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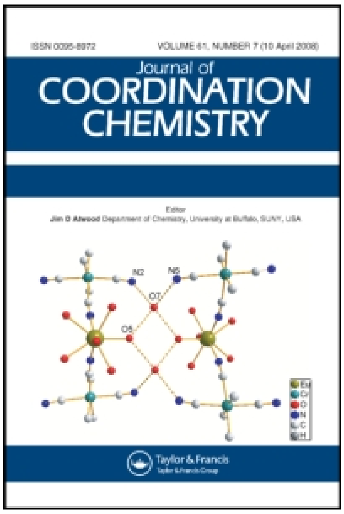

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Synthesis and characterization of a new *vic* -dioxime 6,7- *bis* (hydroxyimino)-9,10-diethylidine-5,8,9,10,11,18-hexahydro-5,8,11,18-tetraazadibenzo[a,e]cyclotetradecane-6,7,12,17-tetraone and its nickel and cobalt complexes

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Synthesis and characterization of a new *vic*-dioxime 6,7-*bis*(hydroxyimino)-9,10-diethylidine-5,8,9,10,11,18-hexahydro-5,8,11,18-tetraazadibenzo[a,e]cyclotetradecane-6,7,12,17-tetraone and its nickel and cobalt complexes

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A new *vic*-dioxime 6,7-*bis*(hydroxyimino)-9,10-diethylidine-5,8,9,10,11,18-hexahydro-5,8,11,18-tetraazadibenzo[a,e]cyclotetradecane-6,7,12,17-tetraone (**H_2L**) and its hydrogen-bridged tetra- and six-coordinate complexes with Ni(II), Co(II), and Co(III) have been synthesized. The six-coordinate complexes of **H_2L** have pyridine and chloride as axial ligands. Hydrogen-bridge complexes were converted to their BF_2-bridged analogues by reaction with boron trifluoride etherate. Structures of the **H_2L** and its complexes were proposed from elemental analysis, ^1H and ^{13}C NMR, IR and mass spectra.

Keywords: vic-Dioxime; Nickel complexes; Cobalt complexes; Hydrogen-bridge complexes; BF2-bridge complexes

1. Introduction

The reactions of amines or thioles with dichloroglyoxime or cyanogen di-N-oxide give various symmetrically substituted diaminoglyoxime derivatives with some donors towards transition metal ions [1–4]. Potential applications of *vic*-dioximes in medicine [5, 6], bioorganic systems [7], catalysts [8, 9], analytical reagents [10], electrochemical, and electrooptical sensors [11, 12] increase interest. The Tc(V) and Cu(II) complexes of *vic*-dioximes are used as cerebral and myocardial perfusion imaging agents [13–18].

Macrocyclic dioxime complexes of Co(III) in which bridging protons of the *vic*-dioxime are replaced by BF_2^+, stabilizes the macrocyclic structure while removing acidic protons from the O binding sites [19–21]. We report herein the synthesis and characterization of a *vic*-dioxime and its complexes with nickel and cobalt. Nickel and cobalt complexes containing BF_2^+-bridges were also synthesized.

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2. Experimental

2.1. *Materials and measurements*

All reagents were obtained from *Merck* and *Fluka* and used without purification. Melting points were measured with Electrothermal 9200 apparatus and are uncorrected. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer; metal content was determined with a Shimadzu AA-6650 atomic absorption spectrometer in solution prepared by decomposition of the compounds in aqua regia followed by dilution with water. Mass spectra were recorded on a Shimadzu QD 5050 and MALDI-TOF mass spectrometer Bruker Biflex III. ^1H and ^{13}C NMR spectra were measured (DMSO-d_6 as solvents) with a Bruker DRX-500 Avance spectrometer at 500.13 and 125.8 MHz, respectively. The IR spectra were recorded with a Shimadzu 8400 S spectrometer.

