Pacific Northwest Laboratories
Richland, Washington 99352

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IN NITRIC ACID SOLUTIONS**

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By

G. F. Schiefelbein

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BATTELLE
PACIFIC NORTHWEST LABORATORIES
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1.0 INTRODUCTION

The Zero Power Plutonium Reactor (ZPPR) located at the National Reactor Testing Station, Idaho Falls, plays a key role in the Atomic Energy Commission's Liquid Metal Fast Breeder Reactor program. The reactor has been used in experiments critical to the design of the Fast Flux Test Facility and commercial breeder reactors.⁽¹⁾

The ZPPR reactor is fueled with a stainless steel clad ternary alloy of depleted uranium, plutonium and molybdenum.⁽²⁾ This fuel and the scrap from fuel fabrication represent a substantial quantity of plutonium which must be recovered. To facilitate shipping to a recovery location, the declad ZPPR scrap has been burned to form a ternary oxide powder (68.7 wt % UO_2 - 28.0 wt % PuO_2 - 3.3 wt % MoO_3) at the Atomic Energy Commission's Rocky Flats Colorado facility. The burning was carried out by heating the alloy, in air, to a temperature sufficient to cause ignition and sustained burning.⁽³⁾

This study was undertaken to determine the optimum conditions for dissolving the ZPPR oxide for subsequent plutonium recovery in the Hanford Plutonium Reclamation Facility (PRF) operated by the Atlantic Richfield Hanford Company (ARHCO).

2.0 SUMMARY AND CONCLUSIONS

The dissolution properties of burned ZPPR fuel (oxide) in nitric acid solutions were investigated. Experimental variations were limited to those practical for use in the Plutonium Reclamation Facility (PRF).⁽⁴⁾ It appears possible to obtain complete dissolution of ZPPR oxide and simultaneously provide a suitable feed solution for the PRF separations plant. Both the molybdenum trioxide and PuO_2 fractions of the ZPPR oxide are known to be

sparingly soluble in nitric acid especially if they have been heated to high temperatures.^(5,6) In this respect, the conditions under which the ZPPR fuel is burned to form the oxide probably affects the dissolution properties of the oxide. The samples available for this study were not burned under carefully controlled conditions, but were merely heated to a temperature sufficient to cause ignition and sustained burning.⁽³⁾ The maximum temperatures reached during the burning process is dependent on several factors⁽⁷⁾ and the effects of this variable and others on the dissolution properties of the oxide should be examined.

Molybdenum is known to form a nitric acid soluble complex with iron(III).⁽⁸⁾ However, formation of this complex is apparently inhibited at the high nitric acid concentrations (12 M or greater) required to attain complete dissolution of the PuO_2 fraction of the oxide. Nearly complete dissolution of ZPPR oxide was attained when the oxide was exposed to boiling 12 M HNO_3 - 0.24 M NaF - 0.12 M $\text{Fe}(\text{NO}_3)_3$ solution for 24 hours at a loading of 70 grams of oxide per liter. However, this loading is below that required to provide the terminal plutonium concentration of 50-85 grams per liter desired for processing in the PRF.

An alternate approach to complete dissolution involved refluxing the ZPPR oxide in B-acid solution (PRF designation for 15.5 M HNO_3 - 0.65 M HF dissolvent) for three hours and then letting the solids settle prior to decanting the solids-free supernatant liquid. The solids from several batch dissolutions were allowed to accumulate in the dissolver before they were removed. The washed solids (mainly molybdenum oxide) contained only traces of plutonium (0.014 wt % of the original plutonium charged) and the solids can be periodically discarded as solid waste or dissolved in sodium hydroxide solution for disposal. The main objection to this approach is the long (4-8 hours) settling time required for each batch.

The solids can be removed from the dissolver solution by filtration through a cloth filter precoated with solids. However, the small particle size of the solids contributes to low filtration rates (2.8 ml/min/in.²)

even when employing vacuum filtration (20 in. Hg). The filtration rate can be more than doubled by the addition of 100 ppm gelatin, which acts as a flocculating agent, to the dissolver solution. Precoating the cloth filter with ashless filter pulp also increases the filtration rate and is more convenient to use than the flocculating agents.

Samples of the filtered solutions stored for 15 days in polyethylene bottles showed no signs of additional precipitation of solids. Thus, the filtered solutions should be stable during short storage periods at 25 °C. Installation of continuous centrifuges in the PRF (planned for FY-1973) should provide adequate solids removal. The solids settled quite readily when centrifuged in a laboratory centrifuge.

