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Iron(II) Complexes $[Fe(DfgH)_2(3\text{-}CONH_2\text{-}Py)_2]$ and $[Fe(DfgH)_2(4\text{-}COOC_2H_5\text{-}Py)_2]$: Synthesis and Structures

I. I. Bulhac[a], P. N. Bourosh[b], D. Schollmeyer[c], V. E. Zubareva[a], K. Suwinska[d], O. Ciobanica[a], and Yu. A. Simonov[b]

[a] *Institute of Chemistry, Academy of Sciences of Moldova, Chisinau, Moldova*
[b] *Institute of Applied Physics, Academy of Sciences of Moldova, Chisinau, Moldova*
[c] *Institute of Organic Chemistry, Gutenberg University, Mainz, Germany*
[d] *Institute of Physical Chemistry, Polish Academy of Sciences, Warszawa, Poland*
E-mail: simonov.xray@phys.asm.md

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Abstract—The complexes $[Fe(DfgH)_2(3\text{-}CONH_2\text{-}Py)_2]$ (**I**) and $[Fe(DfgH)_2(4\text{-}COOC_2H_5\text{-}Py)_2]$ (**II**), where $DfgH_2$ is α-benzyl dioxime, were obtained and examined by X-ray diffraction analysis. The equatorial planes of the coordination octahedra of the metal ions consist of two monodeprotonated α-benzyl dioxime residues united through intramolecular hydrogen bonds O–H···O into a pseudomacrocyclic system. The neutral molecules 3-$CONH_2$-Py and 4-$COOC_2H_5$-Py are coordinated to the Fe^{2+} ion through the N atom of the heterocycle. Structure **I** is layered and structure **II** is molecular. Intermolecular interactions N–H···O are responsible for the formation of layers in crystal structure **I**.

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Solid-state iron dioximates with α-dioximes and pyridine were first synthesized by Chugaev [1]. Similar complexes with pyridinecarboxylic acid derivatives have not been obtained, probably because of the lower pK_a values of these ligands compared to that of pyridine.

Yet iron(II) dioximates containing pyridinecarboxylic acid derivatives were obtained according to a slightly different procedure [1], with sodium acetate as a weak base [2, 3]. The IR spectra of iron complexes with nicotinic acid derivatives (nicotinamide, N,N-diethylnicotinamide, ethyl nicotinate, etc.) suggested that the ligands are coordinated through the heterocyclic N atom. When pyridinecarboxylic acid derivatives contain, apart from the pyridine N atom, other electron-donating groups, their way of coordination to metal atoms can vary with the solvent, the complexing metal, and competitive ligands [4]. According to data from the Cambridge Crystallographic Data Collection [5], the ligand of this type is coordinated to a metal ion mainly through the N atom of the heterocycle. An alternative, bidentate fashion of coordination is found in some transition metal complexes with pyridinecarboxamides: through the heterocyclic N atom and the amide O atom. However, IR spectra are insufficient for comprehensive analysis of the structures and ligand coordination in such complexes because complexation through the carbonyl O atom not always shifts the ν(C=O) band to the higher frequencies [6]: cleavage of an intermolecular hydrogen bond is compensated by coordination of the carbonyl group to a metal atom.

Such distinctive features of pyridinecarboxylic acid derivatives make be very careful when determining the ligand-to-metal coordination from IR spectroscopic data. Here the complexes $[Fe(DfgH)_2(3\text{-}CONH_2\text{-}Py)_2]$ (**I**) and $[Fe(DfgH)_2(4\text{-}COOC_2H_5\text{-}Py)_2]$ (**II**) (where DfgH is the α-benzyl dioxime monoanion) were obtained and examined by X-ray diffraction analysis to reveal the coordination of nicotinic and isonicotinic acid derivatives in iron(II) complexes.

EXPERIMENTAL

Synthesis of complex I. A solution of α-benzyl dioxime (0.30 g, 1.25 mmol) and nicotinamide (0.17 g, 1.39 mmol) in DMF (15 ml) and a solution of sodium acetate trihydrate (0.17 g, 1.25 mmol) in methanol (10 ml) (for creation of weakly basic conditions) were added in an alternating way to a solution of $FeCl_2 \cdot 4H_2O$ (0.13 g, 0.66 mmol) in methanol (20 ml). Argon was bubbled through the reaction mixture for stirring and creation of an inert atmosphere (to prevent oxidation of iron). Then the reaction vessel was hermetically closed. After 5–6 h, dark brown single crystals of complex **I** formed as thickened hexagonal plates.

