RI 8433

Bureau of Mines Report of Investigations/1980

Spectrophotometric Determination of Uranium Using Dibenzoylmethane

By M. M. Jones, J. S. MacDuff, and A. B. Whitehead

UNITED STATES DEPARTMENT OF THE INTERIOR

Report of Investigations 8433

Spectrophotometric Determination of Uranium Using Dibenzoylmethane

By M. M. Jones, J. S. MacDuff, and A. B. Whitehead

UNITED STATES DEPARTMENT OF THE INTERIOR
Cecil D. Andrus, Secretary

BUREAU OF MINES
Lindsay D. Norman, Acting Director

This publication has been cataloged as follows:

Jones, Miriam M

Spectrophotometric determination of uranium using dibenzoylmethane.

(Report of investigations - Bureau of Mines ; 8433)

Bibliography: p. 11-12.

Supt. of Docs. no.: I 28.23:8433.

1. Uranium—Analysis. 2. Spectrophotometry. 3. Dibenzoylmethane. I. MacDuff, J. S., joint author. II. Whitehead, Alton B., joint author. III. Title. IV. Series: United States. Bureau of Mines. Report of Investigations ; 8433.

TN23.U43 [QD181.U7] 622'.08s [546'.431] 79-607983

CONTENTS

ILLUSTRATIONS

TABLES

SPECTROPHOTOMETRIC DETERMINATION OF URANIUM USING DIBENZOYLMETHANE

by

M. M. Jones,[1] J. S. MacDuff,[1] and A. B. Whitehead[2]

ABSTRACT

A procedure for the spectrophotometric determination of uranium has been developed in support of minerals research being conducted by the Bureau of Mines. Uranium is separated from the sample by tributylphosphate extraction from acid solution containing added aluminum nitrate and (ethylene dinitrilo)-tetraacetic acid solutions. The colored uranium-dibenzoylmethane complex is developed in the organic phase by adding an ethanol solution of dibenzoylmethane buffered with triethanolamine. Absorbance is measured at 410 nanometers, and the response is linear up to at least 100 micrograms U_3O_8 in the aqueous aliquot taken for analysis. Aliquots containing as little as 8 micrograms U_3O_8 may be analyzed with relative errors no greater than ±5 percent. For aliquots containing at least 35 micrograms U_3O_8, relative standard deviation at the 95-percent confidence level is about ±2.2 percent. Appropriate procedures are given for analyzing a wide variety of samples.

INTRODUCTION

Research being conducted by the Bureau of Mines, U. S. Department of the Interior, to improve metal and mineral processing technology includes projects in extraction and recovery of uranium from low-grade resources and in improved ion-exchange processing of uranium. These projects, carried out at the Salt Lake City Research Center, generate a wide variety of materials which must be accurately analyzed for uranium.

These analytical requirements were met by use of either a fluorimetric or volumetric method. To achieve reliable results with the fluorimetric method, duplicate determinations for each sample were necessary. In addition, the sensitivity of the fluorimetric method necessitated large dilutions for samples of moderate concentrations of uranium. The volumetric method was very time consuming and was used only when highest accuracy was required. An analytical procedure was needed that would be moderately sensitive, reasonably

---

[1] Chemist.

[2] Supervisory chemist.

All authors are with the Salt Lake City Research Center, Bureau of Mines, Salt Lake City, Utah.

accurate, and readily adaptable to volume production, thus bridging the gap between the extremely sensitive, marginally accurate fluorimetric method and the highly accurate less productive volumetric method.

This need was met by a spectrophotometric method (16)[3] adapted from the procedure of Francois (5), which was an improvement of an earlier method of Yoe, Will, and Black (17) and Pribil and Jelinek (9). The method consisted of separating uranium from its matrix by solvent extraction with tributyl phosphate (TBP) followed by color development in the organic phase using dibenzoylmethane (DBM).

