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# USE OF LEWATIT S-100 FOR ISOLATION OF VANADIUM (V) FROM IRON (III) IN THE DETERMINATION OF VANADIUM IN WATER SAMPLES OF DOYRAN LAKE

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## ИЗВАДОК

Јордановска Вера, Тошев Д. (1984): Употреба на леватит S-100 за одвојување на ванадиум (V) од железо (III) при определување на ванадиум во водата од Дојранско езеро Год. зб. фарм. фак., Скопје

Опишана е метода за определување на содржината на ванадиумот во езерската вода од Дојранското езеро. Од 5 литри вода ванадиумот беше копрецитиран и концентриран со железо (III) хидроксид. Одделувањето на ванадиумот од железото беше изведено со силно кисела катјонска смола Леватит S-100. Како средство за елуирање беше употребен 0.6% $H_2O_2$. Ванадиумот беше определен спектрофотометриски при 550 nm како хелатен комплекс со 8-хидроксихинолин растворен во хлороформ. Количината на ванадиумот во езерската вода од Дајранското езеро се движи од 3.4 до 2.5 mg/l што е блиска до содржината на ванадиумот во морските води.

## ABSTRACT

Jordanovska V., Toshev D. (1984): Use of lewatit S-100 for isolation of vanadium (V) from irin (III) in the determination of vanadium in water samples of Doyran lake.

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A method for determination of vanadium in lake water is described. Iron (III) hydroxide for coprecipitation and concentration of vanadium from water samples was used. The isolation of vanadium from iron was achieved with cation exchange Lewatit S-100 resin. Vanadium was determined spectrophotometrically at 550 nm as complexe with 8-hydroxyquinoline in chloroform. The quantities of vanadium in water samples from Dojransko ezero in Yugoslavia were found to be between 3.4 and 2.5 ug/dm$^3$. The water samples were taken in october of 1977.

Methods for determination of p.p.m. and sub-p.p.m quantities of vanadium in naturally occuring waters are based on spectrophotometry[1—11], atomic-absorption spectrometry[12 13 21] emission spectroscopy[14 15], x-ray fluorescence [16] and neutron activitation analysis[17]. The majority of these techniques can be applied only after preliminary isolation of vanadium.

The preconcentration methods available include: coprecipitation of vanadium with iron (III) hydroxide[2 4 5 7 18] (with subsequent removal of iron by chloroform extraction of iron oxinate[4 5 7] or by cation-exchange[2]); liquid-liquid extractions of chelates of vanadium with 5,7-dichloro-8-hydroxyquinoline in butyl acetate[12], cupferron in isobutyl methyl ketone[13], diethyldithiocarbamate in chloroform[15], ammonium tetramethylene dithiocarbamate in isobutyl methyl ketone[16] and 4-/2-pyridylazo/-resorcional/PAR/ in chloroform[6]; and methods based on the adsorption of vanadium on either cation[2 3 17] or anion[1] exchange resins. With catalytic methods[8—11] it is not necessery to preconcentrate vanadium, but molybdenum interferes strongly.

The present paper presents the results of determination of vanadium in lake water by means of spectrophotometric method. This method includes: coprecipitation of vanadium with iron (III) hydroxide[2], cation-exchange isolation of quinquevalent vanadium with Lewatit S-100 resin and spectrophotometric determination of vanadium as oxinate in chloroform at 550 nm[18].

## EXPERIMENTAL

Apparatus:

The absorbance measurements were made on a Beckman Model DU Spectrophotometer in 1-cm cells.

pH values were obtained on a Ionalyser Orion Research Model 407 A.

Solutions and Reagents:

Cation-exchange resin. The strongly acidic cation-exchange resin Lewatit S-100 hydrogen-form was used. The resin was conditioned in the usual way and 1-cm diameter ion-exchange columns were filled to a 8 cm depth.

Standard vanadium solution (100 mg V/l). Dissolve 0.2296 g of A. R. ammonium metavanadate in water and dilute to 1 1. A working standard solution containing 10 mgV/1 was prepared.

2% 8-hydroxyquinoline as chelating reagent in 1 : 9 acetic acid.

5% malonic acid in water.

Ethyl alcohol-free chloroform[22].

## PROCEDURE

Coprecipitation of vanadium with iron (III) hydroxide. To 5 l lake water sample add 5 g sodium chloride, 20 ml 2 mol/l HCl and 5 ml iron (III) chloride. After adjusting the pH value to 5-7 with 2 mol/l ammonia the solution is left for three days. The precipitate is filtered off, washed with 0.5% water solution of ammonium nitrate and dissolved in 20 ml of 2 mol/l HCl and diluted to 400 ml with water.

