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AEC Research and Development Report

PLATINUM - CATALYZED
HYDRAZINE REDUCTIONS
OF PLUTONIUM (IV)
AND URANIUM (VI)

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PLATINUM-CATALYZED HYDRAZINE REDUCTIONS
OF PLUTONIUM(IV) AND URANIUM(VI)

J. L. Swanson
Chemistry and Metallurgy Division

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BATTELLE
PACIFIC NORTHWEST LABORATORIES
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ABSTRACT

Platinum-catalyzed hydrazine reductions of plutonium(IV) and uranium(VI) were studied for possible application in the separation of plutonium from uranium in the Purex solvent extraction process. Reaction rates, reaction stoichiometry, catalyst stability, and catalyst poisoning were studied. An unidentified catalyst poison was found to be present in the uranium product stream of the Hanford Purex plant.

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PLATINUM-CATALYZED HYDRAZINE REDUCTIONS OF PLUTONIUM(IV) AND URANIUM(VI)

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INTRODUCTION

In the Purex process for the recovery and purification of uranium and plutonium from irradiated reactor fuels, separation of the plutonium from the uranium is generally accomplished by reducing extractable plutonium(IV) to inextractable plutonium(III). The traditional reagent for accomplishing this reduction, ferrous sulfamate, has been used successfully for many years. The use of ferrous sulfamate, however, unavoidably contributes iron and sulfate to the waste stream. These materials may lead to increased corrosion of waste concentration-storage equipment, and also add to the volume of waste which must be stored. Alternate reductants which do not introduce contaminants to the waste stream have been under study for several years. The leading contender has been uranium(IV) but interest in hydroxylamine nitrate has been increasing. Another contender, platinum-catalyzed hydrogen reduction, was scouted by Rainey.⁽¹⁾ In experiments aimed at extending the work of Rainey it was found that reduction of plutonium(IV) and uranium(VI) occurred when solutions containing hydrazine (added as a "holding reductant" to react with nitrous acid) were contacted with platinum catalyst in the absence of added hydrogen. The catalyzed hydrazine reductions appeared to have advantages over the catalyzed hydrogen reductions, especially for reduction within plant streams. The results of the study of the catalyzed hydrazine reductions are the subject of this report.

SUMMARY AND CONCLUSIONS

The platinum-catalyzed hydrazine reductions of plutonium(IV) and of uranium(VI) were studied. The reduction of uranium(VI) was emphasized after preliminary results demonstrated that, at the uranium to plutonium

ratios in the Hanford Purex plant, the bulk of the plutonium reduction would occur via a path involving uranium(IV).

The effects of temperature and of the concentrations of uranium(VI), hydrazine, and nitric acid on the rate of uranium(IV) formation were determined in both beaker-type and column-type experiments. The results demonstrated that rapid generation of uranium(IV) is easily achieved under a variety of conditions.

A major limitation on the use of this process became evident with the discovery that a material which poisons the catalyst was present in plant uranium product solution; subsequent studies indicate that the poison may enter the plant as an impurity in the uranium metal fuel elements and follow the uranium throughout the plant. Attempts to identify this poison were unsuccessful. Certain elements which do act as catalyst poisons were identified but none is thought to be the one present in the plant solutions.

EXPERIMENTAL PROCEDURE

GENERAL

Cylindrical catalyst pellets were obtained from Engelhard Industries in their standard size of 1/8 inch diameter by 1/8 inch high. The pellets contained 0.5 wt% platinum, on an alumina substrate.

Hydrazine was converted to the nitrate form ($\text{N}_2\text{H}_5\text{NO}_3$) before being used in these experiments. Hydrazine (64% in water) was diluted with half its volume of water, cooled in an ice bath, and slowly neutralized with cold concentrated nitric acid.

In determining the solvent extraction behavior of catalyst poisons, runs were made with feeds prepared from both the portion that did not extract and the portion that did extract (after being stripped from the organic and reconcentrated). In the extraction step a 2 M uranyl nitrate solution was contacted with twice its volume of 30% TBP-NPH, giving about 0.5 M U in the organic and 1 M U in the aqueous. Stripping was accomplished with numerous portions of water. In those cases where the path

of a certain element was being followed, the element was added to a purified UNH solution prior to the extraction.

Uranium(IV) was determined spectrophotometrically after dilution under standard conditions to eliminate variations in the molar extinction coefficient.

