Ternary complexes of niobium(V) with nitroderivatives of catechol and tetrazolium salts. Extraction-spectrophotometric investigations

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² Department of Chemistry and Biochemistry, Medical University Plovdiv, 15-A Vassil Aprilov Blvd., 4002 Plovdiv, Bulgaria The formation and solvent extraction of new ion-association complexes between anionic chelates of niobium(V) with nitroderivatives of catechol {NDC: 3,5-dinitrocatechol (3,5-DNC) and 4-nitrocatechol (4-NC)} and tetrazolium cations {2,3,5-triphenyl-2H-tetrazolium (TT⁺) and 3,3'-[3,3'-dimetoxy(1,1'-biphenyl)-4,4'-diyl]-bis[2,5-diphenyl-2H-tetrazolium] (Blue Tetrazolium,BT²⁺)} were studied. Niobium(V) forms two complexes with 4-NC and BT²⁺ and one complex with 3,5-DNC and TT⁺ with suggested formulae (BT²⁺) [NbO(OH)(4-NC)₂] (I), (BT²⁺)₃[NbO(4-NC)₃]₂ (II), and (TT⁺)₂[NbO(OH)(3,5-DNC)₂] (III). The optimum conditions for the extraction of II and III (organic solvent, extraction time, acidity of the aqueous phase, concentration of reagents), some key constants {association constant (β), constant of distribution (K_D), extraction constant (K_{ex})} and analytical characteristics were determined. The molar absorptivities of II and III were calculated to be $\epsilon'_{415} = 4.6 \times 10^4$ L mol⁻¹ cm⁻¹ and $\epsilon'_{400} = 2.5 \times 10^4$ L mol⁻¹ cm⁻¹, respectively. Beer's law was obeyed for Nb(V) concentrations ranging from 0.42 to 1.6 µg/mL (II) and 0.51 to 2.3 µg/mL (III).

Key words: niobium, liquid–liquid extraction, spectrophotometry, nitrocatecholates, tetrazolium salts

INTRODUCTION

Niobium and its compounds have a broad spectrum of applications. They are used for the fabrication of special materials for microelectronics and optics, superconductors, refractory materials, catalysts, and alloys [1–9]. As an important alloy element in steels, niobium can greatly affect the properties of a sample, such as the intensity at high temperature, the ability of tarnish resistance and temper brittleness [6–8]. Niobium steels are valuable constructive materials for the chemical and nuclear industries, jet and rocket engines, gas pipelines and turbines [6–9]. Unalloyed niobium and some niobium alloys are used for the production of surgical instruments, medical devices (e. g., pacemakers), coins and jewellery. The reason for these applications is niobium's physiological inertness and ability to be coloured by anodisation [6, 10].

In nature, niobium almost invariably occurs as complex oxide materials in association with tantalum. Columbite– tantalite minerals are most usually found as accessory minerals in pegmatite intrusions and in alkaline intrusive rocks [11–13]. It should be mentioned that niobium content in geological samples is of particular interest to geochemists [14–16]. This element remains immobile during post-crystallization rock alternation involving hydrous fluids, and the precise determination of ratios such as Nb/U and Nb/Th can give valuable information for the processes taking place during the generation of continental crust from the mantle [16].

Several analytical methods have been used to determine Nb, including spectrophotometry [6, 17–36], high-performance liquid chromatography (HPLC) [7, 14, 15, 37], atomic

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absorption spectrophotometry (AAS) [38], inductively coupled plasma (ICP) optical emission spectrometry (OES) [39], ICP mass spectrometry (MS) [40], neutron activation analysis (NAA) [41]. The main disadvantage of AAS and OES is low sensitivity (because of spectral interference or high background due to the matrix elements), whilst for ICP-MS and NAA it is high costs. The methods involving spectrophotometry are rather popular due to their simplicity, low-cost instrumentation and easy automation [29, 42]. These methods are often based on ion-association systems composed of intensively coloured anionic chelate and bulky organic cation which makes the complex insoluble in water and easily extractable into organic solvents [6, 29–36, 42–52].

In this paper, we study the formation and liquid–liquid extraction (LLE) of ternary ion-association complexes of niobium(V) with nitroderivatives of catechol {NDC: 4-ni-trocatechol (4-NC) and 3,5-dinitrocatechol (3,5-DNC)} and tetrazolium cations {which derive from 2,3,5-triphenyl-2-H-tetrazolium chloride (TTC), and 3,3'-[3,3'-dimetoxy(1,1'-biphenyl)–4,4'-diyl]-bis[2,5-diphenyl–2H-tetrazolium] dichloride (Blue Tetrazolium Chloride, BTC)} as the first step of using these complexes for LLE-spectrophotometric determination of Nb(V).

