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EFFECT OF PLUTONIUM ON THE FLUOROMETRIC DETERMINATION OF URANIUM

by

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CHEMISTRY - TRANSURANIC ELEMENTS
(M-3679, 15th edition)

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ABSTRACT

The fluorometric method for determining microgram quantities of uranium dissolved in concentrated sulfuric acid was reinvestigated for the purpose of measuring any interference caused by plutonium in uranium samples. No plutonium fluorescence, and therefore no positive errors due to plutonium, were observed. Limited transmittance of hexavalent plutonium in sulfuric acid at 513, and especially at 360 millimicrons, produces variable negative errors in uranium determinations.

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INTRODUCTION

The fluorometric method for determining microgram quantities of uranium described by Wise(4) was reinvestigated for the purpose of measuring any interference caused by plutonium in uranium samples. If a concentrated sulfuric acid solution containing plutonium will fluoresce under primary radiation of 350 to 370 millimicrons, then plutonium may cause positive errors when using a Farrand fluorometer and the fluorometric procedure developed by Wise. Interference in this manner, however, was not anticipated. Price(3) was unable to find any condition under which a plutonium inorganic compound gave evidence of fluorescence. One of his samples was observed under conditions similar to those used in the Wise procedure for uranium. Price reported that a solution of Pu (VI) in concentrated sulfuric acid, both at room temperature and at the temperature of liquid nitrogen, gave no indication of fluorescence when the irradiating light was 365 millimicrons in wavelength.

Plutonium may also produce negative errors in uranium fluorometric determinations. If a concentrated sulfuric acid solution of plutonium absorbs light at 360 or 513 millimicrons, the results for uranium will tend to be low with samples containing plutonium. A portion of the primary light normally causing uranium fluorescence may be absorbed by plutonium or interference may arise from reduced transmittance for the secondary radiation from uranyl sulfate, at 513 millimicrons in the

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Wise fluorometric procedure.

Before any effect of plutonium could be measured, it was found necessary to modify the fluorometric procedure, especially in the section dealing with sample preparation. These modifications are described in a separate report(1); only the final revision of the procedure is given in this report.

EQUIPMENT

Fluorometer, photoelectric. Farrand Model A.

Transformer, constant voltage, for ultraviolet lamp in fluorometer. Sola catalog No. 30807.

Galvanometer. Rubicon catalog No. 3406 HA.

Filter, primary. Corning No. 5860, 50 x 50 x 5 mm.

Filters, secondary, for sample and background measurements. Two carefully selected Farrand interference-type filters with maximum transmission near 513 and 470 millimicrons are required. Selection of filters is described under <u>EXPERIMENTS AND DISCUSSION</u> in reference (1).

Heating block. This aluminum block, 25 x 38 x 120 mm., has six 10 x 32 mm. holes drilled into the 25 x 120 mm. side at a 30° angle to the horizontal position for heating 1-ml. volumetric tubes. The aluminum block should be heated with an adjustable electric hot plate, similar to a Fisher Autemp heater.

Tubes, volumetric. The special Pyrex tubes are graduated for one milliliter, 65 - 67 mm. in length with a uniform outside diameter of 10 mm. and a total length of about 105 mm., including a ₮ stopper No. 9. These dimensions are necessary to permit proper placement of the volumetric tube in the fluorometer sample carrier.

Tubes, culture. Pyrex No. 9820, 10 x 75 mm.

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Cap, special, for fluorometer sample carrier. This cap is identical with unit provided with instrument except for a height of 45 mm. instead of 25 mm. The larger cap permits use of the special 1-ml. volumetric tubes in the sample carrier.

Flask, Dewar. Cylindrical flask is 18 cm. in diameter and 30 cm. in height.

Sample support. The support is required for holding sample tubes in a vertical position when placed in the Dewar flask.

Spectrophotometer. Beckman Model DU.

REAGENTS

Uranium metal, high-purity. Uranyl sulfate trihydrate, analytical grade, may be used in place of the metal.

Perchloric acid, 70 per cent, analytical grade.