Complex **I** is diamagnetic. The parameters of the Mössbauer spectra are as follows: at 300 K, IS =

0.47 mm/s and QS = 1.78 mm/s; at 80 K, IS = 0.52 mm/s and QS = 1.78 mm/s (IS (with respect to sodium pentacyanonitrosylferrate) and QS were measured with an accuracy of ±0.04 mm/s). These data confirm the low-spin state of Fe^{2+}.

For $C_{40}H_{34}FeN_8O_6$				
anal. calcd, %:	Fe, 7.17;	C, 61.71;	H, 4.40;	N, 14.39.
Found, %:	Fe, 7.01;	C, 61.42;	H, 4.56;	N, 14.20.

Synthesis of complex II. A solution of α-benzyl dioxime (0.60 g, 2.5 mmol) and ethyl isonicotinate (0.40 ml, 2.65 mmol) in DMF (30 ml) and a solution of sodium acetate trihydrate (0.34 g, 2.5 mmol) in methanol (15 ml) were added in an alternating way to a solution of $FeCl_2 \cdot 4H_2O$ (0.25 g, 1.26 mmol) in methanol (30 ml). The reaction mixture was treated as described for complex **I**. After 5–6 h, dark brown (with a greenish tinge) single crystals of complex **II** formed as truncated prisms.

Complex **II** is diamagnetic. The parameters of the Mössbauer spectra are as follows: at 300 K, IS = 0.45 mm/s and QS = 1.92 mm/s; at 80 K, IS = 0.52 mm/s and QS = 1.93 mm/s. These data confirm the low-spin state of Fe^{2+}.

For $C_{44}H_{40}FeN_6O_8$				
anal. calcd, %:	Fe, 6.67;	C, 63.16;	H, 4.82;	N, 10.04.
Found, %:	Fe, 6.58	C, 63.24;	H, 4.68;	N, 10.26.

X-ray diffraction analysis. The unit cell parameters and a three-dimensional array of reflection intensities for a single crystal of complex **I** were obtained at 298 K on an Enraf Nonius CAD4 diffractometer (CuK_α radiation, ω/2θ scan mode, $2\theta_{max} = 75.0°$). Structure **I** was solved by direct methods (SHELX-86 [7]) and refined by the least-squares method in the anisotropic full-matrix approximation for non-hydrogen atoms (SHELXL-93 [8]).

For complex **II**, experimental data were collected at 100 K on a Nonius Kappa CCD diffractometer (MoK_α radiation, graphite monochromator, ω-2θ scan mode [9]). The unit cell parameters were refined over the whole array of experimental data. The reflection intensities were integrated and reduced to a general scale with the DENZO [10] and SKALEPACK programs [10]. An absorption correction was applied with the XEMP program [11]. Structure **II** was solved by direct methods and refined by the least-squares method in the anisotropic full-matrix approximation for non-hydrogen atoms (SHELX-97 [12]). The H atoms in structures **I** and **II** were located objectively and refined in the rigid-body model.

Crystallographic parameters and a summary of data collection for complexes **I** and **II** are given in Table 1. Selected bond lengths and angles and geometrical parameters of the hydrogen bonds in structures **I** and **II** are listed in Tables 2 and 3, respectively. Atomic coordinates and thermal parameters for structures **I** and **II** have been deposited with the Cambridge Crystallographic Data Collection (nos. 690373 and 690374).

Mössbauer spectra were recorded on a setup of the electrodynamic type in a temporary mode. The ^{57}Co isotope in a chromium matrix at room temperature served as a radioactive source. Pulverized powders of the complexes were used as absorbers (5–10 mg/cm^2 thick in natural isotope composition).

RESULTS AND DISCUSSION

Molecular complexes **I** and **II** structurally relate to the class of compounds with the general formula [M(DioxH)$_2$(A)$_2$] (M is the transition metal ion, DioxH$^-$ is the singly charged residue of α-dioxime, and A is a neutral ligand) described by Chugaev in [1].

