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Synthesis and characterization of zinc(II), palladium(II) and platinum(II) complex with 2'-[1-(2-pyridinyl)ethylidene]oxamohydrazide. The crystal structure of bis{2'-[1-(2-pyridinyl)ethylidene]oxamohydrazido}zinc(II) trihydrate

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Abstract: Complexes of Zn(II), Pd(II) and Pt(II) with 2'-[1-(2-pyridinyl)ethylidene]oxamohydrazide (Hapsox) were synthesized and their structures were determined. All the complexes are of a neutral type with two apsox ligands coordinated to Zn(II) and one apsox ligand coordinated to Pd(II) or Pt(II). In each case, the polydentate was coordinated *via* pyridine and hydrazone nitrogens and α-oxyazine oxygen, forming an octahedral geometry around Zn(II), and a square planar one around Pd(II) and Pt(II). The structure determination was performed by IR, ^{1}H-NMR and ^{13}C-NMR spectroscopy, and for the Zn(II) complex by X-ray structure analysis.

Keywords: Zn(II) complex, Pd(II) complex, Pt(II) complex, 2-acetylpyridine derivative, X-ray analysis, NMR spectra.

INTRODUCTION

As a part of our studies on the influence of the nature of hydrazone/hydrazide ligands and their charge on the structure of transition metal complexes,[1–3] the synthesis of Zn(II), Pd(II) and Pt(II) complexes with a 2'-[1-(2-pyridinyl)ethylidene]oxamohydrazide (Hapsox) ligand is reported here.

In previous work, complexes with 2'2'''-(2,6-pyridinediyldiethylidyne)dioxamohydrazide (H_2dapsox)[1,4,5] were studied in detail, and some information were gathered on complexes with 2,6-bis[1-(methoxycarbonylmethylhydrazono)ethyl]pyridine

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(H_2dapetha).[6] The structure of the H_2dapsox ligand, as well as the charges and radii of the central metal ion favour, in most cases, pentagonal bipyramidal (PBP) geometry of the complexes.[3] On the other hand, with the H_2dapetha ligand, the Cu(II) and Co(II) complexes have trigonal bipyramidal (TBP) geometry.[6]

Both the above-mentioned ligands are very symmetric, having two identical side chains. In the molecule of 2-acetylpyridine and its hydrazone derivatives, one side chain is missing, so that such ligands are less symmetric. Therefore, it was of interest to establish the mode of coordination of 2'-[1-(2-pyridinyl)ethylidene]oxamohydrazide (Hapsox) as an analogue of dapsox but with only one side chain.

The first synthesized complex with a Hapsox ligand was the octahedral complex of Co(III) with two apsox ligands in monoanionic form coordinated meridionally as tridentates. The ligand is coordinated *via* the pyridine and hydrazone nitrogens, as well as the α-oxyazine oxygen, as confirmed by X-ray structure analysis.[7]

As a continuation of the study, two Cu(II) complexes with Hapsox were synthesized and structurally characterized.[8] In one of them two apsox ligands are coordinated in the same manner as in the Co(III) complex, giving a distorted octahedral geometry. The only difference between this complex and the Co(III) complex arose as the consequence of the hydrolysis of the terminal amide group in case of the Cu(II) complex, yielding a carboxylic group.[8] In the second, tetrahedral Cu(II) complex, the ligand is coordinated as a tridentate, forming a six-membered and a five-membered ring around the central metal ion. The fourth coordination site is occupied by a water molecule.[8]

EXPERIMENTAL

Synthesis of the [*Zn(apsox)$_2$*]·*3H$_2$O complex*

$Zn(OAc)_2{\cdot}2H_2O$ (0.11 g, 0.5 mmol) and Hapsox ligand (0.21 g, 1.0 mmol) were dissolved in 100 cm^3 of methanol and the solution was refluxed until it was clear (approx. 15 min). After 48 h, pale yellow crystals precipitated from the solution in 60 % (0.16 g) yield. IR spectrum (KBr; ν/cm^{-1}): 3654(*m*), 3393(*vs*), 3283(*s*), 1700(*s*), 1681(*vs*), 1629(*s*), 1593(*s*), 1573(*s*), 1546(*vs*), 1516(*vs*), 1469(*s*), 1436(*m*), 1374(*m*), 1325(*m*), 1283(*s*), 1166(*w*), 1145(*w*), 1100(*w*), 1072(*m*), 1050(*w*), 1018(*w*), 781(*m*), 743(*m*), 689(*s*), 656(*s*), 569(*m*). Elemental analysis: Found: C 41.06 %, H 4.31 %, N 21.52 %, Calcd. for $[Zn(apsox)_2]{\cdot}3H_2O$, $C_{18}H_{24}N_8O_7Zn$ (M_r = 529.82): C 40.80 %, H 4.57 %, N 21.15 %.