Dissolution rate tests conducted on "Rocky Flats" ZPPR oxide indicate that about 85 wt % of the PuO_2 present in the oxide is dissolved after 10-15 minutes exposure to boiling 15.5 M HNO_3 - 0.65 M HF solution. However, the remaining 15 wt % of the PuO_2 was quite refractory and required 3-4 hours reflux for complete dissolution. This behavior also occurred when the 12.2 M HNO_3 - 0.2 M NaF acid solution designated for use with the Purex dissolution flowsheet was used. These data suggest that substantial increases in oxide throughput could be attained by resorting to a continuous or semi-continuous dissolver operation coupled with a means of solids separation and recycle such as centrifugation.

3.0 EXPERIMENTAL

All operations involving ZPPR oxide were carried out in standard plutonium gloveboxes. ZPPR oxide samples were dissolved in cylindrical dissolver vessels constructed of 304-L stainless steel. Each dissolver was provided with a press-fit Teflon top equipped with a stainless steel water-cooled condenser and sampling port. Heating was provided by a laboratory hot plate. Details of the experimental procedures employed are discussed in sections 3.1 - 3.6 which follow.

3.1 Source of ZPPR Oxide

Burned ZPPR alloy prepared by ARHCO was used in the initial phases of this study. Burned ZPPR fuel scrap from Rocky Flats was utilized as soon as it became available.

3.2 Batch Dissolution Tests

The usual procedure employed in the dissolution tests involved charging a weighed amount of oxide and acid solution to the dissolver vessel, heating the solution to boiling and allowing it to reflux for the desired length of time. The dissolver was then removed from the hot plate and aluminum nitrate solution added slowly (while stirring) to complex fluoride ion and thereby reduce vessel corrosion. Sufficient aluminum nitrate was added to provide 0.70 - 0.75 moles of aluminum per mole of fluoride.

3.3 Batch Dissolution with Solids Accumulation

Difficulties in attaining complete dissolution of the three component ZPPR oxide mixture in nitric acid - hydrofluoric acid solutions prompted an evaluation of an alternate procedure. This alternate procedure consisted of adding fresh ZPPR oxide and 15.5 M HNO_3 - 0.65 M HF acid solution to a heel of settled solids remaining in the dissolver from previous batch dissolutions. This procedure is similar to that used in the PRF.

The mixture was heated to boiling and allowed to reflux for three hours. Heating was then stopped and aluminum nitrate solution (0.9 M) was added (0.75 moles of aluminum per mole of fluoride) to complex fluoride ion. The mixture was then left for 4-6 hours to allow the solids to settle. At the end of this settling period the solids-free supernatant solution was decanted, and the entire process was repeated.

3.4 Dissolution Rate Studies

Dissolution rate studies were performed on ZPPR oxide to determine the time at reflux required for complete dissolution of the plutonium present. The procedure employed consisted of heating the required amount of acid to boiling and adding the ZPPR oxide. Samples of the dissolver solution were taken periodically for determination of uranium, plutonium and molybdenum content. The dissolver solution samples were filtered immediately to prevent further dissolution of any ZPPR oxide particles suspended in the hot liquid. In this case the boiling solution was sparged with a slow stream of nitrogen (0.5 - 1.0 liters/min) to simulate actual PRF operation conditions.

3.5 Filtration Rate Studies

The PRF solvent extraction system requires a feed solution that is essentially solids free. When it became apparent that the twin goals of complete dissolution of ZPPR oxide and a concentration of 50-85 grams per liter plutonium in the terminal dissolver solution could not be met simultaneously, filtration studies were undertaken to determine the filtering characteristics of the ZPPR dissolver solution with and without the acid of flocculating agents.

The apparatus employed for the filtration tests consisted of a Buchner funnel (2.25 inch diameter) fitted with a cloth filter (nominal pore size $\sim 40 \mu$) under a vacuum of 20 in. Hg. The filtration tests were carried out on dissolver solution samples to which aluminum nitrate had been added. Flocculating agents were added to the hot dissolver solution subsequent to aluminum nitrate addition, as 0.5 wt % aqueous solutions. Sufficient flocculating agent was added to provide a concentration of 100 ppm in the dissolver solution. The dissolver solution was agitated during the addition and then allowed to cool to 65 - 70 °C. The solution was again stirred sufficiently to provide a uniform dispersion of solids and the filter was precoated with solids by filtering a 50 ml portion of the slurry without vacuum. A 250 ml portion of the slurry was then filtered under vacuum and the filtration time noted.

