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DETERMINATION OF ALUMINUM
IN PLUTONIUM

by

M. E. Smith

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ABSTRACT

A spectrophotometric method is described for the determination of aluminum in plutonium-aluminum alloys. The alloys are dissolved in perchloric acid and the diluted solution (1M) electrolyzed using a mercury cathode to remove any iron present which would interfere in the subsequent analysis. The plutonium is then separated from the aluminum by precipitation as the insoluble iodate, and the aluminum determined in the supernatant solution using aluminon as a color reagent. By proper choice of absorption cells and use of 20-milligram samples, aluminum in alloys containing approximately 0.02 to 0.04 percent aluminum by weight may be determined with a standard deviation of 5 percent or better, whereas alloys containing approximately 0.04 to 0.4 percent aluminum may be determined with a standard deviation of 3 percent. Reduction of the sample size is allowable for the analysis of alloys containing more than 0.4 percent aluminum.

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INTRODUCTION

Spectrophotometric methods for the determination of microgram quantities of aluminum have been used to a large extent for the analysis of various alloys. The use of these methods for the determination of aluminum in iron and steel is discussed by Rönnholm⁽¹⁸⁾ and an extensive bibliography is included. The determination of aluminum in thorium⁽¹³⁾ and zirconium,⁽⁶⁾ using oxine (8-hydroxyquinoline) as a color reagent, and the determination of aluminum in zinc,⁽¹⁶⁾ titanium,⁽³⁾ lead, antimony, and tin,⁽¹¹⁾ using aluminon as a color reagent, have recently been reported. Directions for the use of these reagents may be found in Sandell.⁽¹⁹⁾ Eriochrome Cyanine as a color reagent for aluminum has been recently studied by Clemser *et al.*⁽²⁾ Several other color reagents for aluminum described in the literature are hematoxylin,⁽¹⁷⁾ alizarin S,⁽¹⁾ and quinalizarin.⁽¹⁰⁾

Of the two color reagents most commonly used (aluminon and oxine), neither could be used for determining aluminum in plutonium without a preliminary separation of the aluminum. Aluminon was chosen for a color reagent rather than oxine since it did not require an extraction and seemed to involve less manipulation. This reagent was first suggested by Hammett and Sutty⁽⁷⁾ and since then its use has been described by many others.^(4,9,11,12,15,17,19,20,21) Neither reagent is highly commendable in that recovery problems are associated with the oxine extraction⁽¹¹⁾ and the use of aluminon requires rigid adherence to ex-

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perimental conditions which is often the case with lake-forming reagents. Also, the aluminon reagent is subject to many interferences as indicated in the works of previous investigators. The interference of iron, magnesium, rare earths, titanium, thorium, gallium, scandium, indium, and beryllium are described by Sandell.⁽¹⁹⁾ Craft and Makepeace showed that small amounts of manganese, nickel, chromium, molybdenum, vanadium, and copper interfere seriously.⁽⁴⁾ Pellowe et al. found that under certain conditions zinc and cadmium do not interfere when present in moderately large amounts.⁽¹⁶⁾

The method described in this report is an empirical one, but if the conditions are followed with reasonable care satisfactory precision may be obtained. Plutonium is separated from the aluminum by precipitation as the insoluble iodate and the aluminum is then determined in the supernatant using aluminon as the color reagent.

APPARATUS

1. Spectrophotometer, Beckman Model DU, equipped with holders for absorption cells of 1- to 10-cm. light path.
2. Electrolysis cells, Melaven type,^(8,14) consisting of a 15 x 125 mm. test tube, the bottom of which is sealed to a three-way stopcock (oblique bore, 1 mm.). The mercury reservoir is constructed from a 15-mm. centrifuge tube by sealing on the bottom a short piece of 7-mm. glass tube which is connected to the stopcock by means of a tygon tube. The anode consists of a piece of perforated platinum sheet (approximately 1 square inch). Connections are made to the mercury pool cathode by means of a tungsten wire immersed in the mercury reservoir. A source of direct current producing 200 milliamperes at 4 volts is used as a power supply for four of these cells connected in parallel.
3. Centrifuge, clinical, 110-volt, 60-cycle, equipped with plastic

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tubes of 40-ml. capacity (glass tubes may be used).