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2.2. *Synthesis of H_2L*

To a stirring solution of N,N′-*bis*(2-aminophenyl)phethalimide (**1**) (3.46 g, 10 mmol) in methanol (100 mL) a solution of dichloroglyoxime (1.57 g, 10 mmol) in methanol (30 mL) was added dropwise at 25°C. The mixture was stirred at room temperature for 2 days, and then the solvent was removed by evaporation. The residue was washed with cold ethylacetate, diethylether, and dried in vacuum.

Yield: 3.20 g (74%); m.p. 182–184°C. Color: brown. IR (KBr): υ_{max} (cm^{-1}): 3475–3371(OH), 3413(NH), 3292(NH), 3054(Ar–H), 1652(C=O), 1639(C=N), 1608–1500 (C=C), 983(N–O). ^1H NMR: δ 11.88(s, 2H, OH), 9.59(s, 2H, NH), 7.80(s, 2H, NH), 7.90–7.14(m, 12H, C_6H_4. ^{13}C NMR: δ 168.87, 166.95, 152.77, 145.24, 143.78, 143.44, 133.59, 132.46, 130.81, 130.20, 129.59, 129.10, 128.63, 128.06, 125.35, 124.89, 124.52, 123.54, 122.69, 122.37, 121.46, 120.48. The EI-MS, *m*/*z*: 429$(M-1)^+$, 368, 352, 296, 220. Anal. Calcd for $C_{22}H_{18}N_6O_4$: C, 61.39; H, 4.22; N, 19.53. Found: C, 61.34; H, 4.20; N, 19.50.

2.3. *Synthesis of $[Ni(LH)_2]$ (3)*

A solution of $NiCl_2 \cdot 6H_2O$ (0.059 g, 0.25 mmol) in methanol (15 mL) was added dropwise with stirring to a solution of $\mathbf{H_2L}$ (0.22 mg, 0.50 mmol) in methanol (25 mL) at 40°C during 30 min. The color of the solution immediately changed and the pH of the solution decreased to about 1. The resulting mixture was stirred at 60°C for 2 h. An equivalent amount of triethylamine (0.1 M) was added to increase the pH to 6–6.5. The mixture was refluxed for 2 h and then the brown solution was evaporated to 20 mL under reduced pressure. The mixture was filtered and the orange precipitate washed with water, ethanol, and acetone, and dried.

Yield: 0.15 g (64%); m.p. 218°C. Color: orange. IR (KBr) υ_{max} (cm^{-1}): 3319, 3296(NH), 3060(Ar–H), 1772(O–H $\cdots$ O), 1653(C=O), 1648(C=N), 1602–1585 (C=C), 1054(N–O). ^1H NMR ppm: 16.01(s, 2H, O–H $\cdots$ O), 8.33(s, 4H, NH), 8.32(s, 4H, NH), 8.41, 8.40(dd, $^3J = 4.85$ Hz, 4H), 7.78, 7.76(dd, $^3J = 1.68$ Hz, 4H), 7.36(dd, $^3J = 1.62$ Hz, 4H), 7.40–7.33(m, 8H), 7.14, 7.12(dd, $^3J = 2.05$ Hz, 8H), ^{13}C NMR ppm: 168.57, 163.01, 145.57, 140.97, 130.95, 128.80, 128.39, 127.56, 126.74, 122.92, 122.59. The EI-MS, *m*/*z*: 918 $(M+1)^+$, 917$(M)^+$, 916$(M-1)^+$. Anal. Calcd for $C_{44}H_{34}N_{12}NiO_8$: C, 57.60; H, 3.74; N, 18.32; Ni, 6.40. Found: C, 57.55; H, 3.67; N, 18.30; Ni, 6.36.

