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New (*o*-phenylenediaminglyoximato)-(*o*-phenylenediaminglyoxime)zinc(II) chloride. Synthesis and crystal structure

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Abstract. The interaction of dichloroglyoxime and *o*-phenylenediamine with zinc(II) acetate dihydrate leads to the formation of a new compound of Zn(II) with *o*-phenylenediamineglyoxime (*o*-FDH$_2$), very little studied ligand. The X-ray study, on the basis of which the crystal structure of this compound was determined, established that this oxime ligand was obtained from the condensation of organic components and coordinates to the metal atom like most vic-dioximes – bidentate-chelate through two nitrogen atoms, forming metallic rings of five atoms. The presented mononuclear complex compound is of a molecular type, the coordination polyhedron of Zn(II) being a pyramidal-tetragonal one is formed by a neutral *o*-FDH$_2$ ligand, a monodeprotonated *o*-FDH$^-$ one and a chloride anion.

Keywords: mononuclear complex, Zn(II), dioxime ligand, chloride anion.

O nouă clorură de (*o*-fenilendiaminglioximato)-(*o*-fenilendiaminoximă)zinc(II). Sinteză și structură cristalină

Rezumat. La interacţiunea diclorglioximei şi o-fenilendiaminei cu acetatul de zinc(II) dihidrat a fost obţinut un compus nou al Zn(II) cu *o*-fenilendiaminglioxima (*o*-FDH$_2$), un ligand foarte puţin cercetat. Studiul cu raze X, în baza căruia a fost determinată structura cristalină a acestui compus, a stabilit că acest ligand oximic s-a obţinut la condensarea componentelor organice şi coordinează la atomul de metal ca şi majoritatea vic-dioximelor – bidentat-chelat prin doi atomi de azot, formând cicluri metalice din cinci atomi. Compusul complex mononuclear prezentat este unul de tip molecular, poliedrul de coordinare al Zn(II), fiind piramidal-tetragonal, este format de un ligand neutru *o*-FDH$_2$, unul monodeprotonat *o*-FDH$^-$ şi de un anion clorură.

Cuvinte-cheie: complex mononuclear, Zn(II), ligand dioximă, anion de clorură.

1. INTRODUCTION

vic-Dioximes and their complexes represent an important class of compounds with versatile reactivity [1-3]. With reporting dimethylglyoxime as a gravimetric reagent for the quantitative determination of nickel(II) by Tschugaeff [4], vicinal dioximes with chelating ability and diprotic nature have become an indispensable part in various research fields, including analytical chemistry, supramolecular chemistry, biochemistry and biomedical applications [5]. Currently, the Cambridge Structural Database includes more than 2000 entries on the structure of transition metal dioximates, the formation of which is determined by several factors: nature of the complexing agent, the properties of the dioxime itself, pH of the solution, etc.

vic-Dioximes can form coordination compounds in which the dioximes are entrained in molecular [6, 7], monodeprotonated [8-11] and bis-deprotonated [12] forms. Nickel(II), palladium(II) and platinum(II) dioximates with the planar-square coordination polyhedron [13], binuclear copper(II) dioximates with tetragonal structure [14], as well as octahedral-shaped iron(II) [15], iron(III) [16], cobalt(II) [2] and cobalt(III) [17, 18] dioximates are well-known in scholarly sources. In most of the complex compounds known, the central atom coordinates two dioxime ligands as monoanions located approximately in a same plane and being linked together by strong intramolecular O–H···O hydrogen bonds.

Recent studies report Zn(II), Mn(II) and Ni(II) complexes with polyhedron structure in which one dioxime ligand coordinates to the metal atom in a neutral form [6, 19], while in the octahedral Co(III) complexes to which already coordinate two oxime ligands, both are mostly monodeprotonated [20], although there are cases when one of them exists in a neutral, while the other in a bideprotonated form [21].

The aim of this paper was considering a Zn(II) compound with *o*-phenylenediamine glyoxime obtained by condensing of dichloroglyoxime with *o*-phenylenediamine and complexing it with zinc(II) ion, together with its crystal structure. *o*-phenilendiaminglioxime ligand is not sufficiently studied as the Cambridge Structural Database contains only data on its structure and a single Fe(II) compound, which coordinates a macrocyclic ligand consisting of two fragments of diphenylglioxime and one of *o*-phenilendiaminglioxime, these being joined by BF_2 [22, 23].