4. Flasks, quartz, 25-ml. capacities. (Quartz flasks were readily available and were therefore used to avoid aluminum contamination. Since the procedure described in this report deals only with acid solutions, acid-cleaned pyrex flasks should serve equally well.)

5. Pipets, volumetric, inter-joint with syringe, 2-, 4-, and 10-ml. capacities.

6. Steam bath (deep enough to allow complete immersion of the 25-ml. volumetric flasks).

REAGENTS

1. Aluminon (aurin tricarboxilic acid) reagent. Composite reagent described by Craft and Makepeace⁽⁴⁾ and Codell and Norwitz; dissolve 0.35 gram of aurin tricarboxilic acid ammonium salt (Eastman Kodak Co., practical grade) in 200 ml. of water and add 70 ml. of 10 percent benzoic acid in methanol. Dilute to 417 ml. with distilled water and add 350 ml. of ammonium acetate buffer solution (described below) and 350 ml. of 1 percent gelatin solution. This solution is stored in a brown glass bottle and allowed to stand for 3 days before using. The solution is stable for a period of at least 3 months.

2. Ammonium acetate buffer solution. Mix 184 ml. of concentrated ammonium hydroxide with 166 ml. of glacial acetic acid. After cooling, adjust the pH to 6.65 (dilution of 1.25 ml. of this solution should give an approximate pH of 5.3).

3. Potassium iodate solution (0.3 M). Dissolve 128.8 grams of potassium iodate (Merck, reagent grade), in distilled water and make up to 2 liters.

4. Standard aluminum sulfate stock solution. Dissolve 0.6175 \pm 0.0001 gram of aluminum sulfate hydrate ($\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{ H}_2\text{O}$) in distilled water, add a few drops of concentrated sulfuric acid, and dilute the solution to 250 ml. The solution used was found to contain 199.6 mg./ml. of aluminum by gravimetric analysis.

5. Plutonium perchlorate stock solution. Dissolve 1.00 \pm 0.01 gram of pure plutonium metal in 10 ml. of 70 percent perchloric acid and dilute to 100 ml. with distilled water. (It is advisable to cool the perchloric acid during the dissolving process by immersing the flask in a beaker of cold water and taking care that the metal is covered with the acid at all times. If the metal is finely divided, it should be added in small quantities.)

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RECOMMENDED PROCEDURE

The procedure described below is for relatively pure plutonium-aluminum alloys. If interfering constituents such as iron, nickel, copper, etc.; are present, it is advisable to use only sulfate or perchlorate solutions in order to facilitate their removal by mercury cathode electrolysis. An aliquot sample of 20 milligrams of the alloy is recommended for samples containing 0.02 to 0.4 percent aluminum, whereas 1-milligram amounts may be used for samples containing 0.4 to 5 percent aluminum. An original sample size of 250 milligrams is used in the following procedure merely as a matter of convenience. The final aliquot sample should, however, be approximately 1 N in acid. If glass equipment is used, a pretreatment with 50 percent hydrochloric acid has been recommended. (16)

1. Weigh accurately 250 ± 1 mg. of the plutonium-aluminum alloy and add to 2.5 ml. of 70 percent perchloric acid in a 25-ml. volumetric flask. The flask should be immersed in cold water during the solution of the metal and care taken to keep the metal covered with the acid. Finely divided samples should be added a little at a time.
2. After dissolving the sample, dilute the solution to 25 ml. with distilled water, mix well, and transfer a 2-ml. aliquot to the mercury cathode electrolysis cell.
3. Electrolyze the solution for 30 minutes, using a current of 200 milliamperes. Drain the solution from the cell into a 25-ml. volumetric flask, rinsing off the anode with distilled water and flushing the cell at least 3 times with distilled water to ensure complete transfer of the solution. The total volume after washing should not exceed 13 ml.
4. Add 10 ml. of 0.3 M potassium iodate solution to the solution in the flask. Mix, and make up to the 25-ml. mark with distilled water.