Sulfuric acid, organic-free, 91.2 to 93.8 percent with optimum concentration of 92.4 percent. The concentration can be determined with sufficient accuracy by measuring the specific gravity at room temperature. The corresponding specific gravities for the above concentration range are 1.820 and 1.831. Before dilution of 96 percent, reagent-grade acid to the optimum concentration, it should be made organic-free by adding one milliliter of perchloric acid, 70 percent, to 100 milliliters of the sulfuric acid and heating the mixture to boiling until it is colorless. This organic-free acid should be cooled, diluted, and retained in the same container, preferably one with an inverted ₸ stopper, to prevent contamination.

Carbon dioxide, solid, commercial material.

Methanol, technical grade.

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REVISED PROCEDURE

Sample Preparation

Samples should be in a form which permits transfer of the contained uranium to 1-ml. volumetric tubes where the material is dissolved in sulfuric acid before making fluorometric measurements. Water or volatile acids are satisfactory solvents for samples. Only traces of non-volatile substances should accompany the uranium. To achieve this condition the uranium may be extracted with diethyl ether, the solvent removed by evaporation, and the residue dissolved in a small volume of nitric acid, normally not exceeding one milliliter. This reagent then serves as the medium for transfer of the uranium to a 1-ml. volumetric tube.

1. Transfer the sample, as an aqueous or nitric acid solution containing no more than trace amounts of non-volatile substances, to a 1-ml. volumetric tube having a diameter and length which permit placement of the tube in the fluorometer sample carrier.
2. Add one drop of 70 percent perchloric acid and one drop of concentrated sulfuric acid to each sample.
3. Place tubes in holes in an aluminum heating block on an adjustable hot plate, and evaporate samples to near dryness at maximum temperature but without loss by spattering.
4. Increase temperature of heating block to about 225° C. until perchloric acid has been removed by fuming.
5. Remove tubes from aluminum block and cool to room temperature.
6. Repeat steps 2 to 5 if there is any question of not having removed all organic matter.

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7. Add one drop of 70 percent perchloric acid and sufficient organic-free sulfuric acid to dilute each sample to exactly one milliliter. Use a narrow pipet in adding the acid so that no liquid is above the graduation mark.

8. Replace tubes in aluminum heating block on an adjustable hot plate and heat at 225° C. for one hour.

9. After removal of tubes from heating block and cooling to room temperature, centrifuge tubes to return all sulfuric acid on the walls to the lower portion of the tube. If necessary, add organic-free sulfuric acid to any tubes where liquid level is below the 1-ml. graduation mark.

10. Close tubes with Ŧ stoppers and make fluorometric determinations according to directions under Operation.

Operation

Prepare the Farrand fluorometer for use in a semi-darkened room by turning on the switch controlling the mercury vapor lamp and fan, but leave the phototube switch turned off and keep the instrument shutter in the down position. Allow at least 10 minutes for the lamp to reach maximum intensity. During this time it is possible to proceed with chilling of samples described in step 1 below.

Make certain that the primary filter holder, containing a Corning No. 5860 filter, is in proper position in the fluorometer. Place the sample-measuring interference filter (513 millimicrons) in the secondary filter holder, and insert holder into instrument with clear side of filter next

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to the sample carrier. The background-measuring interference filter should be available in another secondary filter holder for later use. Place the sample carrier into instrument and cover with cap.

Turn on galvanometer lamp and phototube switch. Depress zero button and carefully note the resulting galvanometer reading. Release zero button and, by turning the Dark Current coarse and fine knobs, adjust the galvanometer to the reading just noted. This procedure balances the fluorometer for dark current. Now bring the galvanometer spotlight to zero reading by placing both thumbs gently against the glass scale, one at each end, and sliding it back and forth to attain the desired position. Normally, the coarse zero adjustment lever on the top of the galvanometer is not employed in bringing the spotlight to zero reading. After the scale has been positioned it is well to repeat the balancing of the fluorometer for dark current as described above. Dark current knobs should not be disturbed during the measurement of samples.