Syntheses of the [*Pd(apsox)Cl*]·*H$_2$O and* [*Pt(apsox)Cl*]·*H$_2$O complexes*

Hapsox ligand (0.14 g, 0.7 mmol) was dissolved in a minimum amount of ethanol and an aqueous solution containing an equimolar amount of $K_2[PdCl_4]$ (0.23 g, 0.7 mmol) or $K_2[PtCl_4]$ (0.29 g, 0.7 mmol) was added. The mixture was refluxed until it was clear (approx. 2 h).

[*Pd(apsox)Cl*]·*H$_2$O*. The microcrystalline product precipitated after cooling the reaction mixture to room temperature. After filtering, the crystals were washed with ethanol. Yield: 0.13 g (51 %). IR spectrum (KBr; ν/cm^{-1}): 3411(*vs*), 3293(*m*), 3235(*m*), 3169(*m*), 1708(*vs*), 1598(*s*), 1520(*vs*), 1463(*m*), 1438(*m*), 1381(*w*), 1335(*w*), 1276(*s*), 1154(*w*), 1079(*w*), 1038(*w*), 783(*w*), 759(*w*), 708(*m*), 585(*m*), 551(*m*), 456(*w*). Elemental analysis: Found: C 30.03 %, H 2.84 %, N 15.34 %, Calcd. for $[Pd(apsox)Cl]{\cdot}H_2O$, $C_9H_{11}N_4O_3ClPd$ (M_r = 365.09): C 29.61 %, H 3.04 %, N 15.35 %.

[*Pd(apsox)Cl*]·H_2O. An orange microcrystalline precipitate separated after keeping the reaction mixture in a refrigerator for 24 h. After filtration, the crystals were washed with ethanol. Yield: 0.18 g (58 %). IR spectrum (KBr; ν/cm^{-1}): 3414(*s*), 3307(*m*), 3237(*m*), 3168(*m*), 1709(*s*), 1645(*w*), 1599(*m*), 1512(*s*), 1465(*m*), 1437(*m*), 1381(*w*), 1272(*s*), 778(*w*), 712(*m*), 594(*w*), 556(*m*). Elemental analysis: Found: C 24.25 %, H 2.03 %, N 12.67 %, Calcd. for [Pt(apsox)Cl]·H_2O, $C_9H_{11}N_4O_3ClPt$ (M_r = 453.78): C 23.82 %, H 2.45 %, N 12.35 %.

Physical measurements

Elemental C, H, N analysis was performed by the standard micromethod in the Centre for Instrumental Analysis, Faculty of Chemistry, University of Belgrade. The ^{1}H-NMR spectra were recorded on a Varian Gemini 2000 spectrometer at 200 MHz, and ^{13}C-NMR spectra were recorded on the same instrument at 50 MHz. The IR spectra were recorded on a Perkin-Elmer FTIR 1726X spectrophotometer using the KBr technique. The molar conductivity of a DMF solution of the complex (1×10^{-3} mol dm^{-3}) was measured at room temperature on a Jenway - 4009 digital conductivity meter.

X-Ray structure determination of the Zn(II) complex[*]

Single-crystal diffraction data was collected using MoKα radiation on a Bruker SMART APEX system with a 2K resolution CCD deterctor. The data collection was computed using the SMART software;[10] integration of the collected frames and cell refinement were done using SAINT.[11] Space-group determination, numerical absorption correction and structure solution/refinement were all performed using the SHELXL-97 program package.[12] All H atoms were found in the difference Fourier map and were refined isotropically. Determination of hydrogen bonds was performed using the program PLATON.[13] Data were collected first at 293 K and then at 110 K due to problems with refining the crystal water. For the data collection at 110 K, the crystal had to be "shock cooled" since it dried and cracked within minutes if exposed to a dry gas flow at room temperature. There were no significant structural changes between the two temperatures.

