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**The Spectrophotometric Measurement
of Rhenium in Plutonium-Rhenium Alloys**

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by

J. W. Dahlby
G. R. Waterbury

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THE SPECTROPHOTOMETRIC MEASUREMENT OF RHENIUM IN PLUTONIUM-RHENIUM ALLOYS

by

J. W. Dahlby and G. R. Waterbury

ABSTRACT

Microgram quantities of rhenium in 100-mg. samples of plutonium-rhenium alloys are measured spectrophotometrically as the furil dioxime complex following separation from plutonium by a double-distillation from sulfuric-hydrochloric acid solution. Furil dioxime, acetone, and stannous chloride are added to the distillate to form the colored complex, and the absorbance is measured at a wavelength of 532 m μ . The measured molar absorptivity of the complex is 40,100. Based upon a total of 42 determinations of 13.4, 134.4, and 201.6 μ g. (134 ppm, 0.1344%, and 0.2016%) of rhenium in 100-mg. samples, relative standard deviations of 1.4, 0.34, and 0.47%, respectively, were obtained. The method was applied successfully to measurement of rhenium concentrations as high as 8%. Of 35 ions investigated, Cu²⁺, Ga³⁺, Hg²⁺, Mo⁶⁺, NO₃¹⁻, Pd²⁺, Pt⁴⁺, Ru²⁺, Sb³⁺, and Ta⁵⁺ caused an interference of greater than 2% when present in 50-mg. quantities.

INTRODUCTION

Metallurgical investigation of the solubility of rhenium in plutonium metal created a need for a method that measured low concentrations of rhenium in these alloys. A search of the literature showed that thiocyanate^(2, 3, 12) and several substituted dioximes^(1, 7, 8, 10, 11, 13) are suitable color-forming reagents for the spectrophotometric measurement of rhenium. Of these reagents, furil dioxime forms the most highly colored rhenium complex, having a molar absorptivity of about 40,000. Unfortunately, nitric acid interferes seriously with the formation of this complex. As nitric and hydrochloric acids generally are used in the complete dissolution of plutonium-rhenium alloys,

either removal of the nitrate ion from the solution or the development of some other method of dissolution was necessary. Volatilization is the usual method of removing nitric acid, but this introduces some risk of losing rhenium as the volatile heptoxide. Therefore, another dissolution method for plutonium-rhenium alloys was sought.

In an effort to bypass the use of nitric acid and at the same time take advantage of the volatility of rhenium oxide to separate it from plutonium, combined dissolution-distillation methods were investigated. The distillation of rhenium can be carried out in several ways. The most frequently used method is steam distillation from sulfuric acid solutions at 270 to 290°C.⁽⁴⁾ In other methods,

rhenium is distilled from a solution containing either hydrochloric or hydrobromic acid with sulfuric acid, sulfuric-phosphoric acids, or perchloric-phosphoric acids.^(5,6) Of these solvents, hydrochloric-sulfuric acid seemed the most amenable to the dissolution and distillation of plutonium-rhenium alloys. At the elevated temperature necessary to distill rhenium oxide, the plutonium-rhenium alloys dissolved completely. As described here, this separation was successfully coupled with the spectrophotometric measurement of rhenium as the furil dioximate to develop a method for determining 130 parts per million to 0.2 percent of rhenium in alloys with plutonium.

APPARATUS AND REAGENTS

APPARATUS

Burner, Bunsen.

Distillation unit, borosilicate glass, consisting of a hydrochloric acid vaporizing pot, a still pot, and a condenser as shown in Figure 1. An air supply line having a needle valve control and a flow meter is required with this apparatus.

Fig. 1. Distillation Unit.

Heater, Precision, Electric, having exposed heating coils.

Miscellaneous equipment. Normal laboratory glassware including beakers, pipets, and volumetric flasks.

Spectrophotometer, Beckman Model DU or similar instrument with cells having 1-cm. light paths.

REAGENTS

Acetone, reagent grade.

Furil Dioxime, 4.5% solution. Prepare the solution just before use by dissolving 0.450 grams of Eastman White Label furil dioxime in 10 ml. of acetone.