2. MATERIALS AND METHODS

Synthesis of [Zn(o-FDH$_2$)(o-FDH)Cl]·6,5H$_2$O. A mix of *o*-phenylenediamine ($C_6H_4(NH_2)_2$) (0.022 g, 0.2 mmol), dichlorglyoxime (0.031 g, 0.2 mmol) and $Zn(CH_3COO)_2 \cdot 2H_2O$ (0.022 g, 0.1 mmol) was dissolved in DMF – methanol (8 mL) in 1:4 ratio (vol./vol.). A yellowish sediment was formed. After adding 3 drops of concentrated HCl, the sediment disappeared, but a yellowish solution was formed. The solution was filtered and left at room temperature for crystallization. In 11 days, colourless cubic crystals were formed. The product is soluble in DMSO, insoluble in methanol, water, ethanol, DMF and diethyl ether. 0.037 g were formed. Yield – 31%.

Found, %: C, 32,01; H, 4,73; N, 18,67; Zn, 10,89;

For $C_{16}H_{28}ClN_8ZnO_{10,5}$

Calculated, %: C, 31,96; H, 4,69; N, 18,63; Zn, 10,88.

IR spectrum (ν, cm^{-1}): 3112 s, 2873 w, 2790 w, 2738 w, 1692 w, 1631 v.s., 1607 v.s, 1492 s, 1448 s, 1366 a, 1318 a, 1264 a, 1240 sh, 1157 w, 1123 w, 1007 v.s, 950 a, 930 a, 853 w, 749 s, 712 w, 655 a, 618 a, 593 a, 524 a, 503 a, 453 a. (relative intensity of the absorption bands: v.s – very strong, s – strong, a – average, w – weak, sh – shoulder).

C, H and N contend in the synthesized compounds was determined using the elemental analyser Elementar Analysensysteme GmbH Vario El III.

Quantitative determination of metal in the coordination compound was performed on the Shimadzu AA-7000 atomic absorption spectrophotometer.

FTIR (ATR) spectra were recorded on Spectrum-100 Perkin-Elmer FTIR spectrometer with ATR accessory (attenuated total reflection) in the 4000-650 cm^{-1} range and in Vaseline oil in the 4000-400 cm^{-1} range in the Department of Physical and Inorganic Chemistry of the Institute of Chemistry, MSU.

X-ray study. Crystal structure was determined using single-crystal X-ray diffraction on Xcalibur E diffractometer with a CCD detector and a graphite monochromator with MoKα radiation source with $\lambda = 0.71073\ \mathring{A}$. Determination of unit cell parameters and analysis of experimental data were performed using CrysAlis Oxford Diffraction Ltd [27]. The SHELXS97 and SHELXL2014 program packages were used to solve and refine the structure in the anisotropic full-matrix variant for non-hydrogen atoms and the hydrogen atoms in an isotropic environment [28, 29]. Bonded H to C atoms were located in the positions calculated using a rigid model with Uiso(H)=1.2Ueq(C) and Uiso(H)=1.5Ueq(C), while H atoms of water molecules and amino groups were found from Fourier syntheses in intermediate refinement steps, using geometric restraints. Occupancy was determined for 10 molecules of water of crystallization and ranged between 1 and 0.5. The crystallographic data obtained for the compound in the crystalline phase are shown in Table 1. The crystal data for compound was deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nr. 2345901, respectively; deposit@ccdc.cam.ac.uk, http://www.ccdc.cam.ac.uk).

3. Results and Discussions

As a result of reaction between dichloroglyoxime, *o*-phenylenediamine and zinc(II) acetate dihydrate in a 1:2:1 molar ratio a mononuclear Zn(II) complex was obtained, in which the ligand obtained from condensation of the two organic proligands coordinates to the metal atom. The reaction proceeded according to the scheme from Figure 1.

Parameters	Compound
Formula	$C_{16}H_{28}ClN_8ZnO_{10.5}$
Mr	601.28
Crystal system	monoclinic
Space group	*Cc*
$a/Å$	28.3571(19)
$b/Å$	13.1707(9)
$c/Å$	7.1199(3)
$\beta/°$	96.140(5)
$V/Å^3$	2643.9
Z	4
$\rho_{calc.}$/ g/cm^3	1.511
μ/mm^{-1}	1.095
$F(000)$	1244
Crystal sizes, mm	0.3x0.3x0.05
Collected reflexions / Independent reflexions	4540/3155
Completeness to theta/ % (θ = 25.05)	99.7
Parameters	337
GOOF	1.004
R_1, wR_2 final	R_1 = 0.0628, wR_2 = 0.1762
R index (overall data)	R_1 = 0.0836, wR_2 = 0.1952

Table 1. Crystallographic data, experimental characteristics, and structure refinement parameters for Zn(II) compound.

