laundry waters are discharged from Rocky Flats. Experiments are being performed to determine if reverse osmosis may be a practical process for purifying these waters for potable and/or industrial uses.

The process waste waters are collected in holding tanks in the buildings where they are generated. When the tank is full, a sample is taken and analyzed. When the analytical results have been received, the tank is emptied by pumping the water to the area dictated by the analytical results. In the present study, 55-gallon drums were filled with waters while the tanks were being emptied. These samples were pumped through an experimental 200-gallon per day reverse osmosis unit. The product water was sampled and sent to the analytical laboratory for complete analysis. Between 50 and 55 separate determinations were made on the reverse osmosis feed and product waters. With a few exceptions, rejection ratios have been at the 90% level or
better, for the substances for which both the feed and product waters have been analyzed. The exceptions are boron (34%), cadmium (88%), copper (83%), and chloride (36%).

Table 17 shows the drinking water standards as recommended by the United States Public Health Service (USPHS), followed by the concentration range of these impurities in the product waters, the average concentration of the impurities, and the rejection rate of these impurities by the reverse osmosis unit. These data are for six drums of process waste waters from two process waste streams.

**Table 17. Reverse Osmosis Product Waters Data.**

<table>
<thead>
<tr>
<th>Substances</th>
<th>Max. Std. Conc. (USPHS)</th>
<th>Product Waters</th>
<th>Rejection Rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Conc. Range</td>
<td>Average Conc.</td>
<td></td>
</tr>
<tr>
<td>Alkyl benzene sulfonate (ABS)</td>
<td>0.5</td>
<td>0.29 – 1.21</td>
<td>0.62</td>
</tr>
<tr>
<td>Arsenic (As)</td>
<td>0.01</td>
<td>0.003 – 0.005</td>
<td>0.004</td>
</tr>
<tr>
<td>Barium (Ba)</td>
<td>1.0</td>
<td>0.0005 – 0.006</td>
<td>0.0027</td>
</tr>
<tr>
<td>Beryllium (Be)</td>
<td>None</td>
<td>&lt;0.0001 – 0.0006</td>
<td>0.0003</td>
</tr>
<tr>
<td>Boron (B)</td>
<td>(5.0)</td>
<td>0.007 – 0.01</td>
<td>0.009</td>
</tr>
<tr>
<td>Cadmium (Cd)</td>
<td>0.01</td>
<td>0.001 – 0.003</td>
<td>0.002</td>
</tr>
<tr>
<td>Calcium (Ca)</td>
<td>(200.0)</td>
<td>0.01 – 1.5</td>
<td>0.30</td>
</tr>
<tr>
<td>Chloride (Cl)</td>
<td>250.0</td>
<td>9.0 – 18.0</td>
<td>14.</td>
</tr>
<tr>
<td>Chromium (Cr VI)</td>
<td>0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>1.0</td>
<td>0.005 – 0.06</td>
<td>0.021</td>
</tr>
<tr>
<td>Carbon chloroform extract (CCE)</td>
<td>0.2</td>
<td>&lt;0.1 – 1.2</td>
<td>0.3</td>
</tr>
<tr>
<td>Cyanide (Cn)</td>
<td>0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Fluoride (F)</td>
<td>0.6 – 1.7</td>
<td>&lt;0.1</td>
<td>&lt;0.1</td>
</tr>
<tr>
<td>Iron (Fe)</td>
<td>0.3</td>
<td>0.004 – 0.008</td>
<td>0.006</td>
</tr>
<tr>
<td>Magnesium (Mg)</td>
<td>(130.0)</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Manganese (Mn)</td>
<td>0.05</td>
<td>&lt;0.0001 – 0.0004</td>
<td>0.0002</td>
</tr>
<tr>
<td>Mercury (Hg)</td>
<td>(0.05)</td>
<td>0.003 – 0.005</td>
<td>0.005</td>
</tr>
<tr>
<td>Lead (Pb)</td>
<td>0.05</td>
<td>0.0002 – 0.0008</td>
<td>0.0004</td>
</tr>
<tr>
<td>Nitrate (NO₃)</td>
<td>45.</td>
<td>&lt;0.5 – 0.5</td>
<td>&lt;0.5</td>
</tr>
<tr>
<td>Phenols⁸</td>
<td>0.001</td>
<td>0.035 – 2.30</td>
<td>2.6</td>
</tr>
<tr>
<td>Phosphate (PO₄)</td>
<td>(0.2)</td>
<td>1.4</td>
<td>3.9</td>
</tr>
<tr>
<td>Selenium (Se)</td>
<td>0.01</td>
<td>&lt;0.010</td>
<td>&lt;0.010</td>
</tr>
<tr>
<td>Silver (Ag)</td>
<td>0.05</td>
<td>&lt;0.00006 – 0.0001</td>
<td>0.0001</td>
</tr>
<tr>
<td>Sulfate (SO₄)</td>
<td>250.0</td>
<td>0.4 – 19.3</td>
<td>4.1</td>
</tr>
<tr>
<td>Thorium (Th)</td>
<td>None</td>
<td>0.0006 – 0.001</td>
<td>0.001</td>
</tr>
<tr>
<td>Total dissolved solids (TDS)</td>
<td>500.0</td>
<td>42. – 67</td>
<td>52.</td>
</tr>
<tr>
<td>Uranium (U)</td>
<td>(5.0)</td>
<td>0.03 – 0.5</td>
<td>0.05</td>
</tr>
<tr>
<td>Zinc (Zn)</td>
<td>5.0</td>
<td>&lt;0.003 – 0.005</td>
<td>0.004</td>
</tr>
</tbody>
</table>