The crystal data for the [Zn(apsox)$_2$]·3H_2O complex are shown in Table I.

TABLE I. Crystal data and structure refinement details for the [Zn(apsox)$_2$]·3H_2O complex

Empirical formula	$C_{18}H_{24}N_8O_7Zn$
Formula weight	529.82
Temperature	110(2) K
Wavelength	0.71073 Å
Crystal system; space group	monoclinic; $P2_1/n$
Unit cell dimensions	a = 13.651(1) Å α = 90º b = 9.5100(8) Å β = 94.568(2)º c = 17.363(1) Å γ = 90º
Volume	2247.1(3) Å
Z, Calculated density	4, 1.566 g/cm^3
Absorption coefficient	1.151 mm^{-1}
F(000)	1096
Crystal size	0.4× 0.3× 0.2 mm
Theta range for data collection	1.83 to 28.29º

* Deposition number: CCDC 209204 (room-temperature) and 209205 (low-temperature)

TABLE I. Continued

Limiting indices	$-17 \le h \le 17, -12 \le k \le 12, -11 \le l \le 21$
Reflections collected / unique	13425 / 5106 [R(int) = 0.0412]
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5106 / 0 / 403
Goodness-of-fit on F^2	0.894
Δ/σ_{max}	0.02
T_{max}/T_{min}	0.3534 / 0.2618
Final R indices [I > 2σ(I)]	R1 = 0.0343; wR2 = 0.0591
R indices (all data)	R1 = 0.0510; wR2 = 0.0629
Largest diff. peak and trough	0.566 and –0.422 e. $Å^{-3}$

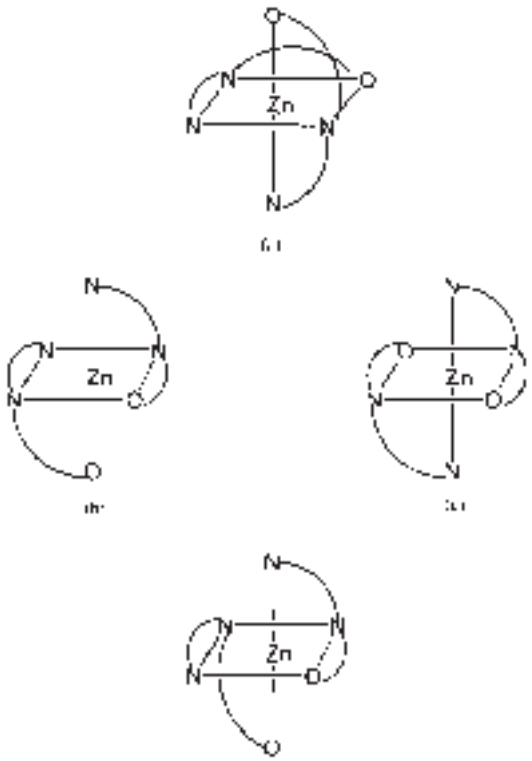
RESULTS AND DISCUSSION

The first complex described in this work is the Zn(II) complex with 2'-[1-(2-pyridinyl)ethylidene]oxamohydrazide (Hapsox). The complex was obtained in methanol solution from $Zn(OAc)_2 \cdot 2H_2O$ and Hapsox ligand (mole ratio 1:2) in 60 % (0.16 g) yield, as a yellow single crystal product. The structure was assumed on the basis of elemental microanalysis, IR and ^{1}H-NMR spectra, and confirmed by X-ray structure analysis.

In the IR spectrum of the Zn(II) complex, bands at 3393 cm^{-1} (*vs*) and 3283 cm^{-1} (*s*) attributed to the ν(NH) vibrations of the primary amide are observed. The most significant feature of the spectrum is the presence of only one band at 1700 cm^{-1} (*s*) due to ν(C=O) vibrations, in contrast to two such bands in the spectrum of the free ligand (1729 cm^{-1} (*vs*) and 1706 cm^{-1} (*vs*)). The loss of one band is attributed to the deprotonation of –NH–CO– group and electron delocalization in the corresponding anion. The absorption band of the coordinated –N=C–O^- group is at 1283 cm^{-1} (*s*). Bands between 1600 and 1400 cm^{-1} are attributed to skeletal vibrations of the pyridine ring and ν(C–N) vibrations.

