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AN ION EXCHANGE-PHOTOMETRIC METHOD
FOR THE DETERMINATION OF URANIUM IN STEEL

TECHNICAL REPORT WAL TR 823/3

BY

THOMAS A. FERRARO

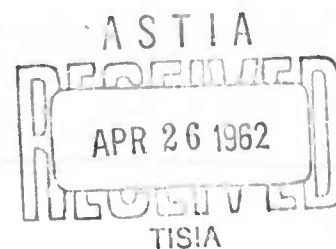
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WALTER F. CZYRKLI

DATE OF ISSUE - MARCH 1962

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RESEARCH OF MATERIALS FOR HIGH TEMPERATURE USE
D/A PROJECT 593-32-008

WATERTOWN ARSENAL
WATERTOWN 72, MASS.



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Chemistry, analytical
Ion exchange - photometric
Uranium - steel

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FOR THE DETERMINATION OF URANIUM IN STEEL

ABSTRACT

An ion-exchange separation scheme was developed for the determination of uranium in steel. Anion exchange from a nitric acid solution is employed. Uranium is determined by the basic peroxide photometric method after its separation. A detailed procedure and data supporting the method are given.

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INTRODUCTION

The determination of uranium generally requires the absence or prior separation of many elements.¹ This is true whether fluorometric, gravimetric, volumetric, photometric, polarographic or other techniques are employed for its determination. The separation of uranium prior to its determination has been accomplished by precipitation,²⁻⁷ electrolytic,⁸⁻¹⁰ solvent extraction,¹¹⁻¹⁷ and ion-exchange^{10,18-26} techniques. Separation by precipitation of uranium may lead to some loss of uranium as well as to incomplete removal of uranium from some elements. Mercury cathode electrolysis does not separate vanadium, aluminum, titanium, etc., and the separation of several other elements is not complete.

Solvent extraction of uranyl nitrate with ethyl ether, methyl isobutyl ketone, or ethyl acetate has been commonly employed for the separation of uranium. Continuous extraction is required with ethyl ether. The procedure is involved as well as potentially hazardous.⁸ Extraction with methyl isobutyl ketone or ether results in incomplete separation of uranium from iron when iron is present in large amounts. Uranium is usually determined photometrically with thiocyanate following this extraction technique.^{13,16} The thiocyanate method can tolerate a small amount of iron if it is reduced to the bivalent state²⁷ whereas other commonly employed photometric methods require its complete removal.^{1,8} The uranium thiocyanate color is not very stable unless developed in the presence of acetone or butyl Cellosolve. Careful control of thiocyanate concentration and acidity is required.¹⁷ Furthermore, vanadium, which is also extracted to some extent, interferes with this determination.

Extraction of uranyl nitrate with ethyl acetate has been used extensively^{11,12,14,17} and offers a cleaner separation from elements such as iron, vanadium; etc. Following this separation, the uranium can be determined photometrically with peroxide or dibenzoylmethane, fluorometrically, or by any other convenient method. A disadvantage of this extraction is that quantitative separation of uranium requires a concentration of 10 grams of hydrated aluminum nitrate per 10 milliliters of sample solution. Crystallization may occur and heating of the separatory funnel in hot water is necessary to dissolve the salts. Variations in the amount of aluminum nitrate added or crystallization can lead to erroneous results.⁸

Another commonly employed technique for the separation of uranium is extraction of cupferrates away from hexavalent uranium.¹⁸⁻¹⁹ The uranium in the aqueous phase must then be separated from those elements not extracted. This is usually accomplished by reduction of uranium to the quadrivalent state and extraction of the uranium cupferrate which is then formed.^{1,8,9} Two such extractions are required at this point to remove the uranium completely from the aqueous phase. When ferric iron is present in appreciable amounts, it is desirable to remove it by ether or ethyl acetate extraction from dilute hydrochloric acid before applying cupferron separations.⁸

The use of ion exchange in the isolation of uranium is now well established. According to Vita et al,²⁵ when the proportion of other elements such as iron, nickel, copper, or aluminum is high compared to uranium, ion exchange offers the best method for the quantitative separation of uranium.

