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PORPHYRINS. 40*. CHEMISTRY OF OXIMES OF METAL COMPLEXES OF *meso*-FORMYL-OCTAALKYLPORPHYRINS. SYNTHESIS, MOLECULAR AND CRYSTAL STRUCTURE OF NICKEL COMPLEXES OF "TRIPYRROLYLISOXAZOLES"

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The synthesis has been carried out of nickel complexes of oximes of two isomers of meso-formyletioporphyrin-II and their conversion in the heterophase system methylene chloride–water into nickel complexes of tripyrrolylisoxazoles has been studied. The structure of one of them was established by X-ray structural analysis.

Keywords: isoxazole, linear polypyrroles, porphyrin metal complexes, oximes of *meso*-formylporphyrins, etioporphyrin-II.

Oximes of *meso*-formylporphyrins may be obtained by heating the appropriate formyl derivatives in pyridine solution with hydroxylamine hydrochloride [3, 4]. The interaction of hydroxylamine hydrochloride with iodomethylates of azomethine derivatives of porphyrins at room temperature is more efficient [5]. Such oximes were used previously to obtain the corresponding *meso*-cyanoporphyrins [6,7].

Systematic investigations carried out by us of the chemical properties of metal complexes of oximes of *meso*-formylporphyrins showed that the latter are readily transformed under conditions of interphase hydrolysis at room temperature in a methylene chloride–water mixture into 1,2-isoxazinochlorins [8,9] and/or into linear polypyrrole compounds [2] depending on the structure of the peripheral substituents in the macrocycle and the presence of a central metal atom.

The aim of the present work is to establish the structure of the polypyrrole compounds formed.

In order to simplify the problem and to exclude the emergence of isomeric mixtures on transforming oximes of *meso*-formylporphyrins, isomers of the oximes of the nickel complex of *meso*-formyletioporphyrin-II **1** and **2**, in which the *meso* substituent is in the vicinity of two identical β-pyrrole groups, ethyl (for isomer **1**) and methyl (for isomer **2**), were selected as subjects of the investigation.

The synthesis of the required oximes **1** and **2** was effected starting from the Schiff's bases **3** and **4** described previously [10]. Refluxing the latter in dichloromethane in the presence of MeI led to the formation of the corresponding iodomethylates **5** and **6** in high yield.

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* For Part 39 see [1], preliminary communication see [2].

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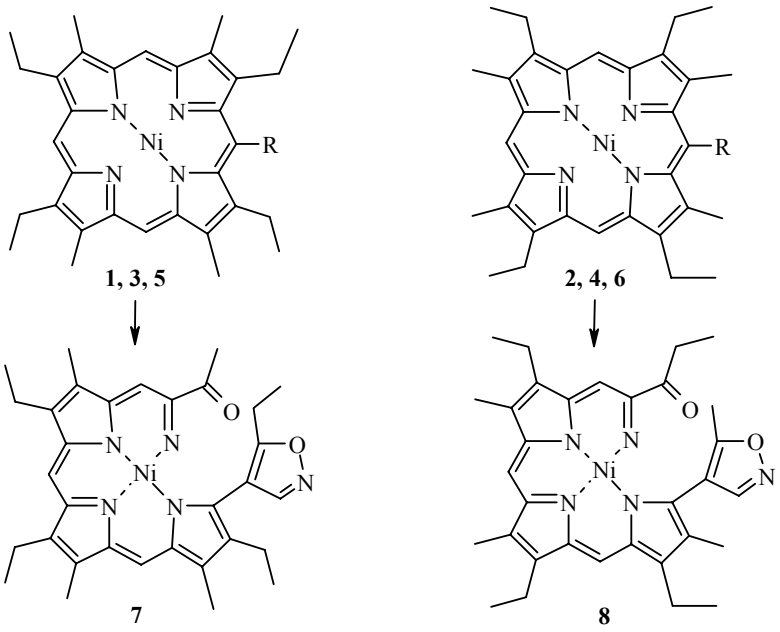
Treatment of iodomethylates **5** and **6** with an excess of $NH_2OH \cdot HCl$ in pyridine at room temperature led to the formation of oximes **1** and **2**, which were isolated as bright red crystalline solids on gradually adding water to the reaction mixture. The oxime crystals, which were filtered off, washed with water, and dried in the air, were perfectly stable. For plotting the electronic spectra of the oximes we used a mixture of methylene chloride with triethylamine, which stabilizes the oximes in solution. However we were unsuccessful in obtaining high quality ^1H NMR spectra for these compounds since even short term storage of the oximes in deutero-chloroform leads to their decomposition. Peaks were observed in the electron impact mass spectra of the oximes for molecular ions and [M-18] fragments of approximately equal intensity, corresponding to the fission of a molecule of water from the molecular ions of the oximes, as in the case of the molecular ions of the corresponding *meso*-nitriles, which arise as a result of thermolysis of the oximes in the ion source of the mass spectrometer in the process of plotting the spectra [11].