^{1}H-NMR spectrum of the $[Zn(apsox)_2] \cdot 3H_2O$ complex was recorded in DMSO-d_6 with TMS as an internal standard. The spectral data of the free ligand (Hapsox)[7] and the complex are given in Table II. The ^{13}C-NMR spectrum of the complex was not recorded because of the low solubility of the complex.

Comparing the spectrum of the ligand with that of the complex, it can be seen that the chemical shifts of the methyl group (C7) and C3 protons are higher in the complex than in the ligand, due to electron withdrawal from the nitrogens N1 and N2 by zinc. The electronic effects arising from coordination of the pyridine nitrogen are more prominent for the *para* (C3) and *ortho* (C1) hydrogens, than for the *meta* (C2 and C4) ones.



Scheme 1.

Final confirmation of the structure of the complex was obtained by X-ray structure analysis. The complex is of a neutral type with three molecules of crystal water. Two ligand molecules are coordinated in the monoanionic form, giving a deformed octahedral geometry. The largest distortion is observed for Zn–N6 and Zn–O3 bonds, the length of which are 2.0663(2) and 2.1262(1) Å, respectively. The apsox ligand is coordinated to Zn(II) *via* the pyridine and hydrazone nitrogens as well as *via* the α-oxyazine oxygen. Theoretically, there are four possible geometric isomers (Scheme 1) and the crystal structure of the $[Zn(apsox)_2]\cdot 3H_2O$ complex shows that the apsox ligands are meridionally coordinated (Scheme 1a), as expected since both ligands are tridentates when coordinated in the monoanionic form (Fig. 1). Their coordination in the α-oxyazine form results in the formation of four five-membered rings around Zn(II), which are more planar than in the analogous complexes with the dapsox ligand.[9] Selected bond lengths and angles are given in Table III. As the geometries of both ligands are almost identical, data are shown for one ligand only. The C8–O1, C8–N3 and N2–N3 are typical for deprotonation at N3 and the consequential electron delocalization.

The crystal packing is characterized by strong intermolecular hydrogen bonds involving most of the potential donors and acceptors (Table IV). Hydrogen bond

system in the structure is three-dimensional. The amide nitrogens N8 and N4 are double donors, as are the oxygens O5 and O7 from molecules of water, while the water oxygen O6 is a single donor. The consequence is that the complete structure is multi-layered. Namely, molecules of the complex and water molecules form layers parallel to the ab-plane.

TABLE II. ^{1}H-NMR spectral data of the ligand (Hapsox) and the complexes $[Zn(apsox)_2]\cdot 3H_2O$, $[Pd(apsox)Cl]\cdot H_2O$ and $[Pt(apsox)Cl]\cdot H_2O$ in DMSO-d_6

Assignment (multiplicity, number of H-atoms)	Chemical shift, δ/ppm			
	Ligand	Zn complex	Pd complex	Pt complex
C1 (*d*, 1H)	8.64	7.87	7.87	8.06
C2(*t*, 1H)	7.47	7.49	7.69	7.78
C3 (*t*, 1H)	7.89	complex signal 8.05	8.24	8.23
C4 (*d*, 1H)	8.09		8.40	8.53
C7 (*s*, 3H)	2.44	2.71	2.49	2.37
N3 (*s*, 1H)	10.82	–	–	–
N4a (*s*, 1H)	8.07	7.33	7.69*	7.78*
N4b (*s*, 1H)	8.41	7.67	7.87**	7.98

*The signal overlaps with the signal of C2–H; **The signal overlaps with the signal of C1–H

