Syntheses and Characterization of Zinc, Cadmium, and Mercury(II) Complexes with N-(2-pyridyl)carbonylaniline (L), X-ray Crystal Structure of $[Hg(L)(SCN)_2]$

Ali Morsali*[,a], Ali Ramazani[b], Ali Reza Mahjoub[a], and Ali Akbar Soudi[b]

[a] Tehran / I.R. Iran, Department of Chemistry, School of Science, Tarbiat Modarres University
[b] Zanjan / I.R. Iran, Department of Chemistry, University of Zanjan

Received May 5th, 2003.

Abstract. The reaction of N-(2-pyridyl)carbonylaniline (L) with $Zn(NO_3)_2$, $CdCl_2$ and $Hg(SCN)_2$ gives the following complexes: $[Zn(L)_2](NO_3)_2$, $[Cd(L)_2Cl_2]$, and $[Hg(L)(SCN)_2]$. The new complexes were characterized by elemental analyses and IR-, ^1H-, ^{13}C-NMR spectroscopy. The crystal structure of the $[Hg(L)(SCN)_2]$ was determined by single crystal X-ray analysis. The monomeric complex is built up of a $Hg(SCN)_2$ unit with one N-(2-pyridyl)carbonylaniline (L) ligand coordinated to the Hg atom via the ring pyridinic nitrogen atom and the carbonyl oxygen atom forming a five-membered chelate ring. The Hg atom has a distorted tetrahedral environment. There is π-π stacking interaction between the parallel aromatic rings belonging to adjacent chain as planar species in which the mean molecular planes are close to parallel and separated by a distance of ~ 3.5 Å, close to that of the planes in graphite. The coordinated N-(2-pyridyl)carbonylaniline (L) molecule is involved in hydrogen bonding acting as hydrogen-bond donors with S and N atoms from SCN^- ligand as potential hydrogen-bond acceptors. There is a shortest intermolecular contacts between the S and N atoms. The hydrogen bonding and shortest intermolecular contacts between the S and N atoms yields infinite chains parallel to the crystallographic vector c. Each molecule is bonded to two neighbors.

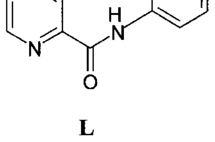
Keywords: Zinc; Cadmium; Mercury; Crystal structure; N-(2-pyridyl)carbonylaniline ligand.

Synthese und Charakterisierung der Zink, Cadmium und Quecksilber(II) Komplexe mit N-(2-pyridyl)carbonylanilin (L), Röntgen-Kristallstrukturanalyse von $[Hg(L)(SCN)_2]$

Inhaltsübersicht. Die Reaktion von N-(2-pyridyl)carbonylanilin (L) mit $Zn(NO_3)_2$, $CdCl_2$ und $Hg(SCN)_2$ ergibt die Komplexe: $[Zn(L)_2](NO_3)_2$, $[Cd(L)_2Cl_2]$ und $[Hg(L)(SCN)_2]$. Die Komplexe wurden mittels Elementaranalyse und IR-, ^1H-, ^{13}C-NMR-Spektroskopie charakterisiert. Die Kristallstruktur von $[Hg(L)(SCN)_2]$ wurde durch Einkristall-Röntgenstrukturanalyse bestimmt. Der monomere Komplex wird von einer $Hg(SCN)_2$-Einheit mit einem N-(2-pyridyl)carbonylanilin-Liganden (L) aufgebaut, der über das N-Atom des Pyridins und das Carbonyl-Sauerstoffatom an das Hg-Atom zu einem fünfgliedrigen Chelatring koordiniert ist. Das Hg-Atom hat eine verzerrt tetraedrische Koordination. Es besteht eine π-π-Wechselwirkung zwischen den parallelen aromatischen Ringen benachbarter Ketten im Abstand von ~ 3.5 Å. Die koordinierten N-(2-pyridyl)carbonylanilin-Moleküle (L) bilden Wasserstoffbrückenbindungen zu den S- und N-Atomen des SCN^--Liganden. Durch die Wasserstoffbrücken und kurzen intermolekularen Kontakte zwischen den S- und N-Atomen werden unendliche Ketten parallel zu *c* gebildet. Jedes Molekül ist an zwei Nachbarmoleküle gebunden.

1 Introduction

Research on the coordination chemistry of carbonylaniline-derived ligands has progressed very rapidly over the last two decades [1−4]. The incorporation of carbonylaniline groups in the design of ligands and the preparation of new complexes allows both the electronic and steric control of the properties of metal complexes. A six-membered heterocycle, like pyridine, and an amide, such as carbonylaniline, as a single ligand system are of particular interest [5−6], because the electronic communication between these two groups can be avoided. The general structure of the ligand is shown below.