Hydrochloric acid, reagent grade, 12N.

Hydrochloric acid, 6N. Dilute 12N hydrochloric acid with an equal volume of water.

Perchloric acid, reagent grade, 70%.

Plutonium solution, 50 mg./ml. Dissolve 10 grams of plutonium metal in 100 ml. of 6N hydrochloric acid and dilute to 200 ml. with water. The total concentrations of the detected impurities were less than 100 ppm in the metal used in this work.

Potassium permanganate, saturated aqueous solution.

Rhenium stock solution. Accurately weigh 200 to 400 mg. of Re metal and dissolve it in hydrochloric-perchloric acids using the sealed-tube method.⁽⁹⁾ Dilute this solution accurately to 200 ml.

Rhenium solution, 40 to 80 $\mu\text{g.}/\text{ml.}$ Dilute 4 ml. of the rhenium stock solution to 100 ml. with 15 ml. of 6N hydrochloric acid and water.

Stannous chloride, 48% solution. Dissolve 1.2 grams of the reagent-grade salt in 2 ml. of 12N hydrochloric acid and dilute to 25 ml. with water. Filter the solution through Whatman No. 42 filter paper. Prepare this solution just prior to using.

Sulfuric acid, reagent grade, 36N.

RECOMMENDED PROCEDURE

Caution: Health safety rules for the handling of plutonium must be rigidly followed, and adequate protection for the operator must be ensured by the use of suitable dry boxes and protective clothing.

1. Add an accurately weighed, 100-mg. sample to the still pot, connect the pot to the still head, and seal the standard-taper joint with 1 drop of 36N sulfuric acid.
2. Connect the water condenser to the still pot and seal the semi-ball joint with 1 drop of 36N sulfuric acid. Insert the delivery tube of the condenser into a 50-ml. volumetric flask.
3. Add 0.5 ml. of 12N hydrochloric acid through the top opening in the still pot, allow the dissolution reaction to subside, and then wash the acid from the entrance tube with a few drops of water. Add 1 ml. of saturated potassium permanganate solution to the still pot, and then 1 ml. of water.
4. Add 2 ml. of 6N hydrochloric acid to the vaporizing pot, connect this pot to the still pot and the air supply line to the top of the vaporizing pot, then adjust the air flow to approximately 20 cc./min.
5. Evaporate the solution in the still pot to dryness by heating it with the electric heater. Allow the still to cool slightly, disconnect the vaporizing pot from the still, add 0.4 ml. of 36N sulfuric acid through the top opening of the still pot, and then reconnect the two pots.
6. Heat the sulfuric acid in the still pot to fuming, and with the Bunsen burner heat the vaporizing pot until the hydrochloric acid is completely evaporated. At this point, nearly all of the sulfuric acid should be distilled from the still pot.
7. Cool the apparatus for 5 minutes, then disconnect the air supply line from the vaporizing pot and the vaporizing pot from the still pot.
8. Add 2 ml. of 6N hydrochloric acid to the vaporizing pot, and 3 ml. of water and 2 drops of 36N sulfuric acid to the still pot. Then reconnect the apparatus.
9. Heat the contents of the still pot until all the water is evaporated, disconnect the vaporizing pot from the still pot, add 0.3 ml. of 36N sulfuric acid to the still pot, and then reconnect the two pots.
10. Again heat the still pot until the sulfuric acid sealing the standard-taper joint fumes; then evaporate the hydrochloric acid in the vaporizing pot to dryness. At this point, no sulfuric acid should be left in the still pot.
11. Rinse the condenser with water into the 50-ml. receiver flask.
12. Allow the still pot to cool for 5 minutes, dissolve the residual plutonium sulfate in water and a few drops of sulfuric acid, and transfer this solution to a residue bottle.
13. Develop the colored complex in the 50-ml. volumetric flask by adding 10 ml. of acetone, 1 ml. of 4.5% furil dioxime, and 1 ml. of 48% stannous chloride, with mixing between each addition.
14. Dilute the solution to volume with water, mix thoroughly, and allow the colored complex to develop for 1 hour.
15. Measure the absorbance of the solution at 532 m μ , using a spectrophotometer equipped with cells having 1-cm. light paths.
16. Prepare a calibration curve by analyzing various amounts (between 13 and 200 μ g. of Re) of the standard rhenium solution added to 100 mg. of plutonium in solution, being sure the quantity of hydrochloric acid in the still pot does not exceed 8 milliequivalents. Calculate the quantity of rhenium in an unknown sample from its measured absorbance and data obtained from the calibration curve.

