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Binuclear Oxidovanadium(IV) Complex with the Bridging Chloranilate Ligand: Synthesis and Magnetic Properties

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Abstract—The binuclear oxidovanadium(IV) complex [VO(Dbbpy)Cl(Ca)Cl(Dbbpy)VO] (**I**) is synthesized by the reaction of [VO(Dbbpy)(H_2O)Cl_2] (Dbbpy is 4,4'-di-*tert*-butyl-2,2'-bipyridyl) with chloranilic acid H_2Ca in acetonitrile in the presence of Et_3N in a yield of 79%. Complex **I** is reduced at $E_{1/2} = -842$ mV (vs. Ag/AgCl), which is shown by cyclic voltammetry for a solid sample using the paste electrode. The EPR spectra and magnetochemical measurements for complex **I** confirm the existence of two paramagnetic vanadium(IV) centers with the total spin $S = 1$ and the antiferromagnetic character of the exchange interaction between the centers.

Keywords: oxidovanadium(IV), chloranilic acid, magnetic susceptibility, voltammetry, EPR spectroscopy
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INTRODUCTION

Coordination compounds of vanadium(IV) with the stable VO^{2+} group compose a wide class of inorganic compounds including thousands examples [1–4]. The studies in this area are presently focused mainly on the biological and catalytic activities of these compounds. These properties are determining for the vanadium compounds as a whole. Catalytic transformations involve most frequently the changes in the oxidation state of vanadium, which is associated with the variety of its available oxidation states. It is known that vanadyl moieties are present in the living nature and are found in the active centers of some enzymes, in particular, haloperoxidases that catalyze the oxidation of halogen-containing compounds by hydrogen peroxide [5–9] and vanadium-dependent nitrogenases involved in nitrogen fixation processes [10, 11]. The oxidovanadium complexes catalyze the oxidation [12–19], oxidative halogenation [20–25], and sulfoxidation of many organic compounds [2, 3, 26–40]. Some vanadyl complexes with the N- and O-donor ligands exhibit an insulin-like effect and anticancer activity and are studied for the possible application in medicine [41, 42].

Another attractive feature of the oxidovanadium(IV) complexes with the d^1 configuration of the central atom is paramagnetism. The mononuclear vanadyl complexes usually behave as ideal paramagnetics in which the paramagnetic centers are isolated and do not interact with each other [43, 44]. Intra- and intermolecular interactions of the antiferromagnetic and ferromagnetic character can occur in the bi- and polynuclear compounds due to either the bridging groups bonding two paramagnetic vanadyl fragments (binuclear complexes) [45–51], or the VO···VO··· interactions between the vanadyl groups of the adjacent molecules (polymer chain compounds) [52–54]. The O-donor ligands, for example, hydroxide, sulfate, and carboxylate, act most frequently as the bridging groups capable of transmitting magnetic interactions in the binuclear compounds. Particularly, in the compounds with the $\{VO-(\mu\text{-}OH)_2-VO\}^{2+}$ core, antiferromagnetic and ferromagnetic exchange interactions can occur, depending on the geometry of the $\{VO-(\mu\text{-}OH)_2-VO\}^{2+}$ fragment [55, 56].

In this work, we report on the synthesis of the new binuclear vanadium(IV) complex [VO(Dbbpy)Cl(μ-Ca)Cl(Dbbpy)VO] (**I**, where Dbbpy is 4,4'-di-*tert*-butyl-2,2'-bipyridyl) in which two vanadyl fragments are linked by the bridging chloranilate ligand. The redox and magnetic properties of this compound are also discussed.

EXPERIMENTAL

Vanadium trichloride VCl_3 (97%, Merck), chloranilic acid H_2Ca (98%, Merck), and 4,4'-di-*tert*-butyl-2,2'-bipyridyl Dbbpy (98%, Merck) were used. The

solvent CH_3CN was distilled over P_2O_5. All experiments were carried out in air.