Crystal structures **I** and **II** are built from the centrosymmetric molecular complexes [Fe(DfgH)$_2$(3-CONH$_2$-Py)$_2$] and [Fe(DfgH)$_2$(4-COOC$_2$H$_5$-Py)$_2$], respectively. The metal ions show a *trans*-octahedral coordination like that found in the transition metal complexes [M(DioxH)$_2$A$_2$] [13, 14]. The coordination polyhedron of the Fe^{2+} ion in these structures consists of six N atoms: four atoms belong to two bidentate monodeprotonated ligands DfgH$^-$ and the other two belong to two 3-CONH$_2$-Py or 4-COOC$_2$H$_5$-Py molecules, respectively, coordinated through the N atom of the heterocycle. It has been demonstrated in [15] that nicotinamide in Mn(II), Fe(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), Hg(II), and Pd(II) complexes is coordinated through the N heteroatom. According to data from the Cambridge Crystallographic Data Collection [5], 3-CONH$_2$-Py is mostly a bidentate ligand for various metal ions, with the pyridine N atom and the carbonyl O atom as binding sites. In crystals, the formation of dimers (e.g., in a Zn(II) complex with diethylnicotinamide [16] and a Cu(II) complex with nicotinamide [17]) or chains is possible (as in Mn(II) and Cu(II) complexes with nicotinamide and its derivatives [18–21]). Different ways of coordination have also been found in transition metal complexes with isonicotinamide and its derivatives. In the complex with dimethylglyoxime, [Cu(DH)$_2$(4-CONH$_2$-Py)] · 4-CONH$_2$-Py · H$_2$O [22], isonicotinamide is coordinated to the metal ion through the N atom of the pyridine ring; in [Ag(4-CONH$_2$-Py)]BF$_4$ [23], the pyridine N and carbonyl O atoms bridge two Ag atoms. The organic ligand 4-CONH$_2$-Py is coordinated through the amide N atom and the carbonyl O atom in a Pt(II) complex [24] and only through the amide N atom in a Ru(III) complex [25].

The Fe(1)–N bond lengths are 1.921(2), 1.897(2), and 2.006(2) Å in structure **I** and 1.903(2), 1.906(2), and 2.007(2) Å in structure **II** (Table 2). In either structure, the coordination polyhedron of the Fe atom is slightly elongated in the direction of the pyridine fragment. The shortening of the Fe–N(dioxime) bond to, on

Table 1. Crystallographic parameters and a summary of data collection for structures **I** and **II**

Parameter	Value	
	I	**II**
M	750.67	836.67
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$
Unit cell parameters:		
a, Å	12.2627(8)	8.6200(1)
b, Å	9.006(1)	19.5180(5)
c, Å	17.0504(8)	12.4740(3)
β, deg	94.487(6)	94.841(2)
V, Å^3	1877.1(3)	2091.20(8)
Z	2	2
ρ_{calcd}, g/cm^3	1.328	1.329
μ, mm^{-1}	3.71 (λCu)	0.421 (λMo)
F(000)	808	872
Crystal size, mm	0.32 × 0.32 × 0.32	0.3 × 0.25 × 0.05
θ scan range, deg	1.5–75.0	2.96–27.50
Ranges of *h*, *k*, and *l* indices	$-15 \le h \le 15$; $0 \le k \le 11$; $0 \le l \le 21$	$-11 \le h \le 11$; $-25 \le k \le 25$; $-16 \le l \le 16$
Number of measured/independent reflections	3857/3857 (R_{int} = 0.0346)	30930/4796 (R_{int} = 0.0790)
Number of reflections with $I > 2\sigma(I)$	3044	3956
Number of parameters refined	257	268
GOOF	1.022	1.027
R factor ($I > 2\sigma(I)$)	R_1 = 0.0487, wR_2 = 0.1365	R_1 = 0.0527, wR_2 = 0.1103
R factor (for all reflections)	R_1 = 0.0681, wR_2 = 0.1591	R_1 = 0.0668, wR_2 = 0.1173
$\Delta\rho_{max}$, $\Delta\rho_{min}$, *e* Å^{-3}	0.29, –0.28	1.030, –0.578

average, 1.907 ± 0.014 Å is probably due to the chelation effect. Two α-benzyl dioxime residues in the equatorial planes of complexes **I** and **II** are coordinated in an N,N-fashion (Figs. 1,2), which gives rise to two chelate rings. Such relative positions of the $DfgH^-$ residues are stabilized by intramolecular hydrogen bond O–H···O (Table 3). In the coordinated α-dioximes, the average N–O, C=N, C–C, and C–C(Ph) bonds (1.357(3), 1.308(4), 1.466(4), and 1.482(4) Å in **I** and 1.356(2), 1.313(3), 1.476(3), and 1.481(3) Å in **II**) are close to those in Fe(II) complexes with monodeprotonated dioximes: α-benzyl dioxime and cyclohexyl isocyanide [26], dimethylglyoxime and imidazole [27], and cyclohexane dioxime and imidazole [28].