After this method was used for a time, the need for a number of improvements became obvious. Iron and thorium, which were extracted into the TBP phase, absorbed at the uranium-DBM wavelength. Means of eliminating these interferences were required. Another problem encountered at intervals was traced to instability of pyridine-buffered chromogenic reagent. Because the uranium-DBM complex formation is optimum in the pH range from 5 to 9, pyridine is added to the DBM solution to neutralize acid extracted by the TBP. A more stable, and possibly less odorous, substitute for pyridine was desired. A third problem that required attention was that low results were sometimes obtained on acidic solutions in which precipitates formed when the pH was adjusted upwards prior to TBP extraction. Even though the precipitates appeared to redissolve upon addition of aluminum nitrate as salting agent, uranium values were occasionally in error.

Investigations were conducted that resulted in the much-improved procedure presented in this report. Addition of the tetrasodium salt of (ethylenedinitrilo)-tetraacetic acid (EDTA) to the aqueous sample before TBP extraction effectively controlled the iron interference and also significantly reduced the thorium interference. By adding the EDTA before the pH adjustment step, the formation of precipitates was prevented, and because of the alkalinity of the EDTA (pH = 11), some of the acid in the sample solution was neutralized. Odorless triethanolamine (TEA) proved to be an effective substitute for pyridine, providing reagent stability and good pH control. In conjunction with EDTA, use of TEA allows acid neutralization steps to be eliminated.

APPARATUS AND EQUIPMENT

Samples are dissolved in 500-milliliter Erlenmeyer flasks. TBP extraction and color formation are carried out in reusable, 40-milliliter glass vials with disposable, plastic-lined caps. Matching 5-centimeter cells are used for absorbance measurements. One-centimeter cells may be used for high concentrations. Absorbance is measured on a Perkin-Elmer (Coleman) 124-D[4] scanning UV/VIS spectrophotometer. Beckman Models B and DU spectrophotometers and a Brinkman PC/1000 probe colorimeter have also been used.

---

[3]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

[4]Reference to specific equipment does not imply endorsement by the Bureau of Mines.

REAGENTS AND STANDARDS

Metacresol purple: 0.5 gram of water-soluble salt per liter in aqueous solution.

EDTA: 200 grams of tetrasodium salt per liter in aqueous solution.

Salting agent: 2.8M aluminum nitrate - 2M ammonium hydroxide, prepared by dissolving 1,050 grams of $Al(NO_3)_3 .9H_2O$ in enough water to make 850 milliliters of solution, then adding 130 milliliters of concentrated ammonium hydroxide, and diluting to 1 liter with water.

Tri-n-butyl phosphate: 5-percent solution by volume in iso-octane (2,2,4-trimethylpentane).

Chromogenic reagent: 0.2 gram of dibenzoylmethane, (1, 3-diphenyl-1, 3-propanedione) and 5 grams of triethanolamine (2,2',2"-trihydroxy-triethylamine) per liter in absolute ethanol.

Standard uranium solution: 1.00 gram of U_3O_8 per liter, prepared by dissolving NBS Standard No. 950a U_3O_8 in 50 milliliters of concentrated nitric acid and diluting to 1 liter. From this solution, a standard containing 20 micrograms of U_3O_8 per milliliter is prepared by dilution with 5 percent HNO_3.

PROCEDURE

Sample Preparation

This method may be applied to a wide variety of materials by varying the sample preparation according to certain criteria.

1. All uranium must exist in the final solution in the +6 state.
2. Organic materials interfere and must be destroyed.
3. Carbonate and bicarbonate must be destroyed.
4. Ores must be completely dissolved to insure that all uranium is released.
5. Acidity of final solutions should be held below the equivalent of 40 volume-percent HNO_3.

Typical procedures for preparing a number of different samples follow.

Ores and Leach Residues

Weigh 0.5 to 2 grams of sample into a 500-milliliter Erlenmeyer flask. Add 10 to 15 milliliters of HCl, 5 to 10 milliliters of HNO_3, and about 5 milliliters of $HClO_4$. Evaporate to small volume, cool somewhat, and add 1

to 5 milliliters of HF, depending on sample size or amount of residue remaining. Heat gently to soft dryness to drive off SiF_4 and excess HF. Cool and add 10 to 15 milliliters of H_2O and 5 to 10 milliliters of HNO_3. Evaporate to soft dryness, cool, and repeat H_2O-HNO_3 evaporation. Add a few milliliters of H_2O and 4 to 10 milliliters of HNO_3, and heat gently for a few minutes. Transfer to a volumetric flask (25 to 250 milliliters), cool, and dilute to mark. Allow any insoluble residue to settle before proceeding with extraction and color development.