Synthesis of complex I. H_2Ca (37 mg, 177 μmol) was dissolved in CH_3CN (20 mL). Triethylamine Et_3N (50 μL, 354 μmol) was added to the obtained solution, after which the color of the solution changed from orange to crimson. This solution was mixed with a solution containing the $[VO(Dbbpy)(H_2O)Cl_2]$ complex (150 mg, 354 μmol) in CH_3CN (10 mL). After mixing, the solution turned brown. The solution was stirred for 10 h, and a weakly colored solution with a brown-green precipitate was formed. The precipitate was filtered off and washed with CH_3CN and Et_2O. The yield was 132 mg (79%).

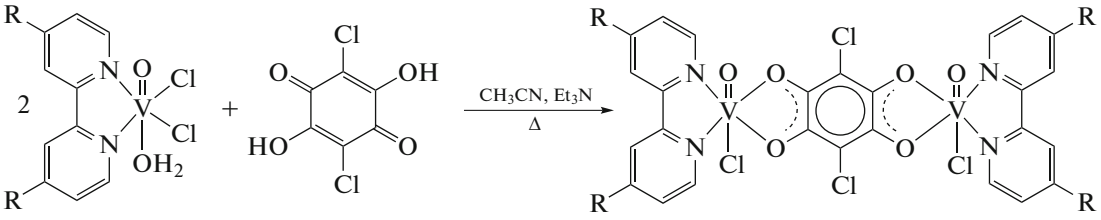
For $C_{42}H_{48}N_4O_6Cl_4V_2$			
Anal. calcd., %	C, 53.3	N, 5.9	H, 5.1
Found, %	C, 53.3	N, 5.6	H, 5.6

IR (KBr; ν, cm^{-1}): 3446 w, 3085 w, 2903 w, 2875 w, 1616 s, 1544 vs, 1487 w, 1406 s, 1373 s, 1306 m, 1288 m, 1249 m, 1202 w, 1119 w, 1037 m, 977 vs, 899 m, 852 s, 750 w, 740 w, 721 w, 624 w, 607 m, 575 w, 557 w, 487 m.

Elemental analyses to C, H, and N were carried out on a Euro EA 3000 Eurovector analyzer. The IR spectrum was recorded on a Scimitar FTS 2000 spectrometer in the 4000–400 cm^{-1} range (pellets with KBr). EPR spectra were measured on a Varian E-109 EPR spectrometer in the X frequency range at 77 and 300 K. The EPR spectrum of DPPH (2,2'-diphenyl-1-picrylhydrazyl) with $g = 2.0036$ was used as a reference for the g factor. The magnetic properties were studied on a Quantum Design PPMS-9 magnetometer, which makes it possible to measure the static magnetic susceptibility in a range of 1.8–350 K in the magnetic fields with the accuracy to ±9 T. The paramagnetic components of the magnetic susceptibility (χ) were determined taking into account the diamagnetic contribution of the sample estimated from Pascal's constants and the contribution of the sample holder. Electrochemical measurements were conducted using a P-30J Elins potentiostat-galvanostat (Russia) at room temperature in a three-electrode cell including the paste electrode, the reference electrode (saturated silver chloride), and the auxiliary platinum electrode. A 0.10 M solution of Bu_4NPF_6 in CH_3CN was prepared for the measurements. Prior to the measurements, the solution was purged with argon. The scan rate was 100 mV/s. A carbon powder, the vanadium compound, and polymethylsiloxane PMS-200 in a ratio of 100 : 14 : 10 (wt/wt/wt) were used for the preparation of the paste electrode. The components were triturated in an agate mortar. The edge of the carbon electrode was filled with the paste obtained after mixing. The electrode was a Teflon tube with an inserted carbon rod tightly matched from the sides to the size of the tube. The rod was connected by a steel current lead screwed into the upper part of the electrode. The paste was deposited onto the electrode edge to a depth of 0.5–1.0 mm and packed on the silicate glass surface [57].