Ion-exchange separation. The above solution (400 ml) is passed through a columm of hydrogen-form Lewatit S-100 cation-exchange resin at a flow rate of about 50 ml/h. Wash the resin with 0.1 mol/l HCl. Vanadium is eluted with 40 ml 0.6% $H_2O_2$. Determination of vanadium. To the vanadium eluate (in silicate basen) add four drops of concentrate perchloric acid. Heat on a steam bath to distroy hydrogen peroxide and its complex with vanadium (V). Then heat on a electrical plate untill white fumes appear. The residue is dissolved in 2.5 ml 2 mol/l $H_2SO_4$, transfer it in a 50 ml separatory funnel with minimal quantities of water, add 1 ml malonic acid solution, then one drop of methylorange indicator-solution and 4 mol/l solution of sodium acetate to a change of color. Add 2 ml of 8-hydroxyquinoline solution, extract with 5 ml of $CHCl_3$ after a 3 minute intensive shaking. Mesure the absorption at 550 nm against blank and determine the concentration of vanadium by means of a standard curve (prepared in exactly the same way as the samples).

## RESULTS AND DISCUSSION

To determine the conditions of absorption of vanadium on Lewatit S-100 the concentration of HCl in vanadium solution was varried. The results are given in Table 1.

TABLE 1

Effect of concentration of hydrochloric acid on the absorption of 10 g of vanadium on cation-exchange resin Lewatit S-100.

| CHCl' mol/l | A550 nm' lgIo/I | μgV/5ml$CHCl_3$ | % |
|---|---|---|---|
| 1. 0.054 | 0.116 | 9.0 | 90 |
| 2. 0.081 | 0.124 | 9.6 | 96 |
| 3. 0.108 | 0.131 | 10.0 | 100 |
| 4. 0.135 | 0.121 | 9.4 | 94 |
| 5. 0.162 | 0.116 | 9.0 | 90 |
| 6. 0.189 | 0.106 | 8.1 | 81 |

The results in Table 1 are in agreement with those known in literature for other strongly acidic cation exchange resins[19].

To determine the quantity of 0.6% $H_2O_2$ for elution of vanadium from the resin different volumes of this solution were used. The results are given in Table 2.

TABLE 2

A dependence of the elution of 20 μg vanadium from Lewatit S-100 of quantities of 0.6% $H_2O_2$.

| ml 0.6% $H_2O_2$ | μgV | % |
|---|---|---|
| 1. 20 | 17.40 | 87.0 |
| 2. 30 | 19.28 | 96.4 |
| 3. 40 | 20.00 | 100.0 |
| 4. 50 | 20.00 | 100.0 |

It can be noticed from the Table 2 that the quantity of 40 ml of 0.6% $H_2O_2$ is enough for quantitative isolation of vanadium.

To determine the elution of vanadium from Lewatit S-100 with 0.6% $H_2O_2$ in the presence of about thousand fold quantity of iron (III) different quantities of vanadium (V) were taken. The results are given in Table 3.

TABLE 3

Elution of vanadium with 0.6% $H_2O_2$ from Lewatit S-100 in the presence of 50 mg of iron (III).

| | μgV added | found | difference in % |
|---|---|---|---|
| 1. | 10 | 10.10 | 1.00 |
| 2. | 20 | 19.90 | 0.50 |
| 3. | 30 | 30.05 | 0.16 |
| 4. | 40 | 40.20 | 0.50 |

Iron (III) does not interfere in thousand fold quantity in determination of vanadium because it is not elute with 0.6% $H_2O_2$.

The depandance of extraction of vanadium (V) with chloroform on concentration of 8-hydroxyquinoline was also determined. The results are given in Table 4.

TABLE 4

Depandance of extraction of 10(I) and 20 (II) μgV with 5 ml $CHCl_3$ from the concentration of 8-hydroxyquinoline.

| | C(8-hydroxyquinol.) ml (mol/l) | μgV found I | II |
|---|---|---|---|
| 1. | 0.2 ($0.55x10^{-3}$) | 8.1 | 17.5 |
| 2. | 0.4 ($1.1x10^{-3}$) | 9.4 | 17.8 |
| 3. | 0.8 ($2.2x10^{-3}$) | 10.0 | 19.0 |
| 4. | 1.2 ($3.3x10^{-3}$) | 10.0 | 19.6 |
| 5. | 1.6 ($4.4x10^{-3}$) | 10.0 | 20.0 |

As can be seen from Table 4 2 ml/5. $5x10^{-3}$ moles) of 8-hydroxyquioline is enough for quantitative determination of vanadium (V) up to 20μg.

The quantities of vanadium in lake water from Doyran lake (South part of Yugoslavia) were determined. The water samples from both surface of the lake and from 5 m-depth were taken. The samples were filtered off before analysing. The results are given in Table 5.

TABLE 5

The contents of vanadium in water samples from Doyran Lake

| area | μgV/l water | |
|---|---|---|
| | surface | 5m-depth |
| 1. Star Dojran | 2.800 | 3.400 |
| 2. R'to | 2.800 | 2.840 |
| 3. Efem | 2.600 | 2.400 |
| 4. Donevo | 2.880 | 2.720 |
| 5. Kaldrma | 2.800 | 2.500 |
| 6. Nov Dojran | 2.880 | 3.100 |

The results presented in Table 5 show that the vanadium contents in Doyran Lake water varry between 3.4 μgV/l and 2.5 μgV/l. These values are similar with that found in sea water.

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