Notes: The substances whose values in the USPHS column are shown in parentheses are not required by the USPHS, but are recommended by the Canadian Water Resources Commission. Substances which could be of concern at Rocky Flats are also shown, and in the USPHS column the word "none" appears for these substances: NA — not available;

⁸the source of the phenolic compounds is unknown. Another analytical procedure is being developed to double-check the phenol results.

KILN INCINERATION

W. E. Domning

A previous report summarized efforts toward the development of a stack-type kiln for the combustion of contaminated waste. A major objective was to develop the concept of neutralizing, within the combustion chamber, any acids, such as HCl, formed during combustion. This should greatly reduce corrosive attack on the incinerator structure and possibly reduce the need for scrubbing of off-gases. Since a large portion of the combustible waste is polyvinyl...
chloride film, a large amount of 2-ethyl hexyl phthalate is produced during the combustion. This material escapes from the burning zone because it forms an aerosol.

An attempt was made to trap this aerosol on a bed composed of crushed metallurgical coke. In these experiments, the off-gas was passed through a bed of coke 2 inches in diameter by one foot deep, at a rate of one liter per minute.

The coke bed proved to be an ineffective absorber. This was demonstrated by using heat to desorb the materials retained by the coke. Table 18 identifies the material desorbed from the bed as a function of temperature.

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>Water</td>
</tr>
<tr>
<td>300</td>
<td>Carboxylic acid, aliphatic ester, phthalate ester.</td>
</tr>
<tr>
<td>400</td>
<td>Long-chain aliphatic hydrocarbon (wax).</td>
</tr>
</tbody>
</table>

The entire amount of material desorbed amounted to only 0.5% of the total weight of the coke. A similar effect was noted when coke was mixed with the pelletized feed.

A gas chromatograph using molecular sieve and Paropac® columns was used to monitor the off-gas during the burning tests. These experiments were run at 645°C at the bottom of the burning zone, 650° in the center of the burning zone, and 345°C above the heated zone. The height of the column of pellets in the kiln was 22 inches. Some selected overhead gas composition data obtained in these tests are given in Table 19, where the CO and CO₂ analyses have been calculated by difference from the measured concentrations of O₂, N₂, and H₂.

<table>
<thead>
<tr>
<th>Time (minutes)</th>
<th>% O₂</th>
<th>% N₂</th>
<th>% H₂</th>
<th>Calc CO and CO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>12.1</td>
<td>78.5</td>
<td>1.7</td>
<td>7.7</td>
</tr>
<tr>
<td>125</td>
<td>5.6</td>
<td>48.5</td>
<td>1.3</td>
<td>44.6</td>
</tr>
<tr>
<td>250</td>
<td>1.8</td>
<td>17.5</td>
<td>0.9</td>
<td>80.6</td>
</tr>
<tr>
<td>315</td>
<td>4.9</td>
<td>50.8</td>
<td>0.3</td>
<td>44.0</td>
</tr>
</tbody>
</table>

The reason for the phthalate ester being in the overhead is probably because of the formation of an aerosol which is transported by the gas stream. Another problem encountered was that the feed pellets stuck to the side walls of the reactor. It is possible that the sticking could be due to the formation of a molten mixture of sodium carbonate and neutralization product sodium chloride during the run, and freezing of the salt during cool-down. The sticking, however, did not prevent the ash produced during the experiments from being easily transported by a screw-type conveyor. To overcome these problems, a new kiln design concept is being evaluated.