RESULTS AND DISCUSSION

Binuclear complex **I** was synthesized by the reaction of $[VO(Dbbpy)(H_2O)Cl_2]$ (**II**) with H_2Ca in acetonitrile in the presence of Et_3N in a yield of 79% (Scheme 1).



(I)

Scheme 1.

The initial mononuclear complex **II** was synthesized by the reaction of VCl_3 with Dbbpy in acetonitrile in the presence of air oxygen, and its structure was confirmed by X-ray diffraction analysis. This approach was successfully used by us for the preparation of a series of the oxidovanadium(IV) complexes with heterocyclic diimines [43, 44, 58]. The IR spectrum of complex **I** exhibits the characteristic bands caused by vibrations of the vanadyl group (977 cm^{-1}) and bipyridyl rings (1618–1034 cm^{-1}). In addition, the intense bands characteristic of the compounds with the bridging chloranilate ion were observed at 1544 and 1373 cm^{-1} [45, 59–63].

The resembling binuclear complex [VO(Bpy)Cl(Ca)Cl(Bpy)VO] (**III**) containing 2,2'-bipyridyl (Bpy) instead of Dbbpy was described. This complex is formed by the reaction of *cis*-$[VO(Bpy)_2Cl]Cl \cdot 2H_2O$ with tetrachlorocatechol H_2TCC in the presence of Bu_4NOH or by the reaction of *cis*-$[VO(Bpy)_2Cl]Cl$ with [(Bpy)ClVO(TCSQ)]

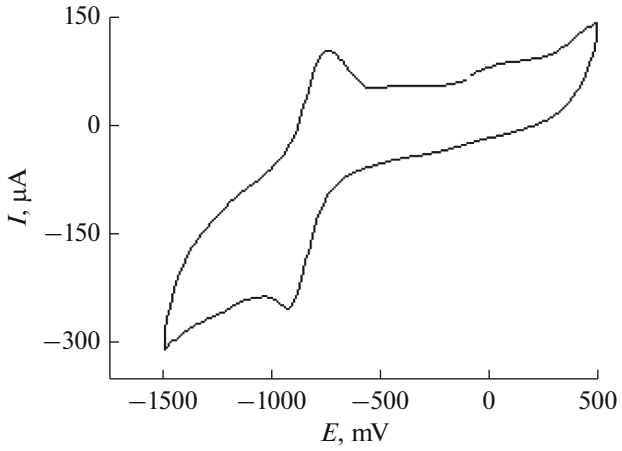


Fig. 1. Cyclic voltammogram of a solid sample of compound **I** (0.1 M solution of Bu_4NPF_6 in CH_3CN as the supporting electrolyte, scan rate 100 mV/s).

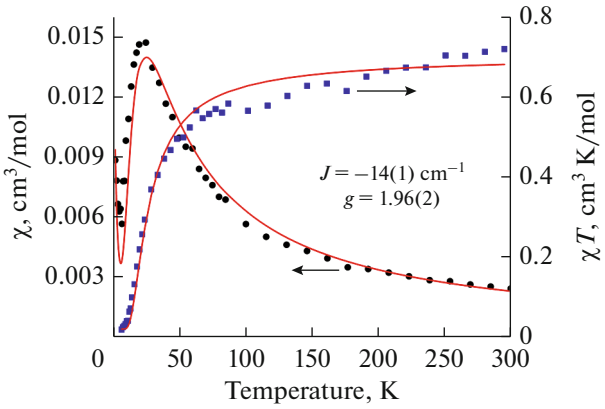


Fig. 2. Temperature dependences (●) χ and (■) χT for complex **I** in an external magnetic field of 5 kOe. Lines are the theoretical dependences with the parameters $J = -14(1)$ cm^{-1} and $g = 1.96(1)$.

(TCSQ is tetrachlorosemiquinone) also in the presence of Bu_4NOH in yields of 39 and 25%, respectively. In these reactions, Cl^- is substituted by OH^- in the tetrachlorocatechol ring to form the chloranilate ion linking two vanadyl fragments $[VO(Bpy)Cl]^+$ into the binuclear structure [45]. The direct method developed in [45] (chloranilic acid was immediately introduced in the reaction as the reactant) results in the twofold increase in the yield of the binuclear complex.