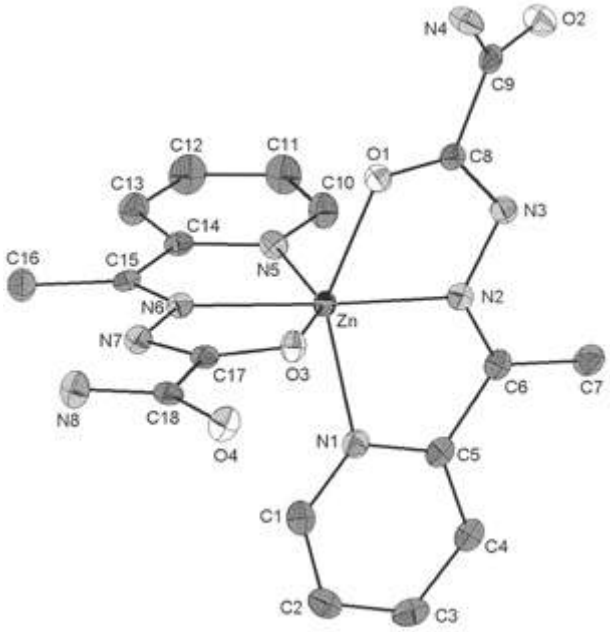


Fig. 1. ORTEP view of the $[Zn(apsox)_2]\cdot 3H_2O$ complex (H atoms are omitted for the sake of clarity).

TABLE III. Relevant bond lengths (Å) and angles(°) for $[Zn(apsox)_2]\cdot 3H_2O$

Bond lengths		*Bond angles*	
Zn–N6	2.066(2)	N6–Zn–N2	160.67(7)
Zn–N2	2.077(2)	N6–Zn–O1	111.44(6)
Zn–O1	2.092(1)	N2–Zn–O1	74.94(6)
Zn–O3	2.126(1)	N6–Zn–O3	75.54(6)
Zn–N1	2.177(2)	N2–Zn–O3	123.46(6)
Zn–N5	2.280(3)	O1–Zn–O3	89.75(5)
Cl–N1	1.335(3)	N6–Zn–N1	101.94(6)
C1–C2	1.385(3)	N2–Zn–N1	74.50(6)
C2–C3	1.370(3)	O1–Zn–N1	146.47(6)
C3–C4	1.385(3)	O3–Zn–N1	95.69(6)
C4–C5	1.379(3)	N6–Zn–N5	73.31(6)
C5–N1	1.357(3)	N2–Zn–N5	88.07(6)
C5–C6	1.491(3)	O1–Zn–N5	96.03(6)
C6–N2	1.287(3)	O3–Zn–N5	148.28(6)
C6–C7	1.492(4)	N1–Zn–N5	96.42(6)
C8–O1	1.276(2)		
C8–N3	1.326(3)		
C8–C9	1.523(3)		
C9–O2	1.231(3)		
C9–N4	1.327(3)		
N2–N3	1.389(2)		

The mode of coordination of the apsox ligand is identical to that of the H_2dapsox ligand. Namely, the two identical side chains in H_2dapsox are coordinated in the same way as the single side chain in apsox.[9]

The Pd(II) and Pt(II) complexes with Hapsox as ligand were prepared by the reaction of the ligand with $K_2[PdCl_4]$ and $K_2[PtCl_4]$, respectively.

The composition of the complexes was determined by elemental microanalysis. The IR spectra of the Pd(II) and the Pt(II) complexes are similar. The bands corresponding to ν(NH) vibrations of the primary amide moiety are at 3411 cm^{-1} (*vs*) and 3293 cm^{-1}(*vs*) for the Pd(II) complex and at 3415 cm^{-1} (*vs*) and 3307 cm^{-1} (*vs*) for the Pt(II) complex. In the spectral region around 1700 cm^{-1}, there are bands (ν(C=O), amide I) at 1708 cm^{-1} (*vs*) in the spectrum of the Pd(II) complex and at 1709 cm^{-1} (*vs*) in the spectrum of the Pt(II) complex. The presence of the bands at 1276 cm^{-1} (*s*) and 1272 cm^{-1} (*s*) in the spectra of pallatium and platinum complexes, respectively, originating from coordinated –N=C–O^- groups, indicates deprotonation of the Hapsox ligand in complexes. Finally, the group of bands at 1600–1400 cm^{-1} corresponds to skeletal pyridine ring and ν(C–N) vibrations.