Solids removal from dissolver solutions in the PRF should be greatly facilitated by the installation of centrifuges now planned for FY-1973.

3.6 Analytical Methods

Samples were analyzed for uranium and plutonium content by x-ray fluorescence spectrophotometry and alpha-energy-analysis techniques respectively.

Molybdenum was separated from uranium and plutonium by adjusting the diluted dissolver solution samples to about pH 14 with 6 N NaOH. The solution was centrifuged to separate the molybdenum bearing supernatant liquid from the precipitated uranium and plutonium. The molybdenum bearing supernatant liquid was decanted and the precipitate redissolved in concentrated nitric acid. The extraction was then repeated and the molybdenum content of the combined supernatant liquids was determined spectrophotometrically.⁽⁸⁾

4.0 RESULTS AND DISCUSSION

The solvents evaluated for the dissolution of burned ZPPR fuel were limited to nitric acid-fluoride mixtures with and without the addition of iron (III). While more suitable solvents for burned ZPPR fuel undoubtedly exist, their use was prohibited because of possible incompatibilities with the PRF solvent extraction system.

4.1 Tests with ARHCO Produced ZPPR Oxide

Initial dissolution experiments were performed with ZPPR oxide prepared in small lots by ARHCO personnel.

4.1.1 Batch Dissolution Experiments

Several batch dissolution tests were carried on ARHCO produced ZPPR oxide to determine the dissolution characteristics of the material. Plutonium dioxide which has been heated to high temperatures is known

to be virtually insoluble in boiling nitric acid, unless fluoride ion has been added to the solution.^(6,10) Molybdenum trioxide is also relatively insoluble in nitric acid.⁽⁵⁾

Previous work has indicated that the solubility of molybdenum in nitric acid solutions is increased significantly in the presence of iron(III),⁽⁸⁾ perhaps through the formation of a negatively charged iron(III)-molybdenum complex. Accordingly, ZPPR dissolution trials were conducted with nitric acid solutions containing sodium fluoride (NaF) and ferric nitrate ($\text{Fe}(\text{NO}_3)_3$). The results of these dissolution trials are presented in Table I.

Trials 1 through 5 indicate that a nitric acid concentration of about 12 M is required to obtain complete dissolution of the PuO_2 fraction of the ZPPR oxide. Complete dissolution of the ZPPR oxide was not obtained in any of the trials. In trial 10, conducted at a loading of 70 grams of oxide per liter of dissolvent, only a small amount of solids remained; and it probably approached the maximum loading at which complete dissolution would be obtained. This loading yields a terminal dissolver solution which contains about 17.5 grams of plutonium per liter which is below the 50-85 grams per liter needed for a high capacity PRF flowsheet.

Thus it appears impossible to attain complete dissolution of the molybdenum fraction at a loading which will provide a high capacity feed to the PRF solvent extraction section. The high nitric acid concentration (12 M) required for complete dissolution of the PuO_2 fraction may inhibit formation of the hypothesized⁽⁸⁾ iron(III)-molybdenum complex $[\text{FeO}_6\text{Mo}_6\text{O}_{15}]^{-3}$, thereby preventing complete dissolution of the ZPPR oxide.

4.1.2 Batch Dissolution with Solids Accumulation

The data presented in the preceding section indicate that it is virtually impossible to obtain complete dissolution of the ZPPR oxide

Table I

DISSOLUTION OF ZPPR OXIDE IN BOILING
 HNO_3 -HF- $\text{Fe}(\text{NO}_3)_3$ SOLUTIONS

Conditions: Twenty-four hours exposure to boiling HNO_3 -0.24 M
 NaF - 0.12 M $\text{Fe}(\text{NO}_3)_3$ solution.

Trial	Loading, grams/liter	Initial HNO_3 , M	Final HNO_3 , M	PuO_2 Dissolved wt %	Comments
1(a)	330	8.4	6.3	44	Solids remaining ^(b) after 5 hours.
2	330	8.4	4.8	60	Solids remaining after 24 hours.
3	330	9.6	4.3	86	Solids remaining after 24 hours.
4	330	11.0	4.5	90	Solids remaining after 24 hours.
5	330	12.2	5.6	98	Solids remaining after 24 hours.
6	220	12.2	5.3	100	Solids remaining ^(c) after 24 hours.
7	140	15.0	6.5	100	Solids remaining after 24 hours.
8	140	12.2	7.3	100	Solids remaining after 24 hours.
9	70	15.0	9.5	100	Small amount of solids remaining after 24 hours.
10	140	(d)	12	100	Solids remaining ^(b) after 5 hours.