EXPERIMENTAL

SEPARATION OF RHENIUM

Volatilization of Rhenium. The first method tried for separating rhenium from plutonium was the volatilization of rhenium oxide by ignition of the

sample in oxygen that contained some hydrogen chloride. In this way the problem of dissolving the sample was avoided entirely. The sample was heated to 1000°C for 30 minutes in a fused silica combustion tube, and oxygen that had been bubbled through hydrochloric acid was passed through the tube. The volatilized rhenium was removed from the effluent gas in a water trap, the solution was diluted to a known volume, and an aliquot containing 13 to 200 micrograms of rhenium was transferred to a 50-milliliter volumetric flask. The rhenium furil dioximate was formed and measured spectrophotometrically as described in the RECOMMENDED PROCEDURE. Application of this method to volatilization and measurement of rhenium in pieces of the pure metal weighing from 3.2 to 7.5 milligrams showed that the rhenium was volatilized and trapped quantitatively. For 15 measurements, the average for the rhenium found was 100 percent and the relative standard deviation was 0.7 percent.

Application of this method to volatilization of rhenium from plutonium - 6 percent rhenium alloy was not successful. The plutonium dioxide remaining in the combustion boat retained 0.5 to 3.4 percent of the rhenium in the alloy. This was proven by dissolving the plutonium dioxide in hydrochloric acid using the sealed-tube method,⁽⁹⁾ and measuring the rhenium spectrophotometrically. Longer combustions, crushing the plutonium dioxide residue, and reheating and breaking the plutonium-rhenium alloy into small pieces prior to combustion did not significantly reduce the quantity of rhenium retained by the plutonium dioxide. When the temperature was increased to 1400°C, more of the rhenium was volatilized, but 0.2 to 0.8 percent still remained with the plutonium dioxide. As this method was unsuccessful in attaining a complete separation of the rhenium, other separation methods were considered.

Distillation of Rhenium. Several methods are reported^(4, 5, 6) in which rhenium is distilled from solutions containing hydrochloric or hydrobromic acid and sulfuric acid, sulfuric-phosphoric acids, or perchloric-phosphoric acids. Perchloric acid was not tried because its boiling point is lower than that of sulfuric acid. Preliminary work show-

ed that rhenium distills from sulfuric and hydrochloric acids, and that phosphoric acid, which precipitates plutonium, need not be used. In order to ensure that the rhenium was carried from the still pot into the condenser, a stream of air flowing at a rate of about 20 cubic centimeters per minute was passed through the distillation apparatus. To maintain a significant concentration of hydrochloric acid in the still pot at the distillation temperature, a vaporization pot was placed in the system immediately before the still pot (see Figure 1). Hydrochloric acid was placed in the vaporization pot which was heated with a Bunsen burner during the distillation to provide a continuing flow of hydrochloric acid into the still pot. Under these conditions most of the rhenium was distilled from plutonium-rhenium alloys. However, analysis of the plutonium sulfate remaining in the still pot following a distillation showed that a significant quantity of rhenium was in the residue. Therefore, the method was modified to include a second distillation. This was done by cooling the still pot following the initial distillation, adding water to dissolve the plutonium residue, and then adding sulfuric acid. The apparatus was reconnected, and a second distillation was made. The plutonium sulfate residues left after the second distillation of alloys containing 4 to 8 milligrams of rhenium were dissolved by the sealed-tube method, and the rhenium in the resulting solution was measured spectrophotometrically. The results showed that 99.9 percent of the rhenium was dissolved and distilled.