2.4. *Synthesis of [Co(LH)$_2$] (4)*

A solution of $CoCl_2 \cdot 6H_2O$ (0.59 g, 0.25 mmol) in methanol (15 mL) was added dropwise with stirring to a solution of **H_2L** (0.22 mg, 0.50 mmol) in methanol (25 mL) at 40°C during 30 min. The color of the solution immediately changed and the pH decreased to about 1. The resulting mixture was stirred at 60°C for 2 h and an equivalent of triethylamine (0.1 M) was added to increase the pH to 6–6.5. The mixture was refluxed for 2 h and the brown solution was evaporated to 20 mL under reduced pressure. The mixture was filtered and the black precipitate washed with water, ethanol, and acetone, and dried.

Yield: 0.17 g (75%); m.p. 212°C. Color: black. IR (KBr) υ_{max} (cm^{-1}): 3352, 3250(NH), 3136(Ar–H), 1708(O–H $\cdots$ O), 1652(C=O), 1643(C=N), 1604–1558(C=C), 1076(N–O). The EI-MS, *m*/*z*: 919 $(M+2)^+$. Anal. Calcd for $C_{44}H_{34}CoN_{12}O_8$: C, 57.58; H, 3.73; N, 18.31; Co, 6.42. Found: C, 57.48; H, 3.66; N, 18.23; Co, 9.34.

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2.5. *Synthesis of [Ni(LH)$_2$PyCl] (5)*

A solution of $NiCl_2 \cdot 6H_2O$ (0.088 g, 0.37 mmol) in methanol (15 mL) was added dropwise with stirring to a solution of **H_2L** (0.32 g, 0.74 mmol) in methanol (40 mL) at 40°C during 30 min. The color immediately changed and pH of the solution decreased to about 1. By addition of NaOH (0.1 M) the pH increases to 6. The resulting mixture was stirred at 60°C for 30 min. Pyridine (0.029 g, 0.37 mmol) in methanol (2 mL) was added to the reaction mixture at 60°C. The mixture was refluxed for 2 h, then the brown solution was evaporated to 20 mL under reduced pressure. The reaction mixture was filtered and the brown precipitate washed with methanol, ethanol, and acetone, and dried.

Yield: 0.24 g (62%); m.p. 225°C. Color: brown. IR (KBr) υ_{max} (cm^{-1}): 3346, 3274(NH), 3152–3072(Ar–H), 1710(O–H $\cdots$ O), 1645(C=O), 1618(C=N), 1604–1450 (C=C), 1070(N–O), 1041(N–O), 750(Ni–Cl). HRMS: *m*/*z*: Calcd for $C_{49}H_{39}ClN_{13}NaNiO_8$: 1053.21; Found: 1054.20. Anal. Calcd: C, 57.78; H, 3.73; N, 17.26; Ni, 5.56. Found: C, 57.69; H, 3.68; N, 17.16; Ni, 5.47.

2.6. *Synthesis of [Co(LH)$_2$PyCl] (6)*

A solution of $CoCl_2 \cdot 6H_2O$ (0.088 g, 0.37 mmol) in methanol (15 mL) was added to a solution of **H_2L** (0.32 g, 0.74 mmol) in methanol (40 mL) at 60°C while bubbling oxygen into the solution. The color of the solution immediately changed and pH decreased to 1. The resulting mixture was stirred at 60°C for 30 min and pyridine (0.029 g, 0.37 mmol) in methanol (5 mL) was added. The mixture was heated and stirred for 6 h with oxygen passing through it. The total volume of the brown solution was decreased to 20 mL under reduced pressure, filtered, and the brown precipitate washed with methanol, ethanol, and acetone, and dried.

Yield: 0.28 g (72%); m.p. 222°C. Color: brown. IR (KBr) $\upsilon_{\max}$ (cm^{-1}): 3357, 3274(NH), 3107–3056(Ar–H), 1712(O–H···O), 1645(C=O), 1619(C=N), 1600–1558 (C=C), 1072(N–O), 1043(N–O), 746(Co–Cl). ^1H NMR ppm: 15.54(s, 2H, O–H···O), 9.23(s, 4H, NH), 7.90(s, 4H, NH), 7.73–7.30(m, 18H). HRMS: *m*/*z*: Calcd for $C_{49}H_{39}ClCoN_{13}iO_8$: 1031.21; Found: 1031.41. Anal. Calcd: C, 57.01; H, 3.81; N, 17.64; Co, 5.71. Found: C, 56.90; H, 3.68; N, 17.56; Co, 5.67.