Anion exchange has found wider application than cation exchange^{18,26} for uranium separation. Anion exchange methods are based on the adsorption of anionic complexes formed by uranium with carbonate,²⁰ sulfate,^{10,21,22} nitrate,^{23,25} and chloride and fluoride anions.¹⁹ The anionic complex formed with ascorbic acid has also been employed.²⁴

The anion exchange of uranyl carbonate is employed chiefly for the separation of uranium from amphoteric elements where hydrolytic precipitation would not be encountered. Uranyl sulfate adsorption is convenient for separation from those elements which do not form anionic sulfate complexes. Iron, however, does form such a complex, which, though not adsorbed as strongly as uranium, is incompletely separated by this method.²⁸ Welford and Sutton,¹⁰ in applying this technique to the analysis of steel, found it necessary to remove residual iron by electrolysis or solvent extraction after the ion-exchange separation.

Anion exchange employing either hydrochloric-hydrofluoric acid, ascorbic acid, or a nitrate medium offers a suitable means for the separation of uranium from iron and many other elements. Although ion-exchange separations are time-consuming, the demands on the analyst's time and attention are relatively small and are comparable in many cases to solvent extraction techniques. Furthermore, ion exchange offers the economical advantage resulting from the fact that ion-exchange resins are re-usable whereas in solvent extraction the solvents employed are often expensive and not conveniently recoverable even after a single extraction.

A separation scheme isolating uranium from as many elements as possible would be useful in providing a method of general applicability for the determination of uranium. Anion exchange from a nitrate medium offered the best promise in this direction.

Ockenden and Foreman²³ proposed a scheme wherein uranium is adsorbed by a strong-base anion-exchange resin from concentrated aluminum nitrate solution. Iron and other elements are eluted with concentrated aluminum nitrate solution and aluminum is next removed with concentrated hydrochloric acid. Uranium is then eluted with dilute hydrochloric acid and determined photometrically with peroxide (milligram amounts) or thoron (microgram amounts).

Vita et al²⁵ found that concentrated hydrochloric acid caused decomposition of some resin when added to the nitrated columns and also led to incomplete separation of iron due to the formation of a strongly adsorbed chloride complex. Since concentrated hydrochloric acid was used by Ockenden and Foreman to remove aluminum, Vita et al modified the procedure

by substituting a saturated sodium nitrate solution for the aluminum nitrate after the sorption step, thereby eliminating the need for concentrated hydrochloric acid.

It has been shown that vanadium, molybdenum, and chromium cause erroneous results in the application of this method.²⁵ Vanadium and molybdenum, when present as vanadate and molybdate, respectively, in 50-mg amounts, caused a slight loss of uranium. The same amount of chromium as chromate interfered seriously with the photometric peroxide determination, causing very high results. The loss of uranium was caused by competition for resin sites between these anions and the uranyl nitrate complex. This resulted in displacement of some uranium. It is likely that increased column length would have avoided such a loss. The adsorption of chromate was shown to be eliminated by reduction with hydrogen peroxide prior to ion-exchange treatment.²⁶

The use of concentrated aluminum nitrate was proposed by Ockenden and Foreman²³ in order to increase the concentration of undissociated uranyl nitrate and thereby enhance its adsorption. These authors felt that this was necessary since they had observed a distribution coefficient of about 10 for the adsorption of uranium from straight nitric acid (8M). (The distribution coefficient of an element is a measure of the ratio of its concentration in the ion-exchange resin to its concentration in the solution with which it is equilibrated.) Ockenden and Foreman felt that a distribution coefficient of 10 was insufficient to effect a clean separation of uranium by a normal column method. It is generally known, however, that species with a distribution coefficient of 10 or over between a given medium and ion-exchange resin do not begin to appear in a column effluent until several column volumes of the same medium have been passed through the ion-exchange resin. Species only slightly or not adsorbed, on the other hand, are often completely removed within two column volumes.

