SPECTROPHOTOMETRIC DETERMINATION OF SCANDIUM WITH ARSENAZO*

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INTRODUCTION

MASTER

Berman, Duval, and Russell¹ have reviewed spectrophotometric methods for scandium. Brudz et al.² have made a comparison of various spectrophotometric reagents and recommended sulfonazo for the determination of scandium. Arsenazo III³, 4-(2-pyridylazo)resorcinol (PAR)⁴, propylfluorone⁵, Xylenol Orange¹, and 8-quinolinol derivatives⁶ have been recently proposed for the spectrophotometric determination of scandium.

After a study on the determination of the rare earths with arsenazo⁷, it was thought worthwhile to attempt a spectrophotometric determination of scandium with the same reagent. Although the color reaction of scandium with arsenazo has been reported by Kuznetsov⁸ and Kuteĭnikov⁹, no procedure seems to have been developed for the determination of this element. The conditions for the determination and the separation from interfering elements are described in the present paper.

APPARATUS

Absorbance measurements were made with a Beckman Model DU spectrophotometer, using 1-cm cells. A Beckman Model G pH meter was used for pH measurements. A Burrell shaking machine with a time switch was used for extractions. Glass tubes (25 cm long,

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1.1 cm i.d.) with coarse, sintered-glass filter disks fused into the bottom were used to hold the anion exchange resin (Dowex 1-X8, supplied in the chloride form, 50-100 mesh). The column of resin was about 10 cm high. For the separation of uranium, the resin was washed with four 10-ml portions of water, two 10-ml portions of ethanol, four 10-ml portions of water, and four 10-ml portions of 8 M hydrochloric acid before use. For the separation of thorium, 8 M hydrochloric acid was replaced by 8 M nitric acid in the above treatment.

REAGENTS

Thenoyltrifluoroacetone (TTA), 0.5 M: 45 g in 400 ml of xylene.

8-Quinolinol: 5.0 g of 8-quinolinol was dissolved in 10 ml of glacial acetic acid and the solution was diluted to 100 ml with water.

Ammonium acetate, 3.5 M: 135 g in 500 ml of water.

Arsenazo: 0.10 g in 100 ml of water. An Eastman product, 3-(2-arsonophenylazo)-4,5-dihydroxy-2,7-naphthalenedisulfonic acid disodium salt (P 7302) was used.

Acetic acid-sodium acetate: Ten ml of 1 M acetic acid was mixed with 320 ml of 1 M sodium acetate (136 g of $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ in 1 liter of water). The pH of the mixture was 6.1.

Standard scandium solution, 1.00 mg Sc/ml: 0.1534 g of Sc_2O_3 was dissolved by heating in 50 ml of concentrated hydrochloric acid. The solution was evaporated to near dryness and diluted to exactly 100 ml with water. The final concentration of

acid was about 0.1 M. Working standard solutions (50 and 10 p.p.m. Sc) were prepared by diluting this stock solution with water.

DETERMINATION OF SCANDIUM

Absorption curve. The maximum absorption of the scandium-arsenazo complex against reagent blank was found at about 570 m μ (Fig. 1).

Effect of pH. The maximum and nearly constant absorbance was obtained at pH 6.1-6.3 (Fig. 2). Acetic acid-sodium acetate and triethanolamine-nitric acid buffer solutions were used for the pH ranges 3.5-6.5 and 7-8, respectively.

Effect of acetate concentration. At pH 6.1, the absorbance of the complex decreased very slightly as the concentration of acetate ion increased from 0.04 to 0.14 M. At 0.08, 0.10, and 0.12 M acetate concentrations, 0.856 p.p.m. Sc gave absorbances of 0.356, 0.350, and 0.349, respectively. A concentration of 0.10 M was chosen.

Effect of arsenazo concentration. For 0.856 p.p.m. Sc, a constant absorbance was obtained by adding 2.0 to 5.0 ml of 0.10% arsenazo solution and diluting to 50 ml.