1, **2** R = CH=NOH; **3**, **4** R = CH=NMe; **5**, **6** R = $CH=N^+Me_2 \cdot I^-$

On treating oximes **1** and **2** in the heterophase system methylene chloride–water (aqueous sodium bicarbonate solution), in the course of several minutes in each case, the formation occurred of compounds with identical electronic spectra, **7** and **8** respectively. They were isolated by column chromatography on silicagel in ~50% yield and proved to be extremely stable in the crystalline state. Their characteristic property on identification by TLC on silicagel plates was the fact that the brown spots gradually turned blue after drying the solvent from the plate.

Well formed prismatic crystals were obtained by crystallization from a methylene chloride–methanol mixture during several days in the cold. These were suitable for investigation by X-ray analysis, which was used to establish the structure of compound **7** obtained from oxime **1** (Fig. 1).

As is seen from Fig. 1 the **7** molecule has an unusual structure since the ligand is a disconnected polypyrrole system. It consists of two main fragments, a tripyrene, i.e. three pyrrole rings linked to one another with methine groups, and a residue of isoxazole, containing an ethyl substituent, added at the α carbon atom of one of the terminal pyrroles. In turn the three pyrrole atoms $N_{(2)}$, $N_{(3)}$, and $N_{(4)}$ and the azomethine $N_{(1)}$ atom form the coordination polyhedron of the nickel atom. For a more graphic representation of the structure of complex **7** a simplified picture of this molecule is given in Fig. 2, in which the peripheral methyl and ethyl groups on the pyrrole rings are not shown for simplicity.

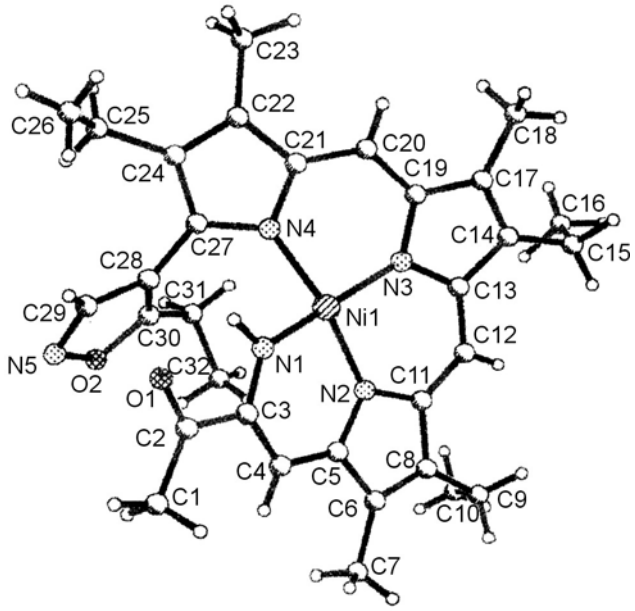


Fig. 1. General form of the molecular structure of **7** with the numbering of the C, N, and O atoms.

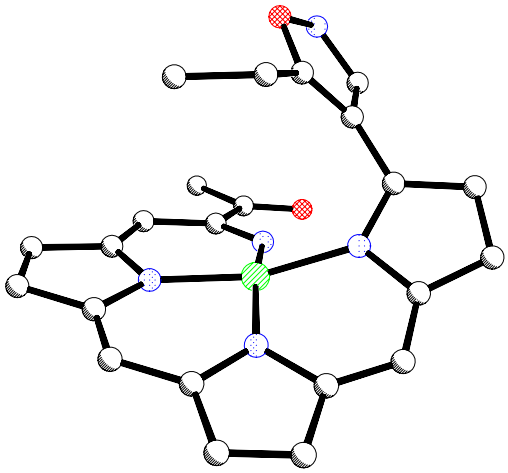


Fig. 2. Molecular structure of complex **7** without the peripheral alkyl substituents.