Effect of Plutonium. The oxidation state of the plutonium in the still pot was found to have a significant effect on the method. If the distillation was done with plutonium in the (III) oxidation state, a precipitate formed during the rhenium-furil dioxime color development, and the results were low. Oxidation of the plutonium to the (IV) or (VI) oxidation state prior to distillation of the rhenium prevented formation of this precipitate. Several oxidizing agents, including ammonium persulfate, potassium persulfate, sodium hypochlorite, iodic acid, cerium (IV), argentic oxide, and potassium permanganate, were tried to ensure oxidation of the plutonium (III). Of these, only potassium permanganate neither caused low results nor formed dif-

difficultly soluble salts with plutonium. Therefore the distillation method was modified to include oxidation of the plutonium by adding 1 milliliter of saturated potassium permanganate solution.

The effects of various quantities of plutonium on the distillation of rhenium were investigated by analyzing solutions containing 44.8 micrograms of rhenium and 100 to 1000 milligrams of plutonium by the RECOMMENDED PROCEDURE. Satisfactory results for rhenium were obtained for solutions containing either 100 or 250 milligrams of plutonium (Table I). Larger quantities of plutonium caused low results for rhenium. However, 100 milligrams is considered the maximum sample size in the existing equipment because larger amounts of plutonium caused violent bumping as the plutonium sulfate solution was heated.

Table I
The Effect of Plutonium on the
Distillation of Rhenium
(44.8 μ g. of rhenium separated)

<u>Pu Added, mg.</u>	<u>Re Found, %</u>
100	101
250	100
500	96
1000	93

Effect of High Rhenium Concentrations. To determine the efficiency of the dissolution and separation of high concentrations of rhenium, 79- to 106-milligram samples of five plutonium alloys that

contained 4 to 8 percent rhenium were analyzed by this method. Each plutonium sulfate residue was then dissolved either in 1N hydrochloric acid, in 6 N hydrochloric acid containing hydrogen peroxide, or by the sealed-tube method ⁽⁹⁾ in 12N hydrochloric acid. The rhenium in these solutions was then measured spectrophotometrically. This was done by forming the rhenium furil dioximate as described in the RECOMMENDED PROCEDURE, and then extracting the complex from the 50-milliliter aqueous phase into 10 milliliters of chloroform ⁽¹⁰⁾ to increase the concentration of the color complex five-fold. The absorbance of the extract was measured at a wavelength of 532 millimicrons. The results showed that less than 0.05 percent of the rhenium did not dissolve and distill (Table II).

To supplement the data in Table II showing that high concentrations of rhenium in alloys with plutonium dissolve and distill quantitatively, two aliquots containing 10.18 milligrams of rhenium and 100 milligrams of plutonium were analyzed by the method to determine the efficiency of the trapping, as well as the distillation, of large quantities of rhenium. Following distillation of the rhenium from each aliquot, a measured fraction of the distillate was taken for the formation of the furil dioxime complex and the spectrophotometric measurement. The average of 99.6 percent for the rhenium found showed that 10.18 milligrams of rhenium may be distilled from 100 milligrams of plutonium and trapped quantitatively. It was concluded from these results and the values in Table II that rhenium concentrations as high as 8 percent

Table II
The Dissolution and Distillation of Plutonium-Rhenium Alloys

<u>Sample Weight, mg.</u>	<u>Re Distilled, mg.</u>	<u>Re Left in Still Pot</u>	
		<u>μg.</u>	<u>%</u>
94.5	7.34	1.0 ^(a)	0.01
106.2	8.70	3.1 ^(a)	0.03
97.0	7.69	3.1 ^(a)	0.04
79.5	4.65	1.8 ^(b)	0.04
79.2	3.28	0.7 ^(c)	0.03

(a) $\text{Pu}(\text{SO}_4)_2$ dissolved in 1N HCl.

(b) $\text{Pu}(\text{SO}_4)_2$ dissolved in 6N HCl and 6 drops 30% H_2O_2 .

(c) $\text{Pu}(\text{SO}_4)_2$ dissolved using the sealed-tube method. ⁽⁹⁾

can be determined reliably in 100-milligram sample of plutonium-rhenium alloys.