Stability of colored solution. Absorbances of the complex and blank solutions remained constant for 3 hours. All the experiments were carried out at room temperature of about 25°.

Calibration curve. The scandium-arsenazo system conforms to Beer's law with concentrations of up to 2.0 p.p.m. Sc in the final solution. The molar absorptivity for scandium was

1.7×10^4 .

Interference study. Table I summarizes the results of the interference study. The following ions give a color with arsenazo; aluminum, chromium, copper, iron(III), rare earths, thorium, titanium, uranium, and zirconium. Fluoride and phosphate ions decrease the absorbance of the scandium complex. An attempt to mask iron(III) with L-ascorbic acid was not successful. Hydrogen peroxide inhibits the reaction of thorium with arsenazo, but it also affects the absorbance of the scandium complex. Microgram quantities of thorium are not masked by sodium sulfate (0.30 mmole).

Determination of scandium and thorium. Although thorium interferes with the determination of scandium, scandium can be determined in the presence of small amounts of thorium by using a similar method that was previously used for the determination of both rare earths and thorium¹⁰. The present method is based on the fact that in a 0.05 M hydrochloric acid medium the absorbance is due to the thorium-arsenazo complex alone, while at pH 6.1 the absorbance is due to both the thorium and scandium complexes. For details of the procedure, the reader is referred to the previous report by Onishi *et al.*¹⁰. The results are shown in Table II.

When the sample contains relatively large amounts of thorium, the separation of scandium by anion exchange is recommended.

SEPARATION OF SCANDIUM

TTA-xylene extraction. Extraction of scandium with TTA in benzene was studied by Bronaugh and Suttle¹¹. The present work has shown that the TTA extraction at pH 1.5 is useful not only in the separation of scandium from the rare earths, but also in the separation from a considerable number of other elements. The data obtained in applying the proposed procedure are collected in Table III.

The first TTA extraction (and back-extraction) gave a recovery of 93% and the second TTA extraction 1% of scandium.

It appears that a large amount of nitrate slightly decreases the recovery of scandium. When 1.6 mmole of nitrate, as magnesium nitrate, was present, 90% of scandium was recovered. When the nitrate was replaced by chloride, 95% of scandium was recovered (Table III). It is of interest that iron(II) is completely separated from scandium, whereas iron(III) is not⁷. Molybdenum(VI), titanium, uranium(VI), and vanadium(V) are considerably extracted with TTA. Thorium cannot be separated from scandium; 96% of thorium was recovered under the conditions described in the procedure. The presence of 0.90 mmole sodium sulfate did not change the recovery of thorium. Zirconium is extracted with TTA, but it is not back-extracted with 2 M hydrochloric acid. Small amounts of fluoride and phosphate do not interfere with the extraction of scandium. This would be very valuable, because fluoride and phosphate cause a negative interference in most of the photometric methods for scandium;

e.g., 4-(2-pyridylazo)resorcinol⁴ and Xylenol Orange¹ methods.

The average recovery of scandium for 24 sets of experiments is 94.6% with a range of 90.6 to 98.5%.

8-Quinolinol-chloroform extraction. Because aluminum, copper, and iron(III) cannot be separated from scandium by the TTA extraction, 8-quinolinol-chloroform extraction has been studied. As shown in Table IV, these interfering elements are extracted as 8-quinolinolates, while scandium remained in the aqueous phase. Molybdenum and titanium also are extracted. Extraction of vanadium is not complete. If the sample contains much vanadium (and iron(III), titanium, and zirconium), cupferron-chloroform extraction¹² would be applicable.

Separation of scandium from uranium. Extraction of uranium(IV) cupferrate with chloroform from 1 M hydrochloric acid was tried without success. Hydroxylamine hydrochloride and hydrazine dihydrochloride were used to reduce uranium(VI) to (IV).