LLE systems involving tetrazolium salts (TS) and orthopolyphenols (including NDC) have been recently applied for the determination of Mo(VI) [44,45], W(VI) [46], V(V) [47,48] and V(IV/V) [49].

EXPERIMENTAL

Reagents and apparatuses

- A stock Nb(V) solution was prepared by dissolving 0.2352 g of Nb₂O₅ (Fluka, p. a.) in 10 mL conc. H₂SO₄ in the presence of 5 g (NH₄)₂SO₄ as described in [52]. After cooling, the obtained transparent mass was carefully collected into a 100 mL calibrated flask and diluted to the mark with 10 mol/L solution of H₂SO₄ [52]. A 1 mL aliquot of this solution was transferred into a 10 mL calibrated flask and diluted to the mark with 17 mol/L solution of H₂SO₄. The concentration of the obtained working solution was 164.4 µg/mL Nb(V).
- 4-NC (Fluka, p. a.), 1.0×10^{-2} mol/L aqueous solution.
- 3,5-DNC (synthesized as described in [53]), 1.0 × 10⁻² mol/L chloroform solution.

- Tetrazolium salts (TS): TTC (Fluka, p. a.) and BTC (Loba Feinchemie, for microscopy), 3 × 10⁻³ mol/L aqueous solutions.
- Chloroform, redistilled.
- A Camspec M508 UV/Vis spectrophotometer, equipped with 1 cm path-length cells.
- A HI 83140 pH meter (Italy) with a combined plastic electrode.
- A Thys 2 shaking machine (Germany).

Procedure

The necessary amounts of Nb(V), H_2SO_4 to adjust the pH of the aqueous medium, NDC (when water-soluble: 4-NC) and TS were placed in separatory funnels or extraction test tubes, diluted to 10 mL with distilled water and extracted with 10 mL of organic solvent (or 10 mL of 3,5-DNC chloroform solution and organic solvent mixture). The samples were shaken, and after phase separation the organic layer was filtered through a paper filter into a 1 cm cell. The absorbance was measured against a blank prepared in the same way.

RESULTS AND DISCUSSION

In sulphuric acid solutions, Nb(V) forms with NDC and TS yellow ion-association complexes which are easily extractable from water into organic solvents [32]. Preliminary investigations on the Nb(V)–4-NC–BTC and Nb(V)–3,5-DNC–TTC ternary systems showed that the formed complexes could be extracted into chloroform, 1,2-dichloroethane, benzene, toluene and tetrachloromethane. Chloroform was used for further investigations because of its better performance. The optimum extraction-spectrophotometric conditions are systematized in Table 1.

Figures 1 and 2 illustrate the saturation curves with NDC and TS at the optimum of other conditions. One can judge that niobium is extracted to the highest degree with a 13-fold excess of TTC and a 25-fold excess of 3,5-DNC (Fig. 1), a 4-fold excess of BTC and a 10-fold excess of 4-NC (Fig. 2). The saturation with TS is easily reached, but the complicated course of curve 2 (Fig. 2) is an indication for a stepwise complex formation in the Nb(V)–4-NC–BTC system.

The Nb(V) : NDC and Nb(V) : TS molar ratios were determined from data presented in Figs. 1 and 2 according

Extraction system Optimum conditions	Nb(V)–3,5-DNC–TTC	Nb(V)-4-NC-BTC	
Organic solvent	Chloroform	Chloroform	
Absorbance maximum	400 nm	415 nm	
Extraction time	5 min	3 min	
Acidity of aqueous phase	0.15–0.28 mol/L H ₂ SO ₄	0.08–0.15 mol/L H ₂ SO ₄	
Concentration of NDC	$5 \times 10^{-4} \text{ mol/L}$	1.2 × 10 ⁻³ mol/L	
Concentration of TS	$2.4 \times 10^{-4} \text{ mol/L}$	$1.0 imes 10^{-4} \text{ mol/L}$	

Table 1. Otimum extraction spectrophotometric conditions



to the methods of Asmus [54] and equilibrium shift [54]. The results show that Nb(V) forms one ternary complex with 3,5-DNC and TTC (with the composition of 1 : 2 : 2) and two ternary complexes with 4-NC and BTC (with the composition of 1 : 2 : 1 and 1 : 3 : 1.5). The suggested equations of complex formation and extraction, based on the mentioned molar ratios and data for the niobium state in sulphuric acid solutions [51, 55] are presented in Schemes 1–3.