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5. Allow the solution to stand for 10 minutes; then centrifuge.
6. Using a pipet, transfer exactly 10 ml. of the supernatant to a clean 25-ml. volumetric flask and add 4 ml. of the composite aluminon reagent.
7. Prepare at this time a reagent blank by adding to a 25-ml. volumetric flask 4 ml. of 0.3 M potassium iodate solution, 0.8 ml. of 1 M perchloric acid and 4 ml. of composite aluminon reagent.
8. Immerse the flasks containing the sample and the reagent blank in the steam bath and heat for 30 minutes.
9. Dissolve the plutonium iodate suspension in concentrated hydrochloric acid and transfer the resulting solution by suction to a container suitable for the storage of radioactive waste solutions.
10. Remove the flasks from the steam bath, cool, and dilute to the 25-ml. mark with distilled water.
11. Transfer a portion of the sample solution to an absorption cell of appropriate length and measure the absorbance at $536 \text{ m}\mu$ versus the reagent blank solution. (The appropriate length for the absorption cell is determined by trial as the one that will give an absorbance reading in the optimum precision range of the spectrophotometer.)
12. Prepare calibration curves for each of the absorption cells used by treating plutonium-aluminum solutions of known aluminum content according to steps 2 through 11 of the RECOMMENDED PROCEDURE. (It is not necessary to mix the standard plutonium and aluminum solutions before electrolysis; therefore, 2-ml. aliquots of the plutonium perchlorate solution containing 20 mg./ml. of plutonium may be electrolyzed and added to known volumes of the diluted aluminum stock solutions in the 25-ml. flasks. The preparation of the stock solutions is described under REAGENTS.)

EXPERIMENTAL

A series of plutonium-aluminum solutions of known aluminum concentration were prepared and the aluminum determined according to the manner described under RECOMMENDED PROCEDURE. Because of the volume considerations, aliquots of the plutonium stock solution were first

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electrolyzed and then added to known volumes of standard aluminum solution. The plutonium was then precipitated as the iodate and the aluminum determined in the supernatant by the aluminon spectrophotometric method. These supernatants were found to be quite low in plutonium, containing approximately 0.2 microgram per milliliter. Data were obtained for the aluminum concentration range of approximately 0.02 to 0.4 weight percent. Absorption cells of 3 different light paths (1-, 2-, and 10-cm.) were used and the absorbance measured at 536 millimicrons versus a reagent blank. Each solution analyzed contained 20 milligrams of plutonium.

Similar absorption data were also obtained on aluminum iodate solutions. These solutions were prepared by treating known aliquots of a standard aluminum sulfate solution (see REAGENTS) according to steps 4, 6, 7, 8, 10 and 11 of the RECOMMENDED PROCEDURE.

Plutonium-Aluminum Alloys (\sim 0.2 to 0.4 percent aluminum)

Data were obtained on plutonium perchlorate solutions containing 0 to 0.35 percent aluminum using absorption cells with a 1-cm. light path. These data are shown in Table I and the average absorbance values are plotted versus the aluminum concentration in Figure 1. In this and subsequent figures the aluminum concentration is expressed in micrograms per milliliter. The conversion to these units may be made by multiplying the value for the total micrograms of aluminum shown in the tables by 0.016. This factor allows for the dilution made by aliquoting and

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the final adjustment of the volume to 25 milliliters. The line drawn through the points was drawn according to the statistical method of least squares.

Table I

ABSORBANCE OF ALUMINUM-ALUMINON COLOR IN
PLUTONIUM IODATE SUPERNATANTS
($\lambda = 536 \text{ m}\mu$, 1-cm. cells, 20 mg. Pu/sample)

$\mu\text{g. of Al}$	0	10	20	30	40	50	60	70
Absorbance	0.013	0.094	0.200	0.283	0.384	0.475	0.575	0.660
	0.006	0.090	0.194	0.274	0.386	0.432	0.561	0.697
	0.004	0.080	0.193	0.280	0.363	0.470	0.567	0.615
	0.007	0.092	0.196	0.276	0.363	0.461	0.560	0.620
Average Absorbance	0.008	0.089	0.196	0.278	0.374	0.460	0.566	0.648

Absorbance data for aluminum iodate solutions were also obtained and the results are shown in Table II. The average absorbance values versus the aluminum concentration are plotted in Figure 1. The line through the points was drawn according to the statistical method of least squares.

