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# DETERMINATION OF CADMIUM IN SOLUTIONS CONTAINING URANIUM, NEPTUNIUM, AND PLUTONIUM

L. W. GRAY, L. A. OVERMAN, and H. F. HODGENS



E. I. DU PONT DE NEMOURS AND COMPANY  
SAVANNAH RIVER LABORATORY  
AIKEN, SOUTH CAROLINA 29801

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT AT(07-2)-1

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**L. W. GRAY, L. A. OVERMAN, and H. F. HODGENS**

Approved by

J. L. Womack, Superintendent  
Separations Technology Department  
Savannah River Plant

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## ABSTRACT

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An analytical method was developed for determining cadmium in solutions containing fissionable materials and high alpha activities. The solutions were first extracted with 30% tributyl phosphate in dodecane to separate the actinide elements from the cadmium. This extraction step removed interfering ions and reduced the alpha activity. The cadmium concentrations were determined by atomic absorption analysis. The method has been applied to solutions containing 20 to 30 g Cd/L, about 10 g Pu/L (38%  $^{238}\text{Pu}$ ), about 15 g U/L (66%  $^{235}\text{U}$ ) and about 0.05 g Np/L with a standard deviation for cadmium of 2.65%. The fissionable materials were generally reduced from about 25 g/L to about  $10^{-7}$  g/L in the solutions analyzed, i.e., about 0.5 picogram of fissionable material was introduced to the atomic absorption flame.

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## **DETERMINATION OF CADMIUM IN SOLUTIONS CONTAINING URANIUM, NEPTUNIUM, AND PLUTONIUM**

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### INTRODUCTION

In the chemical processing of fissionable nuclear material, a neutron absorbing material such as cadmium is sometimes added to the process solutions to maintain the system subcritical by an adequate margin. The Savannah River Plant (SRP) has used cadmium as a neutron "poison" for the recovery of materials from scrap containing varying amounts of enriched uranium, plutonium-238, plutonium-239, and neptunium. To maintain critically safe process control, a rapid and reliable analytical method for cadmium was needed for process solutions containing very high levels ( $10^8$  to  $10^{12}$  dis/(min-mL) of alpha activity. Because of the biological hazard presented by the solutions containing a plutonium alpha content in this range,<sup>1,2</sup> the volume of sample must be kept to a minimum even though the samples are handled in glove boxes.

This report describes a method that was developed in which 30% tributyl phosphate (TBP) in dodecane was used to extract the actinide elements and decontaminate the alpha activity so that atomic absorption analysis could then be used for cadmium determination.

Because prevention of a nuclear accident sometimes depends upon a sufficient amount of neutron-capturing material, e.g., cadmium, being present in the process tanks,<sup>3</sup> a rapid method that also required a minimum of analytical technique was essential for determining the concentration of neutron-absorbing material in solution. Many methods have been developed for the determination of cadmium.<sup>4</sup> The high alpha content of the SRP samples, however, complicated the use of these methods. Direct dilution of the samples followed by atomic absorption could not be used because of the biological hazard.<sup>1,2</sup> Classical precipitation<sup>5</sup> such as CdS, CdMoO<sub>4</sub>, or CdNH<sub>4</sub>PO<sub>4</sub>·H<sub>2</sub>O generally required about 24 hours per determination<sup>6</sup> because of interferences. Chelometric titrations<sup>4,5</sup> were also eliminated because of interferences. Ion-selective electrodes were rapidly poisoned<sup>6</sup> in these solutions and therefore could not be used.

Solutions generated in the SRP scrap recovery lines vary with the type of scrap being recovered. Typical dissolving solutions contain varying concentrations of nitric acid, aluminum nitrate, and hydrofluoric acid. Depending upon the source of the scrap, the final solution may contain natural, depleted,

or slightly enriched to fully enriched uranium in the concentration range of 1 g/L to about 100 g/L; plutonium which ranges from <1 to 90 wt %  $^{238}\text{Pu}$  and from <5 to 93 wt %  $^{239}\text{Pu}$  in the concentration range of from <1 to about 200 g/L; and neptunium in the range of <1 to 50 g/L. Because the alpha content of these solutions frequently range as high as  $10^{12}$  dis/(min-mL), a small sample size was desirable.

## DEVELOPMENT OF METHOD

### Reagents and Solutions

All chemicals, except standards, were either technical grade or prepared from regular mill-run grade products. The 30% TBP solutions were used either in the virgin state or after pre-equilibration with nitric acid solutions.

