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ION EXCHANGE-PHOTOMETRIC DETERMINATION OF TANTALUM IN VANADIUM ALLOYS

TECHNICAL REPORT WAL TR 823/2

BY

THOMAS A. FERRARO, JR.

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TITLE

ION EXCHANGE-PHOTOMETRIC DETERMINATION OF TANTALUM IN VANADIUM ALLOYS

ABSTRACT

A method was developed for the determination of tantalum in vanadium alloys. Tantalum is determined photometrically with pyrogallic acid following a separation by ion exchange. A detailed procedure is given.

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INTRODUCTION

A series of vanadium alloys containing tantalum in the range from 0.04 to 1 percent was submitted to the Analytical Chemistry Branch of Watertown Arsenal Laboratories for analysis. Since there was no information available for this determination, it was necessary to develop an analytical procedure for determining tantalum in vanadium.

Spectrographic determination of the tantalum was first considered, but the very low spectral sensitivity of tantalum made this approach impractical.

Photometric methods for the determination of tantalum employing pyrogallic acid,[1-10] hydrogen peroxide,[11] phenylfluorone,[12] catechol,[13] and methyl violet,[14] and gravimetric methods involving hydrolysis,[15,16] or precipitation with tannic acid,[17] N-benzoyl-N-phenylhydroxylamine,[18-20] phenylarsonic acid,[21] N-cinnamoyl-N-phenylhydroxylamine and cinnamylhydroxylamine acid[22] have been reported.

All of the gravimetric methods would require at least a double precipitation in order to separate tantalum completely from large amounts of vanadium and, in most cases, complete separation would be extremely difficult.[17-22] Moreover, ignition of the precipitate to the pentoxide would be required in each case due to its lack of definite composition or thermal stability. Thus the accurate determination of low amounts of tantalum would not be feasible because of the unfavorable gravimetric factor of the pentoxide, i.e., 0.819.

Of the photometric methods, only the phenylfluorone method[12] showed no interference due to vanadium when present in equal amounts with tantalum. However, when the ratio of vanadium to tantalum is greatly increased, interference due to vanadium is very likely and a preliminary separation of these elements would be necessary. Since the photometric measurements are made in a hydrofluoric-boric acid medium, etching of the photometer cells could occur.

Marzys[3] reported interference of vanadium in the pyrogallic acid method to result in 100 percent error when the weight ratio of vanadium pentoxide to tantalum was 100 to 1. Kidman, et al,[10] reported the interference of 0.1 percent vanadium in steel to be equivalent to 0.04 percent tantalum. Thus a quantitative separation of these two elements prior to photometric measurement is necessary.

A preliminary separation of the tantalum from the vanadium would also be required prior to the use of any of the other photometric methods. Of all the photometric methods, the pyrogallic acid method[1-10] appeared to be the most promising. The method had already been employed by the laboratory in the certification analysis of a National Bureau of Standards high-temperature alloy steel 19-9DL, spectrographic standard No. 1134. In this work, the method of Hague and Machlan[9] employing ion-exchange separations was applied.

Various means of separation were considered; a gravimetric procedure would require at least a double precipitation, with some loss of tantalum likely with each treatment.[16] Solvent extraction of tantalum with methyl isobutyl ketone[33-35] and with cyclohexanone[6] has been employed to separate tantalum from columbium, uranium, zirconium and several other elements. However, no mention is made of the behavior of vanadium. It was shown in this laboratory that vanadium is not extracted to any great extent by methyl isobutyl ketone under conditions similar to those reported,[33-35] thereby indicating that a separation of tantalum from vanadium could very likely be achieved by this method. However, these extractions are made from a hydrofluoric acid medium, and since the only commercially available separatory funnels are made from glass, this practice would not be practical. Furthermore, some loss of tantalum in the methyl isobutyl ketone extraction procedure has been reported.[35] Tributyl phosphate has also been employed for the extraction of tantalum from a hydrofluoric acid solution[36] but vanadium would also be extracted to a great extent under these conditions.[27]

Ion exchange has been employed to separate tantalum prior to its determination.[4,10,28,29] Separation from titanium alloys containing up to 1.5 percent vanadium has been accomplished by the ion-exchange procedure recommended by the Panel on Methods of Analysis of the Metallurgical Advisory Committee on Titanium[28] and has been accepted as a tentative method by the American Society for Testing Materials.[29]