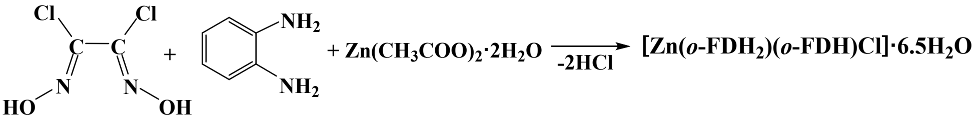

Figure 1. Reaction scheme for the synthesis of compound [Zn(o-FDH$_2$)(o-FDH)Cl]·6,5H$_2$O

A broad band of high intensity is observed at 3112 cm^{-1} in the IR spectrum of the compound, which includes the oscillations: $\nu(\mathrm{OH})_{\mathrm{water}}$ of water of crystallization molecules, $\nu(\mathrm{OH})_{\mathrm{oxime}}$ and ν(NH). The bands at 2873, 2790 and 2738 cm^{-1} are attributed to ν(CH) oscillations. The intense absorption bands at 1631 and 1007 cm^{-1} might be attributed to the ν(C=N) and ν(N–O) vibrations of the oxime groups, respectively [24]. Planar vibrations of the C=C skeleton appear as absorption bands at 1607, 1492, 1448 cm^{-1} [25].

The $\delta(\mathrm{CH})_{\mathrm{nonpl.}}$ absorption band at 749 cm^{-1} corresponds to 1,2-substituted aromatic ring (presence of 4 adjacent hydrogen atoms) [26].

Structure of the Zn(II) compound represents a molecular complex compound, in which oxime ligands coordinate at the central atom, both being formed as a result of condensation of dichlorglyoxime with *o*- phenylenediamine. The formula of this compound is [Zn(*o*-FDH$_2$)(*o*-FDH)Cl]·6.5H$_2$O, in which one of the organic ligands coordinates in neutral form (*o*-FDH$_2$), and the other – in mono-deprotonate form (*o*-FDH), zinc charge being compensated by that of the chloride ion (Figure 2).

Zn(II) compound crystallizes in *Cc* monoclinic space group (Table 1). In the asymmetric part of the unit cell, the metal atom and the two oxime ligands being dentate-chelate coordinated via nitrogen atom while a chlorine atom and 10 water molecules are located in general positions.

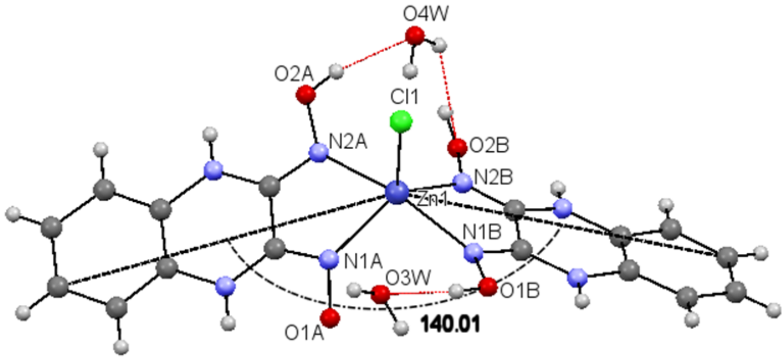

Figure 2. Structure of the mononuclear complex of the Zn(II) compound highlighting the positioning of the oxime ligands coordinated to the metal ion and the intermolecular hydrogen bonds with the involvement of water of crystallization molecules.

The coordination polyhedron of the Zn1 atom in the complex takes shape of a tetragonal pyramid and consists of four nitrogen atoms of the two oxime ligands, located at the base, with the C11 atom located at the apex. As a result, the metal atom is pentacoordinated,

Bonds	(*Å*)
Zn(1)–N(1A)	2.043(10)
Zn(1)–N(2A)	2.184(10)
Zn(1)–N(1B)	2.171(9)
Zn(1)–N(2B)	2.070(11)
Zn(1)–Cl(1)	2.274(3)
Angles	(°)
N(1A)– Zn(1)–N(2A)	75.9(3)
N(1A)– Zn(1)–N(1B)	96.1(3)
N(1A)– Zn(1)–N(2B)	126.0(4)
N(1A)– Zn(1)–Cl(1)	117.2(3)
N(2A)– Zn(1)–N(1B)	162.2(3)
N(2A)– Zn(1)–N(2B)	95.9(4)
N(2A)– Zn(1)–Cl(1)	98.8(2)
N(1B)– Zn(1)–N(2B)	75.7(4)
N(1B)– Zn(1)–Cl(1)	99.0(2)
N(2B)– Zn(1)–Cl(1)	116.8(3)

Table 2. Bond Lengths (*Å*) and Angles (°) in Coordination Metal Environment of Zn(II)

ZnN_4Cl being the coordinating node (Figure 2). The Zn–N interatomic distances in the coordination polyhedron of the complex vary in the range of 2.049–2.184 *Å*, and the Zn–Cl distance is 2.274 *Å* (Table 2).

