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THE MODIFIED TITRIMETRIC DETERMINATION OF PLUTONIUM USING PHOTOMETRIC END-POINT DETECTION

by

W. J. Baughman and J. W. Dahlby

ABSTRACT

A method used at the Los Alamos Scientific Laboratory for the accurate and precise assay of plutonium metal was modified for the measurement of plutonium in plutonium oxides, nitrate solutions, and in other samples containing large quantities of plutonium in oxidized states higher than +3. In this modified method, the plutonium oxide or other sample is dissolved using the sealed-reflux dissolution method or other appropriate methods. Weighed aliquots, containing approximately 100 mg of plutonium, of the dissolved sample or plutonium nitrate solution are fumed to dryness with an HClO_4 — H_2SO_4 mixture. The dried residue is dissolved in dilute H_2SO_4 , and the plutonium is reduced to plutonium(III) with zinc metal. The excess zinc metal is dissolved with HCl, and the solution is passed through a lead reducer column to ensure complete reduction of the plutonium to plutonium(III). The solution, with added ferroin indicator, is then titrated immediately with standardized ceric solution to a photometric end point. For the analysis of plutonium metal solutions, plutonium oxides, and nitrate solutions, the relative standard deviations are 0.06, 0.08, and 0.14%, respectively. Of the elements most likely to be found with the plutonium, only iron, neptunium, and uranium interfere. Small amounts of uranium and iron, which titrate quantitatively in the method, are determined by separate analytical methods, and suitable corrections are applied to the plutonium value.

I. INTRODUCTION

We have modified a method to determine plutonium in plutonium oxides, nitrate solutions, and other materials containing large quantities of plutonium to meet the increased needs of the Los Alamos Scientific Laboratory (LASL) for the chemical characterization of reactor fuels. Other methods^{1,2} that are applicable to the analysis of plutonium oxide use relatively small samples, rang-

ing from 5 to 35 mg. For greater precision, the method described here uses 100-mg samples and could analyze 250-mg samples. Also, instead of the electrode end-point detection system, we use a photometric end-point detection system that is less affected by sample impurities.

The current method for assaying metals and metal alloys that dissolve to produce plutonium(III) is based upon the oxidation of plutonium(III) to plutonium(IV) by cerium(IV) in a sulfuric acid

solution.^{3,4} In our modified method, the plutonium oxide sample is dissolved using the sealed-reflux dissolution method⁵, and other sample materials are dissolved by appropriate methods. Weighed aliquots of the dissolved sample or plutonium nitrate solution containing approximately 100 mg of plutonium are fumed to dryness with an HClO_4 — H_2SO_4 mixture. The dried residues are dissolved in approximately 3.6N H_2SO_4 , and the plutonium is reduced to plutonium(III) with zinc metal. The excess zinc metal is dissolved in concentrated HCl, and the solution is passed through a lead reductor column to ensure complete reduction of the plutonium. Sulfuric acid is added to complex the oxidized plutonium(IV) and thereby reduce the potential of the plutonium(III)—(IV) couple to about 0.7 V. Otherwise plutonium(VI) and possibly plutonium(V) might be formed during the oxidation of plutonium(III). Before air oxidation of the plutonium can occur, the plutonium(III) is titrated immediately (within 15 min) to plutonium (IV) using a standardized cerium(IV) solution. Ferroin indicator is added before the titration, and the end point is detected photometrically at 510 nm as the iron(II) in the indicator is oxidized and loses its red color. With the use of a simple zero-setting and a single-beam photometer, a maximal reading of about 80 to 85% transmittance is taken as the end point; this conforms to the background absorption of the cell and solution.

II. APPARATUS AND REAGENTS

A. Apparatus

Balance: analytical, 0.1-mg sensitivity.

Beaker: electrolytic, 180-ml Pyrex, without a spout.

Bottle: polyethylene, 125-ml capacity, used in taking the sample aliquots.