SPECTROPHOTOMETRIC DETERMINATION OF RHENIUM

The spectrophotometric measurement of rhenium is very similar to the method described by Meloche, Martin, and Webb. ⁽⁸⁾ As the reliability of this method has been proved also by other investigators, ^(1, 7) the concentrations of acetone, furil dioxime, and tin (II) recommended by Meloche et al. were used without further testing, and the absorbance of the colored complex was measured at the recommended wavelength of 532 millimicrons. However, coupling the distillation separation with the spectrophotometric measurement changed the acidity for the formation of the colored complex. In addition, the maximum quantity of rhenium that can be measured by this method was not known. Therefore, the effects on the method of various acid concentrations and of large quantities of rhenium were determined.

Effect of Acid Concentration. In the original work, ⁽⁸⁾ the colored complex was developed in 0.8N hydrochloric acid, but following the distillation separation, the color-forming solution contains a mixture of approximately equinormal concentrations of hydrochloric and sulfuric acids. The approximate concentration of each acid when the distillate is diluted to 50 milliliters is 0.6N, but the total acidity may vary slightly for successive distillations. In order to determine the effect of various acid concentrations on this method, the total acidity for the color development was varied from 0.8 to 1.8N. In each case the quantity of rhenium was 44.8 micrograms and the concentrations of the two acids were approximately equal. Table III shows that acid concentrations from 0.8 to 1.4N do not adversely affect the color development, but higher acidities produce low results for rhenium. The usual acidity of 1.2N following a distillation is within the range that is acceptable in the spectrophotometric measurements.

Table III

The Effect of Total Acid Concentration
on the Determination of Rhenium
(44.8 μ g. Re present in each sample)

Acid Concentration, <u>N</u>	Re Found, %
0.8	100
1.0	100
1.2	100
1.4	99
1.6	96
1.8	91

Effect of Large Quantities of Rhenium. The range of the method was determined by pipetting aliquots containing 202 to 336 micrograms of rhenium into 50-milliliter flasks, and then measuring the rhenium spectrophotometrically as described in the RECOMMENDED PROCEDURE. It was found (Table IV) that 224 micrograms of rhenium is the maximum amount tolerated in the spectrophotometric measurement. However, it was shown previously (Effect of High Rhenium Concentrations) that as much as 10 milligrams of rhenium can be determined provided an aliquot of the distillate containing less than 224 micrograms of rhenium is taken for the spectrophotometric measurement.

Table IV

Range of the Spectrophotometric
Determination of Rhenium

Re Taken, μ g.	Re Found, %
202	100
224	100
246	98.6
269	97.2
291	96.0
336	90.5

RELIABILITY

In the absence of homogeneous standard samples of plutonium-rhenium alloys, the reliability of this method was determined by analyzing solutions hav-

ing known plutonium and rhenium concentrations. The rhenium was distilled from each solution and then measured spectrophotometrically as described in the RECOMMENDED PROCEDURE. For 14 determinations each of 13.4, 134.4, and 201.6 micrograms of rhenium, relative standard deviations of 1.4, 0.34, and 0.47 percent were obtained (Table V). From these absorbances, a molar absorptivity of 40,100 was calculated.

The measured absorbances (Table V) were compared with those obtained when the colored complexes were formed directly with known quantities of rhenium (Table VI). As the absorbances for equal quantities of rhenium are about the same, it is obvious that the distillation does not introduce a bias in the method. The relative standard deviations in Tables V and VI also are about equal, showing that the precision of the method is dependent on the reproducibility of the spectrophotometric measurement rather than the distillation. It was concluded from these data, and also from the measurements made on plutonium-rhenium alloys as described previously, that the method is unbiased.

EFFECTS OF OTHER IONS

The effects of other ions on this method were determined by analyzing solutions containing 134.4 micrograms of rhenium, 50 milligrams of plutonium, and 50 milligrams of the ion being tested. The ion was added in the form shown in Table VII. Duplicate determinations of rhenium in the presence of each of 35 ions showed that 10 ions caused an interference greater than 2 percent. Of these 10 interfering ions, only seven interfered when the quantity added was reduced to 1 milligram in a solution containing 100 milligrams of plutonium and 134.4 micrograms of rhenium (Table VII). It is obvious that alloys containing significant quantities of volatile metals or metals that form volatile chlorides cannot be analyzed for rhenium by this method.