(a) Five-hour exposure time.

(b) X-ray analysis indicated solids were primarily Pu and Mo.

(c) X-ray analysis indicated solids were primarily Mo.

(d) B-acid (15.5 M HNO_3 - 0.65 M HF) was used, iron(III) was omitted.

and still obtain the desired plutonium concentration in the dissolver solution. Therefore, an alternate approach was evaluated wherein the solids from each batch were allowed to settle and only the solids-free supernate was removed from the dissolver vessel. In this approach the solids were allowed to accumulate as a dissolver heel which was retained from batch to batch.

An important question regarding this approach was how much plutonium would be retained with the solids. If the solids were primarily composed of MoO_3 with little plutonium content, then the solids could be disposed of as such, or dissolved in a sodium hydroxide solution and disposed of as liquid waste. Accordingly, dissolution experiments were conducted in which eight consecutive batches of ZPPR oxide, each at a loading of 285 grams of oxide/liter, were cycled through the dissolver by this procedure to determine the magnitude and composition of the accumulated solids. This loading should yield a final plutonium concentration of 70 grams/liter. The results of these experiments are exhibited in Table II.

Table II
ACCUMULATED ZPPR SOLIDS

<u>Oxide Lot No.</u>	<u>No. Batches Dissolved</u>	<u>Total Oxide Throughput</u>	<u>Total Solids Collected</u>	<u>Wt % of Total Oxide</u>	<u>Solids Comp.</u>	<u>Wt % Pu Dissolved</u>
ZP-608	8	275 grams	17.3 grams (airdried 24 hrs @ 25 °C)	6.3	Pu 0.056 wt% U 0.032 wt% Mo ~ 60 wt% Fe ~ 0.5 wt% Mn Cr } Small Ti } Amounts Ni }	100
ZP-604	8	381	5.7 (oven- dried 24 hrs @ 120 °C)	1.5		100

These data indicate that very little plutonium remains associated with the solids. A third batch of solids was accumulated from lot ZP-604 ZPPR oxide in like manner. These solids were washed with nitric acid to remove residual plutonium and successively contacted with 200 ml portions of 50% and 10% sodium hydroxide solution at room temperature. The solids were essentially completely dissolved by this treatment.

4.2 Tests with Rocky Flats ZPPR Scrap

When burned ZPPR fuel scrap from Rocky Flats became available additional testing was conducted to further characterize its dissolution properties.

4.2.1 Dissolution Rates - PRF Flowsheet

In view of the data presented in Table I, a B-acid flowsheet was selected as the most promising candidate for processing ZPPR oxide in the PRF.

Dissolution rate experiments were conducted at a loading of 285 grams of ZPPR oxide per liter of B-acid to determine the time at reflux required for complete dissolution of the PuO_2 fraction. These data are summarized in Table III. The data suggest that 3 to 4 hours at reflux are required to attain complete PuO_2 dissolution. In both trials (A&B) about 85% of the PuO_2 fraction was dissolved after only 10-15 minutes at reflux. The remaining 15% of the PuO_2 was apparently quite refractory and required 3 to 4 hours at reflux for complete dissolution. This suggests that substantial economies could be affected by resorting to continuous or semi-continuous dissolver operation coupled with a means of removing and recycling solids. This mode of operation should have real advantages for a facility like PRF, where critically safe dissolvers are already available.

Also of interest is the behavior of molybdenum. Note that initially (after 10 minutes) about 10% of the available molybdenum was in the

TABLE III

ZPPR OXIDE DISSOLUTION RATE DATA - PRF FLOWSHEET

Trial A

<u>Time at Reflux, min</u>	<u>Uranium in Solution, g/l</u>	<u>Plutonium in Solution, g/l</u>	<u>Molybdenum in Solution, g/l</u>
15	157	48	0.62
30	159	49	0.50
60	163	50	0.36
90	166	50	0.31
120	171	52	0.32
150	---	53	0.27
180	176	55	0.16
210	179	56	0.14
270	179	56	0.15

Trial B

10	165	60	0.70
35	165	62	0.43
60	168	63	0.36
120	167	66	0.28
180	168	69	0.17
300	168	70	0.17

solution, but with continued refluxing the amount in solution gradually fell to about 2.5%. This may have been due to the gradual polymerization of soluble isopolymolybdic acids to form insoluble MoO_3 .