Distillation Apparatus: for preparing pure H_2SO_4 . The apparatus is a closed system made from Pyrex or equivalent glass. It consists of a 2000-ml boiling flask (enclosed in a heating jacket), a 30-cm reflux column, a 40-cm condenser, and a receiving flask with an arm on it for the vacuum. The apparatus is illustrated in Fig. 1. The boiling flask is heated to 550°C, and a vacuum pump is used to evacuate the system to no less than -40 kPa (-12 in.

of Hg). A hollow 2-mm-i.d. glass rod, about 150 cm long and sealed at one end, is used in the boiling flask instead of glass beads to prevent bumping. It must be cleaned before each distillation to empty it of any liquid. The vacuum pump and gauge are protected by a trap containing CaCl_2 to remove the acid vapors. The first 25 ml of acid is discarded, and about 500 ml of acid can be distilled in about 5 h.

Fuming Apparatus: One multiple-unit, electric hot plate, about 61 x 11.4 cm and set at 110 to 120°C, with four infrared heat lamps mounted 8-10 cm above the hot plate (a Lindberg "Sola Basic" hot plate or equivalent). Also, one single-unit, electric hot plate, thermostatically controlled at 250°C, with one heat lamp mounted 8-10 cm above the hot plate [a Thermolyne (Model HF-a1915b) or equivalent].

Lead Reductor Column: consists of a 20-cm-long piece of 2.5-cm-o.d., heavy-walled glass tubing sealed to a 3-mm-bore Teflon stopcock on the bottom and to an upper reservoir bell constructed from 4.5-cm-diam, 5-cm-long, heavy-walled tubing. See Fig. 2. A small plug of glass wool in the bottom of the column retains the reagent-grade lead that fills the column to a depth of 15 cm. The lead is cleaned in the column with 12N HCl then rinsed with water. The reductor column is filled with 0.1N HCl when not in use.

Normal Laboratory Glassware and Polyethyleneware: includes beakers, storage bottles, wash bottles, etc.

Photometer: a modification^{3,4} of G. A. Rost's instrument design.⁶ The sensing unit contains a light source (a GE 46 light bulb or equivalent), a 510-nm interference filter with a 10-nm half-bandwidth, a cell compartment for a 180-ml, electrolytic beaker without a spout, and a cadmium sulfide photocell, such as an RCA 7163 or equivalent. The output is measured on a long-scale 200- μA meter.

Pipet: 0.200-ml, Eppendorf automatic pipet or equivalent.

Sealed-Reflex Dissolution Apparatus: includes a heating block, clamps, hot plates, rubber stoppers, thermocouples, tubes, and assorted laboratory equipment listed in Ref. 5.

Weighing Buret: 60-ml Friedman and LaMer as shown in Fig. 3.

Weight Pipet: transfer, constructed of pyrex glass with a waxed tip as shown in Fig. 4.