1. Chill all samples thoroughly in a slush of dry ice and methanol in a Dewar flask. In addition to samples of unknown uranium content in 1-ml. volumetric tubes, calibration samples in sealed tubes, prepared according to directions under Calibration Curve, and containing either 2.5 or 25 micrograms of uranium, must be chilled to be available for adjustment of the fluorometer sensitivity control to one of the two ranges within which measurements are to be made.
2. Remove either the 2.5- or the 25-microgram calibration sample from

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the chilling mixture, wipe off with a lintless cloth, place in sample carrier, and cover with carrier cap.

3. Quickly raise instrument shutter and adjust sensitivity control so that the galvanometer has a reading of 10.00 centimeters (see Note 1). Lower shutter to down position and replace sample in chilling mixture.

4. Repeat steps 2 and 3 after calibration sample is thoroughly rechilled.

5. Remove an unknown sample from chilling mixture, wipe off 1-ml. volumetric tube, place it in sample carrier, and place cap on carrier (see Note 2).

6. Quickly raise instrument shutter and observe galvanometer when reading is momentarily constant. Record this reading, lower shutter to down position, and return sample to chilling mixture.

7. Repeat steps 5 and 6 for each additional unknown sample (see Note 3).

8. Repeat steps 2 and 3 with the same calibration sample used previously in these steps, and readjust the sensitivity control to a 10.00 cm. galvanometer reading. If this adjustment is more than ±0.20 cm., the instrument has drifted, data recorded under step 6 should be discarded, and all samples should be rerun (see Note 4).

9. Turn off phototube switch, replace secondary filter holder containing the sample-measuring interference filter with the holder containing the background-measuring filter, and turn on phototube switch.

10. Repeat steps 5 and 6 for each unknown sample.

11. Replace secondary filter holder containing the background-measuring

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filter with the holder containing the sample-measuring filter. Remove all samples from chilling mixture, returning the sealed calibration tubes to the calibration sample holder.

12. Turn off galvanometer lamp, phototube switch, and the mercury vapor lamp if the instrument is not to be used for further observations within the next hour.

Calculations

For each unknown sample

1. Subtract galvanometer reading obtained with background-measuring filter (step 10) from the reading obtained with the sample-measuring filter (step 6).
2. Use this difference to read the quantity of uranium in each sample, in micrograms, from a calibration curve. This curve should be prepared according to directions under Calibration Curve with a set of calibration samples which includes the sample used in step 2 under Operation.

Notes

1. If the adjustment of the sensitivity control is not done quickly, the calibration sample tube becomes fogged, and correct adjustment is impossible. To avoid this difficulty and provide time for careful adjustment, chilling of the calibration sample may be omitted and it is placed in the sample carrier at room temperature. Naturally, a galvanometer reading of 10.00 cm. is no longer used since the fluorescence

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intensity is temperature-dependent. Experience indicates that a calibration sample (25 micrograms) which gives a reading of 10.00 cm. when the sample is chilled will give a reading near 3.00 cm. when the sample has warmed to room temperature and no change is made in the instrument adjustment. Consequently, all subsequent adjustments of the sensitivity control could be made with the calibration sample at room temperature and with a galvanometer reading at some arbitrary value near 3.00 cm.

2. If the sulfuric acid solution freezes and forms crystals while in the chilling mixture, it will be impossible to use the sample for a fluorometric determination. Freezing results when the sulfuric acid concentration is above 93.8 percent or below 91.2 percent. A new sample should be prepared.

3. The instrument shutter may be maintained in the raised position continuously during steps 6 and 7 under Operation.

4. If at any time erratic motion of the galvanometer is noticed, or if a sudden decrease in sensitivity is experienced, check each battery of the internal phototube battery supply with a voltmeter which draws a small current (not a vacuum tube voltmeter). Replace any unit battery not measuring full rated voltage.

Calibration Curve

1. Prepare a standard solution containing 50 micrograms of uranium per milliliter (Solution A) by dissolving either 50 mg. of high-purity

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metal in 3 ml. of 6N HCl or 88.2 mg. of $UO_2SO_4 \cdot 3H_2O$ in distilled water, transfer the resulting solution quantitatively to a 1000-ml. volumetric flask, and dilute to volume with distilled water.