In structures **I** and **II**, the apical positions are occupied by two nicotinamide molecules and two ethyl isonicotinate molecules, respectively. The dihedral angles between the planes of the aromatic rings of the latter and the equatorial planes of the corresponding coordination polyhedra are 86.6° and 84.1°. A similar arrangement of α-dioximates and neutral N-containing ligands is found in iron(II) complexes with imidazole [27], β-picoline [29], and pyrazine [30]. The bond lengths and angles for the coordinated 3-$CONH_2$-Py molecules in structure **I**, as well as for 4-$COOC_2H_5$-Py in structure **II**, do not differ from those in Fe(II) complexes with these ligands [31, 32].

Packing of the complexes in crystal structure **I** is shown in Fig. 3. The structure is layered; the complexes are united through hydrogen bonds into a two-dimensional network perpendicular to axis *a* of the crystal. Both amide N atoms of the coordinated nicotinamide

Table 2. Selected bond lengths and angles in structures **I** and **II**

Coordination polyhedron of Fe(II)			Coordinated ligands		
Bond	*d*, Å		Bond	*d*, Å	
	I	**II**		**I**	**II**
Fe(1)–N(1)	1.921(2)	1.903(2)	C(5)–C(8)		1.500(3)
Fe(1)–N(2)	1.897(2)	1.906(2)	C(8)–O(4)		1.333(3)
Fe(1)–N(3)	2.006(2)	2.007(2)	O(4)–C(9)		1.467(3)
Angle	ω, deg		C(9)–C(10)		1.472(4)
	I	**II**	Angle	ω, deg	
N(1)Fe(1)N(2)	80.90(9)	81.02(8)		**I**	**II**
N(1)Fe(1)N(3)	80.03(9)	90.88(7)	O(1)N(1)C(1)	121.7(2)	120.7(2)
N(1)Fe(1)N(2)$^{\#1}$	99.10(9)	98.98(8)	O(2)N(2)C(2)	118.2(2)	119.4(2)
N(1)Fe(1)N(3)$^{\#1}$	90.97(9)	89.12(7)	N(2)C(2)C(1)	113.5(2)	112.0(2)
N(2)Fe(1)N(3)	90.27(9)	91.69(7)	N(1)C(1)C(11)	125.1(2)	122.7(2)
N(2)Fe(1)N(3)$^{\#1}$	89.73(9)	88.31(7)	C(2)C(1)C(11)	122.9(2)	124.6(2)
Coordinated ligands			N(2)C(2)C(21)	123.2(2)	124.6(2)
Bond	*d*, Å		C(1)C(2)C(21)	123.4(2)	123.3(2)
	I	**II**	C(3)N(3)C(7)	116.8(2)	117.1(2)
N(1)–O(1)	1.337(3)	1.342(2)	N(3)C(7)C(6)	123.0(2)	123.3(2)
N(2)–O(2)	1.378(3)	1.371(2)	C(7)C(6)C(5)	119.5(2)	119.1(2)
N(1)–C(1)	1.319(3)	1.317(3)	C(4)C(5)C(6)	118.3(2)	118.7(2)
N(2)–C(2)	1.298(4)	1.310(3)	C(5)C(4)C(3)	118.6(2)	118.9(2)
C(1)–C(2)	1.466(4)	1.476(3)	N(3)C(3)C(4)	123.8(2)	123.0(2)
C(1)–C(11)	1.479(4)	1.478(3)	C(5)C(4)C(8)	125.2(2)	
N(3)–C(3)	1.340(3)	1.353(3)	C(3)C(4)C(8)	116.2(2)	
N(3)–C(7)	1.350(3)	1.349(3)	O(3)C(8)N(4)	122.6(3)	
C(7)–C(6)	1.381(4)	1.382(3)	O(3)C(8)C(4)	119.7(2)	
C(6)–C(5)	1.388(4)	1.387(3)	N(4)C(8)C(4)	117.7(2)	
C(5)–C(4)	1.380(4)	1.385(3)	O(3)C(8)O(4)		125.9(2)
C(4)–C(3)	1.391(4)	1.390(3)	O(4)C(8)C(5)		110.9(2)
C(4)–C(8)	1.507(4)		C(8)O(4)C(9)		118.1(2)
C(8)–O(3)	1.229(4)	1.210(3)	O(4)C(9)C(10)		109.3(2)
C(8)–N(4)	1.321(4)				

* The symmetry operation codes are: $^{\#1}$ $-x + 1, -y, -z + 1$ (**I**) and $^{\#1}$ $-x + 1, -y, -z$ (**II**).