2.7. *Synthesis of [Ni(LBF$_2$)$_2$PyCl] (7)*

A suspension of **5** (0.21 g, 0.2 mmol), in methanol (25 mL) was refluxed under argon. Boron-trifluoride ethyl etherate (0.1 mL, 0.4 mmol) was added with stirring converting to a red solution. By addition of NaOH (0.1 M) the pH increases to 6. The solution was refluxed for 5 h and then allowed to cool to room temperature. The solvent was concentrated to 10 mL under reduced pressure, filtered and the precipitate washed with distilled water, acetonitrile, ethanol, and diethyl ether, and dried in vacuum.

Yield: 0.1 g (43%), m.p. 257°C. Color: orange. IR (KBr) $\upsilon_{\max}$ (cm^{-1}): 3442, 3379(NH), 1677(C=O), 1647, 1631(C=N), 1600–1570(C=C), 1152(B–O) 1072(B–F), 1070(N–O), 1033(N–O), 752(Ni–Cl). ^1H NMR ppm: 8.92(m, 4H, NH), 7.90(m, 4H, NH), 7.63–6.42(m, 24H). ^{13}C NMR ppm: 166.66, 148.72, 132.82, 130.66, 128.23, 127.13, 123.13, 121.73, 118.30, 115.38, 113.08. HRMS: *m*/*z*: Calcd for $C_{49}H_{37}B_2ClF_4N_{13}NaNiO_8$: 1149.19; Found: 1150.20. Anal. Calcd: C, 51.15; H, 3.24; N, 15.82; Ni, 5.10. Found: C, 51.08; H, 3.20; N, 15.75; Ni, 5.09.

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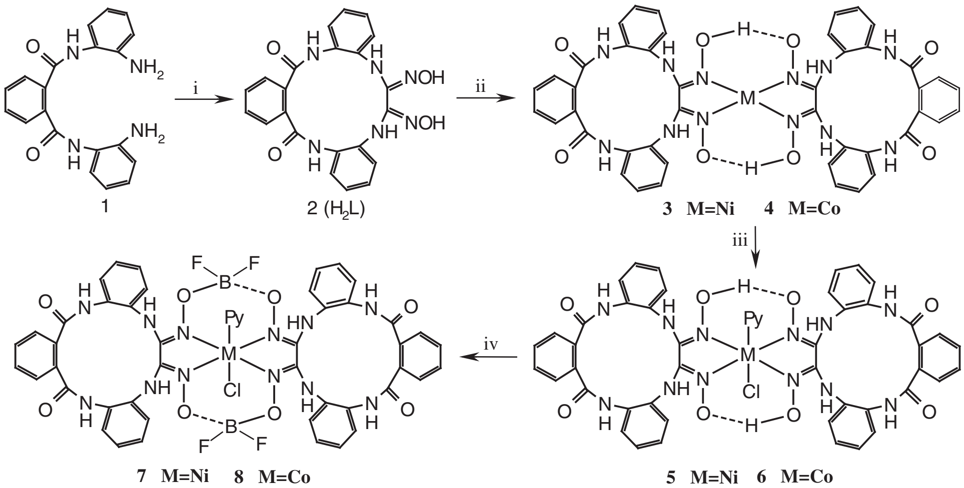
2.8. *Synthesis of [Co(LBF$_2$)$_2$PyCl] (8)*

A suspension of **6** (0.21 g, 0.2 mmol), in methanol (25 mL) was refluxed under argon. Boron-trifluoride ethyl etherate (0.1 mL, 0.4 mmol) was added with stirring to the reaction mixture, which immediately changed to a red solution. The reaction was refluxed for 5 h and then allowed to cool to room temperature. The solvent was concentrated to 10 mL under reduced pressure, filtered and the precipitate washed with methanol, ethanol, and diethyl ether, and dried in vacuum.