Compound **I** is poorly soluble in many organic solvents, which impedes its characterization by other methods, in particular, by X-ray diffraction analysis. Based on the data of the IR and EPR spectra and the results of elemental analysis and magnetochemical measurements, we believe that complex **I** has the same structure as the structurally characterized complex **III** in which two paramagnetic fragments are bound by the chloranilate ligand.

The redox properties of a solid sample of complex **I** were studied by cyclic voltammetry (CV) using the paste electrode. The CV curve in the range from −1500 to 500 mV is presented in Fig. 1. The negative range exhibits a redox process for which the peak potentials are $E_a = -740$ mV and $E_c = -920$ mV and the half-wave potential is $E_{1/2} = -830$ mV. The difference between the anodic and cathodic peaks is 180 mV, which significantly exceeds the value (59 mV) characteristic of the reversible one-electron process. Thus, the observed reduction process is quasi-reversible and, probably, is attributed to the V(IV)/V(III) pair. The participation of the redox-active bipyridyl ligand in this process cannot be excluded, since its properties are noninnocent [64–68]. The quasi-reversible reduction (V(IV)/V(III)) in the range from −0.60 to −0.73 V vs. calomel electrode was also observed for the dialkyldithiocarbamate vanadyl complexes $V_2O_2S_2(R_2Dtc)$ with the binuclear structure [69, 70]. The tetranuclear compounds ($[Li_2(VO)_2(\mu\text{-}Piv)_6(Bpy)_2]$ and $[Li_2(VO)_2(\mu\text{-}Tfac)_6(Bpy)_2]$ undergo reduction of the V(IV)/V(III) type at the potential about −1 V vs. Ag/AgCl/KCl [47]. As a whole, metallocentered reduction processes are characteristic of the oxidovanadium(IV) complexes [1].

The measurements of the magnetic properties of complex **I** in a range of 2–300 K showed that at 300 K $\chi T = 0.72$ cm^3 K/mol, which is insignificantly lower than the theoretical value for two noninteracting magnetic ions with the spin $S = 1/2$ (0.76 cm^3 K/mol). A decrease in temperature results in a monotonic decrease in χT to a minimal value of 0.02 cm^3 K/mol at 2 K (Fig. 2). This magnetic behavior is due to the antiferromagnetic interactions between the paramagnetic V(IV) ions ($S = 1/2$) of one molecule with the total spin of the ground state $S = 1$. To determine the parameter of the exchange interaction (J) between the vanadium ions, the obtained data on $\chi T(T)$ were approximated by the Bleaney–Bowers equation using the PHI program [71]. The best approximation of the theoretical dependence to the experimental data (solid lines in Fig. 2) was obtained for the parameters $J = -14(1)$ cm^{-1} and $g = 1.96(2)$. The observed magnetic behavior of complex **I** is consistent with the literature data obtained for complex **III**. The values of χT are 0.71 and 0.01 cm^3 K/mol at 300 and 5 K, respectively, and the parameters are $J = -22(1)$ cm^{-1} and $g = 1.97(2)$ [45]. Note that the chloranilate bridging ligand is efficient for the exchange antiferromagnetic interactions between two paramagnetic V(IV) centers remote even at long distances (8.153 Å). The antiferromagnetic exchange with the parameter $J = -14.8(4)$ cm^{-1} was also observed in $[(VO)_2(\mu_2\text{-}SO_4)_2\text{-}(Dbbpy)_2\text{-}(CH_3OH)_2] \cdot 4CH_3OH$ [46].