Table 3. Geometrical parameters of the intramolecular and intermolecular hydrogen bonds in structures **I** and **II**

D–H···A	Distances, Å			Angle DHA, deg	Coordinates of atoms A
	D–H	H···A	D···A		
			I		
O(2)–H···O(1)	1.12	1.48	2.567(3)	163	$-x+1, -y, -z+1$
N(4)–H(1)···O(1)	0.92	2.02	2.923(3)	168	$-x+3/2, y-1/2, -z+1/2$
N(4)–H(2)···O(3)	0.83	2.07	2.237(4)	163	$-x+3/2, y-1/2, -z+1/2$
			II		
O(2)–H···O(1)	0.82	1.74	2.530(2)	161	$-x+1, -y, -z$

molecules act as donors and the oxime and carbonyl O atoms act as acceptors in the hydrogen bonding (Table 3). Molecular structure **II** shows only weak C–H···O interactions (Fig. 4). The other intermolecular contacts in these two structures correspond to the sums of the van der Waals radii of the respective atoms.

Thus, the coordination in complexes **I** and **II** is typical of *trans*-octahedral complexes of transition metals:

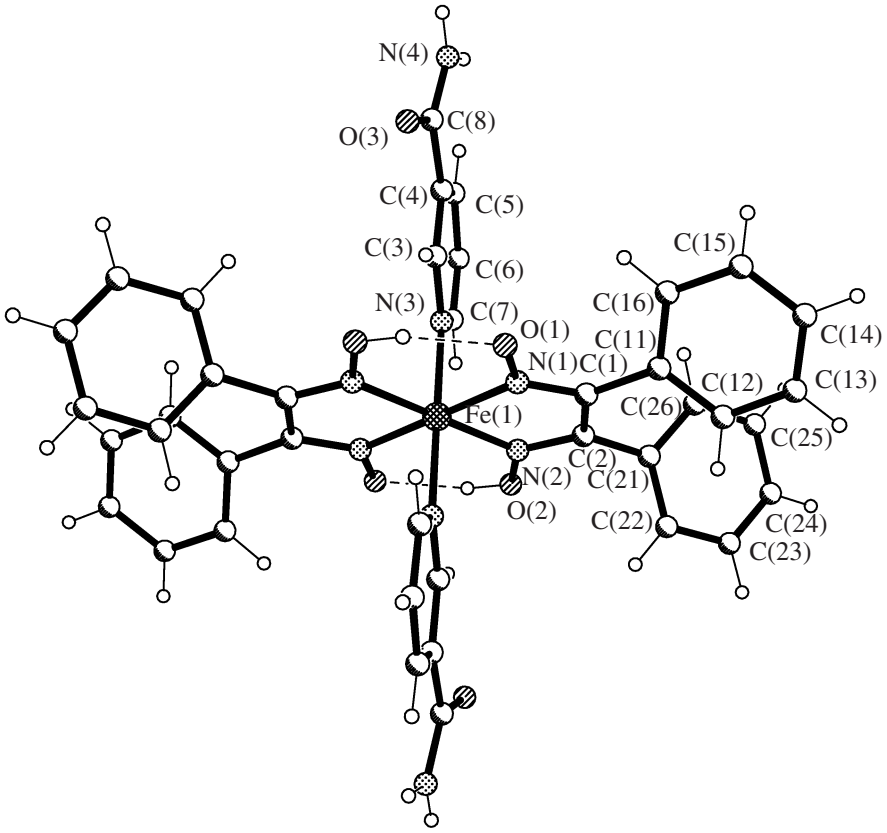

Fig. 1. Structure of $[Fe(DfgH)_2(3\text{-}CONH_2\text{-}Py)_2]$ (**I**).