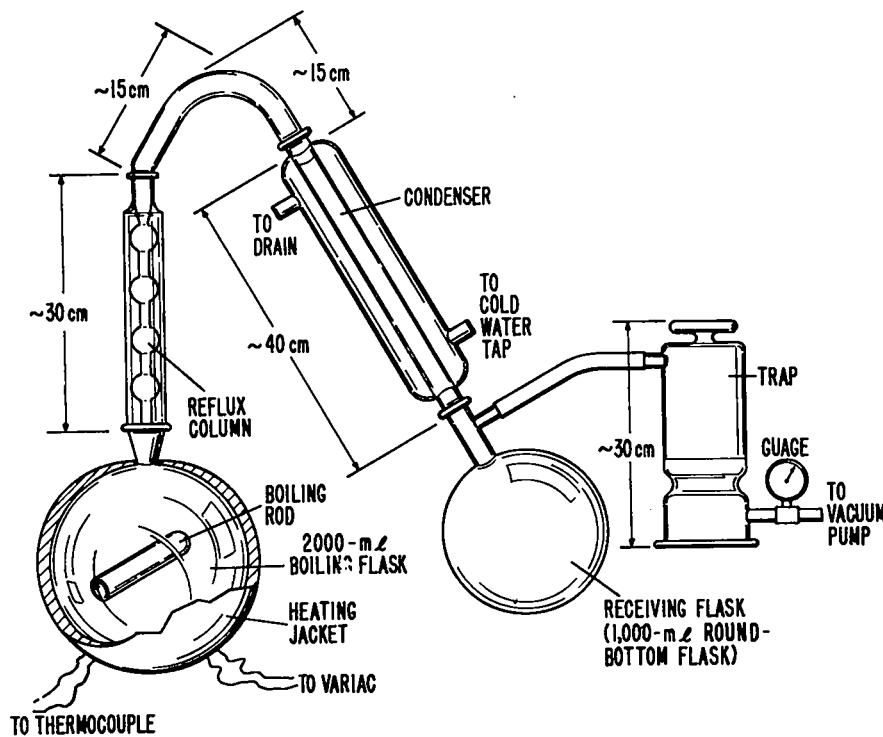


Fig. 1.
Distillation apparatus.

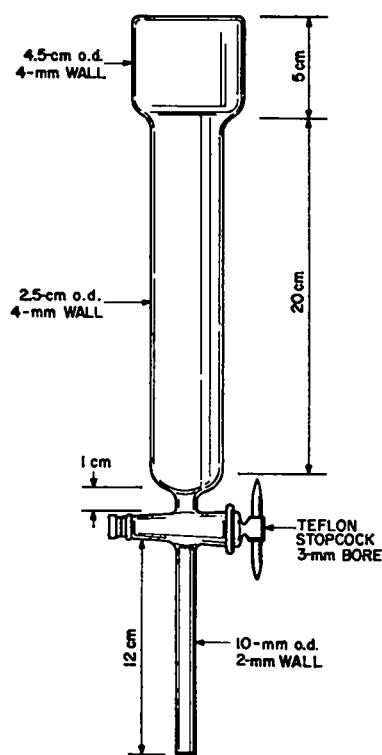


Fig. 2.
Lead reductor column.

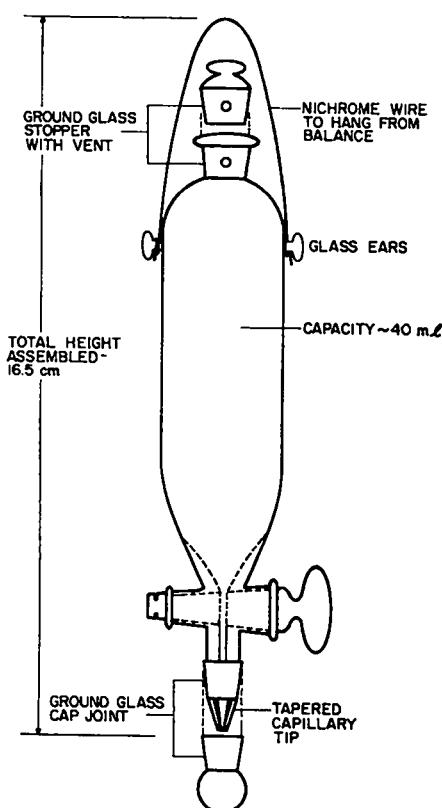


Fig. 3.
Weighing buret.

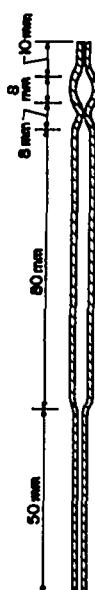


Fig. 4.
Transfer weight pipet.