The ^{1}H-NMR spectral data of the [Pd(apsox)Cl]·H_2O and [Pt(apsox)Cl]·H_2O complexes are given in Table II. As with the Zn complex, deprotonation of nitrogen N3 upon complexation can be seen by loss of the NH signal at 10.82 ppm. By deprotonation, the electron density at oxygen O1 is increased [–N$^-$–C=O ↔ –N=C–O$^-$] making it a better electron donor for coordination. Other changes in the chemical shifts are rather similar to the [Zn(apsox)$_2$]·3H_2O complex indicating that the same atoms participate in the coordination.

The significant chemical shift changes of methyl C7, carbonyl C8 and pyridine ring carbon atom signals in the ^{13}C-NMR spectrum upon complexation with platinum (Table V) confirm that the coordination sites are pyridine nitrogen, hydrazone nitrogen and α-oxyazine oxygen. Based on elemental analysis and the spectral data for the Pd(II) and Pt(II) complexes, it can be concluded that the ligand is coordinated as a tridentate and the fourth coordination site is occupied by a chloride ion. The geometry of the complexes is square planar, characteristic for Pd(II) complexes with coordination number 4. Analogous complexes with H_2dapsox could not be obtained, possibly because of steric reasons.

Since metal complexes of 2-acetylpyridine semicarbazones and thiosemicarbazones are biologically active,[14–17] the antimicrobial and antitumor activities of the synthesized complexes will be the subject of further research.

TABLE IV. List of hydrogen bonds for [Zn(apsox)$_2$]·3H_2O

D – H	D ... A	H ... A	D – H ... Acceptor
O5 – H19	O5 ... O2(i)	H19 ... O2(i)	O5 – H19 ... O2(i)
0.84(3)	2.751(3)	0.84(3)	154(3)
O5 – H23	O5 ... O6	H23 ... O6	O5 – H23 ... O6
0.78(3)	2.848(3)	2.08(3)	168(3)
O6 –H20	O6 ... O5(ii)	H20 ... O5(ii)	O6 – H20 ... O5
0.87(3)	2.817(3)	1.95(3)	170(2)
O7 – H21	O7 ... O4(iii)	H21 ... O4(iii)	O7 – H21 ... O4(iii)
0.82(2)	2.839(2)	2.07(2)	156(2)
O7 – H22	O7 ... O5(iiii)	H22 ... O5(iiii)	07 – H22 ... O5(iiii)
0.76(3)	2.905(3)	2.15(3)	173(2)
N8 – H12	N8 ... O7(iv)	H12 ... O7(iv)	N8 – H12 ... O7(iv)
0.81(2)	2.959(3)	2.20(2)	156(2)
N8 – H13	N8 ... O3(ivv)	H13 ... O3(ivv)	N8 – H13 ... O3(ivv)
0.86(2)	3.056(2)	2.34(2)	141.3(19)

TABLE IV. Continued

D – H	D ... A	H ... A	D – H ... Acceptor
N4 – H3	N4 ... N7(is)	H3 ... N7(is)	N4 – H15 ... O4(is)
0.845(19)	3.039(3)	2.230(19)	160.3(19)
N4 – H15	N4 ... O4(iss)	H15 ... O4(iss)	N4 – H15 ... O4(iss)
0.86(2)	2.992(2)	2.20(2)	153.5(19)

Symmetry operations: (i) $x, -1+y, z$; (ii) $1/2-x, -1/2+y, 3/2-z$; (iii) $x, -1+y, -1+z$; (iiii) $1/2+x, 1/2-y, -1/2+z$; (iv) $1/2-x, 1/2-y, 3/2-z$; (ivv) $1/2-x, -1/2+y, 5/2-z$; (is) $x, 1+y, z$; (iss) $1/2-x, 1/2+y, 5/2-z$.