The structure of **7**.0.5 CH_2Cl_2 contains two crystallographically independent molecules having the same structure (Fig. 3). The molecules are nonplanar, the most flattened tricyclic fragment is formed by the atoms of two pyrrole rings and one chelate ring $Ni_{(1)}N_{(2)}C_{(5)}C_{(6)}C_{(8)}C_{(11)}C_{(12)}C_{(13)}C_{(14)}C_{(17)}C_{(19)}N_{(3)}$ (mean deviation from this mean square plane (Pl. 1) is ±0.089 Å. The pyrrole $N_{(4)}C_{(21)}C_{(22)}C_{(24)}C_{(27)}$ and oxazole $C_{(28)}C_{(29)}N_{(5)}O_{(2)}C_{(30)}$ rings are planar (Pl. 2 and Pl. 3 respectively), but are not coplanar with the tricyclic fragment. The dihedral angles between planes Pl. 1/Pl. 2 and Pl. 2/Pl. 3 are 25.4 and 55.8° respectively. The chelate ring $N_{(2)}C_{(5)}C_{(4)}C_{(3)}N_{(1)}$ (Pl. 4) only insignificantly deviates from the tricyclic fragment and forms a dihedral angle of 9.6° with Pl. 1. The acetyl group $C_{(1)}C_{(2)}O_{(1)}$ is turned along the $C_{(3)}$–$C_{(2)}$ bond by 8.8°. The chelate rings containing atoms $N_{(1)}$

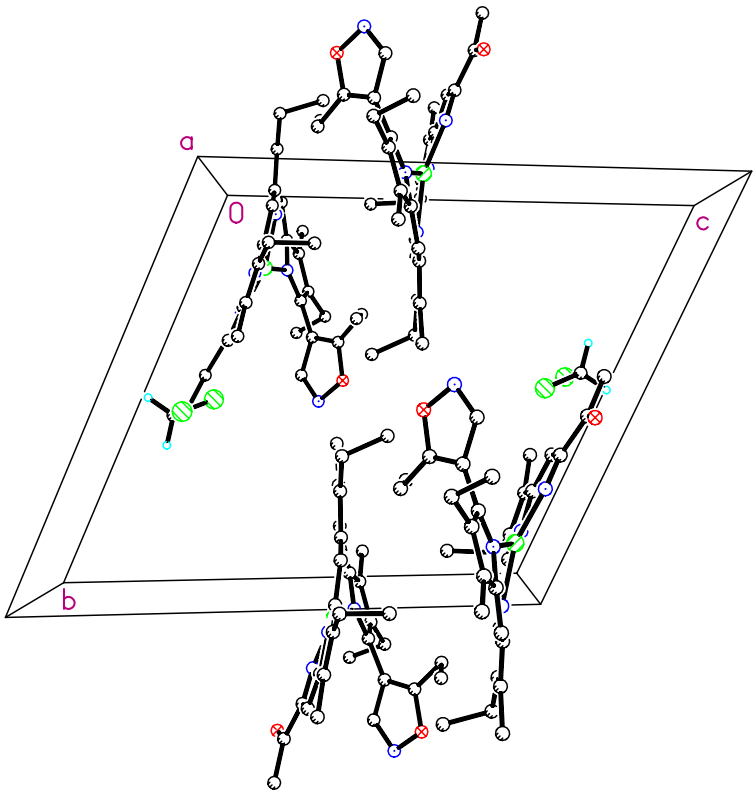


Fig. 3. Disposition of the molecules of complex **7** in the crystal unit cell.

(Pl. 4) and $N_{(4)}$ (Pl. 2) deviate from the tricyclic fragment (Pl. 1) on opposite sides. The methyl groups $C_{(10)}$ and $C_{(16)}$, unlike the $C_{(26)}$ group, are oriented on one side relative to Pl. 1, they form the porous outer surface of the molecule. Probably this also leads to the dimeric disposition of the molecules in the crystal (Fig. 3).