The oxime ligands are positioned at an angle of 140.01° in the complex and the deviation of the four coordinated nitrogen atoms from their mean plane is ±0.29 *Å*, and the Zn1 metal atom is displaced from this plane towards the Cl1 atom by 0.635 *Å*. The same data are also obtained for the pentacoordinated compounds of Zn(II), with the same donor set of N_4Cl atoms, the four nitrogen atoms belonging to macrocyclic or open-loop tetracoordinated ligands [30, 31].

As a result, no intramolecular hydrogen bonds are formed between the two oxime ligands in the zinc(II) compound, but in the crystal a complicated system of O_{oxime}–H. . . O intermolecular hydrogen bonds is formed via water molecules located in the outer sphere; the donor-acceptor distance varies in the range of 2.67(1)–2.94(2) *Å* (Table 3).

The mononuclear complexes in the crystal are connected in chains by N–H···Cl intermolecular hydrogen bonds and in layers by fine intermolecular C–H···π interactions

Table 3. Hydrogen Bond Distances (*Å*) and Angles (°) in crystal

D–H···A	d(H···A)	d(D···A)	∠(DHA)	Symmetry transformation for acceptor
N(3A)–H(1)···Cl(1)	2.56	3.348(10)	153	x, –y+1, z+1/2
N(4A)–H(2)···O(2W)	2.30	3.12(2)	161	x, y, z
N(3B)–H(3)···O(1W)	2.47	3.28(2)	158	x, y, z
N(4B)–H(4)···Cl(1)	2.57	3.377(10)	157	x, –y, z+1/2
O(2A)–H(2)···O(4W)	1.90	2.70(2)	165	x, y, z–1
O(1B)–H(1)···O(3W)	1.86	2.67(1)	173	x, y, z–1
O(2B)–H(2)···O(4W)	2.16	2.94(2)	157	x, y, z–1
O(2W)–H(2)···O(7W)	2.38	2.94(3)	124	x, –y+1, z–1/2
O(3W)–H(1)···O(7W)	1.88	2.70(3)	162	x, y–1, z
O(3W)–H(1)···O(9W)	2.25	2.90(3)	136	x, y–1, z
O(3W)–H(2)···O(7W)	2.09	2.76(3)	136	x, –y+1, z–1/2
O(3W)–H(1)···O(9W)	2.03	2.83(3)	157	x, –y+1, z–1/2
O(4W)–H(1)···Cl(1)	2.68	3.48(3)	147	x, y, z+1
O(4W)–H(2)···O(2B)	2.34	2.94(2)	124	x, y, z+1
O(4W)–H(2)···O(5W)	2.43	3.19(3)	142	x, y, z
O(5W)–H(1)···O(4W)	2.51	3.10(3)	128	x, –y+1, z+1/2

(Figure 3). The complexes are additionally interconnected with water molecules of co-crystallization.

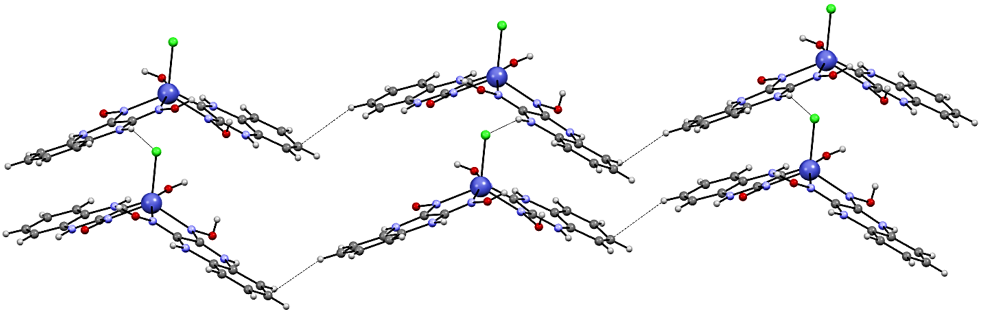

Figure 3. Formation of layers in crystal with entrainment of mononuclear complexes.

4. Conclusions

Upon dichlorglyoxime and *o*-phenylenediamine interaction with zinc(II) acetate dehydrate, a complex molecular compound with the formula [Zn(*o*-FDH$_2$)(*o*-FDH)Cl]·6.5H$_2$O was obtained, in which the organic *o*-phenylenediamineglyoxime (*o*-FDH$_2$) ligand is very little studied. The crystal structure of this compound revealed that this new oxime ligand was obtained by condensation of two organic components and coordinates to the metal atom like most *vic*-dioximes - bidentate-chelate through two nitrogen atoms, forming metallic rings of five atoms. The complex compound has a molecular type, as it is formed by one neutral *o*-FDH$_2$ ligand, one mono-deprotonated *o*-FDH$^-$ ligand and a chloride anion.

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