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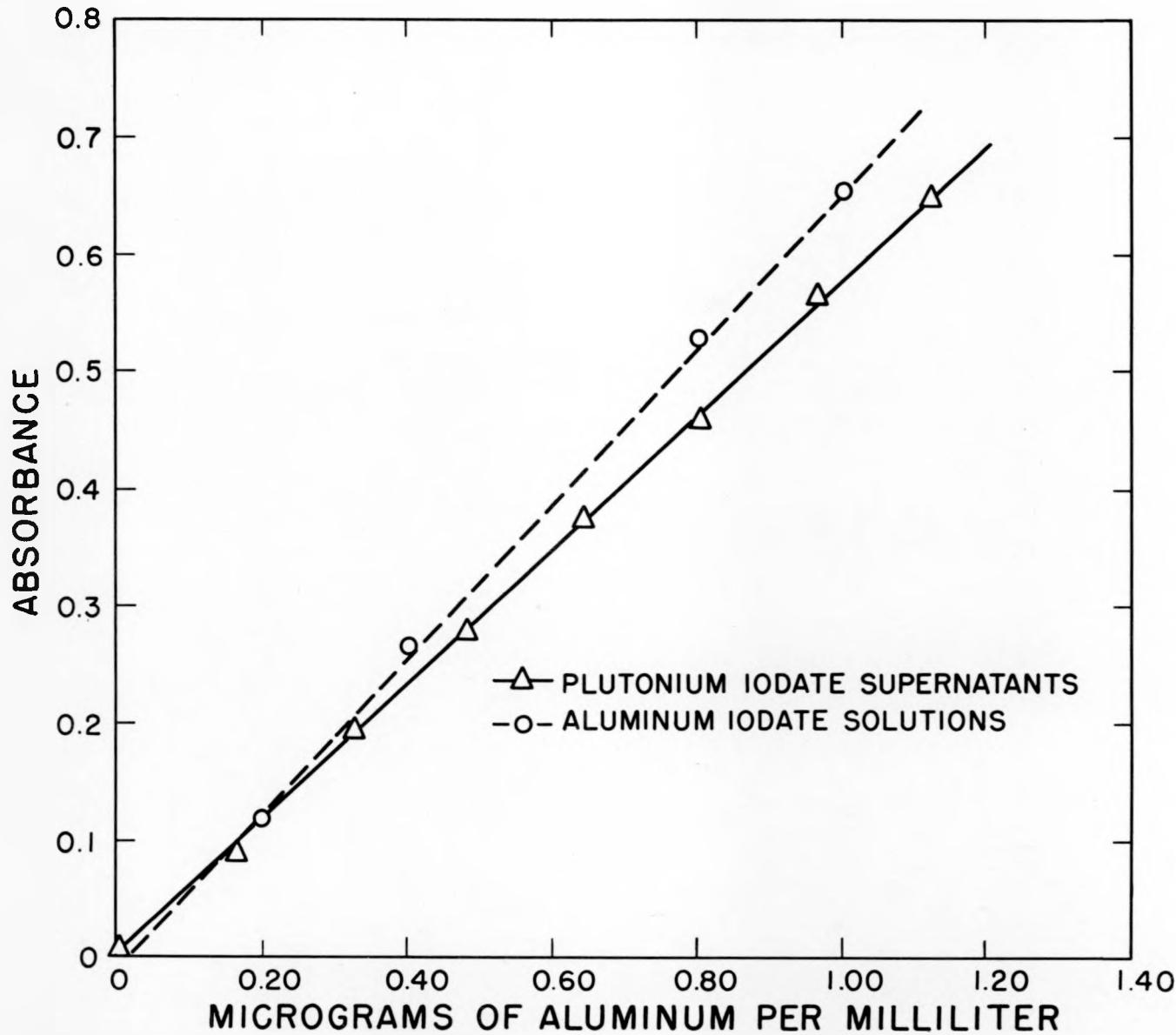


Figure 1. Absorbance of aluminon-aluminum color (1-cm. cells)

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Table II

ABSORBANCE OF ALUMINUM-ALUMINON COLOR IN
ALUMINUM IODATE SOLUTIONS
($\lambda = 536 \text{ m}\mu$, 1-cm. cells)

$\mu\text{g. of Al}$	<u>12.5</u>	<u>25.0</u>	<u>50.0</u>	<u>62.5</u>
Absorbance	0.123	0.265	0.540	0.681
	0.115	0.270	0.510	0.610
	0.118	0.264	0.528	0.658
	0.116	0.270	0.544	0.657
	0.125	0.269	0.548	0.658
	<u>0.119</u>	<u>0.271</u>	<u>0.523</u>	<u>0.654</u>
Average Absorbance	0.119	0.268	0.532	0.653

Plutonium-Aluminum Alloys (~ 0.04 to 0.2 percent aluminum)

Data were obtained for the aluminum concentration range of 0.024 to 0.191 percent using absorption cells with a 2-cm. light path. These data are shown in Table III and the average absorbance values versus the aluminum concentration are plotted in Figure 2. The line through the points was drawn according to the statistical method of least squares.