Hydrazine concentrations were determined by measuring (spectrophotometrically) the amount of nitrous acid remaining after addition of a known amount of sodium nitrite to an aliquot of the sample (plus HNO_3 to establish standard conditions). Measurement was made immediately after mixing so that oxidation of uranium(IV) did not have time to occur. Results obtained in this way agreed well with those employing the p-dimethylaminobenzaldehyde method;⁽²⁾ the latter method was not used because its great sensitivity required large dilution factors with the high hydrazine concentrations used here.

Ammonia concentrations were determined by titration after distillation from basic solution. Prior destruction of the hydrazine was required to avoid interference. This was accomplished with alkaline permanganate or bromine.⁽³⁾

Nitric acid concentrations were determined by titration after complexing the uranium with fluoride.

PLUTONIUM(IV) REDUCTION

Plutonium solutions were stirred in a glass beaker containing catalyst pellets for a measured time period and the spectrum of the solution was then measured to determine the extent of reduction. The solution was then returned for another time period and the spectrum run again. Argon was swept through the beaker to exclude air. These experiments were run at ambient temperature (about 25°C).

URANIUM(IV) FORMATION - BEAKER EXPERIMENTS

Solutions were stirred in contact with catalyst pellets in a stainless steel beaker fitted with a stainless steel cover having ports to

allow stirring, sampling, etc. The beaker rested on a thermistor-controlled hot plate, with the thermistor immersed in the reactant solution. To start an experiment a solution containing the uranium and nitric acid was brought to temperature in the reaction beaker and the hydrazine solution was then added. The hydrazine solution was brought to temperature in a separate beaker before being added. These experiments normally used about 5 grams of catalyst pellets and 50 to 100 ml of solution.

Temperature control during an experiment was good to within 0.5°C . Variations of several degrees from one experiment to another sometimes occurred, however, due to poor set-point reproducibility. For precise comparisons, data were normalized to the same temperature using the experimentally determined activation energy.

URANIUM(IV) FORMATION - COLUMN EXPERIMENTS

The small column used in most of this work was made from a stainless steel pipe with end caps tapped to accommodate fittings for passage of a metal thermometer and solution inlet and outlet lines. The inlet line was at the bottom of the column. The maximum bed height was about 1.5 inches; the bed diameter increased gradually from 0.2 inch at the bottom to 1 inch at a height of 0.75 inch and was 1 inch the remainder of the way. About 13 grams of catalyst pellets were used in this arrangement. The bottom of the outlet tube was near the top of the pellet bed to minimize the hold-up of solution. The volume of solution held in the column was about 9 ml, based on the observation that the half-time for uranium wash-out was about 9 minutes at flow rate of 1 ml/min.

The column and contents were heated in an oil bath. The reaction temperature, which was measured with a thermometer positioned within the bed, depended not only on the temperature of the oil bath but also on the rate of solution flow and the rate of the exothermic reaction. In runs where the rate of the reaction varied drastically, the reaction temperature could vary by several degrees at the rate extremes.

A few experiments were done with a column containing 270 grams of pellets in a bed 1 inch in diameter by 20 inches high to verify that comparable results were obtained on a significantly larger scale.

EXPERIMENTAL RESULTS

PLUTONIUM(IV) REDUCTION

The platinum-catalyzed hydrazine reduction of plutonium(IV) was investigated only briefly. Results obtained in the presence and in the absence of uranium are given in Table I and are plotted in Figure 1. More rapid reduction is seen to occur with uranium present; in these experiments the effect of 0.12 M UNH was to increase the rate of reduction of plutonium(IV) to the extent of about 0.0002 M/min. In a similar experiment in the absence of plutonium, the rate of uranium(IV) formation was found to be 0.0001 M/min. These results indicate that the reduction of plutonium(IV) proceeds via two paths at these uranium(VI) concentrations; the catalyzed hydrazine reduction of plutonium(IV) itself and the catalyzed hydrazine reduction of uranium(VI) to uranium(IV), which then rapidly reduces plutonium(IV).