B. Reagents

Ceric Sulfate: approximately 0.025*N* in 1*N* H₂SO₄. Prepare the solution by diluting 2 *l* of approximately 0.5*N* ceric sulfate [Ce(HSO₄)₄] solution* to 19 *l* with 1*N* H₂SO₄. Stir for 24 h by bubbling filtered air through the solution. Standardize the solution against high-purity National Bureau of Standards, Standard Reference Material (NBS SRM) plutonium metal at least once a month as follows. Prepare the standard by dissolving a clean portion of the SRM metal in 6*N* HCl in a 100-mL beaker covered with a watch glass, transferring the solution to a clean, dry, tared 125-mL polyethylene wash bottle, and weighing aliquots containing approximately 100 mg of plutonium. The procedure as described in steps 12 to 18 of Sec IV is followed, except that the blanks are made from 5 mL of 6*N* HCl diluted to 40 mL and passed through the reductor column. Five mL of 6*N* HCl must be added to the plutonium aliquots before they are passed through the reductor column to prevent hydrolysis. Wash the column with 40 mL of 0.1*N* HCl and 40 mL of water, catching all the effluent solutions in a 180-mL, electrolytic beaker containing 5 mL of 18*N* H₂SO₄ and 0.200 mL of ferroin indicator. Calculate the titer, which is defined as the mg of plutonium

*A solution from G. Frederic Smith Chemical Company or equivalent.

equivalent to 1 g of ceric sulfate solution, in the following way.

$$T = \frac{(\text{wt\% Pu})(\text{mg Pu metal})(\text{g Pu soln aliquot})}{(\text{Total g Pu soln}) [\text{Net g Ce(IV) soln used}]} \quad (100)$$

Ferroin Indicator: prepare by dissolving 0.98 g of ferrous ammonium sulfate [FeSO₄ · (NH₄)₂ · 6H₂O · SO₄] and 1.487 g of 1,10-phenanthroline monohydrate (C₁₂H₈N₂ · H₂O) in 100 mL of water.

Hydrochloric Acid: 12*N*, analytical reagent grade.

Hydrochloric Acid: 6*N*, prepare by diluting 12*N* HCl 1:1 with distilled water.

Hydrochloric Acid: 0.1*N*, prepare by diluting 17 mL of 6*N* HCl to 1 *l* with distilled water.

Hydrofluoric acid: 1.3*N*, dilute 5 mL of 48% HF to 100 mL with distilled water.

Lead: granular, analytical reagent grade.

Nitric Acid: 15.6*N*, analytical reagent grade.

Perchloric Acid: 72%, 11.7*N*, analytical reagent grade.

Plutonium Metal: NBS-SRM-949.

Sulfuric Acid: 36*N*, distilled from borosilicate glassware.

Sulfuric Acid: 18*N*, prepare by diluting the distilled 36*N* H₂SO₄ 1:1 with distilled water.

Sulfuric Acid: 6*N*, prepare by diluting 167 mL of the distilled 36*N* H₂SO₄ to 2 *l* with distilled water.

Zinc: granular, analytical reagent grade.

III. SAMPLE PREPARATION

Caution: *Health and safety rules for the handling of plutonium must be rigidly followed, and adequate protection for the operator must be ensured with suitable dryboxes, protective clothing, and radiation monitoring equipment. All work with solid plutonium, especially oxides, must be done in a glove box, but plutonium solutions may be handled in a fume hood or glove box with the gloves removed.*

1. Dry the plutonium oxides at 950°C and pretreat other plutonium materials as required.
2. Accurately weigh a PuO₂ sample containing 650 to 700 mg of plutonium and place it in a clean, dry, sealed-reflux tube.*

3. Add 3 drops of 15.6N HNO₃, 5 drops of 1.3N HF, and 5 mL of 12N HCl to the tube and seal with a rubber stopper and clamp.
4. Place the tube in an aluminum holder on a hot plate and heat to 150°C.
5. Hold that temperature until the sample is dissolved—4 to 24 h depending on the sample.
6. Dissolve other plutonium-containing materials by the sealed-reflux technique or by other appropriate techniques.
7. Transfer the dissolved sample to a tared polyethylene bottle.
8. Determine the sample concentration by weight and transfer weighed aliquots that contain about 100 mg of plutonium into 30-mL beakers.
9. Inspect the plutonium nitrate sample solutions to ensure that there is no precipitate present, then transfer weighed aliquots containing about 100 mg of plutonium into 30-mL beakers using the transfer weight pipet shown in Fig. 4.