The coordination polyhedron of the nickel atom is a tetrahedrally distorted square. The N–Ni–N valence angles are equal to 88.7-93.0°, the Ni–N bond lengths differ significantly, the shortest is $Ni_{(1)}$–$N_{(1)}$ at 1.828(4) Å, the longest is $Ni_{(1)}$–$N_{(4)}$ at 1.905(4) Å, and the two others ($Ni_{(1)}$–$N_{(2)}$ and $Ni_{(1)}$–$N_{(3)}$ are in between [mean 1.873(4) Å]. For comparison, the smallest mean-statistical value of the Ni–N bond length for a 4-coordinate Ni^{2+} atom (1.888 Å) was obtained in complexes with Schiff's bases (Ni→N[R]=C) [12], in pyrrole complexes these values are equal to 1.906 Å, and in porphyrins 1.943 Å.

The mean values of the bond lengths and some valence angles are given in Table 1.

From an analysis of the X-ray structural data on complex **7** we made a conclusion on the structure of complex **8** obtained from oxime **2**. After this a correct assignment became possible for all the other spectral data for this family of compounds. We have given the trivial name tripyrrolylisoxazoles to structures like complexes **7** and **8** [2].

A distinctive feature of the electronic spectrum of complex **7** (Fig. 4) and of all the other tripyrrolylisoxazoles obtained by us [2] is the extremely broad long wave band in the region of 700-900 nm with a maximum at about 800 nm, which is linked most probably with the presence of a conjugated polypyrrole chain.

In the IR spectra the band at 3300 cm^{-1} belongs to the stretching vibration of N–H in the C=N–H group. The coordination of the imine nitrogen atom with the central nickel atom practically does not affect the frequency of the vibrations of the N–H bond. The intense band at 1682 cm^{-1} corresponds to the stretching

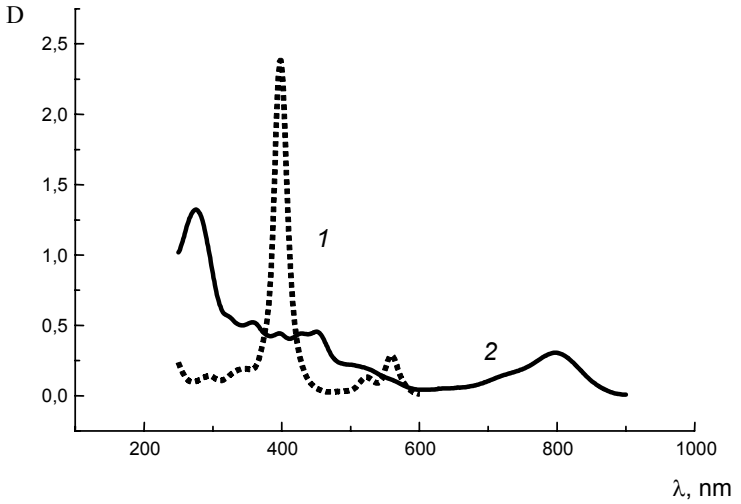


Fig. 4. Electronic spectra: 1) oxime **1** in methylene chloride + 1% Et_3N; 2) compound **7** in methylene chloride.