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Table III

ABSORBANCE OF ALUMINUM-ALUMINON COLOR IN
 PLUTONIUM IODATE SUPERNATANTS
 ($\lambda = 536 \text{ m}\mu$, 2-cm. cells, 20 mg. Pu/sample)

μg of Al	<u>4.87</u>	<u>18.44</u>	<u>28.14</u>	<u>38.16</u>
	(0.073	0.320	0.537	0.757
	(0.083	0.325	0.547	0.757
Absorbance	(0.080	0.319	0.536	0.753
	(0.080	0.320	0.526	0.756
	(<u>0.087</u>	<u>0.322</u>	<u>0.530</u>	<u>0.745</u>
Average Absorbance	0.081	0.321	0.535	0.754

Data were also obtained on aluminum iodate solutions using absorption cells with a 2-cm. light path. These data are shown in Table IV, and the average absorbance values versus the aluminum concentration are plotted in Figure 2. The line through the points was drawn according to the statistical method of least squares.

Table IV

ABSORBANCE OF ALUMINUM-ALUMINON COLOR IN
 ALUMINUM IODATE SOLUTIONS
 ($\lambda = 536 \text{ m}\mu$, 2-cm. cells)

$\mu\text{g.}$ of Al	<u>10</u>	<u>20</u>	<u>30</u>	<u>40</u>
	{ 0.156	0.371	0.597	0.837
Absorbance	{ 0.158	0.362	0.601	0.826
	{ <u>0.162</u>	<u>0.392</u>	<u>0.610</u>	<u>0.806</u>
Average Absorbance	0.159	0.375	0.603	0.823

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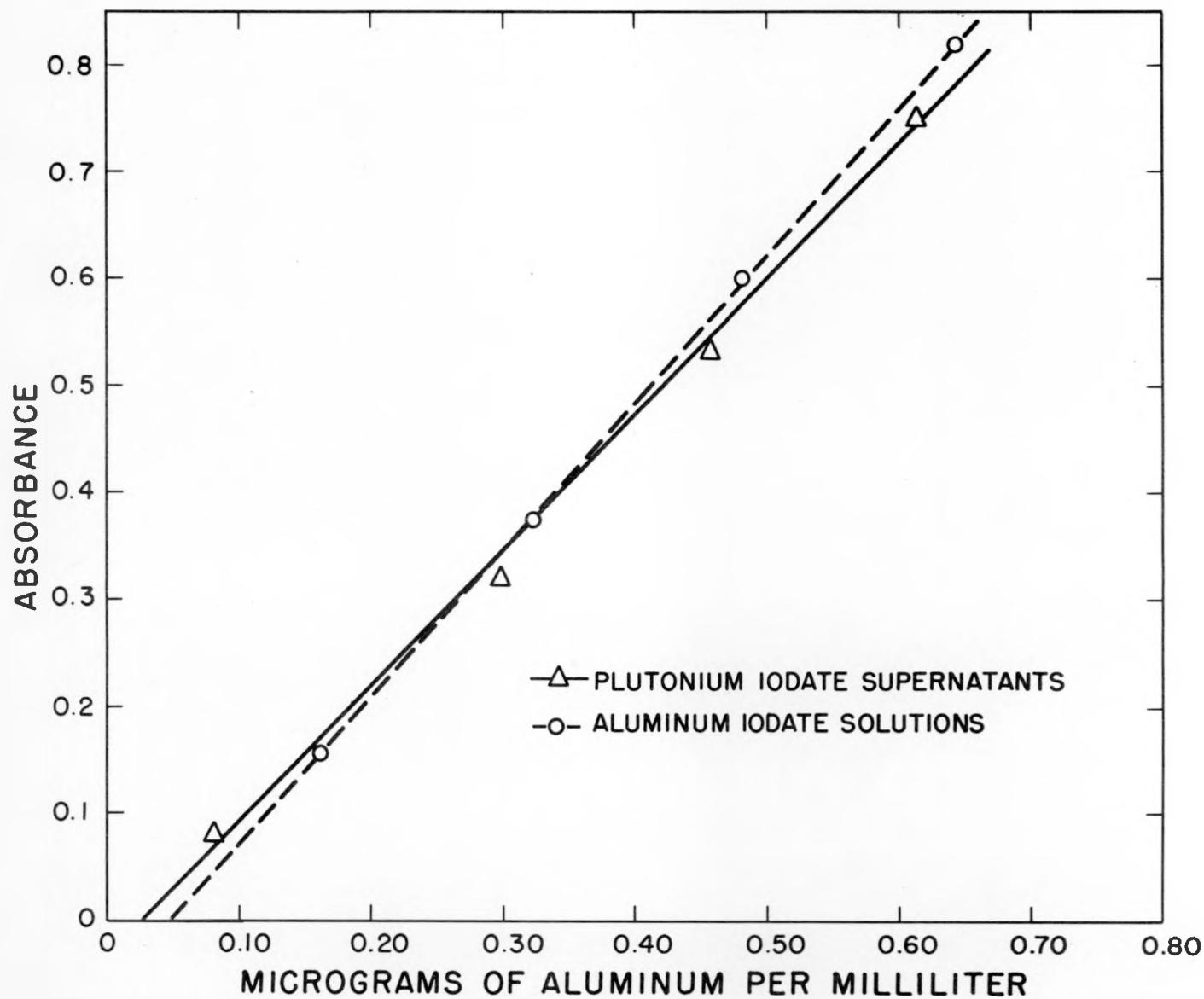