In view of this and the fact that Buchanan and Faris²⁹ and Kraus and Nelson³⁰ reported a distribution coefficient of between 10 and 20 for uranium in 8M nitric acid, thus confirming the value found by Ockenden and Foreman, it appears that column separation of uranium from nonadsorbed and slightly adsorbed elements is indeed feasible in nitric acid.

Although a distribution coefficient of over 2000 was found for uranium from an 80 percent saturated solution of aluminum nitrate (0.5M in nitric acid),²⁶ it is to be expected that some other elements will also show an increase in adsorption from such a medium compared to their behavior in straight nitric acid. This would account for the adsorption of chromium, molybdenum, and vanadium from aluminum nitrate solution as reported by Vita et al. The adsorption of about 60 elements from nitric acid solutions and about 35 elements from ammonium nitrate solutions has been studied by Buchanan and Faris.²⁹ It was found that for some elements adsorption from ammonium nitrate solutions showed substantial increase over that found from a nitric acid medium. A distribution coefficient of

10 or greater was found for molybdenum at all concentrations of ammonium nitrate of low acidity whereas only slight adsorption was found from nitric acid solutions. In the presence of aluminum nitrate rather than ammonium nitrate the increase in adsorbance is generally much greater.

All of the elements likely to be present in steels show negligible or no adsorption from nitric acid solutions. Chromium and vanadium, for example, are not adsorbed. In fact, of the elements studied by Buchanan and Faris, only palladium, gold, bismuth, thorium, protoactinium, neptunium, and plutonium show a distribution coefficient of 10 or greater from 8M nitric acid. Thus, the separation of uranium from many elements by anion exchange from a nitric acid medium not only appears feasible but offers considerable improvement over existing methods.

In this investigation this technique was employed to develop an analytical procedure for the determination of alloying amounts of uranium in steel. Uranium was determined photometrically by the alkaline peroxide method following ion-exchange separation.

EXPERIMENTAL WORK

Preliminary Ion-Exchange Work

An ion-exchange column was prepared from a polystyrene tube 12 inches long and 5/8-inch inside diameter. The bottom of the column was closed with a rubber stopper with a 3/16-inch hole in which was inserted a 3-inch length of rigid polyethylene tubing of 3/16-inch outside diameter and 1/16-inch bore. To this was attached a short length of Tygon-R tubing and a hosecock which controls the flow through the column.

A layer of acid-resistant vinyl-chloride plastic wool was placed on the bottom of the column. A suspension of Dowex-1, strong-base anion-exchange resin, 180 to 270 mesh, 8- to 10-percent cross linkage, in dilute hydrochloric acid was added to the column until a settled column of resin 3-1/2 inches high was obtained. The resin bed volume was estimated to be about 16 cc.

A solution was prepared containing 2 grams of a low alloy steel (SAE 4340, which contains nominally 0.4% C, 0.7% Mn, 0.3% Si, 1.8% Ni, 0.8% Cr, and 0.2% Mo) and 37.3 mg of pure uranium metal in 22 ml of 8M HNO₃. The column was conditioned with about 25 ml of 8M HNO₃ and the sample transferred to the column. After washing the sample container and column above the resin with 8M HNO₃, and allowing the washings to drain, 30 to 35 ml of 8M HNO₃ was passed through the resin. A spot test with thiocyanate indicated that no iron was present in the effluent at this point.

The effluent was discarded and the receiving beaker was replaced with a clean one. The column above the resin was washed three times with

1M HCl, allowing the solution to drain each time. Elution with 1M HCl was then conducted until about four column volumes (64 ml) were collected. The receiving beaker was replaced and elution with 1M HCl continued until an additional 50 ml of eluate was collected.

The two portions of the 1M HCl effluent were treated with 5 ml of H_2SO_4 and evaporated to heavy fumes of SO_3 . The solutions were cooled, diluted, and cooled again, transferred to 100-ml volumetric flasks, and diluted to volume. Aliquots of 20 ml were analyzed for uranium by the basic peroxide photometric method. The uranium content was ascertained by reference to a calibration curve prepared from a reference solution of pure uranium. Details on the procedure and materials used are given in the Appendix.