TABLE 1. Bond Lengths (*d*) and Valence Angles (ω) in the **7** Molecule

Bond	*d*, Å	Bond	*d*, Å	Angle	ω, deg.
$Ni_{(1)}–N_{(1)}$	1.828	$C_{(8)}–C_{(9)}$	1.500	$N_{(1)}–Ni_{(1)}–N_{(3)}$	162.7
$Ni_{(1)}–N_{(3)}$	1.870	$C_{(9)}–C_{(10)}$	1.516	$N_{(1)}–Ni_{(1)}–N_{(2)}$	91.00
$Ni_{(1)}–N_{(2)}$	1.876	$C_{(11)}–C_{(12)}$	1.367	$N_{(3)}–Ni_{(1)}–N_{(2)}$	93.00
$Ni_{(1)}–N_{(4)}$	1.905	$C_{(12)}–C_{(13)}$	1.415	$N_{(1)}–Ni_{(1)}–N_{(4)}$	88.7
$O_{(1)}–C_{(2)}$	1.206	$C_{(13)}–C_{(14)}$	1.441	$N_{(3)}–Ni_{(1)}–N_{(4)}$	91.8
$O_{(2)}–C_{(30)}$	1.358	$C_{(14)}–C_{(17)}$	1.364	$N_{(2)}–Ni_{(1)}–N_{(4)}$	165.2
$O_{(2)}–N_{(5)}$	1.414	$C_{(14)}–C_{(15)}$	1.494		
$N_{(1)}–C_{(3)}$	1.314	$C_{(15)}–C_{(16)}$	1.520		
$N_{(2)}–C_{(5)}$	1.365	$C_{(17)}–C_{(19)}$	1.441		
$N_{(2)}–C_{(11)}$	1.387	$C_{(17)}–C_{(18)}$	1.500		
$N_{(3)}–C_{(13)}$	1.366	$C_{(19)}–C_{(20)}$	1.381		
$N_{(3)}–C_{(19)}$	1.392	$C_{(20)}–C_{(21)}$	1.392		
$N_{(4)}–C_{(27)}$	1.353	$C_{(21)}–C_{(22)}$	1.420		
$N_{(4)}–C_{(21)}$	1.390	$C_{(22)}–C_{(24)}$	1.387		
$N_{(5)}–C_{(29)}$	1.301	$C_{(22)}–C_{(23)}$	1.498		
$C_{(1)}–C_{(2)}$	1.505	$C_{(24)}–C_{(27)}$	1.423		
$C_{(2)}–C_{(3)}$	1.520	$C_{(24)}–C_{(25)}$	1.500		
$C_{(3)}–C_{(4)}$	1.400	$C_{(25)}–C_{(26)}$	1.532		
$C_{(4)}–C_{(5)}$	1.385	$C_{(27)}–C_{(28)}$	1.477		
$C_{(5)}–C_{(6)}$	1.457	$C_{(28)}–C_{(30)}$	1.350		
$C_{(6)}–C_{(8)}$	1.360	$C_{(28)}–C_{(29)}$	1.423		
$C_{(6)}–C_{(7)}$	1.493	$C_{(30)}–C_{(31)}$	1.485		
$C_{(8)}–C_{(11)}$	1.464	$C_{(31)}–C_{(32)}$	1.536		

vibration of the carbonyl group in the acyl residue of the molecule. The presence in the spectrum of bands at 1616, 1608, 1599, and 1576 cm^{-1} probably corresponds to the vibrations of the numerous C=N bonds in the molecule.

In comparison with porphyrins or metalloporphyrins the signals of all the protons in the ^{1}H NMR spectra of tripyrrolylisoxazoles **7** and **8** (both *meso*-H and the peripheral methyl and ethyl substituents) were displaced significantly (1.5-2.0 ppm) towards high field, which is linked with the absence of ring current in these molecules. At lowest field are the singlet signals of the imine protons (CH=N) in isoxazoles. The broadened signals at 6.97 (for compound **7**) and 7.12 ppm (for compound **8**) correspond to the imine fragment (C=NH···Ni), and the doublet at 6.46 and 6.48 ppm with coupling constant 2.2-2.4 Hz corresponds to the allylic proton [=C<u>H</u>–C(COR)=NH···Ni].

The mechanism of conversion of nickel complexes of oximes of *meso*-formyloctaalkylporphyrins into tripyrrolylisoxazoles has not finally been clarified, but it probably consists of several stages, among which is an intramolecular cyclization of the *meso* substituent with the formation of intermediates unstable to oxidation by the oxygen of chlorin or phlorin systems. The oxidative degradation of porphyrins to linear tetrapyrrole compounds (biliverdins and bilirubins) with fission at the *meso* carbon atom is known as a main stage of catabolism occurring under mild fermentative conditions, and fission of the macrocycle at a β,β-pyrrole bond may occur only under the action of strong oxidizing agents (for example the formation of *seco*-chlorins [13, 14]). Consequently the formation of linear polypyrrole structures of the tripyrrolylisoxazole type under mild conditions discovered by us forces reconsideration of the general opinion on the high chemical stability of the aromatic tetrapyrrole porphyrin cyclic system.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker AC 250 spectrometer (operating frequency 200 MHz) in $CDCl_3$. The IR spectra were recorded on a Nicolet Magna 750 instrument in KBr disks. Electronic spectra were obtained on HP model 8453 and Hitachi 320 spectrophotometers. Recording of spectra was effected without dilution of solutions at concentrations of approximately 1.2-1.7 optical density in the region of the Soret band, the relative intensities of the absorption maxima are given in the description of spectra. Mass spectra (electron impact) were obtained on a Finnigan MAT instrument and MALDI spectra on a Vision 2000 instrument.