Organic-Bearing Ores

Weigh 0.5 to 2 grams of sample into a 500-milliliter Erlenmeyer flask and add 10 to 15 milliliters of HNO_3 and 5 to 10 milliliters of $HClO_4$. Heat at heavy fumes of $HClO_4$ until volume is small. If solution is still dark, cool slightly, add about 10 milliliters of HNO_3 and 5 milliliters of $HClO_4$, and repeat evaporation. Repeat these steps until solution is light-colored after evaporation to about 5 milliliters. (CAUTION: Do not allow evaporation to proceed to dryness if organics are still present, as evidenced by dark solution. An explosion may result.) Cool, add 10 to 15 milliliters of HCl, evaporate to small volume, and continue with HF treatment as described for ores and leach residues.

Acid Solutions

Pipet 1 to 5 milliliters of sample into a 250-milliliter beaker and add 10 to 15 milliliters of HNO_3 and 10 milliliters of $HClO_4$. Evaporate carefully to near dryness. Let cool somewhat, add 10 to 15 milliliters of HNO_3, and evaporate to 5 milliliters or less. Do not take to dryness. Add 10 to 15 milliliters of H_2O, boil to dissolve any solids, transfer to a volumetric flask (25 to 250 milliliters), cool, and dilute to mark.

Carbonate-Bicarbonate Solutions

Pipet 1 to 5 milliliters of sample into a 250-milliliter beaker and carefully add 2 to 3 milliliters of HNO_3. If the sample is exceedingly high in carbonates or if CO_2 is released too vigorously, some H_2O should be added before the HNO_3. When gas evolution is complete, add 10 milliliters of HNO_3 and 10 milliliters of $HClO_4$. Proceed as for acid solutions.

Organic Solutions

Pipet 1 to 5 milliliters of sample into a 250-milliliter beaker. Highly viscous liquids should be sampled by weight. Carefully evaporate to dryness, then continue heating at increased temperatures until fumes are no longer evolved. Cool, add 10 to 15 milliliters of HNO_3 and 5 to 10 milliliters of $HClO_4$, and continue as described for organic-bearing ores.

Extraction and Color Development

Pipet a 1- to 5-milliliter aliquot of the clear solution into a clean vial. If less than 5 milliliters are taken, add H_2O to make the volume approximately 5 milliliters. Add 1 drop of indicator and 2.0 milliliters of EDTA.

If the color is pink and the aliquot contains 2 milliliters or less of HNO_3, proceed with the addition of aluminum nitrate. If there is more than 2 milliliters of acid in the aliquot, add NH_4OH until the orange transition point of the indicator is reached. If this point is exceeded, add a drop or so of HNO_3 to return to the orange stage. If the initial color is yellow, add HNO_3 until the color is orange. In any case, it does not matter if the orange point is exceeded slightly on the pink or acid side. Add 10.0 milliliters of aluminum nitrate salting agent, mix, and add 5.0 milliliters of TBP. Close the vial with a new cap and shake for 30 seconds. Pipet a 2.0-milliliter aliquot of the organic extract into a second, dry vial. Take care not to touch the pipet to the aqueous phase when drawing the aliquot because very small amounts cause serious error in development of the colored complex. Add 15.0 milliliters of dibenzoylmethane reagent, mix thoroughly, and measure the absorbance at 410 nanometers in a 5-centimeter cell with a reagent blank in the reference cell. Measure a standard with each set of samples. Calculate the amount of U_3O_8 in the aliquot according to standard spectrophotometric practice.

Precision and Accuracy

The procedure has been used for analysis of uranium ores submitted monthly under the Analytical Comparison Program (ACP) administered by the U. S. Department of Energy laboratories at Grand Junction, Colo. Analytical results on some of these samples are shown in table 1. For comparison, the average value of all results reported by all participating laboratories are included. Excellent precision is indicated for this method. The average of the relative standard deviations given in the table is 2.1 percent. Agreement between Bureau of Mines' values and the ACP data was better than 2 percent relative for all samples. Further indication of accuracy of the method is given in table 2, where results on several different sample types are compared with plasma spectrometric and delayed neutron activation analysis data.