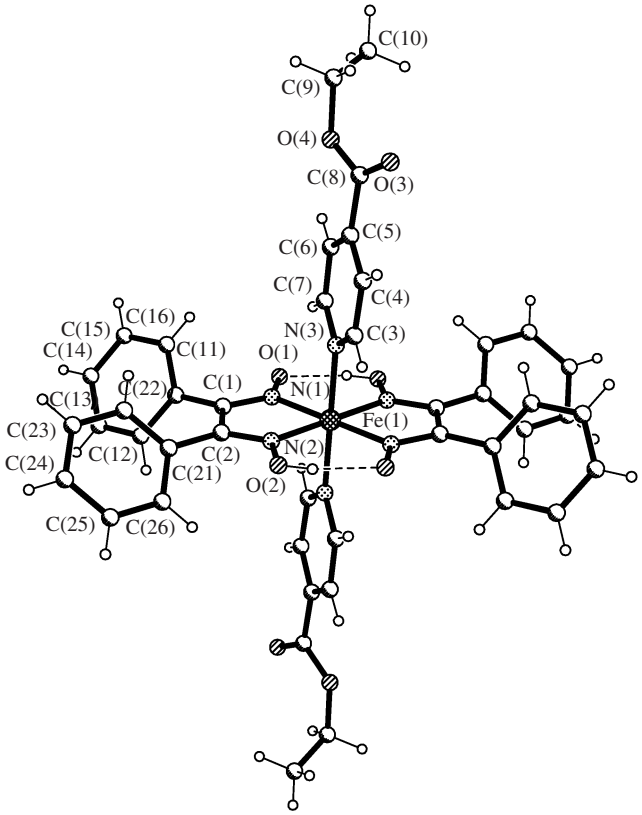

Fig. 2. Structure of [Fe(DfgH)$_2$(4-COOC$_2$H$_5$-Py)$_2$] (**II**).

the equatorial planes are made up of two α-benzyl dioxime residues coordinated in an N,N-fashion, which gives rise to two five-membered chelate rings, and the apical positions are occupied by neutral nicotinamide molecules in structure **I** and by ethyl isonicotinate molecules in structure **II**. The latter in these two complexes are coordinated to the central metal atom through the N atom of the aromatic ring. This way of coordination of the apical ligands to the central metal atom depends on several factors (metal nature, reaction medium, and sto-

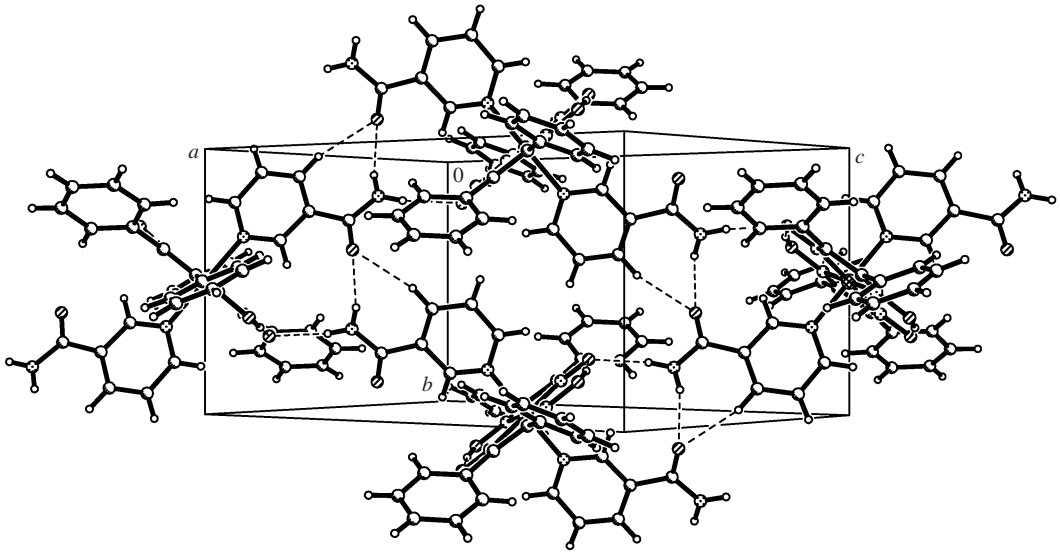

Fig. 3. Fragment of crystal structure **I**.