Figure 2. Absorbance of aluminon-aluminum color (2-cm. cells)

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Plutonium-Aluminum Alloys (~0.02 to 0.04 percent aluminum)

Absorbance data for the aluminum concentration range of 0.022 to 0.034 percent aluminum were obtained using absorption cells with a 10-cm. light path. These are shown in Table V and the average absorbance values plotted versus the aluminum concentration in Figure 3. The line through the points for the aluminum concentration range of 0.070 to 0.108 micrograms of aluminum per milliliter was drawn according to the statistical method of least squares.

Table V

ABSORBANCE OF ALUMINUM-ALUMINON COLOR IN
PLUTONIUM IODATE SUPERNATANTS
($\lambda = 536 \text{ m}\mu$, 10-cm. cells, 20 mg. Pu/sample)

$\mu\text{g. of Al}$	<u>0.954</u>	<u>0.975</u>	<u>2.61</u>	<u>2.81</u>	<u>4.35</u>	<u>4.69</u>	<u>5.21</u>	<u>5.85</u>	<u>6.58</u>	<u>6.73</u>
	(0.070	0.098	0.127	0.195	0.286	0.336	0.383	0.479	0.515	0.571
Absorbance	{									
	(0.064	0.094	0.136	0.207	0.288	0.361	0.343	0.469	0.540	0.580
	{									
	(<u>0.118</u>	<u>0.097</u>	<u>0.195</u>	<u>0.205</u>	<u>0.304</u>	<u>0.375</u>	<u>0.378</u>	<u>0.473</u>	<u>0.545</u>	<u>0.579</u>
Average										
Absorbance	0.084	0.096	0.152	0.202	0.293	0.357	0.368	0.474	0.533	0.577

Similar data for aluminum iodate solutions are shown in Table VI and the average absorbance values versus the aluminum concentration are plotted in Figure 3. The line through the points was drawn according to the statistical method of least squares.