$$NbO(OH)_{3} + 2H_{2}L \rightarrow [NbO(OH)(L)_{2}]^{2-} + 2H_{2}O + 2H^{+} (1)$$
$$[NbO(OH)(L)_{2}]^{2-} + 2TT^{+} \leftrightarrow (TT^{+})_{2}[NbO(OH)(L)_{2}] (2)$$
$$\{(TT^{+})_{2}[NbO(OH)(L)_{2}]\}_{aq} \leftrightarrow \{(TT^{+})_{2}[NbO(OH)(L)_{2}]\}_{org} (3)$$

Scheme 1. Formation and extraction of the ternary complex of Nb(V) with 3,5-DNC (H,L) and TTC

$NbO(OH)_{3} + 2H_{2}L \rightarrow [NbO(OH)(L)_{2}]^{2-} + 2H_{2}O + 2H^{+}$	(4)
$[NbO(OH)(L)_2]^{2-} + BT^{2+} \leftrightarrow (BT^{2+})[NbO(OH)(L)_2]$	(5)
$\{(BT^{2+})[NbO(OH)(L)_2]\}_{aq} \leftrightarrow \{(BT^{2+})[NbO(OH)(L)_2]\}_{org}$	(6)

Scheme 2. Formation and extraction of the ternary complex of Nb(V) with 4-NC (H,L) and BTC at deficiency of the reagents

$NbO(OH)_3 + 3H_2L \rightarrow [NbO(L)_3]^{3-} + 3H_2O + 3H^+$	(7)
$2[NbO(L)_3]^{3-} + 3BT^{2+} \leftrightarrow (BT^{2+})_3[NbO(L)_3]_2$	(8)
$\{(BT^{2+})_3[NbO(L)_3]_2\}_{aq} \leftrightarrow \{(BT^{2+})_3[NbO(L)_3]_2\}_{org}$	(9)

Scheme 3. Formation and extraction of the ternary complex of Nb(V) with 4-NC (H,L) and BTC at the optimum extraction spectrophotometric conditions

Constants	System	Nb(V)–3,5-DNC–TTC	Nb(V)–4-NC–BTC
Log β		8.9 ± 0.8	7.8 ± 0.8
Log K _D		1.02 ± 0.01	0.538 ± 0.008
Log K _{Ex}		9.9 ± 0.8	8.3 ± 0.8
Recovery		(91.1 ± 0.1)%	(77.3 ± 0.1)%





Fig. 3. Determination of the constant of association (β) and true molar absorptivities ($\epsilon = 1/b$) by the Komar–Tolmatchev method [54] The following straight line equations (Y = aX + b) were obtained: $1 - Y = 2.379 \times 10^{-5}X + 3.974 \times 10^{-5}$; $2 - Y = 1.541 \times 10^{-5}X + 2.085 \times 10^{-5}$. The constants required for the quantitative assessment of the extraction equilibria (Table 2) were calculated as follows: the association constant β , characterizing the equilibriums 2 and 8, by the Komar–Tolmachev method [54]; the distribution constant K_p , characterizing the equilibriums 3 and 9, by comparing the absorption for single extraction in the optimum conditions (A₁) to that for triple extraction (A₃) in equal volumes (25 mL) $K_D = [Nb]_{org} / [Nb]_{aq} = A_1 / (A_3 - A_1)$; and the recovery factor R and extraction constant K_{Ex} from the equations R% = $K_D 100 / (K_D + 1)$ and $LogK_{Ex} = LogK_D + Log\beta$, respectively [44, 46–48, 56].

The Komar–Tolmachev method allows also to calculate the true molar absorptivities (ε) of the complexes (Fig. 3, $\varepsilon = 1/b$): $\varepsilon = (2.5 \pm 0.17) \times 10^4$ L mol⁻¹ cm⁻¹ {for (TT⁺)₂[NbO(OH) (3,5–DNC)₂]} and $\varepsilon = (4.8 \pm 0.3) \times 10^4$ L mol⁻¹ cm⁻¹ {for (BT²⁺)₃[NbO(4-NC)₃]₂}. The mentioned values agree very well with these obtained from Beer's law (ε ') (Table 3). This is an indication for the correctness of investigations and absence of serious side-reactions [54]. Only one complex is extracted in the Nb–4-NC–BTC system in the optimum operation conditions (Scheme 3). The NDC to Nb molar ratio in this complex (3 : 1) is higher than that reported for other complexes with similar reagents (Table 4). The significantly higher molar absorptivity of (BT²⁺)₃[NbO(4-NC)₃]₂ with respect to the complex of Nb(V) with 4-NC and tetrazolium violet [32] (Table 4, 3rd row) could be attributed to this fact.