TABLE 1. - Comparison of results on ore samples

Sample	Number of determinations	Bureau of Mines		Analytical Comparison Program[1]	
		U_3O_8,[2] percent	RSD,[3] percent	U_3O_8,[2] percent	RSD,[3] percent
1.....	6	0.124±0.0027	2.2	0.127±0.006	4.7
2.....	18	.361± .0078	2.2	.363± .010	2.8
3.....	7	.359± .0076	2.2	.361± .009	2.5
4.....	9	.452± .0090	2.0	.457± .013	2.8
5.....	5	.175± .0042	2.4	.175± .008	4.6
6.....	9	.396± .0048	1.2	.402± .016	4.0
7.....	5	.167± .0048	2.8	.167± .008	4.8

[1]Average results obtained by 15 laboratories using a variety of methods.
[2]Includes standard deviation at 95-percent confidence limit.
[3]Relative standard deviation at 95-percent confidence limit.

TABLE 2. - Comparison of results on miscellaneous samples

Sample	Sample type	U_3O_8, grams per liter	
		Dibenzoylmethane	ICAP[1]
1.....	Ion-exchange eluates	0.29	0.31
2.....	.....do.....	1.74	1.73
3.....	.....do.....	4.93	4.88
4.....	.....do.....	8.90	8.75
5.....	.....do.....	15.6	15.8
		U_3O_8, percent	
6.....	Ion-exchange resins.	2.65	2.66
7.....	.....do.....	4.82	4.94
8.....	.....do.....	10.7	10.5
9.....	.....do.....	11.7	11.9
10.....	.....do.....	2.79	2.76
		DBM	Delayed neutron activation
11.....	Leach residues.....	0.0010	0.0009
12.....	.....do.....	.0008	.0008
13.....	.....do.....	.0004	.0004

[1]Inductively coupled argon plasma spectrometer.

EXPERIMENTAL RESULTS AND DISCUSSION

Interferences

Consistently inaccurate material balances obtained by metallurgists on leaching tests of certain ores were traced to unusually high iron concentrations in the ores. Some of the iron was extracting into the TBP, and on addition of the DBM solution, an orange rather than yellow color formed. This type of interference has been suppressed by a variety of masking agents, such as fluoride (5, 14-15), acetate (5, 15), sulfite (5), bisulfate (15), tartrate (3), nitrilo-triacetic acid (1), (1,2-cyclohexylene-dinitrilo) tetraacetic acid (4), EDTA (3, 6, 12), and various forms of EDTA including calcium EDTA (13), disodium EDTA (11), and tetrasodium EDTA (15). Tetra-sodium EDTA, a complexing agent in common use in this laboratory, was tested qualitatively and was effective in preventing the orange color from forming. The effect was further demonstrated by analyzing aliquots of aqueous standards containing 100 micrograms U_3O_8 and various amounts of iron. Solutions containing as much as 50 milligrams of iron did not exhibit any significant positive error when 400 milligrams of tetrasodium EDTA was added prior to TBP extraction. When 75 milligrams of iron was present, a positive relative error of about 3 percent was observed. If a 1-gram sample were to be dissolved and diluted to 10 milliliters, and a 5-milliliter aliquot were to be taken--conditions chosen to produce the lowest detection limit for uranium--the sample would have to contain 15 percent iron to produce 75 milligrams of iron in the aliquot. Ore samples containing this much iron have not been encountered in this laboratory, and since incorporating EDTA into the procedure, iron has not been a problem.