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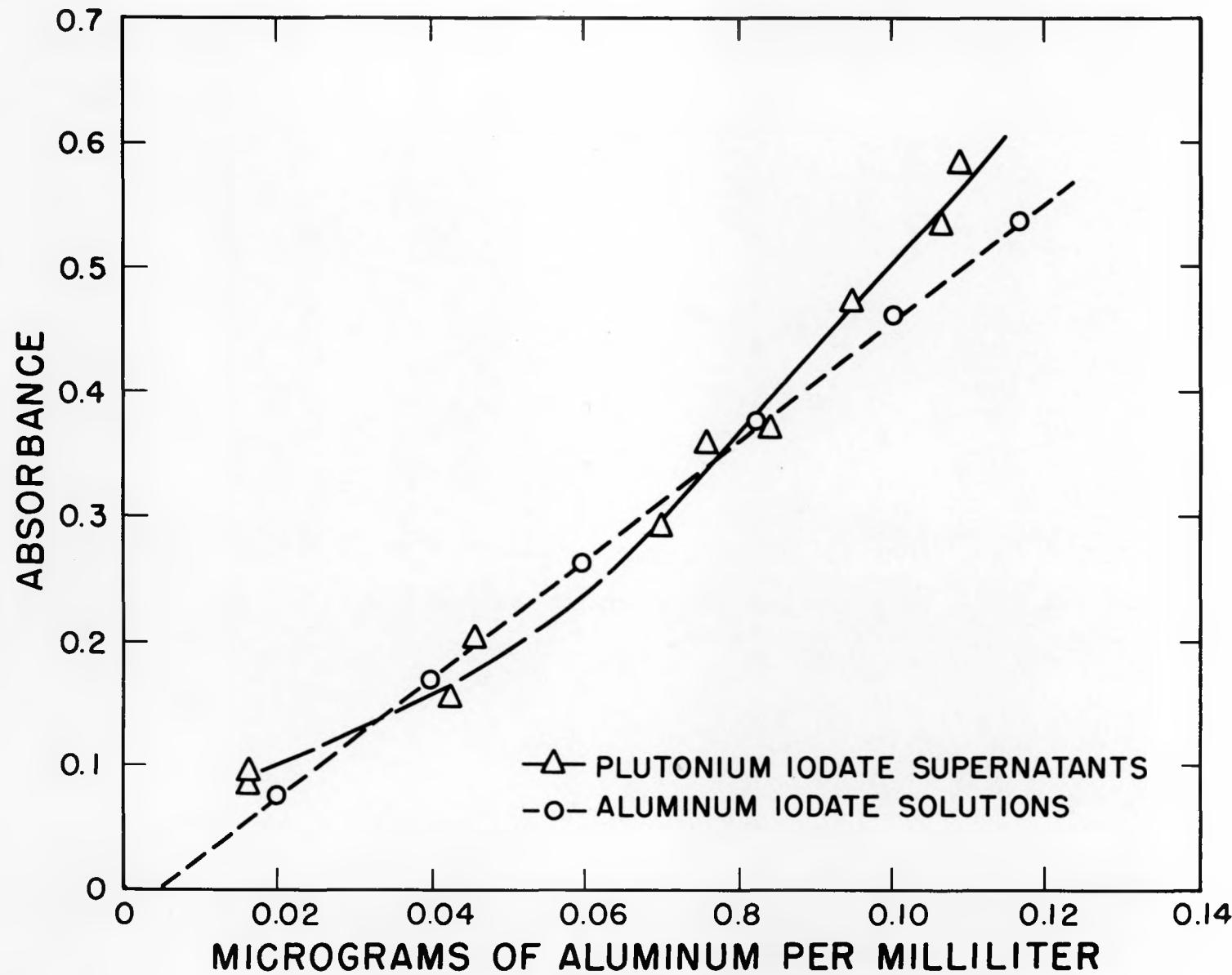


Figure 3. Absorbance of aluminon-aluminum color (10-cm. cells)

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Table VI

ABSORBANCE OF ALUMINUM-ALUMINON COLOR IN
ALUMINUM IODATE SOLUTIONS
($\lambda = 536 \text{ m}\mu$, 10-cm. cells)

$\mu\text{g. of Al}$	<u>1.23</u>	<u>2.50</u>	<u>3.73</u>	<u>5.14</u>	<u>6.25</u>	<u>7.30</u>
Absorbance	(0.059	0.152	0.245	0.359	0.447	0.516
	(0.099	<u>0.195</u>	<u>0.285</u>	<u>0.397</u>	<u>0.477</u>	<u>0.552</u>
Average Absorbance	0.079	0.174	0.265	0.378	0.462	0.534

STATISTICAL RESULTS

It is apparent from the general experimental technique and from the comparison of the absorbance curves for the supernatants and pure solutions, especially those plotted in Figure 3, that the method is strictly empirical. Evaluation of the method depends on the selection of the range which will give satisfactory precision. In order to do this the data for the three aluminum concentration ranges (Tables I, III, V) were given statistical treatment and a standard deviation calculated for each aluminum concentration used based on all of the data in the particular range considered.⁽⁵⁾ The results are shown in Tables VII through IX.