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Table V

Determination of Known Quantities of Rhenium in 100 Milligrams of Plutonium

Re Taken, $\mu\text{g.}$	Number of Determinations	Average Absorbance	Absorbance per mg. of Re	Relative Std. Dev., %
		532 $m\mu$		
0	13	0.002	-	20
13.4	14	0.058	4.3 ₂	1.4
134.4	14	0.578	4.30	0.34
201.6	14	0.868	4.30	0.47

Table VI

Spectrophotometric Measurement of Rhenium

Re Taken, $\mu\text{g.}$	Number of Determinations	Average Absorbance	Absorbance per mg. of Re	Relative Std. Dev., %
		532 $m\mu$		
13.4	18	0.055	4.0 ₉	1.6
26.9	16	0.116	4.30	0.71
89.6	16	0.389	4.34	0.45
179.2	16	0.775	4.32	0.49

Table VII

The Effects of Other Ions on the Determination of Re
(Ions added in the form shown)

50 mg. of Ion Added ^(a)		1 mg. of Ion Added ^(b)	
Non-Interfering Ions	Interfering Ions ^(c)	Non-Interfering Ions	Interfering Ions ^(c)
Mn ²⁺ (MnSO ₄), Sn ²⁺ (SnCl ₂),	Hg ²⁺ (HgCl ₂)	Ta ⁵⁺ (TaCl ₅)	Hg ²⁺ (HgCl ₂)
Ni ²⁺ (NiCl ₂), Cr ²⁺ (CrCl ₂),	Ga ³⁺ (metal) (d)	Sb ³⁺ (metal) (d)	Ga ³⁺ (metal) (d)
Mg ²⁺ (MgSO ₄), Bi ³⁺ (metal), (d)	Cu ²⁺ (CuSO ₄)	Cu ²⁺ (CuSO ₄)	Ru ²⁺ (RuCl ₂)
Na ¹⁺ (NaCl), K ¹⁺ (KCl),	Sb ³⁺ (metal) (d)		Pd ²⁺ (PdCl ₂)
Co ²⁺ (CoSO ₄), VO ²⁺ (VOSO ₄),	Ru ²⁺ (RuCl ₂)		Pt ⁴⁺ (H ₂ PtCl ₆)
Cd ²⁺ (CdCl ₂), Pb ²⁺ (PbCrO ₄),	Pd ²⁺ (PdCl ₂)		Mo ⁶⁺ ((NH ₄) ₆ Mo ₇ O ₂₄)
TiO ²⁺ (TiOSO ₄), Ag ¹⁺ (Ag ₂ SO ₄),	Pt ⁴⁺ (H ₂ PtCl ₆)		NO ₃ ¹⁻ (NH ₄ NO ₃)
Fe ²⁺ (FeSO ₄), Zn ²⁺ (metal), (d)	Ta ⁵⁺ (TaCl ₅)		
Zr ⁴⁺ (ZrOCl ₂), Ti ¹⁺ (TiCl),	Mo ⁶⁺ ((NH ₄) ₆ Mo ₇ O ₂₄)		
La ³⁺ (LaCl ₃), Nb ⁵⁺ (Nb ₂ (SO ₄) ₅),	NO ₃ ¹⁻ (NH ₄ NO ₃)		
Ge ⁴⁺ (GeO ₂), SO ₄ ²⁻ (MgSO ₄),			
F ¹⁻ (NaF), Cl ¹⁻ (KCl),			
ClO ₄ ¹⁻ (NaClO ₄)			

(a) 50 mg. Pu and 134.4 μ g. Re in each determination.

(b) 100 mg. Pu and 134.4 μ g. Re in each determination.

(c) The Re found differed from the Re added by more than 2 percent.

(d) Dissolution of the metal in hydrochloric acid formed the ion shown.

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