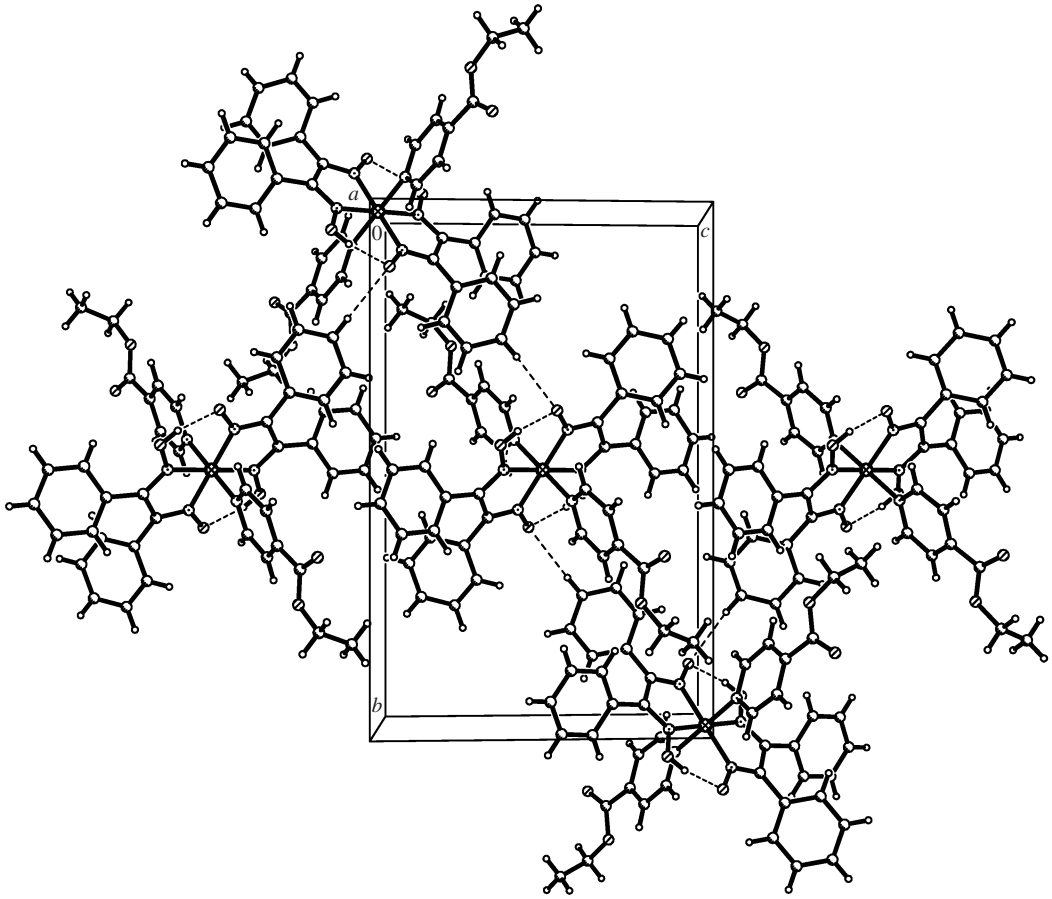

Fig. 4. Fragment of crystal structure **II**.

ichiometry of the reagents). Apparently, the rigid equatorial fragment in transition metal dioximates, which favors the octahedral coordination of the metal, causes nicotinamide or ethyl isonicotinate to coordinate through the aromatic N heteroatom only.