Complexes **3** and **4** were obtained by the procedure of [15].

Nickel Complex of *meso*-(N-Methylformaldimino)etioporphyrin-II Iodomethylate (5). A solution of complex **3** (81 mg, 141 μmol) in dichloroethane (18 ml) was refluxed with methyl iodide (0.4 ml). After 1 h methyl iodide (0.2 ml) was added and the mixture refluxed for a further 30 min. The solvent was then evaporated and isomer **5** (93 mg, 92%) was obtained. ^{1}H NMR spectrum, δ, ppm: 12.14 (1H, br. s, C<u>H</u>=N$^+$); 9.44, 9.34 (1H, 2H, two s, *meso*-H); 4.52, 2.41 [6H, two s, =N$^+$(C<u>H</u>$_3$)$_2$]; 3.8-3.60 (8H, overlapping q, 4 × C<u>H</u>$_2$CH$_3$); 3.40, 3.31 (6H, 6H, two s, 4 × CH$_3$ ring); 1.68, 1.63 (6H, 6H, two t, 4 × CH$_2$C<u>H</u>$_3$). Electronic spectrum in dichloromethane, λ_{max}, nm (I_{rel}): 386 (7.33), 456 (10.8), 604 (1.06), 712 (1.0). IR spectrum: $\nu_{C=N^+}$ 1650 cm^{-1}. Found, %: C 57.31; H 5.88; N 9.67. $C_{34}H_{39}IN_5Ni.0.5H_2O$. Calculated, %: C 57.33; H 5.66; N 9.83.

Nickel Complex of *meso*-(N-Methylformaldimino)etioporphyrin-II Iodomethylate (6) was obtained analogously from complex **4** in 95% yield. ^{1}H NMR spectrum, δ, ppm: 11.77 (1H, br. s, CH=N$^+$); 9.36, 9.30 (1H, 2H, two s, *meso*-H); 4.42, 2.19 (3H, 3H, two s, =N$^+$-C<u>H</u>$_3$); 4.0-3.72 (8H, overlapping q, 4 × C<u>H</u>$_2$CH$_3$); 3.28, 3.24 (6H, 6H, two s, 4 × CH$_3$ ring); 1.68, 1.58 (3H, 9H, two t, 4 × CH$_2$C<u>H</u>$_3$). Electronic spectrum in dichloroethane, λ_{max}, nm (I_{rel}): 3.92 (5.75), 458 (11.06), 608 (1.0), 728 (1.0). IR spectrum: $\nu_{C=N^+}$ 1650 cm^{-1}.

Nickel Complex of *meso*-Formyletioporphyrin-II Oxime (1). Hydroxylamine hydrochloride (25 mg) was added to a solution of iodomethylate **5** (101 mg, 141 μmol) in pyridine (4 ml) at room temperature. After 10-15 min water (several drops) was added, the precipitated solid was filtered off, washed on the filter with water, dried at 80°C, and oxime **1** (69 mg, 119 μmol) was obtained. Yield 85%. UV spectrum, λ_{max}, nm (I_{rel}): 294 (1.21), 338 (1.34), 398 (16.33), 521 (1.0). Mass spectrum (EI), *m/z* (I_{rel}, %): 577 (5) [M]$^+$, 559 (5) [M-H$_2$O]$^+$. Found, %: C 66.59; H 6.45; N 12.08. $C_{33}H_{37}N_5NiO.H_2O$. Calculated, %: C 66.46; H 6.59; N 11.74.

Nickel Complex of *meso*-Formyletioporphyrin-II Oxime (2) was obtained analogously to oxime **1** from the iodomethylate of Schiff's base **6** (29 mg, 40 μmol). Yield 20 mg (34 μmol, 85%). UV spectrum, λ_{max}, nm (I_{rel}): 294 (1.20), 338 (1.34), 398 (16.44), 521 (1.0). Mass spectrum (EI), *m/z* (I_{rel}, %): 577 (8) $[M]^+$, 559 (7) $[M\text{-}H_2O]^+$.