A synthetic stock solution (I) was prepared by diluting a nominal (but not primary standard) 100 g Cd/L standard and a nominal 100 g U/L solution to yield a solution containing  $\sim 25$  g Cd/L and  $\sim 25$  g U/L in 8M  $\text{HNO}_3$ . This stock solution was submitted to two analysts who made 20 individual extractions and dilutions to prepare the samples for atomic absorption analysis. Because the uranium was in the U(VI) oxidation state, no valence adjustment was necessary.

As Stock Solution I was believed to be about 5% more concentrated than the calculated value, a second set of stock solutions was prepared. A 100.0 g Cd/L stock solution (II) was shown by analysis to contain  $100.00 \pm 0.98$  g Cd/L. Dilution of Stock Solution II with  $\text{UO}_3(\text{NO}_3)_2$  solution and nitric acid yielded a synthetic stock solution (III) containing  $25.00 \pm 0.25$  Cd/L - 25 g U/L in 8M  $\text{HNO}_3$  (Table 1).

TABLE 1  
Composition of Stock Solutions

No.	Cd, g/L	U, g/L	Ratio Cd/U	$\text{HNO}_3$ , M
I	$\sim 100$	$\sim 25$	4:1	8
II	$100 \pm 0.98$	25.0	4:1	8
III	$25.0 \pm 0.25$	25.0	1:1	8



Process samples were drawn from the scrap recovery line which contained about 16.75 g U/L (66%  $^{235}\text{U}$ ) and 9.14 g Pu/L (38%  $^{238}\text{Pu}$ , 56%  $^{239}\text{Pu}$ ) to which about 30 g Cd/L had been added. Because the scrap material was dissolved in an 8M  $\text{HNO}_3$  solution at near boiling temperatures, the valence of plutonium was virtually all in the extractable Pu(IV) and Pu(VI) states. Valence adjustment was therefore not necessary.

## Analytical Methods

Uranium was determined by the isotope dilution mass spectrographic method; plutonium was determined by alpha pulse height analysis; and cadmium was determined by using a Farrell-Ash Model 82-810 spectrophotometer in the atomic absorption mode.

## EXTRACTION OF CADMIUM INTO TBP

### Selection of Organic Extractant

Solvent extraction methods were investigated to isolate the cadmium from the uranium, neptunium, and plutonium. These extraction methods require minimum time, simple equipment, and can be easily performed with small samples. Because tri-n-butylphosphate (TBP) effectively extracts U(VI),  $^{7-9}\text{Np(IV)}$ ,  $^{7-9}\text{Np(VI)}$ ,  $^{7-9}\text{Pu(IV)}$ , and  $^{7-9}\text{Pu(VI)}$  from 3 to 8M nitric acid (Figures 1 and 2), a minimum of valence adjustment and nitric acid concentration adjustment was necessary before extraction. TBP (30 vol % in dodecane) was a logical choice for the extractant. Other extractants, such as thenoyltrifluoroacetone (TTA) or tri-n-butylphosphine oxide (TBPO),  $^{7,8}$  could be used, but these extractants require more effective valence control and more accurate adjustment of the nitric acid concentration for effective isolation of the cadmium.

### Extraction Technique

All extractions were carried out in 4-dram vials. All solutions were introduced to the vials via microliter pipets. After equilibration of the aqueous and organic phases with a vortex mixer for about one minute, the phases were allowed to separate. The organic phase was removed with a slurry pipet and discarded. After three extractions, an aliquot of the aqueous phase was removed for gross alpha determination via counting and for determination of cadmium via atomic absorption. The experimental procedure is outlined in the Appendix.

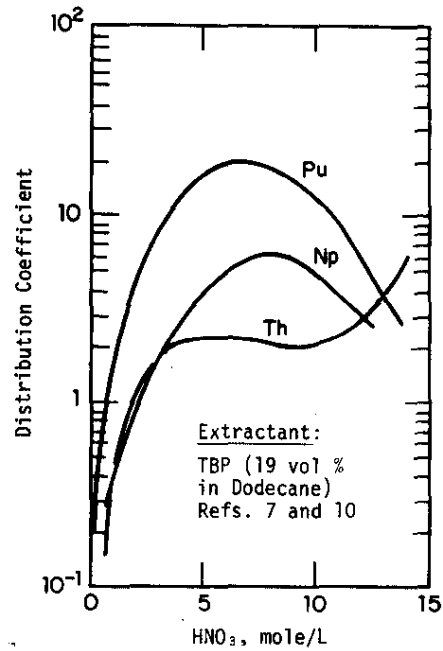


FIGURE 1. Distribution Coefficients for Extracting Plutonium, Neptunium, and Thorium in the Tetravalent State