Absorbance measurements were taken 10 minutes, 90 minutes, and 24 hours after addition of peroxide in order to check the stability of the uranium-peroxy complex. Calibration and stability data are shown in Table I. A straight line calibration curve was obtained with this data and is shown in Figure 1. The color stability was found to be good through the 24-hour period studied.

TABLE I			
CALIBRATION AND STABILITY DATA			
Uranium mg	Absorbance		
	10 min after Addition of H_2O_2	90 min after Addition of H_2O_2	24 hr after Addition of H_2O_2
0.00	0.000	0.000	0.000
2.01	0.098	0.097	0.099
5.04	0.243	0.244	0.242
10.1	0.485	0.485	0.487
15.1	0.721	0.723	0.722
17.6	0.838	0.836	0.834
20.1	0.959	0.961	0.962

Approximately 83 percent of the uranium added was recovered in the first fraction obtained by elution with 1M HCl. No uranium was detected in the next elution fraction. Obviously, some of the uranium had been eluted by the 8M HNO_3 used to remove iron and other elements such as nickel, chromium, manganese, etc.

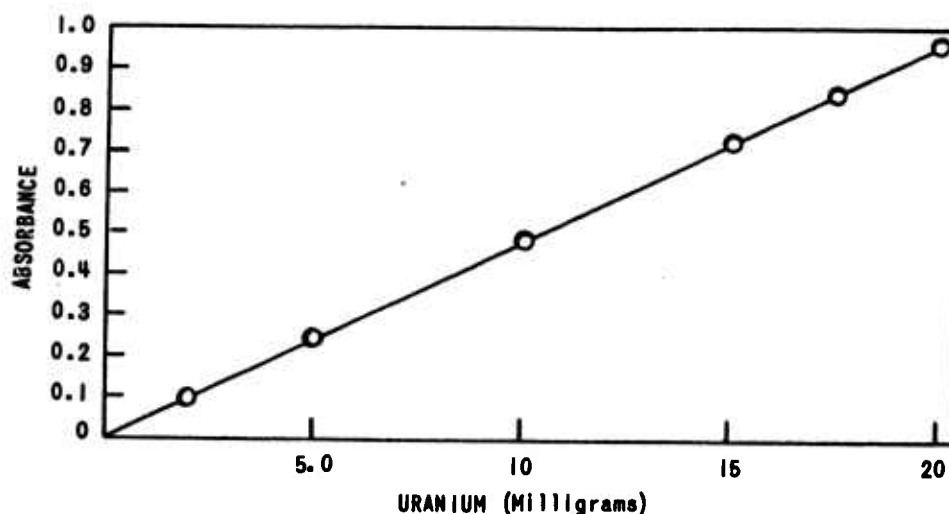


FIGURE 1
URANIUM WITH PEROXIDE
(400 mμ)

When the column technique of ion exchange is employed and overlapping of species to be separated occurs, lengthening of the resin bed is a simple and often successful expedient for eliminating cross contamination. Accordingly, the resin bed employed in the previous experiment was increased from 3-1/2 inches to 4-1/2 inches in height, giving a column volume of 20 ml. The experiment was repeated with an 8M HNO₃ solution containing 2 grams of 4340 steel and 47.6 mg of uranium after conditioning the column with 40 ml of 8M HNO₃. This time quantitative recovery of uranium was achieved.

Due to observed interaction between the nitric acid solution and the rubber stopper at the outlet end of the column, another column was prepared using a standard 50-ml buret of the Mohr type. Details on this are included in the procedure given in the Appendix. It should be noted here, however, that the inside diameter of the new column was smaller, i.e., 7/16 inch, so that, since the same amount of resin was used, the resin bed height was now approximately 8-1/2 inches, almost twice the original height. This enhances further the desired separation of uranium from iron and other elements much in the same manner as would result from increasing the bed height by adding more resin.

Preparation and Analysis of Synthetic Samples

In the absence of suitable standards, synthetic samples were prepared by dissolving 2 grams of 4340 steel and different amounts of pure

uranium metal in dilute HNO_3 (1:3). The choice as to type of steel employed in the preparation of these synthetic samples was based on advanced knowledge that samples of 4340 steel containing up to 6 percent uranium were soon to be received by the Analytical Chemistry Branch for analysis. The synthetic sample solutions were carried through the procedure described in the Appendix.