Table 3. Analytical characteristics

System	Nb(V)–3,5-DNC–TTC	Nb(V)–4-NC–BTC	
Apparent molar absorptivity (ϵ')	$2.48 \times 10^4 L mol^{-1} cm^{-1}$	$4.6 \times 10^4 L mol^{-1} cm^{-1}$	
Adherence to Beer's law	0.51–2.3 μg/mL Nb(V)	0.42–1.6 μg/mL Nb(V)	
Coefficient of correlation	0.9987	0.9986	
Sandell's sensitivity	0.0038 μg/cm ²	0.0020 μg/cm ²	
Limit of detection (LOD)	0.15 μg/mL Nb(V)	0.13 μg/mL Nb(V)	
Limit of quantification (LOQ)	0.51 μg/mL Nb(V)	0.42 μg/mL Nb(V)	

Table 4. Extraction-chromogenic systems for niobium involving ortho-polyphenols and tetrazolium salts

Reagents	Solvent	Composition Nb : R ₁ : R ₂	Molar abs. (ɛ′) [L mol⁻¹ cm⁻¹]	Ref.
Catechol + tetrazolium violet	CHCl₃	1:2:1	0.57×10^4	31
Dihydroxynephtalene + tetrazolium violet	CHCl₃	1:2:1	1.18×10^4	31
4-NC + tetrazolium violet	$C_2H_4Cl_2$	1:2:1	1.59×10^{4}	32
Pyrogallol + tetrazolium violet	$C_2H_4Cl_2$	1:2:1	0.649×10^4	32
Catechol + TTC	CHCl₃	1:2:1	$1.60 imes 10^4$	50
3,5-DNC + TTC	CHCl₃	1:2:2	$\textbf{2.48}\times \textbf{10}^{4}$	This work
4-NC + BTC	CHCl ₃	1:3:1.5	4.6×10^4	This work

It should be mentioned that 4-NC and BTC allow a more sensitive determination of niobium in comparison to many other reagents, namely 5,7-diiodo-8-hydroxyquino-line (4.853 × 10³ L mol⁻¹ cm⁻¹) [23], p-sulphobenzenoazo-4-(2,3-dihydroxypyridine) (5.218 × 10³ L mol⁻¹ cm⁻¹) [24], sulphochlorophenol S + laurylamine (1.8×10^4 L mol⁻¹ cm⁻¹) [25], thiocyanate + tetraphenylarsonium chloride (or tetraphenylphosphonium chloride) (3.3×10^4 L mol⁻¹ cm⁻¹) [17], Xylenol Orange + 3-hydroxy-2-methyl-1-phenyl-4-pyridone (3.72×10^4 L mol⁻¹ cm⁻¹) [18], PAR (3.76×10^4 L mol⁻¹ cm⁻¹) [19], 3-hydroxyflavone (4.088×10^4 L mol⁻¹ cm⁻¹) [20], and tiazolylazo compounds + cetylpyridinum bromide ((2.79-4.46) × 10⁴ L mol⁻¹ cm⁻¹) [27].

Some other characteristics concerning the applicability of NDC and TS as LLE-spectrophotometric reagents for niobium are presented in Table 3.

CONCLUSIONS

1. Nb(V) forms well chloroform-extractable ternary ionassociation complexes with NDC (4-NC, 3,5-DNC) and TS (TTC, BTC). The anionic part of the complexes ensures intensive yellow coloration, and the bulkiness of the cationic part, in its turn, guarantees their poor solubility in water.

2. In the optimum extraction conditions, niobium(V) forms a 1 : 2 : 2 complex with 3,5-DNC and TT⁺ {suggested formula $(TT^+)_2[NbO(OH)(3,5-DNC)_2]$ } and a 1 : 3 : 1.5 complex with 4-NC and BT²⁺ {suggested formula $(BT^{2+})_3[NbO(4-NC)_3]_2$ }.

3. The complex involving 4-NC and BTC has some better characteristics than those with 3,5-DNC and TTC, namely a 1.8 times higher molar absorptivity, 40%-shorter extraction time, 2.4 times lower TS consumption, availability (no need of synthesis) and water-solubility of NDC.

4. The couple 4-NC and BTC could compete successfully with many reagents for the spectrophotometric and LLEspectrophotometric determination of niobium(V).

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TRIGUBI NIOBIO(V) KOMPLEKSAI SU KATECHOLIO NITRODARINIAIS IR TETRAZOLIO DRUSKOMIS. EKSTRAKCINIAI-SPEKTROFOTOMETRINIAI TYRIMAI

Santrauka

Buvo tiriamas naujų niobio(V) anijoninių chelatinių kompleksų su katecholio nitrodariniais ir tetrazolio katijonais susidarymas ir ekstrakcija tirpikliu. Nustatytos optimalios gautų kompleksų ekstrakcijos sąlygos ir kai kurios konstantos. Ištirtos kompleksų spektrofotometrinės savybės.