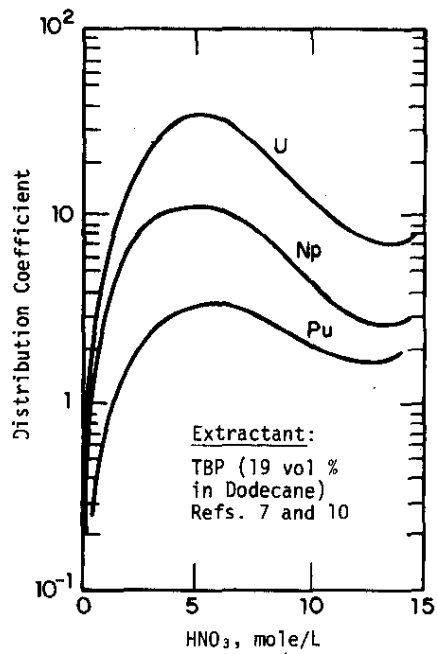


FIGURE 2. Distribution Coefficients for Extracting Uranium, Neptunium, and Plutonium in the Hexavalent State

The general procedure followed was to add 100  $\mu\text{L}$  of sample to 1500  $\mu\text{L}$  of 0.1M  $\text{HNO}_3$  in a 4-dram vial. Then 2.5 mL of 30% TBP pre-equilibrated with 4M  $\text{HNO}_3$  was added to the 4-dram vial. The vial was capped and agitated for 2 minutes on a vortex mixer. The phases were allowed to separate and the organic slurried off with a slurry pipet and discarded. The extraction was repeated twice more. In general, approximately 93 to 97% of the residual uranium, neptunium, and plutonium was extracted each time. This resulted in a decontamination factor for plutonium and uranium of about  $10^4$ , i.e., the alpha content was reduced from about  $1.5 \times 10^{11}$  dis/(min-mL) to about  $1 \times 10^7$  dis/(min-mL). Upon dilution of an aliquot to the range of 0.5 to 2.5  $\mu\text{g Cd/L}$  (a dilution of about  $10^4$ ) the plutonium content of the sample introduced to the atomic absorphotometer flame had been reduced from about 10 g/L to about  $7 \times 10^{-7}$  g/L. Without the extraction step, the plutonium content would have been reduced from about 10 g/L to only about  $7 \times 10^{-3}$  g/L. This concentration would have presented an unacceptable biological risk for introducing specimens into the flame of the atomic absorption spectrophotometer.

#### Effect of Nitric Acid Concentration

In general, the extraction of U(VI), Np(IV), Np(VI), Pu(IV), and Pu(VI) is greatest in the nitric acid range of 3M to 8M (Figures 1 and 2). Therefore, for the most effective decontamination of actual process solutions, the acid must be adjusted to this range before extraction [see Steps 1 and 2 in Procedure (Appendix)]. It was necessary, however, to show that cadmium is not extracted at these nitric acid concentrations.

Two sets of experiments were carried out: one set with virgin 30% TBP, and one set with 30% TBP which had been pre-equilibrated with nitric acid. The first set of extractions were performed with a standard solution containing 102.8 g Cd/L in 4M  $\text{HNO}_3$  and a simulated process solution containing 25.70 g Cd/L and 25.0 g U/L in a 4M  $\text{HNO}_3$  solution. Data (Table 2) show that an average of only  $34.7 \pm 1.9\%$  of the cadmium is extracted into the virgin solvent. Although not investigated further, this loss may be due to entrainment as the virgin solvent absorbs water and acid.

The second set of extractions were performed with standard solutions containing approximately 4.7 g Cd/L. The nitric acid concentration was varied from 1M to 10M. Before each extraction, the 30% TBP solvent was pre-equilibrated with a nitric acid solution of the same concentration as the sample to be extracted. Data (Table 3) show that within experimental error, cadmium is not extracted into 30% TBP over the nitric acid concentration range of 1M to 10M, if the solvent is first pre-equilibrated with nitric acid of approximately the same concentration. Although

not statistically significant, there was a downward trend in the aqueous cadmium determinations as the number of extractions increased, especially at nitric acid concentrations greater than 5M. As this could result in a negative bias, samples should be adjusted to 3M to 5M before the extraction of the uranium, neptunium, or plutonium.