The data presented in Table III also suggest that the burned ZPPR fuel may vary somewhat in composition. Note the difference in terminal uranium and plutonium concentrations between trials A and B. The oxide used in these trials came from different lots. Calculated terminal concentrations are 172 grams/liter uranium and 70 grams/liter plutonium. The lot of ZPPR oxide used in Trial A appears to have contained a higher ratio of uranium to plutonium than the lot of material used in Trial B.

The solids remaining after reflux in Trial B were collected by filtration, washed with 8 M HNO_3 , dried for 24 hours at 120 °C and weighed. The solids amounted to 2.7 wt % of the original ZPPR oxide charge.

4.2.2 Dissolution Rates - Purex Flowsheet

The PRF solvent extraction system has a limited capacity for processing high uranium feeds. Therefore, consideration was given to a plan whereby ZPPR oxide would be dissolved and filtered in the PRF and shipped to Purex for final processing. This flowsheet utilized a 12 M HNO_3 - 0.2 M NaF dissolvent at a loading of 285 grams of ZPPR oxide per liter. Dissolution rate data for this system are shown in Table IV.

Again there is evidence of possible inhomogeneity between different lots of ZPPR oxide. Note that in this trial a dissolvent of lower acid and fluoride concentration was used, and yet more plutonium has been dissolved after 10 minutes reflux than in trials A and B. One would expect the opposite result if the ZPPR oxide were homogenous. Note also that the molybdenum concentration in the terminal dissolver solutions was nearly twice that present with the B acid flowsheet. Presumably this increase in solubility is due to the reduced acidity of the Purex dissolver solution.

TABLE IV

ZPPR OXIDE DISSOLUTION RATE DATA - PUREX FLOWSHEET

<u>Time at Reflux, min</u>	<u>Uranium in Solution, g/l</u>	<u>Plutonium in Solution, g/l</u>	<u>Molybdenum in Solution, g/l</u>
10	159	67.8	0.50
30	163	70.2	0.65
75	167	71.5	0.57
135	166	72.0	----
195	167	73.3	0.59
255	167	72.8	0.42
315	167	73.1	0.35

The solids were separated, washed, dried for 24 hours at 120 °C and weighed. They amounted to 3.1 wt % of the ZPPR oxide charged to the dissolver. They contained about 0.5 wt % plutonium, 0.2 wt % uranium and 65 wt % molybdenum. The amount of plutonium in the solids thus amounted to about 0.06 wt % of the total present in the original oxide.

4.3 Filtration Rate Studies

The molybdenum-bearing solids remaining in the terminal dissolver solution require a prohibitively long settling time to obtain a reasonable volume of solids-free supernatant liquid. The small particle size of the solids results in rapid filter blinding when the dissolver solution is filtered through a 40 μ filter cloth under vacuum. Accordingly, the effect of various flocculating agents and filter-aids toward improving filtration rates were evaluated. Results of this evaluation are presented in Table V.

TABLE V

EFFECT OF VARIOUS AGENTS ON ZPPR
DISSOLVER SOLUTION FILTRATION RATES

Agent	Concentration, (1) ppm	Filtering Rate (2) ml/min/in ²
None	---	2.8
PURIFLOC 502 ⁽⁴⁾	100	4.6
PURIFLOC 501 ⁽⁴⁾	100	3.9
SEPERAN 2610 ⁽⁴⁾	100	5.0
Gelatin	100	6.3
Ashless Filter Pulp	(3)	5.0

1. Additive concentration in final dissolver solution.
2. Calculated on the basis of the time required to pass 250 ml of a homogeneous mixture of solids-bearing dissolver solution through a 2.25 in. diameter filter under a 20 in. Hg vacuum. The filter (40 μ cloth) had been precoated as described in section 3.5.
4. Applied to filter cloth as a precoat.

Gelatin was the most effective flocculating agent tested. Ashless filter pulp is probably the most convenient filter aid since it eliminates both the addition of reagents to the dissolver solution and precoating the filter cloth with solids from a portion of the dissolver solution.

4.4. Post Precipitation of Solids

As described previously (Section 3.5) the filtration experiments were performed with the dissolver solution at 65-70 °C. During filtration the solution cooled to about 45 °C. The possibility existed therefore, for additional precipitation of solids from the filtered dissolver solution as it cooled further. Samples of filtered dissolver solution prepared by the PRF and by the Purex flowsheets were stored in polyethylene bottles for 15 days. At the end of this time the

solutions were filtered through membrane filters (1.2 μ pore size) to determine if any post precipitation of solids had taken place. No solids were observed on the filters.

5.0 ACKNOWLEDGEMENTS

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