TABLE V. ^{13}C-NMR spectral data of the ligand (Hapsox) and of the complex $[Pt(apsox)Cl]\cdot H_2O$ in DMSO-d_6

Assignment	Chemical shift, δ/ppm	
	Ligand	Complex
C1	149.0	141.0
C2	124.9	130.6
C3	137.0	149.2
C4	120.8	129.7
C5	154.7	157.4
C6	162.1	160.0
C7	12.2	16.7
C8	157.5	151.2
C9	156.8	155.0

Supplementary materials: Cambridge Crystallographic Data Centre, CCDC 209204 (room-temperature) and 209205 (low-temperature) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge *via* www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

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ИЗВОД

СИНТЕЗА И КАРАКТЕРИЗАЦИЈА КОМПЛЕКСА ЦИНКА(II), ПАЛАДИЈУМА(II) И ПЛАТИНЕ(II) СА 2´-[1-(2-ПИРИДИНИЛ)ЕТИЛИДЕН]ОКСАМОХИДРАЗИДОМ. КРИСТАЛНА СТРУКТУРА БИС{2´-[1-(2-ПИРИДИНИЛ)ЕТИЛИДЕН]ОКСАМОХИДРАЗИДО}ЦИНК(II) ТРИХИДРАТА

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У овом раду описана је синтеза и структурна карактеризација комплекса Zn(II), Pd(II) и Pt(II) са 2´-[1-(2-пиридинил)етилиден]оксамохидразидом (Hapsox). Сва три добијена комплекса су неутралног типа са два координована apsox лиганда у случају комплекса Zn(II), односно једним apsox лигандом у случају Pd(II) и Pt(II) комплекса. Координација полидентата је у свим комплексима остварена преко пиридинског азота, хидразонског азота и α-оксиазинског кисеоника градећи октаедарску геометрију око Zn(II), односно квадратно планарну геометрију око Pd(II) и Pt(II). Потврду за овакве структуре добили смо из ИЦ, ^{1}H-NMR и ^{13}C-NMR спектара, док је за Zn(II) комплекс урађена и рендгенска структурна анализа.

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REFERENCES

1. I. Ivanović, K. Anđelković, V. Beljanski, B. Prelesnik, V. M. Leovac, M. Momirović, *J. Coord. Chem.* **42** (1997) 335
2. G. Pelizzi, A. Bacchi, I. Ivanović-Burmazović, M. Gruden, K. Anđelković, *Inorg. Chem. Commun.* **4** (2001) 311
3. A. Bacchi, I. Ivanović-Burmazović, G. Pelizzi, K. Anđelković, *Inorg. Chim. Acta* **313** (2001) 109
4. K. Anđelković, I. Ivanović, B. V. Prelesnik, V. M. Leovac, D. Poleti, *Polyhedron* **15** (1996) 4361
5. I. Ivanović-Burmazović, A. Bacchi, G. Pelizzi, V. M. Leovac, K. Anđelković, *Polyhedron* **18** (1998) 119
6. K. Anđelković, R. Tellgren, S. Niketić, D. Sladić, D. Poleti, *J. Chem. Crystallography* **29** (1999) 575
7. K. Anđelković, J. Howing, R. Tellgren, D. Jeremić, I. Ivanović-Burmazović, D. Sladić, *J. Coord. Chem.* **56** (2003) 611
8. A. Bacchi, D. Jeremić, G. Pelizzi, D. Sladić, M. Gruden-Pavlović, K. Anđelković, *Transition Metal Chemistry* (in press)
9. M. Šumar, I. Ivanović-Burmazović, I. Hodžić, K. Anđelković, *Syn. Reac. Inorg. Met. Org. Chem.* **32** (2002) 721
10. *SMART: Brucker's CCD Data Collection Program*, Madison, Wisconsin, USA
11. *SAINT: SAX, Brucker's Data Processing Program*, Madison, Wisconsin, USA
12. G. Sheldrick, *SHELXL-97, Program for Structure Refinement*, University of Göttingen, Germany, 1997
13. L. A. Spek, *PLATON/PLUTON, Acta Cryst.* **C34** (1990) A46
14. N. Fahmi, D. K. Shama, R. V. Singh, *Syn. Reac. Inorg. Metal-Org. Chem.* **24** (1994) 377
15. D. X. West, A. E. Liberta, S. B. Padnye, R. C. Chikale, P. B. Sonawane, A. S. Kumbhar, R. G. Yerande, *Coord. Chem. Rev.* **123** (1993) 49
16. E. Bermejo, R. Carballo, A. Castiñeiras, R. Dominguez, *Eur. J. Inorg. Chem.* **6** (1999) 965
17. H. L. Singh, M. Sharma, A. K. Varshney, *Syn. React. Inorg. Metal-Org. Chem.* **29** (1999) 817.