REFERENCES

1. Chugaev, L.A., *Izbrannye trudy* (Selected Works), Moscow: Akad. Nauk SSSR, 1954.
2. Batyr, D.G. and Bulgak, I.I., *Izv. Akad. Nauk Moldav. SSR, Ser. Biol. Khim. Nauk*, 1971, no. 1, p. 77.
3. Batyr, D.G. and Bulgak, I.I., *Izv. Akad. Nauk Moldav. SSR, Ser. Biol. Khim. Nauk*, 1971, no. 3, p. 71.
4. Khakimov, Kh.Kh., Khodzhaev, O.F., and Azizov, T.A., *Kompleksy perekhodnykh metallov s tsiklicheskimi amidami* (Transition metal Complexes With Cyclic Amides), Tashkent: FAN Uzbekskoi SSR, 1984.
5. Allen, F.H., *Acta Crystallogr., Sect. B: Struct. Sci.*, 2002, vol. 58, nos. 3–4, p. 380.
6. Uhlig, E. and Neugelauer, V., *Z. Anorg. Allg. Chem.*, 1967, vol. 351, nos. 5–6, p. 286.
7. Sheldrick, G.M., *Acta Crystallogr., Sect. A: Found. Crystallogr.*, 1990, vol. 46, no. 6, p. 467.
8. Sheldrick, G.M., *SHELXL-93 Program for the Refinement of Crystal Structure*, Göttingen (Germany): Univ. of Göttingen, 1993.
9. Hooft, R., *Collect. Nonius*, Delft (Netherlands), 1980.
10. Otwinowski, Z. and Minor, W., *Macromolecular Crystallography. Methods in Enzymology*, Carter C.W., Sweet R.M, Eds., New York: Academic, 1997, vol. 276, Pt A, p. 307.
11. XEMP. Version 4.2. Siemens Analytical X-ray Inst. Inc., 1990.
12. Sheldrick, G.M., *SHELX-97 Program for the Refinement of Crystal Structure*, Göttingen (Germany): Univ. of Göttingen, 1997.
13. Simonov, Yu.A., Dvorkin, A.A., Gulya, A.P., et al., *Dokl. Akad. Nauk SSSR*, 1989, vol. 305, no. 3, p. 635.
14. Simonov, Yu.A., Kravtsov, V.Kh., Gerbeleu, N.V., et al., *Zh. Neorg. Khim.*, 1999, vol. 44, no. 9, p. 1468 [*Russ. J. Inorg. Chem.* (Engl. Transl.), vol. 44, no. 9, p. 1390].
15. Wright, W.B. and King, G.S.D., *Acta Crystallogr.*, 1954, vol. 7, no. 3, p. 283.
16. Davies, G., El-Toukny, A., Onan, K.D., and Veidis, M., *Inorg. Chim. Acta*, 1984, vol. 84, no. 1, p. 41.
17. Valigura, D., Moncol, J., Korabik, M., et al., *Eur. J. Inorg. Chem.*, 2006, p. 3813.

18. Bigoli, F., Braibanti, A., Pellinghelli, M.A., and Niripicchio, A., *Acta Crystallogr., Sect. A: Cryst. Phys., Diffr., Theor. Gen. Crystallogr.*, 1973, vol. 29, no. 1, p. 39.
19. Tsintsadze, G.V., Tsivtsivadze, T.I., Orbeladze, P.V., et al., *Nauch. Tr. Gruz. Politekh. Inst. im. Lenina*, 1985, p. 66.
20. Cakir, S., Bulut, I., and Aoki, K., *J. Chem. Crystallogr.*, 2003, vol. 33, no. 11, p. 875.
21. Davies, G., El-Toukny, A., Onan, K.D., and Veidis, M., *Inorg. Chim. Acta*, 1985, vol. 98, no. 1, p. 85.
22. Bishop, M.M., Lee, A.H.W., Lindoy, L.F., et al., *Supramol. Chem.*, 2005, vol. 17, nos. 1–2, p. 37.
23. Bhogala, B.R., Thallapally, P.K., and Nangia, A., *Cryst. Growth. Des.*, 2004, vol. 4, no. 2, p. 2115.
24. Sakai, K., Shiomi, M., Tsubomura, T., et al., *Acta Crystallogr., Sect. A: Cryst. Phys., Diffr., Theor. Gen. Crystallogr.*, 2003, vol. 59, no. 8.
25. Chou, M.H., Szalda, D.J., Creutz, C., and Sutin, N., *Inorg. Chem.*, 1994, vol. 33, no. 8, p. 1674.
26. Simonov, Yu.A., Dvorkin, A.A., Bulgak, I.I., et al., *Koord. Khim.*, 1979, vol. 5, no. 12, p. 1883.
27. Bowman, K., Gaughan, A.P., and Dori, Z., *J. Am. Chem. Soc.,* 1972, vol. 94, no. 3, p. 727.
28. Prout, C.K. and Wiseman, T.J., *J. Chem. Soc., Sect. A*, 1964, no. 1, p. 497.
29. Dvorkin, A.A., Simonov, Yu.A., Malinovskii, T.I., et al., *Dokl. Akad. Nauk SSSR*, 1977, vol. 234, no. 6, p. 1372.
30. Kubel, F. and Strahle, J., *Z. Naturforsch., B: Chem. Sci.*, 1983, vol. 38, no. 2, p. 258.
31. Bulgak, I.I., Zubareva, V.E., Turte, K.I., et al., *Koord. Khim.*, 1987, vol. 13, no. 7, p. 960.
32. Cakir, S., Bicer, E., Aoki, K., Coskun, E., *Cryst. Res. Technol.*, 2006, vol. 41, no. 3, p. 314.