Yield: 0.13 g (56.6%); m.p. > 260°C. Color: orange. IR (KBr) $\upsilon_{\max}$ (cm^{-1}): 3344, 3230(NH), 3100(Ar–H), 1677(C=O), 1635(C=N), 1585–1496(C=C), 1151(B–O), 1021(B–F), 1052(N–O), 1037(N–O), 746(Ni–Cl). ^1H NMR ppm: 9.34(m, 4H, NH), 7.93(m, 4H, NH), 7.69–7.21(m, 24H). HRMS: *m*/*z*: Calcd for $C_{49}H_{37}B_2ClCoF_4N_{13}O_8$: 1126.21; Found: 1126.47. Anal. Calcd C, 52.18; H, 3.31; N, 16.14; Co, 5.23. Found: C, 52.09; H, 3.22; N, 16.08; Co, 5.15.

3. Results and discussion

The new *vic*-dioxime 9,10-diethylidine-5,8,9,10,11,18-hexahydro-5,8,11,18-tetraazadibenzo[a,e]cyclotetradecane-6,7,12,17-tetraone-6,7-dioxime was prepared from reaction of N,N′-*bis*-(2-aminophenyl)phethalimide (**1**) and dichloroglyoxime in methanol (scheme 1). Compound **1** was obtained from reaction of 2-phenylenediamine and 1,2-phthallic anhydride [22]. In the ^1H NMR spectrum of $\mathbf{H_2L}$ the deuterium-exchangeable protons of the =NOH and NH groups appeared at 11.88, 9.59, and



Scheme 1. Synthesis of $\mathbf{H_2L}$ and its mono-, di-, and trinuclear complexes. (i) MeOH, dichloroglyoxime. (ii) MeOH, MCl_2. (iii) MeOH, pyridine, (iv) MeOH, BF.

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7.80, respectively. In the ^{13}C NMR spectrum the C=O and C=NOH carbons were observed at 168.87 and 152.77 ppm. In the IR spectrum, characteristic stretching vibrations of OH, NH, C=O, C=N, and N–O groups were observed at 3475–3371, 3413, 3292, 1652, 1639, and 983, respectively. The EI-mass spectrum of $\mathbf{H_2L}$ shows a molecular ion peak at $m/z = 430$ $[M]^+$, confirming formation of the desired compound [1, 8].

Complex **3**, $[Ni(LH)_2]$, was prepared in 64% yield by treating of $NiCl_2 \cdot 6H_2O$ with $\mathbf{H_2L}$ in methanol. This complex has a metal : ligand ratio of 1 : 2 according to the elemental analysis and mass spectral data. In the ^1H NMR spectra of **3** the chemical shifts belonging to the OH of $\mathbf{H_2L}$ disappeared and a new deuterium exchangeable resonance appeared at lower field ($\delta = 16.01$ ppm), which could be interpreted by formation of intramolecular $O–H \cdots O$ bridges. The ^{13}C NMR spectrum of **3** is similar to that of the free $\mathbf{H_2L}$ but with slight shifts. In the IR spectrum of **3**, O–H stretching vibrations are absent and bending vibrations assigned to $O–H \cdots O$ bridges observed at $1727\,cm^{-1}$. The mass spectrum of **3** displays a molecular ion peak at $m/z = 917$ confirming formation of this complex. A lowering of the vibration frequency of the C=N absorption in the H-bonded nickel(II) complex (relative to the free ligand) indicated that coordination occurred through the N,N′ moieties in agreement with the proposed structure in scheme 1 [23–30]. Similarly **4**, $[Co(LH)_2]$, was obtained in 75% yield from reaction of $\mathbf{H_2L}$ with $CoCl_2 \cdot 6H_2O$.