Tripyrrolylisoxazole 7. An aqueous solution of $NaHCO_3$ (0.5 M) was added to a solution of oxime **1** (15 mg, 26μmol) in methylene chloride (5 ml) at room temperature. The mixture was stirred for 5-10 min until the end of the reaction (chromatographic check), the organic layer was then separated, evaporated, and chromatographed on a column (1.5 × 12 cm) of silica gel, eluting with methylene chloride. The fraction containing a brown substance was evaporated, crystallized from methylene chloride–methanol, and compound **7** (7.5 mg, 13 μmol, 50%) was obtained. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 8.36 (1H, s, C<u>H</u>=N isoxazole); 7.34, 7.00 (2H, two s, *meso*-H); 6.97 [1H, br. s, =CH-C(COMe)=N<u>H</u>···Ni]; 6.47 [1H, d, *J* = 2.1, =C<u>H</u>–C(COMe)=NH···Ni]; 3.08-2.94 and 2.87-2.71 (8H, overlapping q, 4 × C_2H_5); 2.51, 2.44, 2.42, 2.34 (12H, all s, 4 × Me); 1.29, 1.23, 1.05, 0.95 (12H, all t, *J* = 7.5, 4 × $CH_2C\underline{H}_3$). UV spectrum, λ_{max}, nm (I_{rel}): 276 (4.28), 357 (1.06), 398 (1.44), 453 (1.51), 796 (1.0). Mass spectrum (MALDI), *m/z* (I_{rel}, %): 582.4 (100) [M+H]. Found, %: C 64.46; H 6.67; N 11.34. $C_{32}H_{37}N_5NiO_2.H_2O$. Calculated, %: C 64.02; H 6.55; N 11.66.

Tripyrrolylisoxazole 8 was obtained analogously from oxime **2** (42 mg, 73 μmol). Yield of crystalline substance was 22 mg (38 μmol, 52%). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 8.50 (1H, s, C<u>H</u>=N isoxazole); 7.29, 6.97 (2H, two s, *meso*-H); 7.12 [1H, br. s, =CH–C(COEt)=N<u>H</u>···Ni]; 6.46 [1H, d, *J* = 2.1, =C<u>H</u>–C(COEt)=NH···Ni]; 3.00-2.66 (8H, overlapping q, 4 × C_2H_5); 2.50, 2.37, 2.31, 2.00 (12H, all s, 3 × Me on pyrrole rings, CH_3 isoxazole), 1.30, 1.27, 1.24, 1.18 (12H, all t, 4 × C_2H_5). UV spectrum, λ_{max}, nm (I_{rel}): 276 (4.25), 357 (1.03), 398 (1.43), 453 (1.50), 796 (1.0). Mass spectrum (MALDI), *m/z* (I_{rel}, %): 582.3 (100) [M+H].

X-ray Structural Investigation of a Monocrystal of Compound 7. Brown crystals of **7**·0.5CH_2Cl_2 were triclinic, at -163°C *a* = 14.799(18), *b* = 14.863(18), *c* = 6.503(21) Å; α = 106.40(2); β = 107.48(3), γ = 108.12(2)°; *V* = 2995(6) Å^3; d_{calc} = 1.386 g/cm^3; *Z* = 2; space group *P*-1. The intensities of 13653 independent reflections were obtained on a Bruker SMART 1000 CCD automatic diffractometer [λ(MoKα), graphite monochromator, ω scanning with a 0.3° step, exposure time 10 sec] at -163°C. Processing of the experimental data was carried out with the aid of SAINT [16] and SADABS [17] programs. The structure was solved by the direct method making all the nonhydrogen atoms apparent and refined according to F^2_{hkl} by the full matrix least squares method in an anisotropic approach for the nonhydrogen atoms. The positions of the hydrogen atoms of the methyl and ethyl substituents were calculated geometrically and refined by the rider model. The remaining hydrogen atoms were revealed objectively by Fourier difference syntheses and were refined in an isotropic approach. The final values for the reliability factors were $R_1 = 0.065$ [calculated according to F_{hkl} for 6231 reflections with $I > 2\sigma(I)$], $wR_2 = 0.1857$ (calculated according to F^2_{hkl} for all 10393 reflections). All calculations were carried out with the SHELXTL-97 (PC version) program [18].

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