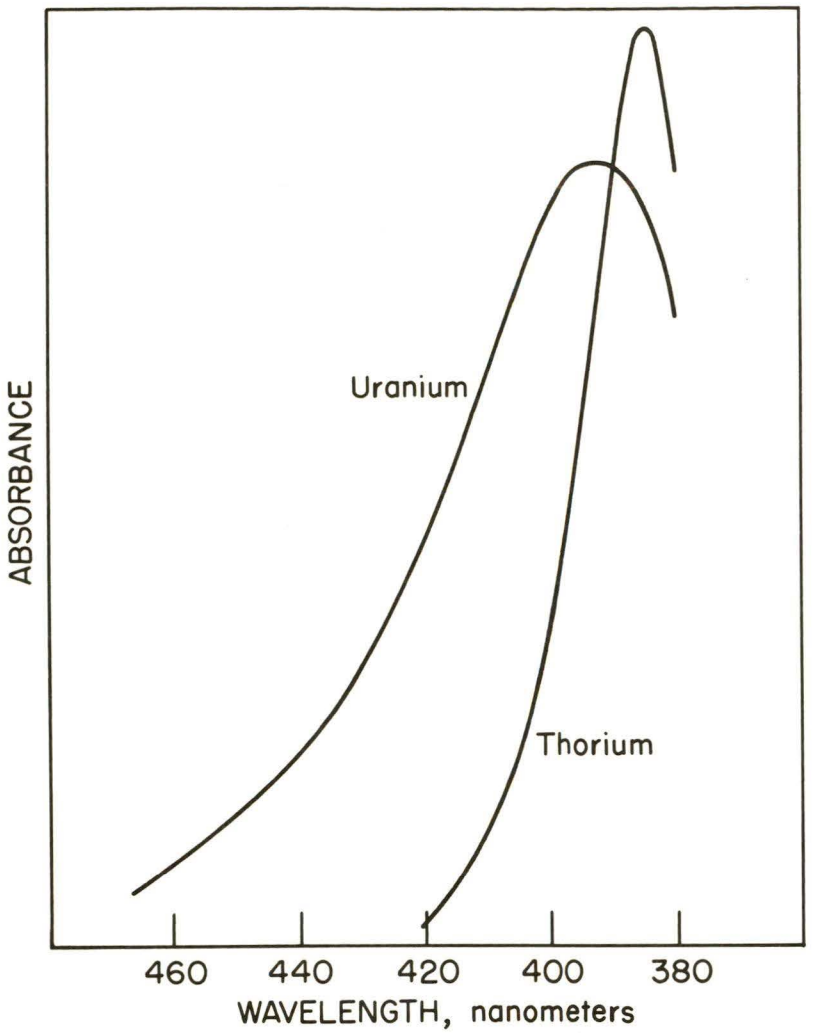

FIGURE 1. - Partial absorption spectra of uranium and thorium complexes with DBM.

Thorium also interfered, absorbing strongly with DBM at a wavelength just below that of uranium (fig. 1). By adding varying amounts of thorium to aqueous standards containing 100 micrograms of U_3O_8 and analyzing the standards with and without EDTA, the effect of the EDTA on the interference from thorium was determined. Typical data are shown in table 3 for solutions containing 0, 100, and 1,000 micrograms of thorium. As much as 1,000 micrograms of thorium can be tolerated if relative errors of 7 percent are permissible. However, the absorbance may be measured on the high-wavelength shoulder of the uranium-DBM peak, further reducing the contribution of the thorium-DBM absorbance. The values for U_3O_8 in table 3 were derived from measurements at 410 nanometers. The same solutions were measured at 420 nanometers, and in all cases, the magnitude of the interference was significantly reduced. At this wavelength with EDTA, there was no detectable interference from 1,000 micrograms of thorium, as shown by table 4. To produce 1,000 micrograms in a 5-milliliter aliquot, a sample prepared for lowest detection limit as described above in the discussion of iron interference would have to contain 0.2 percent thorium. This level of thorium is not rare, and other means must be used for the determination of uranium in such samples.

TABLE 3. - Effect of EDTA on thorium interference

Thorium added, micrograms	U_3O_8, relative percent found[1]	
	No EDTA	400 milligrams EDTA
0.....	99	98
100.....	117	101
1,000.....	207	107

[1] All solutions contained 100 micrograms U_3O_8.

TABLE 4. - Thorium interference at different wavelengths

Thorium added, micrograms	U_3O_8, relative percent found[1]	
	410 nanometers	420 nanometers
0.....	98	101
100.....	101	99
1,000.....	107	101

[1]All solutions contained 100 micrograms U_3O_8 and 400 milligrams EDTA.