Faris,[30] working with hydrofluoric acid solutions, showed the possibility of separating tantalum from vanadium by means of anion exchange. Kallmann, et al,[4] working with titanium alloys containing up to 5 percent vanadium, used a nitric-hydrofluoric acid mixture to separate all alloying elements, including columbium, from tantalum in a single step. This latter approach seemed to offer the best promise and was selected for the following reasons:

1. Vanadium-tantalum alloys are readily dissolved in mixtures of nitric and hydrofluoric acids.

2. The separation from columbium is desirable, especially at the 1 percent tantalum level, since columbium does interfere, although only slightly, in the pyrogallic acid photometric method.[10]

3. Tantalum is strongly adsorbed from the nitric-hydrofluoric acid mixture[4] whereas vanadium shows only slight adsorption from hydrofluoric acid solutions[30] and no adsorption from nitric acid solutions.[31] It is to be expected, therefore, that vanadium will be eluted along with columbium by the nitric-hydrofluoric acid mixture, while the tantalum remains on the resin.

EXPERIMENTAL WORK AND DISCUSSION

Photometric Method

Many variations in the pyrogallic acid photometric method for tantalum have appeared in the literature and various combinations of mineral acids, complexing agents, and pyrogallic acid concentrations have been employed. Different wavelengths for absorbance measurements have also been recommended.

In general the method described by Hague and Machlan[9] was employed for this work. These authors recommended that photometric measurements be made 10 minutes after the addition of the pyrogallic acid. Past experience in this laboratory with this method has shown that on standing over a long period of time a slow but definite increase in absorbance occurs. Theodore,[35] working with somewhat different conditions, found that a waiting period of 30 minutes was necessary for full color development. In view of these considerations, a study of absorbance readings versus time was made. An initial study was made employing a solution containing 1 mg of tantalum. A subsequent series of measurements involved two solutions which contained 0.1 mg and 1 mg of tantalum, respectively. The results are shown in Table I.

TABLE I

ABSORBANCE READINGS VERSUS TIME

Time after Addition of Pyrogallic Acid	Absorbance		
	1 mg Ta	0.1 mg Ta	1 mg Ta
5 min	-	0.183	0.279
10 min	0.287	0.188	0.283
20 min	-	0.193	0.288
30 min	0.297	0.193	0.291
45 min	-	0.197	0.293
65 min	0.303	0.203	0.299
80 min	0.306	0.205	0.302
3 hr	-	0.210	-
4 hr	-	-	0.310
5 hr	-	0.212	-
7 hr	-	-	0.310
20 hr	0.310	-	-

The color stability study of the tantalum-pyrogallic acid complex indicates a definite change in absorbance with time. However, this change is so slow that measurements made soon after the addition of the pyrogallic acid, if compared with standards run at the same time, should yield results which are comparable with those obtained by allowing the solution to stand overnight.

This was shown to be true by measuring the color of three standard tantalum solutions prepared with varying amounts of tantalum pentoxide (as described in the appendix) and two solutions of pure tantalum metal. The measurements were made 1/2 hour after adding the pyrogallic acid and again after letting them stand overnight. Results are shown in Table II.

TABLE II

EFFECT OF TIME ON TANTALUM RECOVERY

Tantalum Present (mg)	Absorbance		Factor $\left(\frac{\text{mg Ta}}{\text{absorbance}}\right)$		Tantalum Found (mg)	
Standard Solutions	1/2 hr*	24 hr*	1/2 hr*	24 hr*	1/2 hr*	24 hr*
0.253	0.071	0.074	3.56	3.42		
0.505	0.143	0.147	3.54	3.44		
1.010	0.286	0.291	3.53	3.47		
		average	3.54	3.44		
Tantalum Metal Solutions						
8.33	0.238	0.245			8.43	8.43
8.88	0.250	0.257			8.85	8.84

*Time elapsed after addition of pyrogallic acid solution.

Since it is more convenient to have a calibration curve which can be used over a period of time with a given lot of reagents, rather than to prepare a standard calibration curve with each set of runs, it was decided that all solutions, after development of the tantalum-pyrogallic acid color, should stand overnight prior to taking photometric measurements.

EVALUATION OF METHOD

Since suitable standards were not available, the preparation of synthetic standards was necessary in order to evaluate the proposed analytical procedure. These were prepared by dissolving weighed portions of vanadium and tantalum metals in a nitric-hydrofluoric acid mixture. These metals were the same as those used to prepare the alloys to be tested.