The EPR spectrum of a polycrystalline sample of complex **I** at 77 K represents a single line with $g =$

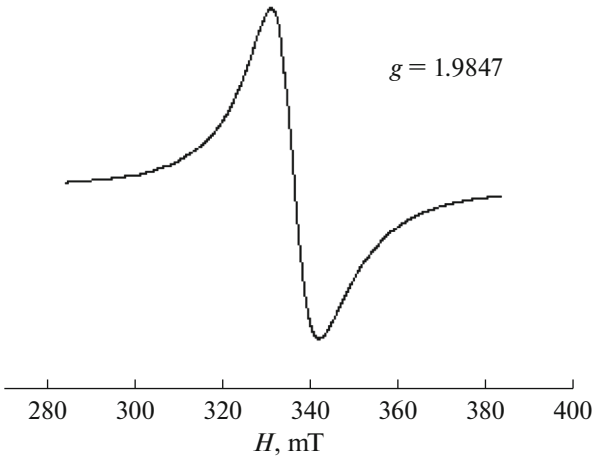


Fig. 3. EPR spectrum of a solid sample of complex **I** at 77 K.

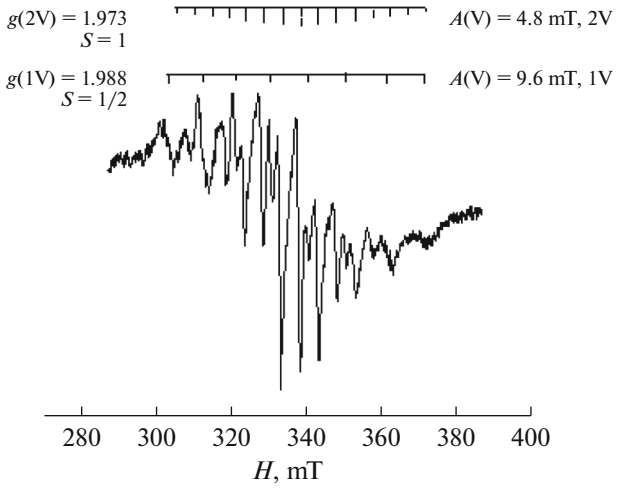


Fig. 4. EPR spectrum of complex **I** in CH_2Cl_2 at 300 K.

1.9847 and ΔH = 20.0 mT (Fig. 3). The spectrum is typical of magnetically concentrated samples and can be due to the exchange interactions between the V(IV) atoms both inside the dimers and between them.

As mentioned above, compound **I** is poorly soluble in organic solvents. A weakly colored solution with a low concentration of complex **I** was obtained by its prolong stirring in CH_2Cl_2. The complicated EPR spectrum of this solution at room temperature is caused by the superposition of the spectra of the compounds with one and two V(IV) centers. The spectrum of one of them exhibits the hyperfine structure (HFS) from one V(IV) ($I = 7/2$ for ^{51}V) with the spin $S = 1/2$ and HFS constant A(V) = 9.6 mT. These values are typical of the compounds with one vanadyl center [43, 44, 72]. The second spectrum has the HFS from two equivalent V(IV) atoms with the halved HFS constant ($A = 4.8$ mT) (Fig. 4). It can be concluded that the second EPR spectrum is due to the formation of a dimer with $S = 1$; i.e., by the interaction of two lone electrons of two vanadyl fragments. This agrees with the data of magnetic measurements for complex **I**. The presence in the solution of one more paramagnetic species with $S = 1/2$ and the HFS characteristic of one V(IV) atom can be explained by the oxidation of one vanadium atom in complex **I** due to its prolong dissolution in air and the formation of the binuclear V(IV)–V(V) system.

Thus, the new vanadium(IV) complex bearing two vanadyl fragments $[VO(Dbbpy)Cl]^+$ linked by the bridging chloranilate ligand into the binuclear structure [VO(Dbbpy)Cl(Ca)Cl(Dbbpy)VO] (**I**) was synthesized. The cyclic voltammogram of compound **I** (for a solid sample) exhibits the quasi-reversible electron transfer attributed, probably, to the V(IV)/V(III) pair. The paramagnetic nature of compound **I** was proved by EPR spectroscopy and magnetic measurements. The antiferromagnetic exchange between the paramagnetic V(IV) centers of the dimeric molecule was found.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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