Buffering of the Chromogenic Reagent

Because the colored uranium-DBM complex forms optimally within a limited pH range, acid which extracts into the TBP phase must be controlled. This range is generally agreed to be between pH 5 and 9, with some variation for different media. Early workers (2, 10, 17) using an alcohol-water medium adjusted the pH with dilute acid or base to the desired "apparent pH" using a pH meter. Francois (5) simplified the pH adjustment by adding pyridine as buffer to the chromogenic reagent. Other buffers have been used, including tris (hydroxymethyl) aminomethane (8) and 2,6-dimethyl-pyridine (15). Ethanolamine, diethanolamine, benzylamine, di-n-butylamine, alcoholic KOH (7), and lithium and sodium acetates (15), have all been tested as substitutes for pyridine, but none was effective.

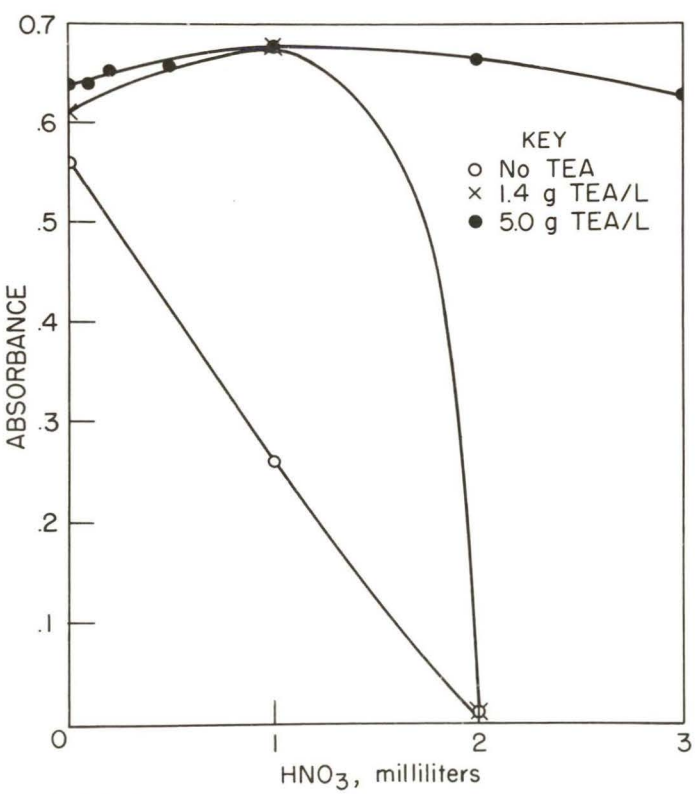

FIGURE 2. - Effect of HNO_3 on absorbance with varying amounts of TEA.

The odorless organic base, triethanolamine (TEA), was tested for use as buffer. Chromogenic reagent solutions were prepared containing various amounts of TEA. These were reacted with TBP extracts obtained from aqueous uranium solutions to which varying amounts of HNO_3 had been added. EDTA was present but pH adjustment was not made. Absorbances were measured and plotted against amount of HNO_3 added to the aqueous solution. Typical behavior is shown in figure 2. The need for acidity control during the color-forming step is domonstrated by the rapid decrease of absorbance with increasing acid content when no

TEA is present, or when the amount of TEA is insufficient, as in the case for a TEA concentration of 1.4 grams per liter when 2 milliliters HNO_3 is present. A TEA concentration of 5 grams per liter was effective in controlling all levels of unneutralized acid studied; the absorbance value of 3 milliliters HNO_3 differed from the average by only 3.2 percent.

These data suggest that aqueous aliquots containing as much as 3 milliliters HNO_3--60 to 100 percent by volume depending on aliquot size--may be analyzed with no pH adjustment beyond that afforded by the EDTA and 5 grams TEA per liter. To test this concept, real ore samples were prepared according to the procedures described above. Three additional solutions were prepared for each sample, each solution containing a different amount of HNO_3. These four solutions, containing 0, 30, 60, and 100 volume-percent of HNO_3, were then analyzed in triplicate with and without pH adjustment. For analyses with pH adjustment, aliquots showing yellow after addition of EDTA were adjusted back to orange with HNO_3, while NH_4OH was used to bring highly acid solutions from pink to orange. Results of these tests are given in table 5.