## ACCURACY AND PRECISION

Cadmium results of duplicate analyses of a series of actual process samples are given in Table 4. The relative deviation for cadmium for this series was 2.65%. The RSD for the 100 g Cd/L (standard) was 1.23% with a bias of -1.50%. The RSD for the 25 g Cd/L (synthetic) was 0.68% with a positive bias of 0.36% (Table 5). These data confirmed that cadmium could be determined in actual process solutions with a precision at the 95% confidence level of  $\pm 2.4\%$  and with a positive bias of no more than 5%.

TABLE 2

Loss of Cadmium to Virgin 30% TBP Solution

	<i>Synthetic, 25.70 g Cd/L</i>		<i>Standard, 102.8 g Cd/L<sup>a</sup></i>	
	<i>Cd Found, g/L</i>	<i>Recovered, %</i>	<i>Cd Found, g/L</i>	<i>Recovered, %</i>
After first extraction	16.2	63.0	64.8	63.0
After second extraction	17.0	66.1	67.2	65.4
After third extraction	17.3	67.3	68.8	66.9

a. Stock Solution I (Table 1).

TABLE 3

Nitric Acid and Cadmium Analysis of Aqueous Phases after Successive Extractions

<i>Original</i>		<i>After First Extraction</i>		<i>After Second Extraction</i>		<i>After Third Extraction</i>		<i>Average</i>
<i>H<sup>+</sup>, M</i>	<i>Cd, g/L</i>	<i>H<sup>+</sup>, M</i>	<i>Cd, g/L</i>	<i>H<sup>+</sup>, M</i>	<i>Cd, g/L</i>	<i>H<sup>+</sup>, M</i>	<i>Cd, g/L</i>	
1.03	4.8 $\pm 0.26^a$	1.02	4.7 $\pm 0.17$	1.01	4.79 $\pm 0.17$	1.01	4.79 $\pm 0.17$	4.80 $\pm 0.17$
2.77	4.84 $\pm 0.11$	2.77	4.79 $\pm 0.17$	2.77	4.79 $\pm 0.17$	2.79	4.72 $\pm 0.10$	4.79 $\pm 0.13$
5.13	4.79 $\pm 0.25$	5.13	4.70 $\pm 0.24$	5.13	4.68 $\pm 0.28$	5.09	4.55 $\pm 0.17$	4.67 $\pm 0.23$
7.54	4.68 $\pm 0.28$	7.54	4.68 $\pm 0.28$	7.57	4.45 $\pm 0.06$	7.59	4.45 $\pm 0.06$	4.56 $\pm 0.21$
9.55	4.74 $\pm 0.25$	9.55	4.68 $\pm 0.28$	9.57	4.55 $\pm 0.17$	9.57	4.55 $\pm 0.17$	4.63 $\pm 0.22$

a. The  $\pm$  values are 1 $\sigma$  values for duplicate analysis obtained on four separate dilutions of each sample. The relative standard deviation for this set of experiments was 4.69%.

TABLE 4

## Precision Study "A" for Cadmium

Sample	Cadmium Concentration, g Cd/L			
	Analyst A		Analyst B	
	Dilution A	Dilution B	Dilution A	Dilution B
1	28.2	28.1	29.3	29.2
2	29.7	29.4	29.5	29.6
3	32.8	35.2	33.2	34.4
4	34.2	33.6	33.2	32.5
5	31.6	32.2	30.4	32.4
6	31.5	31.7	32.0	31.5
RSD, %	2.41		3.11	
RSD, % (total)	2.65			

TABLE 5

## Precision Study "B" for Cadmium

Sample	Standard Solution, <sup>a</sup> 100 g Cd/L	Synthetic Solution, <sup>b</sup> 25 g Cd/L - 25 g U/L
1	97.3	25.15
2	97.7	25.28
3	99.1	24.88
4	99.9	25.05
Average	98.5	25.09
1 $\sigma$	1.21	0.17
RSD, %	1.23	0.68
Bias, %	-1.50	+0.36

a. Stock Solution II (Table 1).

b. Stock Solution III (Table 1).