RESULTS AND DISCUSSION

The results obtained on the synthetic samples, as shown in Table II, are satisfactory. In every case the amount of uranium found is within one percent of the amount added. The method was next applied to the determination of uranium in actual samples of 4340 steel for which this analysis was requested. Some typical results covering the range from 0.02 to 6.0 percent are shown in Table III.

TABLE II ANALYSIS OF SYNTHETIC SAMPLES			
Uranium Added (mg)	Absorbance (1/4 Aliquot)	Uranium Found (mg)	Recovery (%)
9.5	0.115	9.56	100.6
14.8	0.179	14.9	100.7
24.4	0.294	24.5	100.4
40.2	0.482	40.1	99.8
40.7	0.487	40.5	99.5
47.6	0.569	47.2	99.2

TABLE III DETERMINATION OF URANIUM IN ACTUAL STEEL SAMPLES				
Sample	Sample Size (g)	Absorbance (1/4 Aliquot)	Nominal U Content (%)	U Found (%)
V1-O	2.00	0.006	0.00	0.02
V1-M	2.00	0.004	0.00	0.02
V2-O	2.00	0.220	1.00	0.92
V2-M	2.00	0.168	1.00	0.70
V3-O	2.00	0.967	4.00	4.02
V3-M	2.00	0.947	4.00	3.94
V10-O	1.036	0.739	6.00	5.94
V10-M	1.039	0.712	6.00	5.70

CONCLUSIONS

The ion exchange-photometric method presented here for the determination of uranium in steel has been shown to give quantitative recovery of uranium in the range covered. The method is straightforward and suitable for routine application since it lends itself readily to handling a number of samples through the simultaneous operation of several ion-exchange columns.

APPENDIX

PROCEDURE FOR THE DETERMINATION OF URANIUM IN STEEL

SCOPE

1. This method covers the determination of uranium in concentrations from 0.1 to 6.0 percent.

SUMMARY OF METHOD

2. The sample is dissolved in nitric acid and transferred to an anion-exchange resin column. Interfering elements are eluted with 8M nitric acid while the uranium remains in the column. Uranium is removed by elution with 1M hydrochloric acid. After fuming with sulfuric acid, a suitable portion of the eluate is made alkaline and the uranium complex formed with hydrogen peroxide. Photometric measurement is made at approximately 400 mμ.

CONCENTRATION RANGE

3. The recommended concentration range is from 1.0 to 20.0 mg in 100 ml of solution, using a cell path of 1 cm (Note 1).

Note 1 - This method has been written for cells having a 1-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

STABILITY OF COLOR

4. The color develops in 10 minutes and is stable for at least 24 hours.

INTERFERENCES

5. Provision has been made for the removal of all interfering elements.

APPARATUS

6. Ion-Exchange Column. - The column is constructed of a standard 50-ml buret of the Mohr type with a short length of polyvinyl tubing attached at the bottom. The flow is controlled by a hosecock on the polyvinyl tubing.

REAGENTS

7. (a) Hydrogen Peroxide (30 percent). - Concentrated hydrogen peroxide (H_2O_2).

(b) Ion-Exchange Resin. - Strongly basic anion-exchange resin*, 180 to 270 mesh, 8- to 10-percent divinylbenzene cross linkage. Prepare a suspension of the material in HCl (1:19), allow the coarser fraction to settle 10 to 15 minutes, and remove the fines by decantation. Repeat the process several times until most of the very fine material has been removed and discarded.

(c) Sodium Carbonate Solution (100 g per liter). - Dissolve 100 grams of sodium carbonate (Na_2CO_3) in 850 ml of water and dilute to 1 liter.

(d) Sodium Hydroxide Solution (300 g per liter). - Dissolve 300 grams of sodium hydroxide (NaOH) in 500 ml of water and dilute to 1 liter.