2. Prepare another standard solution containing 5 micrograms of uranium per milliliter (Solution B) by transferring exactly 10 ml. of Solution A to a 100-ml. volumetric flask and diluting to volume with distilled water.

3. Prepare volumetric tubes graduated at 1 ml. by careful addition of exactly 1 ml. of water to Pyrex culture tubes (10 x 75 mm.). The water should be added from a precision pipet so that no splashing occurs. Mark the liquid level on the glass tubes with a diamond marking pencil.

4. For the range of 1.0 to 25 micrograms of uranium, transfer to each 1-ml. volumetric tube an aliquot of Solution A (volumes from 20 to 500 microliters) with a micropipet. Mark the amount of uranium added to each tube with a diamond marking pencil just above the graduation mark.

5. Repeat step 4 for a series of aliquots taken from Solution B for the range of 0.1 to 2.5 micrograms of uranium (volumes of 20 to 500 microliters).

6. Place the volumetric tubes in a small beaker, cover with an inverted larger beaker, and place in an oven at 95° C. until dry.

7. After allowing the tubes to cool to room temperature, add to each one drop of 70 percent perchloric acid and sufficient organic-free sulfuric acid to dilute to exactly one milliliter. Use a narrow pipet in adding acid so that no liquid is above graduation mark.

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8. Repeat step 7 with volumetric tube to which no uranium was added, to prepare a blank calibration sample.

9. Place tubes in holes in an aluminum heating block on an adjustable hot plate and heat at 225° C. for one hour.

10. After removing tubes from the heating block and cooling to room temperature, if necessary, add organic-free sulfuric acid to any tube where liquid level is below 1-ml. graduation mark.

11. Immediately seal the open end of the tubes with an oxygen-gas flame so that the total length of the closed tube is about 80 mm.

12. Place sealed calibration sample tubes in suitable container to prevent loss from breakage.

13. Obtain calibration data by making observations with sealed samples according to steps 1 to 10 under <u>Operation</u>, and under step 1 of <u>Calculations</u>.

14. Prepare a calibration curve from these data with galvanometer readings in centimeters as ordinate and micrograms of uranium as abscissa.

PLUTONIUM FLUORESCENCE

To verify the conclusion that plutonium does not introduce a positive error into fluorometric measurements of uranium, a solution was prepared by transferring approximately 10 mg. of pure plutonium, as a hydrochloric acid solution of Pu (III), to a culture tube (10 x 75 mm.). After a few drops of perchloric acid were added, the tube was heated to fumes with an infrared lamp to remove the chloride and oxidize plutonium to Pu(VI). When the tube was cooled, one milliliter of concentrated sulfuric acid and one drop of 70 percent perchloric acid were added. The tube was reheated sufficiently to remove any organic matter and drive off excess perchloric acid. After cooling, the acid solution was transferred to a clean culture tube which was immediately sealed in an oxygen-gas flame. Two blank solutions were given identical treatment except for the omission of plutonium. The tube containing plutonium and all similar samples in subsequent operations were handled with sufficient care so that the sealed tubes showed no radioactivity.

After the plutonium sample and the two blanks were chilled in a mixture of dry ice and methanol, observations made according to the revised procedure led to the data in Table I. For comparative purposes the data in Table II were obtained with uranium samples and blank solutions with one change in procedure. After the tubes containing sulfuric and perchloric acids were heated, the culture tubes were immediately sealed without the additional step of transferring the solutions to clean tubes. Likewise in the preparation of all subsequent samples, the solutions were sealed in tubes in which final heating was performed.

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Table I

INTENSITY OF FLUORESCENCE FOR Pu (VI) AND BLANK SOLUTIONS

Solution No.	1	2	3
Pu added, Milligrams	0	9.52*	0
Galvanometer readings, Cm.			
Filter W-512	1.00	0.16	1.32
Filter W-470	0.77	0.12	1.06
Difference	0.23	0.04	0.26

* By radiochemical analysis Solution No. 2 contained 0.87 milligram of Pu (III) and (IV) and 8.65 milligrams of Pu (VI).