This method can be used for a wide range of plutonium concentrations, but it is usually better to titrate about 100 mg of plutonium. The end points will differ slightly if the plutonium content varies too much.

IV. SAMPLE ANALYSIS

1. To two accurately weighed aliquots of each sample in 30-mL beakers, add 0.2 mL of 72% HClO₄ and 0.5 mL of 6N H₂SO₄. Also add 0.2 mL of 72% HClO₄ and 0.5 mL of 6N H₂SO₄ to each of two or more 30-mL beakers to be used as blanks.
2. Dry the solutions on the fuming apparatus set between 100-110°C. **Caution:** All hoods where HClO₄ is fumed should be thoroughly washed at least weekly.
3. Transfer the beakers to the smaller hot plate set at 250°C to ensure complete dryness.
4. Cool, add approximately 2 mL of 1N H₂SO₄ to each beaker, and repeat steps 2 and 3. (When analyzing plutonium nitrate solutions, repeat step 4 one more time.)
5. Cool, add approximately 2 mL of distilled water, then 2 mL of 18N H₂SO₄, and dilute to approximately 10 mL with distilled water.
6. Add one heaping scoop (approximately 0.7 g) of zinc metal to each beaker and immediately cover the beaker with a watch glass.
7. Warm the solutions on a hot plate set between 100-110°C for 30 min.
8. Remove the solutions from the hot plate and cool for 15 min.
9. To dissolve excess zinc, add 12N HCl to each beaker in the following manner.
 - a. Add 2 mL through the beaker spout.
 - b. After the reaction subsides, add 3 mL to wash down the walls.
 - c. Add 5 mL to finish mixing the solution.
10. Warm each solution on a hot plate set between 100-110°C for 15 min.
11. Remove each solution from the hot plate and cool for 1 h.
12. Flush the lead reductor column, first with 200 mL of 12N HCl then with 800 mL of water.
13. Dilute 5 mL of 6N HCl to 40 mL with water, pour this solution through the reductor column followed by 40 mL of 0.1N HCl and 40 mL of water. Discard the effluent solutions.
14. Repeat step 13 once.
15. Add 5 mL of 18N H₂SO₄ and 0.200 mL of ferroin indicator to a 180-mL, electrolytic beaker.
16. Wash, with water, the watch glass and walls of a 30-mL beaker containing a blank solution. Pour that solution through the lead reductor column, rinse the walls of the beaker with 2 to 3 mL of water and also pour this solution through the column. Wash the column with 40 mL of 0.1N HCl then with 40 mL of water, catching all the effluent solutions in the beaker described in step 15.
17. Within 15 min or less, put the beaker and solution into the photometer compartment, start the stirrer, and turn on the photometer's lamp. Weigh the 60-mL weight buret containing standardized ceric sulfate solution. Cautiously add ceric sulfate solution one drop at a time to the beaker until there is no further movement of the microammeter needle.*
18. Reweigh the weight buret and obtain by difference the g of ceric sulfate solution used in titrating the reagent blank.
19. Repeat steps 15 through 18, and the two results should compare within ± 0.005 g.

*The voltage applied to the lamp should be adjusted to keep the meter needle on scale at the end point. In practice, samples with similar amounts of plutonium and similar volumes will have nearly constant end-point readings. Determine the actual voltage setting and the end-point reading on the first sample each day.

20. Repeat steps 15 through 18 for each sample solution, adding the ceric sulfate solution in larger increments, and weigh the buret to obtain the g of ceric sulfate solution used in titrating the sample.

21. Calculate the wt% of plutonium in the sample as follows.

$$\text{wt\% Pu} = \frac{(T) (Ce_s - Ce_{RB}) (100)}{\text{mg of sample}}$$

T = the titer of the ceric sulfate solution, g Pu/g solution.

Ce_s = g of ceric sulfate solution needed to titrate the sample.

Ce_{RB} = average g of ceric sulfate solution needed to titrate the blanks.