(e) Uranium, Standard Solution (1 ml = 1.000 mg U). - Dissolve 1.000 grams of pure uranium in 10 ml of HCl (1:1) and oxidize with HNO_3 dropwise. When dissolution is complete, add 30 ml of H_2SO_4 (1:1) and evaporate to dense white fumes. Cool, transfer to a 1-liter volumetric flask and dilute to volume.

PREPARATION OF ION-EXCHANGE COLUMN

8. Place a wad of glass wool at the bottom of the buret to support the resin. Add the resin, which is in the form of a slurry, in small portions to obtain a settled column 20 ml in volume. Place a wad of glass wool above the resin in order to prevent disturbance of the resin on the addition of solutions to the column.

PREPARATION OF CALIBRATION CURVE

9. (a) Calibration Solutions. - Transfer 1.0, 5.0, 10.0, 15.0, and 20.0 ml of the uranium solution (1 ml = 1.000 mg U) to 150-ml beakers. Adjust the solution to a pH of 5 to 6 with the NaOH solution, using dilute H_2SO_4 to adjust the acidity, if necessary. Add 10 ml of the Na_2CO_3 solution and mix, 25 ml of the NaOH solution and mix again. Cool and transfer the solutions to 100-ml volumetric flasks.

(b) Reference Solution. - Prepare a reference solution by transferring an equivalent amount of dilute H_2SO_4 to a 150-ml beaker and carrying it along with the calibration solutions.

(c) Color Development. - To each flask add 1 ml of H_2O_2 (30 percent), dilute to volume and mix. Allow to stand for 10 minutes.

(d) Photometry. - Transfer a suitable portion of the reference solution to an absorption cell with a 1-cm light path and adjust the photometer to the initial setting, using a light band centered at

* Dowex 1-X10 anion-exchange resin was used.

approximately 400 mμ. While maintaining this adjustment, take the photometric readings of the calibration solutions containing 1.0 to 20.0 mg of uranium.

(e) Calibration Curve. - Plot the photometric readings of the calibration solutions against milligrams of uranium per 100 ml of solution.

PROCEDURE

10. (a) Test Solutions.

(1) Transfer a sample containing no more than 60 mg of uranium to a 250-ml beaker. Add 22 ml of HNO_3 (1:3) for each gram of sample taken, and place on a hot plate until dissolution is complete. Evaporate solution to about 10 ml. Remove from heat, cool, and add 10 ml of HNO_3 .

(2) Condition the column by passing 50 ml of 8M HNO_3 through the resin.

(3) Transfer the sample solution to the column in 5-ml increments, allowing each portion to drain to the top of the resin bed before adding the next. Wash the beaker with three 3- to 4-ml portions of 8M HNO_3 , allowing the washings to pass through the resin. Finally wash the column above the resin three times with 3- to 4-ml portions of the same solution, allowing the solution to drain each time.

(4) Continue to elute with 8M HNO_3 until a negligible test for iron is obtained with NaSCN on a spot plate. Usually less than 30 ml will be required. Discard this fraction.

(5) Place a clean 250-ml beaker beneath the column. Wash the column above the resin three times with 3- to 4-ml portions of 1M HCl, allowing the solution to drain each time. Pass 80 ml of 1M HCl through the column and reserve the eluate for the determination of uranium.

(6) Add 10 ml of H_2SO_4 (1:1) to the uranium-bearing eluate and evaporate to dense white fumes. Cool, transfer to a 100-ml volumetric flask, cool again, and dilute to volume. Transfer a suitable aliquot containing from 1 to 20 mg of uranium to a 150-ml beaker and proceed, beginning with the adjustment of the pH in Section 9 (a).

(b) Reference Solution. - Prepare a reagent blank as described in Section 9 (b).

(c) Color Development. - Develop the color as described in Section 9 (c).

(d) Photometry. - Take the photometric reading of the test solution as described in Section 9 (d).

CALCULATION

11. Convert the photometric reading of the test solution to milligrams of uranium by means of the calibration curve. Calculate the percentage of uranium as follows:

$$\text{Uranium, percent} = \frac{A}{B \times 10}$$

where

A = milligrams of uranium found in 100 ml of final solution,

and

B = grams of sample represented in 100 ml of final solution.

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