Uranium(VI) forms anionic chloride complexes in 4-12 M hydrochloric acid and it is adsorbed on a strong-base anion-exchange resin¹⁴. On the other hand, scandium is slightly adsorbed in 12 M hydrochloric acid¹⁴. These form the basis for the development of the proposed procedure. As shown in Table V, as much as 3 mg of uranium can be completely removed. Under the conditions described in the procedure, the following elements also would be adsorbed on the resin almost quantitatively¹⁴; Mo(VI), W(VI), Fe(III), Ru(IV), Os(IV), Co(II), Ir(IV), Pd(II),

Pt(IV), Cu(II), Au(III), Zn(II), Cd(II), Hg(II), Ga(III), In(III), Tl(III), Ge(IV), Sn(IV and II), As(III), Sb(V and III), and Bi(III).

Separation of scandium from thorium. Thorium(IV) forms anionic nitrate complexes in nitric acid and it is adsorbed on an anion-exchange resin (Dowex 1)^{15,16,17}. On the other hand, scandium does not form adsorbable nitrate complexes^{17,18}. As shown in Table V, 27 μg of scandium is separated from as much as 1 mg of thorium. This method is simpler than the method that involves the removal of thorium by precipitation as the iodate¹².

PROCEDURE

TTA-xylene extraction. Evaporate the sample solution which contains 10 to 50 μg of scandium in dilute hydrochloric acid to dryness. Dissolve the residue in 0.9 ml of 0.5 M hydrochloric acid, add 4 ml of water, and transfer the solution to a 60-ml separatory funnel. Wash the vessel used for the evaporation with two 5-ml portions of water. If necessary, confirm that the pH of the solution is 1.5 ± 0.2 . Shake the solution for 5 minutes with 10 ml of TTA solution. When the layers have separated, drain off the aqueous phase into a second separatory funnel. Add 10 ml of TTA solution to the second separatory funnel. Shake the system for 5 minutes and discard the aqueous phase. Combine the second extract with the first and wash the extracts by equilibrating them for 10 seconds with 10 ml of 0.03 M hydrochloric acid. Discard the aqueous phase.

Shake the organic phase for 5 minutes with 20 ml of 2 M hydrochloric acid. Transfer the aqueous phase to another separatory funnel. Discard the organic phase. Shake the aqueous phase for 10 seconds with 10 ml of xylene. Transfer the aqueous phase to a 50-ml beaker and evaporate to dryness. To the residue add 1 ml of 70% perchloric acid and 1 ml of concentrated nitric acid. Evaporate the solution to dryness. Wash the sides of the beaker with a minimum amount of water and evaporate the solution to dryness. Then proceed as described in Spectrophotometric determination of scandium.

8-Quinolinol-chloroform extraction. To a 10-ml aqueous solution (pH 2-3) add 1.0 ml of 8-quinolinol solution, 1.0 ml of ammonium acetate solution, and 3 ml of water. The pH of the solution should be in the range 4.5-4.7. Add 20 ml of chloroform and shake the system for 1 minute. When the layers have separated, drain off and discard the organic phase. Add 0.3 ml of 8-quinolinol solution and 20 ml of chloroform and equilibrate the phases for 1 minute. Discard the organic phase. Wash the aqueous phase by shaking it for 10 seconds with 20 ml of chloroform and discard the organic phase. Transfer the aqueous phase to a 50-ml beaker and evaporate to dryness. To the residue add 3 ml of concentrated hydrochloric acid and 1 ml of concentrated nitric acid. Cover the beaker with a watch glass and heat the solution. When a vigorous reaction ends, remove and wash the watch glass and evaporate the solution to dryness. Then proceed as described in Spectrophotometric determination of scandium.