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Table VII

STATISTICAL RESULTS FOR THE DETERMINATION OF ALUMINUM
IN PLUTONIUM IODATE SUPERNATANTS
(~0.2 to 0.4% Al, 1-cm. cells)

<u>Al</u> (μ g./ml.)	<u>Al</u> (%)	<u>Standard Deviation*</u> (relative %)
0.16	0.05	17.4
0.32	0.10	8.5
0.48	0.15	5.7
0.64	0.20	4.2
0.80	0.25	3.4
0.96	0.30	2.9
1.12	0.35	2.5

* Based on 32 determinations.

Table VIII

STATISTICAL RESULTS FOR THE DETERMINATION OF ALUMINUM
IN PLUTONIUM IODATE SUPERNATANTS
(~0.04 to 0.2% Al, 2-cm. cells)

<u>Al</u> (μ g./ml.)	<u>Al</u> (%)	<u>Standard Deviation*</u> (relative %)
0.078	0.024	16.1
0.295	0.092	4.0
0.448	0.141	2.7
0.608	0.191	2.0

* Based on 20 determinations.

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Table IX

STATISTICAL RESULTS FOR THE DETERMINATION OF ALUMINUM
IN PLUTONIUM IODATE SUPERNATANTS
(~0.02 to 0.04% Al, 10-cm. cells)

<u>Al</u> (<u>μg./ml.</u>)	<u>Al</u> (%)	<u>Standard Deviation*</u> (relative %)
0.070	0.022	4.7
0.075	0.024	4.3
0.083	0.026	3.8
0.094	0.029	3.4
0.105	0.033	3.1
0.108	0.034	3.0

* Based on 18 determinations.

Seven plutonium-aluminum alloy samples ranging in aluminum content from 0.03 to 0.3 weight percent were analyzed in duplicate according to the method described in this report. Absorption cells with a 2-cm. light path were used. The difference between duplicate analyses for each sample was less than 3 relative percent.

EFFECT OF VARIOUS IONS ON THE PROCEDURE

Cations

In general all ions found previously to interfere with the aluminum spectrophotometric procedure would be expected to interfere

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with the determination of aluminum in plutonium as described in this report. The mercury cathode electrolysis included in the procedure serves to remove some of the most troublesome ions such as iron, copper, nickel, lead, tin, and antimony. Many of the interference studies reported in the literature are based on a specific procedure which includes a preliminary separation, e.g., cupferron precipitation, and care must be taken not to assume that these data are applicable to the method described in this report. Also, the use of reducing agents such as thioglycolic acid to prevent iron interference is not possible in the procedure described in this report due to the presence of iodate in the solution.

The effect of varying the plutonium content of the sample was studied. The results are shown in Table X and indicate that variations of the plutonium to aluminum ratio from 17 to 417 affect the absorbance readings less than 5 percent. Iron was removed from each plutonium sample by electrolysis prior to analysis.

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Table X

EFFECT OF PLUTONIUM ON THE DETERMINATION OF
ALUMINUM IN PLUTONIUM ALLOYS

<u>Pu in sample (mg.)</u>	<u>Al in sample (μg.)</u>	<u>No. of Determinations</u>	<u>Average Absorbance</u>
1	60	2	0.602
5	60	2	0.577
10	60	2	0.595
20	60	2	0.569
25	60	2	0.583

Anions

Anions preventing the proper functioning of the electrolytic separation must be avoided.⁽¹⁴⁾ Sulfate and perchlorate solutions are quite satisfactory and, in addition, do not interfere with the color reagent. Chloride and nitrate ions do not interfere with the color formation but cause difficulty in the electrolytic separation.

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The color of the permanganate ion interferes when present in the final solution in 2 micrograms per milliliter concentrations.⁽⁴⁾ Also phosphate present in 10 micrograms per milliliter concentration begins to inhibit the color formation.⁽⁴⁾

CONCLUSIONS

It is possible by the method described in this report to determine aluminum in plutonium alloys containing approximately 0.02 to 0.4 percent (weight percent) by the choice of the proper length absorption cells. A standard deviation of approximately 3 relative percent or better was calculated from the data obtained in the concentration range of approximately 0.035 to 0.4 percent.

The method is fairly independent of the amount of plutonium present thereby permitting applications over a wide range of aluminum concentrations by the proper selection of sample size.

Elements commonly known to interfere in the aluminon procedure for aluminum in other materials interfere here. Some can be removed by mercury cathode electrolysis.

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