The general procedure used was to add 2000  $\mu\text{L}$  to Stock Solution I and 3000  $\mu\text{L}$  of 0.1M  $\text{HNO}_3$  to a 4-dram vial. Then 5 mL of 30% TBP was pipeted into the 4-dram vial. The vial was capped and then agitated on the vortex mixer for about 2 minutes. After allowing phase separation, the organic phase was slurried out of the tube with a slurry pipet. The extraction was repeated twice more. An aliquot of the extracted aqueous layer was then removed and diluted with 3M  $\text{HNO}_3$ , such that the concentration of cadmium in the diluted sample would be in the range of 0.5 to 2.5  $\mu\text{g/mL}$ . Analyst A used an overall dilution factor of 25,000 to yield a series of 10 samples with a nominal 1.00  $\mu\text{g Cd/mL}$ . Analyst B used an overall dilution factor of 33,333 to yield a series of 10 samples with a nominal 0.75  $\mu\text{g Cd/mL}$ . These 20 samples were then submitted for atomic absorption analysis where all determinations were made by a third analyst. As shown in Table 6, the relative standard deviation (RSD) for Analyst A was 0.89%; for Analyst B, the RSD was 0.69%. However, the bias for Analyst A was +3.11%; for Analyst B, the bias was +4.93%. The combined RSD for the 20 determinations was 1.18%; the combined bias was +4.02%. Stock Solutions II and III were then submitted to a less exhaustive precision study. The same procedure as given above was used to prepare the samples for atomic absorption analysis.

## CONCLUSIONS

The analysis for neutron poisons in process solutions containing gross amounts of uranium, neptunium, and plutonium can be simplified if these radionuclides are first separated from the neutron poison. Once the neutron poison (in this case, cadmium) has been isolated, analyses can proceed by normal methods.

The extraction method presented here was successfully used on actual process solutions. The alpha content of the solutions was reduced from  $10^{11}$  dis/(mL-min) in the original sample to  $10^4$  dis/(mL-min) in the sample aliquots actually introduced to the atomic absorption flame. The overall fissionable material content was reduced from about 25 g/L in the original sample to about  $10^{-7}$  g/L in the sample aliquot actually analyzed. As up to about 5 mL of sample can be introduced to the flame during a typical determination, the sample could introduce up to about 0.5 picograms of fissionable material to the atomic absorption flame.

TABLE 6

## Analysis of Process Samples for Cadmium

Sample	<i>Series A<sup>b</sup></i>		<i>Series B<sup>c</sup></i>	
	<i>Diluted Sample, µg Cd/mL by AA</i>	<i>Original Sample, calculated g Cd/L</i>	<i>Diluted Sample, µg Cd/mL by AA</i>	<i>Original Sample, calculated g Cd/L</i>
1	1.06	26.50	0.80	26.67
2	1.07	26.75	0.80	26.67
3	1.06	26.50	0.81	27.08
4	1.06	26.50	0.81	27.00
5	1.08	27.00	0.81	27.00
6	1.06	26.50	0.81	27.00
7	1.06	26.50	0.81	27.00
8	1.05	26.25	0.81	27.00
9	1.05	26.25	0.81	27.00
10	1.05	26.25	0.82	27.33
Average	1.060	26.50	0.809	26.967
$\sigma$	0.0094	0.2357	0.0057	0.1873
RSD, %		0.89		0.69
Bias, %		+3.11		+4.93
Combined avg				26.734
Combined $\sigma$				0.317
RSD, %				1.18
Bias, %				+4.02

a. Original solution contained 25 g Cd/L and 25 g U/L (Stock Solution II).

b. Dilution factor = 25,000.

c. Dilution factor = 33,333.





## APPENDIX: Experimental Procedure

### Apparatus

Vortex mixer  
Four-dram vials with polyethylene-lined caps  
Micropipets and transfer pipets (as required) plus syringes  
Volumetric flasks, 50 mL and 1000 mL  
Graduated cylinder (50 mL)  
Plastic bottles (50 mL)  
Gross  $\alpha$  and gross  $\lambda$  plates  
Gross  $\alpha$  counter  
Gross  $\beta$  counter  
Atomic absorption spectrometer

### Reagents

Tributyl phosphate (30% by volume in dodecane)  
Nitric acid: 0.1M, .4M, 8M, and 15.7M  
Sodium hydroxide: 0.1M  
KMnO<sub>4</sub>: 5 wt % in water  
Potassium oxalate sol'n: saturated  
Deionized water

### Procedure

Note: All determinations for neutron poisons are handled as Nuclear Safety Samples. Therefore, duplicate samples must be run in duplicate by two different analysts. The two analysts must use different dilution factors. The resulting four results must agree within 5%.