TABLE 5. - Effect of unneutralized HNO_3 on absorbance

Sample	Excess HNO_3 in aliquot, milliliters	Acidity adjusted				Acidity not adjusted			
		Absorbance[1]	Mean	Deviation	Relative deviation	Absorbance[1]	Mean	Deviation	Relative deviation
1	0	0.333		-.004	-1.2	0.340		0.010	3.0
	1.5	.336	0.337	-.001	.30	.337	0.330	.007	2.1
	3.0	.335		-.002	.59	.312		-.018	-5.5
	5.0	.343		.006	1.8	[2] .278		-	-
2	0	.289		-.006	-2.03	.286		-.006	-2.1
	.9	.291	.295	-.004	-1.4	.296	.292	.004	1.4
	1.8	.297		.002	.69	.298		.006	2.1
	3.0	.302		.007	2.4	.289		-.003	1.0

[1] Each value is an average of three absorbances.
[2] Precipitate formed on addition of EDTA. Value was not included in calculation of mean.

As expected, there was little effect on absorbance when acidity was controlled. Relative deviations from the means were within ±2.4 percent, the same order of magnitude as measured precision. When the acidity was not adjusted, absorbances deviated from the mean by as much as -5.5 percent, as shown for 3 milliliters of excess HNO_3 in sample 1. On this basis and despite the fact that the absorbance value for sample 2 with 3 milliliters of HNO_3 was different from the mean by only 1 percent, TEA at 5 grams per liter was not totally adequate in controlling the effects of 3 milliliters of excess HNO_3. However, if the data from acid levels of 3 milliliters are excluded, there is no significant difference between the two sets of data. Reorganizing the data according to increasing acid strength, without regard to sample, allows for easy comparison of mean values. Table 6 summarizes data from table 5 in this fashion, including deviations. It is obvious from these data that up to almost 2 milliliters of unneutralized acid in the aliquots have caused no significant difference in the absorbance readings. It is, therefore, safe to conclude that the pH

adjustment for excess acid (indicator remains pink upon addition of EDTA) may be omitted when the aliquot is known to contain 2 milliliters or less of HNO_3.

TABLE 6. - Effect of acidity adjustment on absorbance

Excess HNO_3 in aliquot, milliliters	Absorbance		Deviation	
	Acidity adjusted	Acidity not adjusted	Absolute	Relative
0	0.289	0.286	-.003	-1.0
0	.333	.340	.007	2.1
.9	.291	.296	.005	1.7
1.5	.336	.337	-.001	-.30
1.8	.297	.298	.001	.34
3	.302	.289	-.013	-4.3
3	.335	.312	-.023	-6.7
5	.343	[1] .278	-.065	19

[1] Precipitates form on addition of EDTA.

Because the effect of too high a pH in the aliquot was not studied, it is recommended in cases where the indicator turns yellow that a few drops of HNO_3 be added to adjust the pH to the orange stage.

The TEA-buffered chromogenic reagent has exhibited no degradation for at least 30 days. Using this reagent, the color of the uranyl-DBM complex forms immediately and is stable for at least 24 hours.

CONCLUSIONS

A much-improved and simplified method has been developed for the spectrophotometric determination of uranium in a variety of metallurgical materials. Use of tetrasodium salt of EDTA effectively controls interference from iron and reduces the interference from thorium. Triethanolamine replaces pyridine as buffer in the dibenzoylmethane solution and is so effective in controlling acid, which extracts into the TBP, that the neutralization of excess acid prior to TBP extraction is unnecessary for aliquots containing as much as 2 milliliters of HNO_3. By eliminating the need for acidity adjustment, the precipitation of salts during addition of NH_4OH is avoided. For occasional samples that may require addition of NH_4OH, such precipitation is prevented by the presence of EDTA. Chromogenic reagent solutions prepared with TEA are stable, and the instability and odor of the pyridine-buffered solution are no longer problems. The method is capable of good precision, the RSD being ±2.2 percent (95-percent confidence limit) when the aliquot contains at least 35 micrograms of U_3O_8. Lower analysis limit of the method is about 5 micrograms of U_3O_8 in the aqueous aliquot, although good agreement with other methods has been obtained at 2.5 micrograms of U_3O_8.