V. RESULTS

We determined the accuracy of this method by titrating 100-mg aliquots of two solutions prepared by dissolving two separate ~2-g portions of pure plutonium metal in 6N HCl. Five aliquots from the first metal solution were titrated on November 30, 1978 using the original photometric method for metals and metal alloys.^{3,4} The weight per cent of plutonium was then determined for each of these aliquots using the formula given in Sec. IV. We found that 5 mL of 6N HCl had to be added to the solution before it was passed through the reductor column to prevent hydrolysis and keep the solution clear. Then eight aliquots, of this same metal solution, were analyzed using the procedure outlined in Sec. IV. Before fuming these aliquots, 0.05 mL of 15.6N HNO₃ and 0.05 mL of 1N HF had to be added to oxidize the plutonium to plutonium(IV) so that the aliquots would sulfate properly. The weight per cent of plutonium in each of the aliquots was then determined using the formula given in Sec. IV. The above procedure was repeated for the second metal solution on January 9, 1979. The results in Table I show that the method is not biased and that the relative standard deviation (RSD) is 0.06% for 13 different solution aliquots from 2 different portions of the same plutonium metal.

We determined the precision of the modified method for titrating plutonium oxides by dissolving six different plutonium oxide samples using the sealed-reflux dissolution method⁵ and analyzing the

aliquots of these oxides following the procedure outlined in Sec. IV. The weight per cent of plutonium found in each aliquot was determined using the formula given in Sec. IV. The results in Table II show that the method has an average RSD of 0.08% for the six plutonium oxides.

We compared our modified method with the controlled-potential coulometric method for assaying plutonium dioxide samples.¹ Each sample was dissolved by the sealed-reflux dissolution method⁵, and six aliquots were weighed from each solution. For each set of six aliquots, four aliquots were analyzed by the modified photometric titration method and two by controlled-potential coulometry. Table III shows the average values obtained for each sample by each method. Relative standard deviations are 0.08% for the modified photometric method and 0.10% for the controlled-potential coulometric method. For thirteen of the samples, the average results by the two methods agreed within 0.10% ($\pm 0.05\%$); eight of these had higher values by the modified photometric titration method than by the controlled-potential coulometric method. Seven samples agreed within 0.18% ($\pm 0.09\%$), but again five of these assays had higher values by the modified photometric titration method. The remaining four samples were not in agreement, having differences of 0.21 to 0.34%; three of the four assays had higher values by the modified photometric titration method. The controlled-potential coulometric titration is much less affected by diverse ions in the sample solutions because of judicious selection of the potentials used in the oxidations and reductions. The modified photometric titration method is affected by any impurity that is reduced by lead or oxidized by cerium(IV). For these reasons, the coulometric titration method is used for samples containing significant levels of impurities, and the modified photometric titration method is used for high-purity, plutonium oxides and other materials.

The modified photometric titration method was evaluated also by assaying a plutonium nitrate solution carefully prepared with a known plutonium content. The reference plutonium nitrate solution was prepared by dissolving a large, accurately weighed portion of high-purity plutonium metal in 6N HCl. The solution was cooled to room temperature and transferred to a tared plastic bottle.

TABLE I

ANALYSIS OF PLUTONIUM USING THE ORIGINAL AND THE
MODIFIED PHOTOMETRIC TITRATION METHODS

Date Analyzed	Aliquot Cut Identification	Original Method, Plutonium (wt%)	Modified Method, Plutonium (wt%)
11/30/78	11	99.95	
"	15	99.99	
"	23	100.04	
"	3	100.09	
"	43	99.94	
"	4		99.93
"	8		99.91
"	12		99.97
"	16		99.99
"	20		99.99
"	24		100.11
"	28		99.91
"	41		100.05
1/9/79	F	100.05	
"	J	99.96	
"	L	99.98	
"	P	100.02	
"	N	99.96	
"	G		99.94
"	K		99.96
"	C		100.05
"	M		100.00
"	A		100.01
"	E		99.96
"	I		100.00
Average = <u>100.00</u>		Average = <u>99.99</u>	
RSD = <u>0.05%</u>		RSD = <u>0.06%</u>	

*A titer of 6.6261 was used.