Table II

INTENSITY OF FLUORESCENCE FOR U (VI) AND BLANK SOLUTIONS

Solution No.	4	5	6	7
U added, Micrograms	0	0	4.2	17.4
Galvanometer readings,Cm.				
Filter W-512	0.12	0.30	1.86	7.07
Filter W-470	0.08	0.22	0.32	0.85
Difference	0.04	0.08	1.54	6.22

There is no indication of plutonium fluorescence from the data in Table I, since the intensity for the plutonium solution is even less than that for the blank solutions. The fact that the blank solutions in Table I have significantly greater intensities than the blank solutions in Table II is attributed to organic material in the supposedly clean tubes to which final transfer was made for solutions 1, 2, and 3.

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The low result for solution 2 in Table I, compared with solutions 1 and 3, may be due to absorption of radiation at 365 or 512 millimicrons by plutonium. If plutonium causes quenching in this manner, it will introduce negative errors in the fluorometric determination of uranium.

QUENCHING BY PLUTONIUM

An attempt to observe quenching of uranium fluorescence by plutonium was made with four uranium samples prepared by transferring weight aliquots of a standard uranium solution to culture tubes (10 x 75 mm.) and carefully evaporating to dryness. One milliliter of concentrated sulfuric and several drops of perchloric acids were added to each tube. Before final heating, two of these samples were altered by adding plutonium. The 10 mg. of Pu (VI) added to solution 10 (Table III) remained in solution after heating to perchloric acid fumes, but the equal amount of plutonium added as a mixture of Pu (III) and (IV) to solution 11 formed a blue precipitate which was converted to a red insoluble solid during the heating. Radiochemical analysis of solution 11 made at a later time revealed that only 0.2 mg. of plutonium remained dissolved in this solution.

The effect of these additions of plutonium to uranium samples is shown in Table III. In the amount added, Pu (VI) almost completely quenches uranium fluorescence. These results were sufficient to indicate a need

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Table III

EFFECT OF PLUTONIUM ON FLUOROMETRIC URANIUM DETERMINATIONS

Solution No.	8	9	10	11
Pu added, Mg.	0	0	10	0.2
U added, Micrograms	1.0	8.3	18.3	18.5
Galvanometer readings, Cm.				
W-512	0.59	3.40	0.53	7.32
W-470	0.17	0.47	0.07	0.95
Difference	0.42	2.93	0.46	6.37
U found, Micrograms*	1.2	8.2	1.3	17.8
U error, Micrograms	+0.2	-0.1	-17.0	-0.7

* The uranium found was calculated by dividing the differences given in this table by 0.358 cm. per microgram, which is the slope of the calibration curve prepared by Wise(4). His calibration sample 25-B was used to adjust the fluorometer.

for data on percent transmittance at various wavelengths for concentrated sulfuric acid containing dissolved plutonium, preferably at both the III or IV and the VI valence states. This information was obtained with a Beckman model DU spectrophotometer and is presented graphically in Figure 1. Concentrated sulfuric acid (approximately 95 percent H_2SO_4 by weight) was used as a reference standard for three Pu solutions. Two of these solutions contained Pu (VI) which was prepared by treating a pure plutonium chloride solution with several drops of perchloric acid and heating to fumes before adding the proper amount of concentrated sulfuric acid. The third solution, indicated in Figure 1 as containing Pu (IV) and (VI), was prepared by attempting to dissolve one milligram of plutonium as Pu (IV) nitrate in one milliliter of