REFERENCES

1. Adam, J., and M. Stulikova. Selective Extraction and Determination of Uranium Using Diphenylacetic Acid. Collect. Czech. Chem. Commun. v. 39, No. 9, 1974, pp. 2576-2580.

2. Adams, J. A. S., and W. J. Maeck. Fluorometric and Colorimetric Microdetermination of Uranium in Rocks and Minerals. Anal. Chem., v. 26, 1954, pp. 1635-1639.

3. Blanquet, P. Dosage Colorimetrique de 1'Uranium par le Dibenzoylmethane (Colorimetric Determination of Uranium with Dibenzoylmethane). Anal. Chim. Acta, v. 16, 1957, pp. 44-56 (in French).

4. Budesinsky, B. W. Selective Spectrophotometric Determination of Uranium. Microchem. J., v. 22, No. 1, 1977, pp. 50-54.

5. Francois, C. A. Rapid Spectrophotometric Determination of Submilligram Quantities of Uranium. Anal. Chem., v. 30, No. 1, 1958, pp. 50-54.

6. Fremeaux and Cattin. Adaptation of Uranium Colorimetric Titration by the Dibenzoylmethane Method Using the Technicon Auto-Analyzer. Chim. Anal. (Paris), v. 50, No. 1, 1968, pp. 34-39.

7. Horton, C. A., and J. C. White. Separation of Uranium by Solvent Extraction with Tri-n-octylphosphine Oxide. Anal. Chem., v. 30, No. 11, 1958, pp. 1779-1784.

8. Long, J. L., and L. F. Grill. A Spectrophotometric Method for Uranium Using Tributyl Phosphate and Dibenzoylmethane. U.S. Atomic Energy Commission, RFP-78, 1957, 20 pp.

9. Pribil, R., and M. Jelinek. Colorimetric Determination of Uranium with Dibenzoylmethane. Chem. Listy, v. 47, 1953, pp. 512-515.

10. Seim, H. J., R. J. Morris, and R. G. Pastorino. Application of Ion Exchange to the Colorimetric Determination of Trace Amounts of Uranium Using Dibenzoylmethane. Anal. Chem., v. 31, No. 5, 1959, pp. 957-958.

11. Shigematsu, T. and M. Tabushi. Separation and Spectrophotometric Determination of Uranium with Dibenzoylmethane. Nippon Kagaku Zasshi, v. 81, 1960, pp. 265-268.

12. Shigematsu, T., M. Tabushi, and T. Tarumoto. Dibenzoylmethane as a Chelating Reagent in Solvent Extraction and Spectrophotometric Determination. Bull. Inst. Chem. Res. Kyoto Univ., v. 40, No. 5/6, 1962, pp. 388-399.

13. Shigematsu, T., M. Tabushi, M. Matsuii, and M. Munakata. Coprecipitation and Spectrophotometric Determination of Uranium as Dibenzoylmethane Chelate. Bull. Chem. Soc. Japan, v. 41, No. 7, 1968, pp. 1610-1614

14. Umezaki, Y. The Spectrophotometric Determination of Uranium With Dibenzoylmethane. Bull. Chem. Soc. Japan, v. 36, No. 7, 1963, pp. 769-773.

15. Wise, W. M. Spectrophotometric Determination of Submilligram Quantities of Uranium With Dibenzoylmethane. U.S. Atomic Energy Commission, MCW-1425, No. 12, 1959, pp. 133-139.

16. Western Nuclear, Inc. Colorimetric or Spectrophotometric Uranium Determination. Laboratory Procedures Manual. March 1963, pp. 82-83.

17. Yoe, J. H., F. Will III, and R. A. Black. Colorimetric Determination of Uranium With Dibenzoylmethane. Anal. Chem., v. 25, 1953, pp. 1200-1204.

☆U.S. GOVERNMENT PRINTING OFFICE: 1980-603-102/32

INT.-BU.OF MINES,PGH.,PA. 24481