Before proceeding with the preparation and analysis of the synthetic standards, the tantalum metal, claimed to be at least 99.5 percent pure, was analyzed in order to ascertain its purity. Gravimetric and photometric methods were employed. The gravimetric method involved ion-exchange separation and subsequent precipitation with cupferron; the cupferrate was ignited to the pentoxide and weighed. The photometric method employing pyrogallic acid was applied directly and also indirectly following

precipitation with cupferron. Results are shown in Table III. Disregarding the results obtained by the direct photometric method, an average of 99.7 percent tantalum was found. The results from the direct photometric method were slightly high due to interference from small amounts of platinum from the crucibles in which samples were dissolved.[26]

TABLE III

DETERMINATION OF TANTALUM IN TANTALUM POWDER

Method	Sample Weight (mg)	Ta Found (%)	Average (%)
Ion exchange-gravimetric	203.4 200.7	99.8 99.8	99.8
Direct Photometric*	10.88 11.04 8.33 8.88 8.91 9.75 8.95	99.1 101.4 100.6 100.5 100.7 101.1 100.1	100.5
Cupferron-photometric	19.30 12.59 10.57	99.5 99.3 99.8	99.5

*High results may be due to interference by platinum from the crucibles.

The standard samples were added to conditioned ion-exchange columns and the vanadium eluted with a nitric-hydrofluoric acid mixture. The nitric acid was then displaced by passing an ammonium chloride-hydrofluoric acid mixture through the columns. The nitric acid must be removed prior to the elution of the tantalum since nitric acid interferes in the precipitation of tantalum with cupferron. The eluates collected up to this point were discarded.

The tantalum was eluted with an ammonium chloride-ammonium fluoride solution, precipitated from the eluates with cupferron, and determined by the pyrogallic acid photometric method.

A detailed description of the procedure, including reagents and materials used, is presented in the appendix. Results obtained on the synthetic samples are shown in Table IV. As can be seen, complete recovery of the added tantalum was achieved. Results on a series of actual alloys submitted for analysis are shown in Table V. The expected or nominal values agree well with the values found.

TABLE IV

ANALYSIS OF SYNTHETIC SAMPLES

Sample	Weight of Vanadium (g)	Ta Added (%)	Ta Recovered (%)	Deviation (%)
A	5.0	0.000	0.001 0.000	+0.001 0.000
B	5.0	0.046	0.046 0.046	0.000 0.000
C	5.0	0.052	0.053 0.053	+0.001 +0.001
D	2.0	0.788	0.795 0.785	+0.007 -0.003
E	2.0	0.820	0.820 0.805	0.000 -0.015
			Average Deviation	±0.003

TABLE V

ANALYSIS OF VANADIUM-TANTALUM ALLOYS

Sample	Ta Expected (%)	Ta Found (%)
MR 804	0.80	0.800
MR 805	0.40	0.393
MR 806	0.12	0.131
MR 807	0.60	0.606
MR 808	0.24	0.252
MR 810	0.16	0.163
MR 811	0.04	0.055

CONCLUSION

The ion exchange-photometric method presented here for the analysis of tantalum in vanadium is satisfactory with regard to convenience, accuracy, and precision. Details of the method, as developed in this study, are given in the appendix.

APPENDIX

ION EXCHANGE-PHOTOMETRIC METHOD FOR THE DETERMINATION OF TANTALUM IN VANADIUM

Scope

This method is applicable to samples of vanadium alloys containing up to 20 mg of tantalum. If more than 20 mg of tantalum is present in the sample taken for test, a gravimetric finish is recommended. An upper limit of 40 mg of tantalum is recommended for an ion-exchange column prepared as described in this report.

Principle of the Method

The sample is dissolved in a hydrofluoric-nitric acid solution and transferred to an anion-exchange column. Vanadium and alloying elements other than tantalum are removed with a hydrofluoric-nitric acid solution. Nitric acid is displaced with an ammonium chloride-hydrofluoric acid solution and the tantalum is then removed in a separate fraction by elution with an ammonium chloride-ammonium fluoride solution. The tantalum is precipitated with cupferron in the presence of zirconium added as a carrier, and determined photometrically with pyrogallic acid.

Interferences

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

Concentration Range

The sample size should be adjusted to contain between 0.1 mg and 20 mg of tantalum.

Apparatus

1. Ion-Exchange Column. - The column, constructed of polystyrene, is approximately 12 inches in length and 1/2 inch in inside diameter. The bottom of the column is closed with a rubber stopper with 3/16-inch hole into which is inserted a 3-inch length of polyethylene tubing of 3/16-inch outside diameter and 1/16-inch bore. This tubing is connected to a short length of Tygon R tubing to which is attached a hosecock to control the flow through the column.