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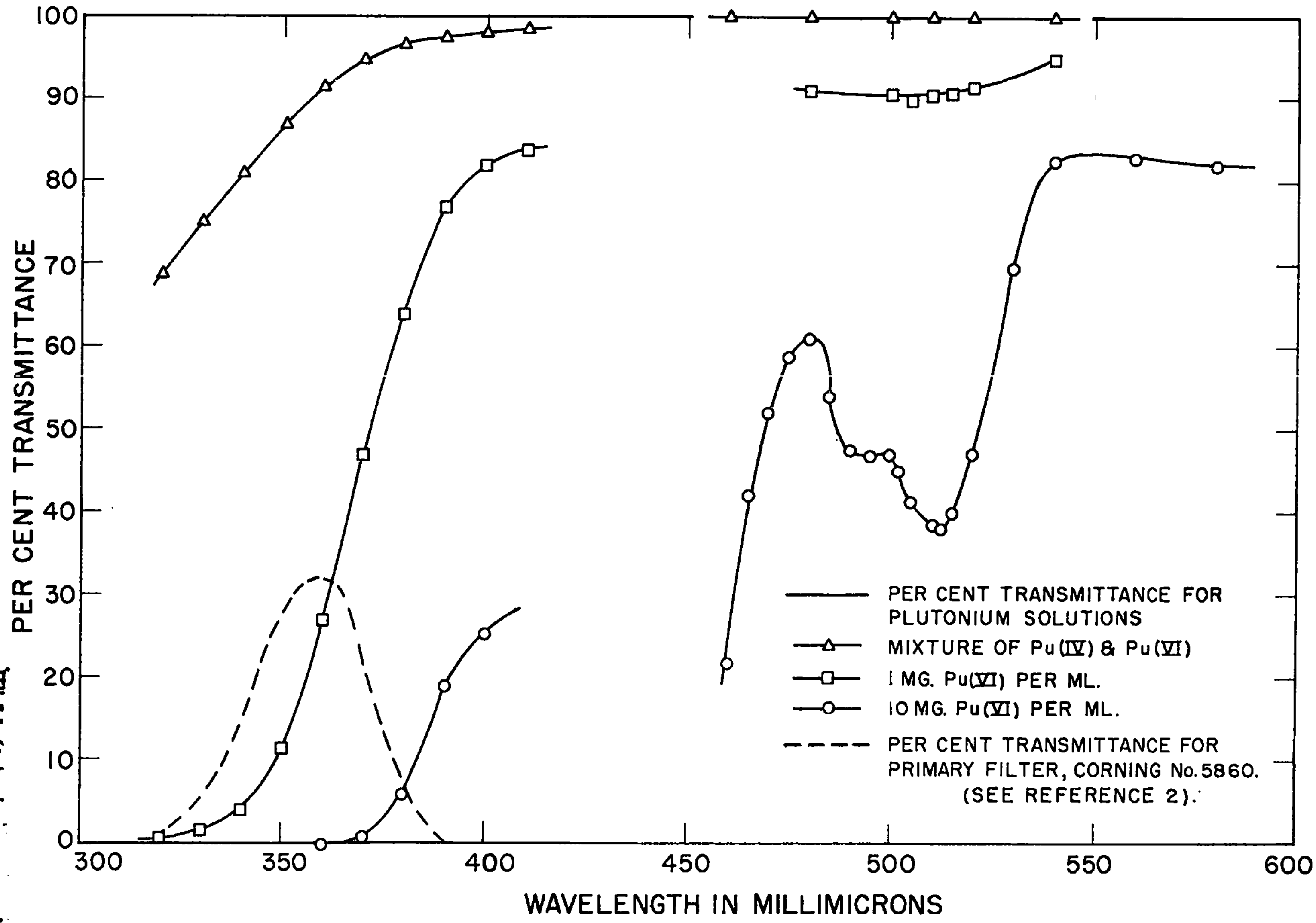

Figure 1. Percent Transmittance for Solutions of Plutonium in Concentrated Sulfuric Acid

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concentrated sulfuric acid. Most of this plutonium did not dissolve and probably the major portion of the dissolved material was present as Pu (VI). Not much significance can be given to the transmittancy data for this solution.