Separation of scandium from uranium(VI). Evaporate the sample solution in a beaker to dryness. Dissolve the residue in 10 ml of 8 M hydrochloric acid. Pass the solution through the prepared resin column at a rate of about 2 ml per minute. Wash the beaker and resin with four 10-ml portions of 8 M hydrochloric acid. Evaporate the effluent in a 100-ml beaker to dryness. Then proceed as described in Spectrophotometric determination of scandium. The uranium can be eluted with water.

Separation of scandium from thorium. Evaporate the sample solution in a beaker to dryness. Dissolve the residue in 10 ml of 8 M nitric acid. Pass the solution through the prepared resin column at a rate of about 2 ml per minute. Wash the beaker and resin with four 10-ml portions of 8 M nitric acid. Evaporate the effluent in a 100-ml beaker to dryness. To the residue add 1 ml of 6 M hydrochloric acid and evaporate the solution to dryness. Then continue the determination as described below. The thorium can be eluted with 0.5 M nitric acid.

Spectrophotometric determination of scandium. Dissolve the residue in 1.0 ml of 0.1 M hydrochloric acid. Add 4 ml of water, mix, and add 2.0 ml of arsenazo solution. Add 2.5 ml of acetic acid-sodium acetate buffer solution and 1.0 ml of 0.1 M ammonium hydroxide. Confirm that the pH of the solution is 6.1-6.3. Transfer the solution to a 25-ml volumetric flask and dilute to the mark with water. Measure the absorbance of the solution in a 1-cm cell at 570 m μ , using water or reagent blank as the reference.

Construct the calibration curve by taking, for example, 0, 10, 20, 30, and 50 μg of scandium, adding 1.0 ml of 0.1 M hydrochloric acid, and proceeding as described above.

A blank determination should accompany any separation step.

SUMMARY

Arsenazo is used for the spectrophotometric determination of scandium in the range 10 to 50 μg . The absorbance is measured at 570 m μ and pH 6.1. A method is proposed for the successive determination of scandium and thorium. Scandium is separated from magnesium, calcium, rare earths, zirconium, fluoride, phosphate, and some other metals by extraction with TTA in xylene. Copper, aluminum, and iron(III) are removed by 8-quinolinol-chloroform extraction. Uranium(VI) is removed by anion exchange using hydrochloric acid. Thorium is separated from scandium by anion exchange using nitric acid.

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TABLE I
INTERFERENCE STUDY

(Final volume: 25 ml)

Addition		Sc taken, μg	Sc found, μg
Al(III)	0.030 mg	0	44
Ca(II)	0.10 mg	26.7	27.3
Cd(II)	0.10 mg	26.7	27.0
Co(II)	0.10 mg	0	4
Cr(III)	0.10 mg	0	35
Cu(II)	0.10 mg	0	18
Fe(III)	0.10 mg	0	25
Gd(III)	0.053 mg	0	18
Mg(II)	0.10 mg	26.7	27.5
Mn(II)	0.10 mg	26.7	27.0
Mo(VI)	0.10 mg	26.7	27.0
Ni(II)	0.10 mg	0	5
Pb(II)	0.10 mg	26.7	25.0
Th(IV)	0.10 mg	0	33
Ti(IV)	0.10 mg	0	34
U(VI)	0.10 mg	0	18
V(V)	0.10 mg	0	4
W(VI)	0.10 mg	26.7	26.7
Zn(II)	0.10 mg	26.7	27.5
Zr(IV)	0.10 mg	0	39
NaCl	0.50 mmole	26.7	26.5
NaClO ₄	0.30 mmole	26.7	26.8

TABLE I (concluded)

Addition		Sc taken, μg	Sc found, μg
NaNO_3	0.50 mmole	26.7	26.8
Na_2SO_4	0.30 mmole	26.7	26.5
F (as NaF)	101 μg	26.7	21.2
P (as KH_2PO_4)	10 μg	26.7	18.3