2. Beckman Model B Spectrophotometer.

Reagents

1. Ammonium Chloride Solution (240 g per liter). - Dissolve 480 g of ammonium chloride (NH_4Cl) in 1600 ml of water and dilute to 2 liters. Filter to remove insoluble material. This solution is used as a stock solution in preparing solutions 2 and 3.

2. Ammonium Chloride-Ammonium Fluoride Solution. - Add to a plastic bottle 600 ml of NH_4Cl solution and 40 ml of HF. Adjust the solution

to a pH of 5 to 6 with NH_4OH (approximately [illegible] to 85 ml will be required) and dilute to 1 liter with water. This solution must be prepared carefully with regard to pH adjustment.

3. Ammonium Chloride-Hydrofluoric Acid Solution. - Transfer 600 ml of the NH_4Cl solution and 40 ml of HF to a plastic bottle and dilute to 1 liter with water.

4. Ammonium Oxalate-Ammonium Citrate Solution. - Add 25 ml of H_2SO_4 to 975 ml of water and mix well. Add 40 g of ammonium oxalate monohydrate ($(NH_4)_2C_2O_4.H_2O$) and 50 g of diammonium citrate ($(NH_4)_2HC_6H_5O_7$) and warm on steam bath to dissolve the salts. Filter through a dry filter paper.

5. Cupferron Solution (60 g per liter). - Dissolve 6 g of cupferron in 90 ml of water, dilute to 100 ml, and filter through a dry filter paper. Prepare as needed and keep cool (5 C).

6. Cupferron Wash Solution. - Add 25 ml of the cupferron solution to 975 ml of cold (5 C) HCl (1:19). Prepare as needed.

7. Ion-Exchange Resin. - Dowex 1X-10, strongly basic anion-exchange resin, 200- to 400-mesh, chloride form. Air-dry the resin as received and pass that fraction which is retained on a No. 270 sieve through a No. 180 sieve. Prepare a suspension of the 180-to 270-mesh resin 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.

8. Nitric-Hydrofluoric Acid Mixture. - Transfer 700 ml of water, 100 ml of HNO_3, and 100 ml of HF to a plastic bottle and dilute to 1 liter with water.

9. Pyrogallic Acid Solution (200 g per liter). - Dissolve 200 g of pyrogallic acid in 800 ml of the ammonium oxalate-ammonium citrate solution, dilute to 1 liter with the same solution, and transfer to a glass-stoppered bottle. Discard when blank photometric reading becomes excessive.

10. Standard Tantalum Solution (1 ml = 0.125 mg Ta). - Transfer 0.0305 g of tantalum pentoxide to a platinum crucible. Add 1 g of potassium pyrosulfate and fuse the oxide over a medium flame until a clear melt is obtained. Leach the cooled melt in 75 ml of warm ammonium oxalate-ammonium citrate solution, stirring continuously until the melt is dissolved. Transfer the cooled solution to a 200-ml volumetric flask and dilute to volume with ammonium oxalate-ammonium citrate solution.

11. Zirconium Sulfate Solution (1 ml = approximately 1 mg Zr). - Dissolve 4 g of zirconium sulfate tetrahydrate ($Zr(SO_4)_2.4H_2O$) in 100 ml of H_2SO_4 (1:19). Transfer to a 1-liter volumetric flask and dilute to volume with H_2SO_4 (1:19).

Preparation of Ion-Exchange Column

Cover the bottom of the ion-exchange column with a layer of acid-resistant vinyl chloride plastic wool, 1/4- to 3/8-inch thick. Add the

resin suspension in small portions to obtain a settled column of the resin 4-1/2 inches high. Wash the column with 25 ml of HNO_3 (1:9) and then run through three elution cycles with alternate additions of 15 ml of HCl (3:1) and HCl (1:9) until the remainder of the fines have been removed. The final wash should be with HCl (1:3). When not in use, the column should be filled with HCl (1:3) to a level above the resin bed.

Procedure

1. Weigh a portion of the sample containing no more than 20 mg of tantalum and transfer to a covered Teflon beaker. Add 10 ml of HF and then add 10-15 ml of HNO_3 in small increments, allowing the reaction to subside somewhat between the additions of the HNO_3. Heat the solution to insure complete dissolution of the sample. Rinse the cover and sides of beaker with water and dilute to approximately 75 ml.