**REFERENCE**


**FLUID BED INCINERATION**


A fluid bed concept for the incineration of combustible wastes with in situ neutralization of produced hydrogen chloride is being tested. Although the stack kiln incineration tests did demonstrate the validity of the in situ neutralization concept, inherent temperature distribution problems caused large amounts of unburned hydrocarbons to be produced. Fluid bed incineration appeared to offer some advantages: such as, bed temperature control with uniform heating throughout.

**INCINERATOR DESIGN AND OPERATION**

A quartz fluid bed reactor, with resistance heating, is used for the incinerator. The off-gas system is all glass, including the caustic scrubbers. A nonmetal system was desired to prevent loss of HCl by reaction with metal. This was necessary for the calculation of a material balance for chloride.

The incinerator system now being tested is shown in Figure 17. Air or an argon-oxygen mixture is first passed through a gas heater and into the bottom of the heated reactor. The bed is fluidized, heated to 500-550°C, and pelletized waste is fed in from the side. Most of the HCl produced by the polyvinyl chloride (PVC) decomposition reacts with the Na₂CO₃ in the bed. The small amount of unreacted HCl, with unburned tars and gases, is then pulled through the overhead Na₂CO₃ static bed to neutralize
The rocking extruder\(^1\) has been used in preparing waste pellets for use in the incinerator tests. Some difficulty was experienced with this extruder. At operating temperature, molten plastic was ejected from the open end of the extruder as it rocked to squeeze the waste through the cutters and extrusion holes. An end-covering flap was added to prevent this, but it was then found that a change in the method of force application was needed. Until this problem is resolved, no larger models of the rocking design will be constructed.

A 3-in., eight-hole ram type cutter-extruder has been fabricated and tested. With a nozzle temperature of about 200\(^\circ\)C, 3 pounds of waste per hour can be extruded. Extruded waste strands are \(\frac{7}{16}\)-in. in diameter and have a specific gravity of about one. A 6-in. diameter, 38-hole extruder is being fabricated to process about 12 pounds of waste per hour.

**REFERENCE**


**FILTER MEDIA PROCESSING**

*W. E. Dunning*

A Hydrofluorination has been proposed as a method for the processing of used High Efficiency Particulate Air (HEPA) filters to recover deposited plutonium. To efficiently feed the rotating kiln-type reactor, the filter media and separators need to be ground. Grinding of this material is facilitated by calcination. The effects of temperature on the media and separator are shown in the thermogravimetric traces in Figure 18. These data indicate degradation that correlates well with observed loss of filtering efficiency of HEPA filters at elevated temperatures.

In addition to runs with catalytic off-gas combustion, other runs were made using a modified laboratory burner as an off-gas after-burner. Results were not encouraging. Problems were probably the result of poor mixing of the off-gas and added air, low gas-stream temperature, and a short gas-residence time in the flame chamber. The oxidation catalysts have performed quite well, although those tested up to this time have been susceptible to HCl attack. Acid-resistant catalysts are now being procured and will be tested.
A much closer agreement was obtained by using the following equation, developed for a particle size distribution:

\[
V_{mf} = \frac{D_p^2 (\rho_s - \rho_g)^{0.9} (\rho_g)^{0.1} g_1}{800\mu}
\]

Where:
- \( V_{mf} \) = gas velocity
- \( \rho_s \) = true solid density
- \( \rho_g \) = gas density
- \( g_1 \) = acceleration of gravity
- \( \mu \) = gas viscosity
- \( D_p \) = mean particle diameter

\( D_p \) is calculated from a sieve analysis of the particles by a sum of the squares method. The calculations suggest that the gas flow rate through the proposed 4-in.-diameter tube should not exceed 1.6 feet per second.

**PLASTIC DRUM LINERS**

*J. W. Lindsay and P. A. Patton*

An improved liner for 55-gallon steel drums has been developed and is in use as an inner, secondary container for Rocky Flats contaminated wastes. To reduce a tendency for the snap-in lid to pop-out in drop tests, a modified design has been evolved and is in the final testing stages. Figure 19 shows the new liner with its screw-on lid. The liner is made of cross-linked polyethylene by a rotational molding process. Lid disengagement upon dropping is prevented by a tongue and groove arrangement between the top of the liner and the lid, and by use of a small quantity of low density urethane foam to stiffen the lid. Closure of the drum is facilitated by six molded recesses to accommodate a lug wrench.

The new drum liner, filled with water, can withstand a 4-foot drop without leaking. Encased in the 55-gallon steel drum, no leakage was observed after a 30-foot drop onto the bottom chime. Drop tests were run with the drum assembly filled with water-ethylene glycol mixture at -40°F. No leakage was observed after a 4-foot drop.

**REFERENCE**

RFP Reports


Publications