TABLE II

DETERMINATION OF THORIUM AND SCANDIUM WITH ARSENAZO

Taken		Found	
Th, μg in 25 ml	Sc, μg in 25 ml	Th, μg in 25 ml	Sc, μg in 25 ml
0	26.7	0.0, 0.0	27.8, 27.5
15.2	10.6	14.5, 14.5	9.8, 10.5
15.2	26.7	14.5, 14.5	26.5, 26.0
15.2	42.7	15.5, 15.5	40.5, 40.5
25.3	10.6	25.0, 25.0	10.0, 10.0
25.3	26.7	25.0, 25.0	26.5, 25.3
50.7	0	50.0, 50.0	0.0, 0.2
50.7	10.6	50.0, 50.0	9.5, 9.8

TABLE III

DETERMINATION OF SCANDIUM AFTER TTA EXTRACTION

Addition	Sc taken, μg	Sc found, ^a μg	Average recovery, %
none	0	0.0, 0.0	-
none	26.7	25.5, 25.5	95.5
0.10 mg Al	0	6.5, 6.5	-
0.10 mg Al	26.7	23.0, 21.2	82.7
21 μg Al	0	0.0, 0.0	-
21 μg Al	26.7	24.8, 25.0	93.2
10 mg As(III)	0	0.5, 0.4	-
10 mg As(III)	26.7	25.5, 25.5	95.5
20 mg Ca	0	0.0, 0.0	-
20 mg Ca	26.7	25.0, 25.0	93.6
10 mg Cd	0	0.0, 0.0	-
10 mg Cd	26.7	24.7, 24.7	92.5
10 mg Co	0	1.3, 1.3	-
10 mg Co	26.7	25.7, 25.5	95.9
10 mg Cr(III)	0	1.3, 1.4	-
10 mg Cr(III)	26.7	22.5, 23.0	85.4
1.0 mg Cr(III)	0	1.3, 1.3	-
1.0 mg Cr(III)	26.7	24.5, 24.5	91.8
1.0 mg Dy	0	0.6, 0.4	-
1.0 mg Dy	26.7	26.0, 26.5	98.5
1.0 mg Fe(II) ^b	0	0.8, 0.7	-
1.0 mg Fe(II) ^b	26.7	25.3, 25.0	94.4

TABLE III (continued)

Addition	Sc taken, μg	Sc found, ^a μg	Average recovery, %
20 mg Mg	0	0.0, 0.0	-
20 mg Mg	26.7	25.5, 25.2	95.1
10 mg Mn(II)	0	0.6, 0.6	-
10 mg Mn(II)	26.7	25.7, 25.2	95.5
9.8 mg Mo(VI)	0	4.5 ^c , 2.5 ^c	-
9.8 mg Mo(VI)	26.7	22.5 ^c , 22.7 ^c	85.0
0.98 mg Mo(VI)	0	0.3, 0.3	-
0.98 mg Mo(VI)	26.7	25.3, 25.3	94.8
10 mg Ni	0	0.2, 0.1	-
10 mg Ni	26.7	24.5, 24.5	91.8
10 mg Pb	0	0.0 ^d , 0.0 ^d	-
10 mg Pb	26.7	25.7 ^d , 25.0 ^d	95.1
97 μg Ti	0	8.5, 8.2	-
97 μg Ti	26.7	18.5, 18.5	69.3
9.7 μg Ti	0	1.3, 1.3	-
9.7 μg Ti	26.7	24.0, 24.3	90.6
10 μg U(VI)	0	1.9, 1.7	-
10 μg U(VI)	26.7	25.0, 25.2	94.0
0.50 mg V(V)	0	4.5, 5.2	-
0.50 mg V(V)	26.7	24.0, 24.0	89.9
0.10 mg V(V)	0	2.6, 2.8	-
0.10 mg V(V)	26.7	24.8, 25.2	93.3
20 μg V(V)	0	0.5, 1.3	-
20 μg V(V)	26.7	25.3, 25.3	94.8

TABLE III (concluded)