Furthermore, **5** was obtained by reaction of a suspension of **3** in methanol with pyridine as an axial ligand [27–29]. This complex has a metal : ligand ratio of 1 : 2 according to the elemental analysis data and mass spectra. The mass spectrum of **5** exhibits a molecular ion peak at $m/z = 1030$, confirming formation of the desired compound. In the IR spectrum of **5** a weak absorption assigned to bending vibrations of the intramolecular hydrogen bonds $O–H \cdots O$ was observed at $1710\,cm^{-1}$. The C=N stretch decreases from $1639\,cm^{-1}$ in the free ligand to $1618\,cm^{-1}$ in **5**. The shifting of C=N and N–O stretches of the complex supports MN_4 coordination [1–4, 25–29].

The Co(III) complex **6** was obtained when O_2 was bubbled through a suspension of the $[Co(LH)_2]$ in methanol in the presence of pyridine. In the ^1H NMR spectrum of **6** the resonance of the intramolecular O–H $\cdots$ O bridged protons is a singlet at 15.54 ppm.

When a solution of boron trifluoride diethylether was added to a refluxing suspension of **5** in methanol, the bridged protons were replaced by BF_2^+ groups; pyridine remained as an axial ligand (**7**). In the ^1H NMR spectrum of **7** the deuterium exchangeable O–H $\cdots$ O bridged protons belonging to the precursor Ni(II) complex is absent. The IR spectrum of **7** shows significant bands for NH, CONH, C=O, C=N, B–O, B–F, and N–O stretching vibrations at 3442, 3379, 1677, 1647, 1631, 1152, 1072, 1070, and 1033 cm^{-1}, respectively. In **7** the bending vibration of O–H $\cdots$ O disappeared and new peaks related to BF_2^+ were seen at 1152 and 1072 cm^{-1} for B–O and B–F bonds, respectively. The mass spectrum of **7** displayed a molecular ion peak at $m/z = 1126$, confirming formation of the desired compound.

The BF_2^+-bridged Co(III) complex (**8**) was obtained in 56.6% yield from the hydrogen-bridged Co(III) complex and a boron-trifluoride etherate linking reagent. This reagent readily reacts with the O–H $\cdots$ O bridge in the precursor molecule, yielding an extremely stable compound containing two O–BF_2^+ $\cdots$ O bridges, while the axial pyridine remained. According to ^1H NMR, IR, and elemental analysis data, **8** is $[Co(LBF_2)_2PyCl]$. In IR spectra of **8**, the C=N stretching frequency can be used to distinguish between the dihydrogen- and the diboron-bridged compounds with BF_2-complex showing about 16 cm^{-1} upfield shift due to presence of strong electron-withdrawing BF_2 groups in **8**. The weak and broad band at 1712 cm^{-1} of the in-plane deformation of the O–H $\cdots$ O hydrogen bond disappeared upon encapsulation of the H-bonded complex. Peaks appeared at 1151 and 1021 cm^{-1} for the B–O and B–F groups, respectively [31]. In the ^1H NMR spectrum of **8**, the deuterium exchangeable O–H $\cdots$ O protons of the H-bonded Co(III) complex are no longer present after formation of the BF_2 bridge [32]. This BF_2-bridged Co(III) complex is also confirmed by its high resolution mass spectrum, which gives a molecular ion peak at $m/z = 1126$ $[M]^+$.

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4. Conclusions

We have synthesized and characterized a new macrocyclic (E,E)-dioxime starting from $\mathbf{H_2L}$ and $MCl_2 \cdot 6H_2O$ with nickel(II), cobalt(II), and cobalt(III) complexes prepared. Complexes have a mole ratio 1 : 2 for metal to ligand and coordination occurred through nitrogens of the *vic*-dioximes.

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