2. Condition the ion-exchange column by passing 20 ml of the nitric-hydrofluoric acid mixture in 5-ml portions through the resin into a plastic beaker.

3. Transfer the sample solution to the column in increments with the hosecock open and allow the solution to pass through into the plastic beaker. When all the sample solution has been transferred, allow the liquid to drain to the top of the resin bed. Wash the beaker and the column above the resin bed with 3 to 5 ml of the nitric-hydrofluoric acid mixture. Allow the liquid to drain as before and repeat the washing two more times.

4. Elute the remaining vanadium by passing another 30 ml of the nitric-hydrofluoric acid mixture through the column. Allow the solution to drain and wash the column with 3 to 5 ml of the ammonium chloride-hydrofluoric acid solution. Repeat the washing of the column two more times, allowing the column to drain completely. Pass 30 ml of the ammonium chloride-hydrofluoric acid solution through the column and drain completely. Discard this eluate, which contains the vanadium and nitric acid.

5. Place a clean, 100-ml plastic beaker or bottle beneath the column. Wash the column above the resin three times with 5-ml portions of the ammonium chloride-ammonium fluoride solution, allowing the solution to drain after each addition. Pass 60 ml of this solution through the column and reserve this eluate for the determination of tantalum.

6. Clean the column by passing 30 ml of HCl (1:3) through the resin in 5-ml increments, draining the column after each addition. This prepares the column for the next sample.

7. Transfer the tantalum-bearing eluate to a 250-ml Pyrex beaker containing 1.5 g of boric acid, rinsing the plastic beaker thoroughly. Add 20 ml of the zirconium sulfate solution and 20 ml of HCl. Heat to dissolve the boric acid, then cool to 5 C.

8. Prepare a blank by adding 20 ml of the zirconium sulfate solution to a 250-ml beaker containing 1.5 g of boric acid. Add 20 ml of HCl, heat

to dissolve the boric acid, and cool to 5 C. Carry this blank along with the sample through the remaining steps of the procedure.

9. Add slowly, with continuous stirring, 20 ml of cold cupferron solution. Add paper pulp, stir well and allow to stand for 10 to 15 minutes at 5 C.

10. Filter through a Whatman No. 42 paper containing some paper pulp and wash well with the cupferron wash solution. Transfer the paper and precipitate to a platinum crucible and ignite at a low temperature (500 to 550 C).

Note. - If the sample contains more than 20 mg of tantalum, the determination can be completed gravimetrically at this point by igniting the cupferron precipitate in a weighed platinum crucible at 1100 C. In this case, the addition of the zirconium sulfate solution in step 7 is not necessary. The factor for converting the Ta_2O_5 to Ta is 0.819.

11. Fuse the mixed oxides with 1 g of potassium pyrosulfate over a medium flame. Leach the cooled melt in 25 to 30 ml of warm ammonium oxalate-ammonium citrate solution. Stir continuously while dissolving the melt to prevent a localized high concentration of tantalum and consequent hydrolytic precipitation.

12. Transfer the solution to a 50- or 100-ml volumetric flask, depending upon the amount of tantalum expected, and dilute to volume with the ammonium oxalate-ammonium citrate solution. Transfer exactly 25.0 ml of the pyrogallic acid solution to a 50-ml volumetric flask. Add a suitable aliquot of the sample solution, not to exceed 1 mg of tantalum, and mix well.

13. Dilute to volume with the ammonium oxalate-ammonium citrate solution, mix well, and allow to stand overnight. Measure the absorbance of the test solution and the blank solution versus water at 400 mμ using 1-cm cells. Correct the absorbance reading of the test solution by subtracting that obtained for the blank.

Preparation of Calibration Curve

To four 50-ml volumetric flasks, add exactly 25.0 ml of pyrogallic acid solution. To three of these flasks, add 2.0, 5.0, and 10.0 ml of the standard tantalum solution (1 ml = 0.125 mg Ta) and carry the fourth flask along as a reagent blank. Dilute to volume with the ammonium oxalate-ammonium citrate solution, mix well, and let stand overnight. Read the solutions at 400 mμ versus water. Correct for the reagent blank and plot the absorbance values versus milligrams of tantalum.

Calculation

Convert the absorption reading of the sample solution to milligrams of tantalum by means of the calibration curve. Calculate the percentage of tantalum as follows:

$$\text{Tantalum, percent} = \frac{A}{B} \times 100$$

where:

A = milligrams of tantalum from calibration curve, and

B = milligrams of sample in aliquot taken.

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