Addition	Sc taken, μg	Sc found, ^a μg	Average recovery, %
10 mg W(VI)	0	0.0,0.0	-
10 mg W(VI)	26.7	24.8,24.5	92.5
0.49 mg Y	0	0.3,0.5	-
0.49 mg Y	26.7	25.2,25.5	95.1
10 mg Zn	0	0.3,0.0	-
10 mg Zn	26.7	25.7,26.5	97.8
1.0 mg Zr	0	0.0,0.0	-
1.0 mg Zr	26.7	26.0,25.8	97.0
0.10 mg F (as NaF)	0	0.0,0.0	-
0.10 mg F (as NaF)	26.7	24.7,24.6	92.5
0.10 mg P (as KH_2PO_4)	0	0.1,0.3	-
0.10 mg P (as KH_2PO_4)	26.7	25.5,25.7	95.9
0.90 mmole Na_2SO_4	0	0.0,0.0	-
0.90 mmole Na_2SO_4	26.7	25.8,26.1	97.4

^a Values obtained with 26.7 μg Sc were corrected for respective blanks.

^b Fe(III) reduced with hydrazine dihydrochloride prior to TTA extraction.

^c Dilute HCl solution was filtered through a plug of glass wool prior to color development.

^d TTA extraction from 0.03 M ($\text{HClO}_4 + \text{HNO}_3$) solution.

TABLE IV
DETERMINATION OF SCANDIUM AFTER 8-QUINOLINOL-CHLOROFORM
EXTRACTION

Addition	Sc taken, μg	Sc found, ^a μg	Average recovery, %
none	0	0.0, 0.0	-
none	26.7	25.7, 25.8	96.6
1.0 mg Al, 1.0 mg Fe(III)	0	0.3, 0.0	-
1.0 mg Al, 1.0 mg Fe(III)	26.7	24.5, 24.5	91.8
1.0 mg Cu	0	0.2, 0.2	-
1.0 mg Cu	26.7	26.0, 25.3	96.3
0.98 mg Mo(VI)	0	0.4, 0.4	-
0.98 mg Mo(VI)	26.7	25.7, 25.3	95.5
0.97 mg Ti	0	0.0, 0.0	-
0.97 mg Ti	26.7	25.5, 25.0	94.8
1.0 mg V(V)	0	2.4 ^b , 2.4 ^b	-
1.0 mg V(V)	26.7	20.0 ^b , 21.0 ^b	76.8

^a Values obtained with 26.7 μg Sc were corrected for respective blanks.

^b Before extraction chloroform was shaken with six separate portions of water¹³.

TABLE V

DETERMINATION OF SCANDIUM AFTER SEPARATION FROM
URANIUM OR THORIUM

Addition	Sc taken, μg	Sc found, ^a μg	Average recovery, %
Anion exchange using hydrochloric acid			
none	26.7	25.8, 27.0	98.9
1.0 mg U	0	0.1, 0.3	-
1.0 mg U	26.7	24.8, 26.2	95.5
3.0 mg U	0	0.3, 0.6	-
3.0 mg U	26.7	25.7, 26.5	97.8
10 mg U	0	3.8, 2.8	-
10 mg U	26.7	25.3, 24.8	94.0
Anion exchange using nitric acid			
none	26.7	25.0, 25.5	94.8
0.207 mg Th	0	0.0, 0.0	-
0.207 mg Th	26.7	25.5, 25.3	95.1
0.518 mg Th	0	0.0, 0.0	-
0.518 mg Th	26.7	26.3, 26.2	98.5
1.03 mg Th	0	0.2, 0.1	-
1.03 mg Th	26.7	25.5, 26.0	96.6

^a Values obtained with 26.7 μg Sc were corrected for respective blanks.

Fig. 1. Absorption curves

- A. Reagent blank against water
- B. 0.856 p.p.m. Sc against reagent blank

Fig. 2. Effect of pH

A. Reagent blank against water

B. 1.71 p.p.m. Sc against reagent blank

END