TABLE I. Reduction of Plutonium(IV)

<u>Pu(IV)</u> <u>in Feed, M</u>	<u>Time in Contact</u> <u>with Catalyst, min</u>	<u>Pu(IV) Reduced, M</u>		<u>Increase</u> <u>Due U(VI)</u>
		<u>In Presence</u> <u>of 0.12 M U(VI)</u>	<u>In Absence</u> <u>of U(VI)</u>	
0.0019	1	0.0007	0.0005	0.0002
	3	0.0017	0.0012	0.0005
	5		0.0016	
0.0047	1	0.0016	0.0014	0.0002
	3	0.0033	0.0028	0.0005
	5	0.0043	0.0038	0.0005
	7		0.0044

FIGURE 1. Reduction of Plutonium(IV)

The relative importance of these two paths of plutonium(IV) reduction would depend on the relative concentrations of uranium(VI) and plutonium(IV). At the concentrations used in these experiments, the catalyzed hydrazine reduction of plutonium(IV) predominated; at sufficiently high uranium(VI) to plutonium(IV) ratios, the path involving uranium(IV) would dominate. Under the experimental conditions used here, where the catalyzed hydrazine reduction of plutonium(IV) itself followed first-order kinetics with a half-time of two minutes and the reduction of plutonium(IV) by the path involving uranium(IV) proceeded at a rate of 0.0002 M/min, the two paths would contribute equally during one minute at a plutonium(IV) concentration of about 0.0006 M (giving a uranium(VI) to plutonium(IV) ratio of 200). Since potential applications of the catalyzed hydrazine reductions in the Hanford Purex plant would involve uranium(VI) to plutonium(IV) ratios at least this high, emphasis of the study was placed on the generation of uranium(IV).

URANIUM(IV) FORMATION

Reaction Stoichiometry

One reasonable way of picturing the catalyzed hydrazine reduction of uranium(VI) is to think of it as a platinum-catalyzed decomposition of hydrazine into nitrogen and hydrogen

followed by the reduction of uranium(VI) by the hydrogen thus formed

The apparent reaction then would be

In actual fact, while about 1.5 moles of hydrogen ion were found to be consumed per mole of uranium(IV) formed under a variety of conditions, the hydrazine consumption was significantly greater than 0.5 mole hydrazine per mole of uranium(IV) formed.

One possible explanation for the hydrazine consumption being greater than that predicted from equation (3) is that some of the hydrazine is being reduced to ammonia by the hydrogen

Data concerning ammonia formation are given in Table II. It is seen that the amount of ammonia formed increases with increasing hydrazine to uranium(VI) ratio; in agreement with the idea of hydrazine competing with uranium(VI) for the active hydrogen. The amount of ammonia found in these solutions was, however, less than the amount predicted from equations (3) and (4), indicating that other reactions are also important.

TABLE II. Ammonia Formation
Beaker-Type Experiments
~53°C

Feed Composition, M			$\frac{\text{N}_2\text{H}_5\text{NO}_3}{\text{U(VI)}}$	Concentration in Product, M		$\frac{\text{NH}_4^+}{\text{U(IV)}}$	Hydrazine Consumption [mole per mole U(IV)]
U(VI)	HNO ₃	$\frac{\text{N}_2\text{H}_5\text{NO}_3}{\text{U(VI)}}$		U(IV)	NH_4^+		
2.0	0.7	0.55	0.28	0.59	0.03	0.05	0.68
2.0	0.7	0.91	0.46	0.49	0.07	0.14	0.67
1.8	0.6	0.71	0.39	0.80	0.06	0.08	0.64
1.5	1.2	0.92	0.61	0.49	0.06	0.12	0.69
0.5	1.2	0.90	1.8	0.31	0.17	0.55	1.42

The gas evolved during reaction was analyzed in one experiment and no hydrogen was found. At the time the sample was taken for analysis the solution still contained large amounts of reducible material (1.8 M uranium(VI) and 0.4 M hydrazine).

Reaction Rate

The effects of varying solution composition and temperature were investigated in both beaker-type and column-type experiments. Comparable results were obtained in the two types of experiments with the exception that the effect of nitric acid concentration depended on the type of experiment employed.

The reaction rate did, of course, vary with the catalytic activity of the catalyst pellets. Decreases in catalytic activity occurred on continued use of the pellets, but the change was very gradual so that comparison of results was valid providing the experiments were done close together. Such was the case with the following results unless otherwise noted.

Data from beaker-type experiments at two different uranium(VI) concentrations are shown in Figure 2. There was essentially no difference in the initial rates of uranium(IV) formation but hydrazine disappeared more rapidly from the solution containing less uranium. Thus, the hydrazine consumption (moles hydrazine reacted per mole uranium(IV) formed) increased with decreasing uranium(VI) concentration.

The rate of uranium(IV) formation was found to increase with increasing hydrazine concentration, as shown in Figure 3. The rate of hydrazine disappearance also increased with increasing concentration, so that little variation in hydrazine consumption was observed.