1. Determine H<sup>+</sup> concentration.
  - a) Adjust saturated potassium oxalate solution to pH 7 with standard 0.1M NaOH or standard 0.1M HNO<sub>3</sub> as required.
  - b) Add sample aliquot to adjusted oxalate solution.
  - c) Titrate solution to pH 7 with standard 0.1M NaOH.

d) Calculate acid concentration.

Note: For most situations, the nominal acid concentrations will be 7 to 9M HNO<sub>3</sub>.

2. Using an aliquot to equal about 25 to 50 mg Cd, calculate the amount of 0.1M HNO<sub>3</sub> necessary to adjust the acidity to 3 to 5M HNO<sub>3</sub>.

Note: Usually 1.5 volumes of 0.1M HNO<sub>3</sub> per volume of sample aliquot will adjust the nitric acid to the proper range.

3. Using microliter pipets, deliver the prescribed aliquots of 0.1M HNO<sub>3</sub> to the four-dram vial (usually 1500 µL for one dilution; 3000 µL for the second dilution).

4. Using microliter pipets, deliver the prescribed aliquots of sample to the four-dram vials (usually 1000 µL and 2000 µL aliquots are used for the two dilutions).

5. Pre-equilibrate the 30% TBP with 4M HNO<sub>3</sub>.

6. Add an equal volume of pre-equilibrated TBP to the four-dram vial. Cap the vial and agitate the vial for 2 minutes on a vortex mixer.

7. Allow the phases to separate. Then remove most of the organic phase (the top layer) with a slurry pipet.

Note: Total removal of the organic phase is desirable but not necessary.

8. Discard the organic layer to waste.

9. Scrub the aqueous layer twice more with pre-equilibrated 30% TBP. Discard the organic layers.

10. Using deionized water, make a primary dilution of the extracted aqueous phase with aliquots to give 5 to 10 mg Cd in 50.0 mL of solution in a volumetric flask.

Note: For nominal 25 g Cd/L samples, aliquots of 500 µL and 750 µL are used.

11. Transfer about 4 mL of the primary dilution to a four-dram vial.

12. Transfer the primary dilution vial (Step 11) from the glove box to a radiobench before making the secondary dilution.

- Using deionized water, make a secondary dilution of the sample using aliquots of 0.04 to 0.08 mg Cd to 50.0 mL in a volumetric flask.

Note: The final dilution must be in the range of 0.5 to 2.5  $\mu\text{g Cd/mL}$ .

- Prepare a gross  $\alpha$  and a gross ( $\beta+\lambda$ ) plate of the final dilutions. Determine gross  $\alpha$  and gross ( $\beta+\lambda$ ) on each dilution.

Note: The gross alpha should not exceed  $1 \times 10^5$  dis/(min-mL); the gross ( $\beta+\lambda$ ) should not exceed  $1 \times 10^7$  dis/(min-mL).

- If the gross  $\alpha$  and ( $\beta+\lambda$ ) counts are within the prescribed limits, transfer at least 25 mL of the final dilution to a plastic bottle. If the gross  $\alpha$  and ( $\beta+\gamma$ ) counts are not within the prescribed limits, skip to Step 22.

Note: Keep the outside of the bottle uncontaminated.

- Bag the final dilution vial from the radiobench. Check the outer container for radio-contamination. Label as radioactive sample.
- Transfer samples to the atomic absorption laboratory.
- Place samples in the radiobench.
- Transfer an aliquot of the sample to a "clean" bottle. Keep the bottle clean!
- Run standards on the atomic absorption spectrometer to span the range of 0.5 to 2.5  $\mu\text{g Cd/mL}$ . Draw calibration curve.
- Run samples on the atomic absorption spectrometer. Determine concentration of cadmium from the calibration curve.
- If the gross  $\alpha$  and gross ( $\beta+\gamma$ ) are not within the prescribed limits, return to the original sample and begin the extractions again. This time, however, after adjusting the acid, add two drops of 5%  $\text{KMnO}_4$  solution to oxidize neptunium and/or plutonium ions to  $\text{Np(VI)}$  and/or  $\text{Pu(VI)}$ . Proceed as written.

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