The plutonium concentration of the solution (by weight) was calculated, and three unequal aliquots were weighed and transferred to 30-ml beakers. The three solutions were evaporated to dryness, 2 ml of 15.6N. HNO₃ was added, and the solutions were evaporated again and fumed to near dryness. Each residue was dissolved in 8N HNO₃, quantitatively washed with 8N HNO₃ into a tared 7-g vial, and weighed. The plutonium concentration of each 8N nitric acid solution was calculated by weight, and six accurately weighed, 600-mg aliquots were taken from each solution. Then the steps in Sec IV were

followed to determine by titration the plutonium concentration in each solution. These results are compared to the calculated concentrations in Table IV. The method is unbiased with an average relative standard deviation of 0.14% for plutonium nitrate solutions. Because this method successfully analyzed plutonium nitrate solutions, it is applicable to assays of plutonium in any oxidation state. Also, aliquots containing from 40 to 120 mg of plutonium were analyzed to test the effect of sample size. The assays were good, but the analyst must be aware that the meter-indicated end point varies

TABLE II

ANALYSIS OF PLUTONIUM OXIDE USING THE
MODIFIED PHOTOMETRIC TITRATION METHOD

Sample No.	No. of Determinations	Plutonium (wt%)	RSD (%)
1	10	87.42	0.076
2	9	87.31	0.068
3	7	87.15	0.081
4	11	87.37	0.082
5	8	87.29	0.073
6	7	87.14	<u>0.081</u>
Average RSD =			0.07, or 0.08

with the density of the solution when different amounts of plutonium are titrated.

The effects of several impurities, usually present in the low ppm range in plutonium, on the original photometric titration method are described in Ref 3. Two other impurities found in various concentrations in plutonium dioxide were investigated to measure their effect on the modified photometric titration method. Up to 0.1% of americium relative to the plutonium did not interfere, but neptunium interfered slightly by blunting the end point. Uranium and iron titrate quantitatively, but both of these impurities can be determined by alternative methods. Corrections can then be applied to the plutonium results.

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TABLE III

COMPARISON OF THE MODIFIED PHOTOMETRIC TITRATION
METHOD AND THE CONTROLLED-POTENTIAL COULOMETRIC
TITRATION METHOD FOR ANALYZING
PLUTONIUM OXIDES

Sample No.	Plutonium ^a (wt%)	Plutonium ^b (wt%)	Difference
1	87.79	88.00	-0.21
2	87.94	87.90	+0.04
3	87.85	87.95	-0.10
4	87.88	87.75	+0.13
5	87.62	87.65	-0.03
6	87.72	87.65	+0.07
7	87.87	87.75	+0.12
8	87.76	87.80	-0.04
9	87.84	87.50	+0.34
10	87.95	87.80	+0.15
11	88.00	87.85	+0.15
12	88.12	88.05	+0.07
13	87.92	87.90	+0.02
14	87.63	87.55	+0.08
15	88.02	87.80	+0.22
16	88.17	87.96	+0.21
17	88.18	88.00	+0.18
18	87.98	88.00	-0.02
19	88.02	87.95	+0.07
20	87.92	88.10	-0.18
21	87.89	87.85	+0.04
22	87.76	87.80	-0.04
23	87.83	88.00	-0.17
24	87.78	87.65	-0.13

^aEach result is an average of four determinations using the modified photometric titration method.

^bEach result is an average of two determinations using the controlled-potential coulometric titration method.

TABLE IV

ANALYSIS OF REFERENCE PLUTONIUM NITRATE SOLUTIONS
BY THE MODIFIED PHOTOMETRIC TITRATION METHOD

Calculated Plutonium Concentration (mg/g)	Measured Plutonium Concentration* (mg/g)	RSD (%)
99.70	99.73	0.15
139.63	139.64	0.14
189.33	189.40	0.12
Average RSD =		0.14

*Six determinations each.

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