Results of beaker-type experiments at two different nitric acid concentrations are shown in Figure 4. These data show acid concentration to have little effect on either the rate of uranium(IV) formation or the hydrazine consumption in beaker-type experiments. In column-type experiments, however, the acid concentration did affect the rate of uranium(IV) formation (higher acid concentration giving lower uranium(IV) formation rates), as shown in Table III. The reason for the different effect in the different types of experiments is not known.

Increasing the temperature resulted in increases in both the rate of uranium(IV) formation and the rate of hydrazine disappearance, as shown in Figures 5 and 6. Up to at least 54°C the increases were proportional to each other so that no change in hydrazine consumption occurred. The activation energy was calculated to be about 14 Kcal. The probable reason for the unexpectedly low rate of uranium(IV) formation in the experiment at 65°C is that a back-reaction involving re-oxidation of uranium(IV) became more important at the higher temperature.

FIGURE 2. Effect of Uranium(VI) Concentration

FIGURE 3. Effect of Hydrazine Concentration

FIGURE 4. Effect of Nitric Acid Concentration in Beaker-Type Experiments

FIGURE 5. Effect of Temperature

FIGURE 6. Temperature Effect

TABLE III. Effect of Nitric Acid Concentration in Column-Type Experiments

Set ^(a)	Feed Composition, <u>M</u>			U(IV) in Product, <u>M</u>	Rate of Reaction (millimole/min)	
	<u>U(VI)</u>	<u>HNO₃</u>	<u>N₂H₅NO₃</u>		<u>U(IV)</u>	<u>N₂H₅⁺</u>
A	1.5	0.6	0.9	0.45	0.41	-0.24
A	1.5	1.2	0.9	0.27	0.24	-0.12
B	1.5	1.2	0.9	0.46	0.43	-0.30
B	1.5	2.4	0.9	0.16	0.16	-0.14
C	0.75	0.6	0.45	0.21	0.20	
C	0.75	1.2	0.45	0.13	0.11	
C	0.75	1.8	0.45	0.09	0.085	
C	0.75	2.4	0.45	0.07	0.067	

a. Data were obtained in three sets, using pellets of varying catalytic activity. Comparison within sets is valid, but not between sets.

CATALYST POISONING

In the beaker-type experiments, little if any difference was observed between solutions prepared from crystallized UNH (uranyl nitrate hexahydrate) or from uranium product solution from the Hanford Purex plant. In the column-type experiments, however, a very important difference was readily apparent--with feed from plant product solution, the rates of reaction decreased as the volume of solution fed to the column increased, as shown by the data in Figure 7.

The poisoning effect was found to depend on the concentration of poison present in the feed, as seen in Figure 8. There is apparently at least a semi-quantitative correlation between the concentration of poison and the rate at which the concentration of uranium(IV) in the product decreases with the amount of feed put through the column. To obtain feeds of varying poison content, a portion of the uranium in a plant product sample was removed by crystallization as UNH. The poison remained in the supernatant solution; its relative concentration was calculated from the fraction of the uranium that had been removed as crystals. Recombination

FIGURE 7. Comparison of Plant and Purified Uranium Solutions

FIGURE 8. Effect of Poison Concentration

of portions of the supernatant and crystalline fractions in known ratios then provided feeds of known poison contents on a relative basis, at least.

The poisoning effect was also found to depend on the catalytic activity of the pellets, as shown in Figure 9. As the pellets became less active, decreases occurred in both the maximum concentration of uranium(IV) and the rate at which this concentration decreased. Because of these changes, quantitative comparisons among many of the experiments were impossible. In the data shown, comparison within a given figure is felt to be valid but comparison of data in one figure with that in another may not be valid because of differing catalytic activities.

Eight samples of plant product solution were tested and all were found to contain poison, though there was some variation in concentration, as seen in Figure 10. Two samples came from storage tanks but the other six were current product. These were taken over a period of time to check the consistency of the amount of poison present. The data in Figure 10 were obtained with three of these samples covering the range of poison content.

FIGURE 9. Effect of Catalytic Activity on Poisoning by Plant Solution

Attempts to identify the poison present in the plant solutions were unsuccessful. Analysis on the spark source mass spectrograph did not disclose the identity, even when the concentration level was increased six-fold (by removing most of the UNH by crystallization). The poison was concluded to be inorganic in nature since it was not removed by passing the solution through columns of activated charcoal or alumina, by extended boiling in HNO₃ solution, or by calcining the UNH to UO₃. The activity of poisoned catalyst was readily restored by washing with nitric acid but not by washing with nitric acid plus hydrazine. This indicates that the poisoning action is caused by a reduced form of the poison element.