It is obvious from Figure 1 that the quenching of uranium fluorescence by Pu (VI) results from absorption of the primary ultraviolet light. The maximum transmission, for the Corning filter No. 5860 which is used in the Farrand fluorometer to absorb visible radiation, is at 360 millimicrons(2). At this wavelength a 10-milligram Pu (VI) per milliliter solution is completely opaque and a 1.0-milligram solution has a transmittancy of 27 percent. The absorption of visible light by Pu (VI) solutions, for example at 512 millimicrons, is less pronounced but still significant. There is some possibility that interference from plutonium could be minimized if the formation of Pu (VI) were prevented and the plutonium separated from the sulfuric acid solution as insoluble Pu (IV) sulfate. This is difficult, however, since the uranium procedure includes heating with perchloric acid to oxidize traces of organic material.

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INTERFERENCE BY PLUTONIUM IN URANIUM DETERMINATIONS

The sulfuric acid solution (containing one milligram of Pu (VI) per milliliter) with which the percent transmittance data in Figure 1 were obtained was also used in a fluorometric determination after adding a known quantity of uranium. A negative error of 28 to 29 percent was observed in samples containing 5 micrograms of uranium. Further measurement of errors caused by plutonium in the range of 10 to 1000 micrograms Pu per milliliter was made with samples, each containing the same quantity of uranium. The various amounts of plutonium, and the uranium as a 100-microliter volume aliquot, were added to culture tubes (10 x 75 mm.) and were carefully evaporated to dryness before being dissolved in concentrated sulfuric acid. The standard uranium solution was prepared from high-purity metal so that 100 microliters contained 11.4 micrograms of the element. The results of these measurements are shown in Table IV.

These data indicate that up to 25 micrograms of plutonium in one milliliter of sulfuric acid solution will have no effect upon uranium determinations made with such solutions. If the amount of plutonium is increased beyond this quantity to as much as one milligram, two effects will be noticed. Low uranium values will be obtained with average errors up to 30 percent, and repeated determinations on aliquots of the same sample will yield results over a wider range as the plutonium concentration is increased. This latter effect may be due to varying

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amounts of plutonium being oxidized to Pu (VI) by perchloric acid during preparation of the sulfuric acid solution of uranium.

Table IV

EFFECT OF PLUTONIUM ON URANIUM DETERMINATION

(Each sample contained 11.4 micrograms of U)

Pu added, Micrograms	0		12	24	60		120	1200
U found in Micrograms*	11.8	12.1	11.3	11.4	9.0	11.0	10.1	7.8
	11.0	11.5	11.9	11.6	9.7	9.5	11.3	7.4
	11.8	11.9	11.7	11.3	11.7	9.6	11.2	8.3
	11.8	11.8	11.6	11.5	10.8	10.2	10.8	8.9
	11.4	11.9	11.5		11.4		10.5	
	11.6	11.3	11.5		11.1		8.3	
	11.5	11.5			10.7		10.3	
	11.1				11.5		10.4	
	11.8				11.5		10.7	
Ave. U found	11.6		11.6	11.5	10.6		10.4	8.1
Standard Dev.	0.3		0.2	0.1	0.9		0.9	0.7
Percent error in Ave. U found	+2		+2	+1	-7		-9	-29

* The uranium found was determined by standardizing the fluorometer with a 10-microgram uranium sample No. 10-A prepared by Wise and described in LA-1098, pages 11 and 18 (Table IV).

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CONCLUSIONS

1. No fluorescence due to plutonium was observed under the conditions required for fluorometric uranium determinations.

2. A solution of hexavalent plutonium in concentrated sulfuric acid has a limited transmittance at 513 millimicrons and an even more restricted transmittance at 360 millimicrons.

3. The transmittance of hexavalent plutonium in sulfuric acid is the reason for interference by plutonium in fluorometric determinations of uranium. Up to 25 micrograms of plutonium in one milliliter of sulfuric acid solution will have no effect upon determinations of uranium in this solution. If the amount of plutonium is increased to one milligram, low uranium values will be obtained with average errors up to 30 percent. Repeated uranium determinations on aliquots of the same sample will yield results over a wider range as the plutonium concentration is increased.

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