One important property of the poison present in plant product solution is that it follows uranium very closely through solvent extraction steps (employing tributyl phosphate extractant). When half of the uranium was extracted, then stripped into water and used as feed to the catalyst column, the poisoning behavior was the same as with feed made from the half that did not extract, indicating that half of the poison had also extracted.

FIGURE 10. Variations in Poison Content of Plant Solutions

The fact that the poison follows uranium so closely on solvent extraction means that the poison could enter anywhere in the process and still end up in the uranium product solution. Evidence was indeed obtained to indicate that the poison enters the process as an impurity in the uranium metal used in the fuel elements. Feed solution prepared by dissolving uranium from an N-Reactor fuel element (non-irradiated) was found to contain poison at a level comparable to the plant product solution samples. The poison present in this dissolver solution was also found to closely follow uranium on solvent extraction, as did the poison present in plant product solution.

In attempting to identify the poison present in plant product solution by a different means, many elements were added to feed solutions to determine experimentally whether or not they poisoned the catalyst. Several elements were found to poison; these are listed in Table IV along with those tested that did not poison, and the concentration level to which the element was added. In these scouting tests, a salt of the added element was boiled with plant product solution for several hours before the solution was used as feed. Later tests showed poisoning also occurred when the salts were simply added to solutions prepared from crystallized UNH.

TABLE IV. Elements Tested for Poisoning Action

<u>Poisons</u>	<u>Non-Poisons</u>
Bismuth	Arsenic (3×10^{-4} <u>M</u>)
Copper	Cobalt (3×10^{-4} <u>M</u>)
Lead	Iron (0.003 <u>M</u>)
Molybdenum	Manganese (3×10^{-4} <u>M</u>)
Thallium	Phosphorus (0.005 <u>M</u> DBP, MBP, or H_3PO_4)
Vanadium	Selenium (0.1×10^{-4} <u>M</u>)
	Sulfur (0.012 <u>M</u>)
	Technetium (0.2×10^{-4} <u>M</u>)
	Tellurium (3×10^{-4} <u>M</u>)
	Tin (3×10^{-4} <u>M</u>)
	Titanium (2×10^{-4} <u>M</u>)
	Tungsten (3×10^{-4} <u>M</u>)

While bismuth, copper, lead, molybdenum, thallium, and vanadium were all found to act as catalyst poisons, none of these is thought to be the one present in plant product solution. Copper was found to be inextractable so was ruled out on that basis. Bismuth, lead and thallium were somewhat extractable but not to the extent of the plant poison. The results obtained with bismuth and lead are shown in Figure 11; results with thallium were essentially identical to those with lead.

Extraction of vanadium and molybdenum was not tested--the elimination of them as suspects is based on the fact that the effect of adding either of these elements to feed solutions was not the same as the effect observed with plant poison. As shown in Figure 12, addition of vanadium or molybdenum did result in an immediate lowering of the concentration of uranium(IV) in the product but little change in the rate at which the uranium(IV) concentration decreased further. Addition of the plant poison resulted in an increase in the rate at which the uranium(IV) concentration decreased, in addition to the initial lowering of the uranium(IV) concentration (as was seen in Figure 8).

CATALYST PELLET STABILITY

Standard platinum-on-alumina pellets purchased from Engelhard Industries were not very resistant to nitric acid--a weight loss of 9% occurred in four days at 50°C in 2 M UNH + 1.6 M HNO₃. Special pellets (employing a higher density alumina) prepared for us by Engelhard were much more resistant to nitric acid (about 1% weight loss in 2 M HNO₃ at 50°C for two months) and were of comparable catalytic activity. These special pellets were used in the work reported here.

Continued use of even the special pellets led to gradual reduction of the catalytic activity, evidently due to erosion of the platinum. In one instance in which the pellets were exposed to 1 to 2 M HNO₃ at about 50°C for 109 days with solution flowing about 60% of the time, the catalytic activity decreased to about one-fourth the initial level. During this time the platinum content decreased from 0.4 to 0.07%.

FIGURE 11. Results with Bismuth and Lead
(added to crystallized UNH solutions)

FIGURE 12. Results with Molybdenum and Vanadium (added to plant U solution)

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