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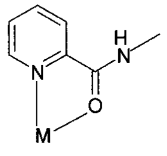

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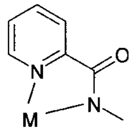

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The presence of a pyridyl nitrogen atom ortho to the amide substituent allows the ligand (L) to chelate a metal ion

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* Ali Morsali
Department of Chemistry, School of Chemistry,
Tarbiat Modarres University
P.O. Box 14155-4839
Tehran / I.R. Iran
E-mail: morsali_a@yahoo.com

DOI: 10.1002/zaac.200300159

in a bidentate fashion forming a five membered chelate ring bonding either to the N atom or to the carbonyl O atom of the amide group. Both types of isomers, O-bonded and N-bonded, are known for the ligand glycinamide, chelated to a metal atom [7−9]. This ligand is interesting not only because of the structural chemistry of its multifunction coordination modes but also because of the formation of complexes similar to metal-peptides [10−11]. The ability of zinc (II), cadmium (II), and mercury (II) salts to form a wide variety of 1:1 and 1:2 complexes with neutral ligands is well-documented [12−16]. The most common neutral donors are phosphine and amine derivatives. Most of these complexes contain halide ions; in contrast, complexes with thiocyanate ions have rarely been reported [17−19]. This ion is an ambidentate ligand and can coordinate metal ions via both the N and S atoms. The type of coordination depends on the nature of the metal center. N-donor atoms are more likely to coordinate zinc in complexes, whereas the S atom is the expected ligand for Hg^{+2} in mercury(II) complexes [20]. In part one of our current study, we reported the preparation and characterization of a series of 1:1 and 1:2 Zn^{II}, Cd^{II}, and Hg^{II} complexes [21−22], which provides ample justification for investigating the coordination chemistry of metals with terminal carbonylaniline-derived ligands. Here, we show the nature of adducts formed between Zn^{II}, Cd^{II}, and Hg^{II} ions and the N-(2-pyridyl)carbonylanilineligand (L).

2 Experimental

2.1 Physical measurment

IR spectra were recorded as Nujol mulls using a Perkin- Elmer 597 and a Nicolet 510P spectrometers. Elementalanalyses were carried out using a Heraeus CHN-O-Rapid analyzer. The ^{1}H- and ^{13}C-solution NMR spectra were recorded on a Bruker DRX-500 AVANCE spectrometer.

2.2 Synthesis of ligand

The ligand N-(2-pyridyl)carbonylaniline (L) was synthesized as previously reported [1].

2.3 Synthesis of the $[Zn(L)_2](NO_3)_2$ complex

The colorless crystals of the $[Zn(L)_2](NO_3)_2$ complex were obtained from the reaction of hydrated $Zn(NO_3)_2$ with N-(2-pyridyl)carbonylaniline in a molar ratio of 1:2 in aqueous solution. The resulting colorless solution was heated and stirred for about 1 h. It was then left to evaporate at room temperature; after a few days, colorless crystals were isolated. Yield: 0.29g, 50 %), m.p. 220 °C.

Analysis for $C_{24}H_{20}N_6O_8Zn$ (585.39); C 40.2 (calc. 41.2); H 3.2 (3.4); N 14. 2 (14.3) %.

IR (KBr, cm^{-1}) ν(N-H) 3220; ν(C-H)$_{ar}$ 3015; ν(C=C), ν(C=N) 1620, 1550; ν(C=O) 1658; ν(NO_3) 1370. **^{1}H NMR** ([D6]Aceton) δ: 7.1-7.2 (m, 1H), 7.3-9.4 (m, 2H), 7.5-7.6 (m, 1H), 7.9(m, 2H), 8.0 (m, 1H), 8.2-8.6 (m, 1H), 10.1 (s, 1H). **^{13}C-{^{1}H} NMR** ([D6]Aceton) δ: 119.3, 121.6, 123.4, 126.2, 128.3, 137.4, 138.1, 147.6, 149.8, 161.4.

Table 1 Crystal data and structure refinement for $[Hg(L)(SCN)_2]$.

Empirical formula	$C_{14}H_{10}HgN_4S_2$
Formula weight	514.97
Temperature	120(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 15.7579(1 5) Å
	b = 8.744(9) Å
	c = 11.2705(11) Å
	β = 104.1738(2)°
Volume	1505.7(16) Å^3
Z	4
Density (calculated)	2.272 Mg/m^3
Absorption coefficient	10.504 mm^{-1}
F(000)	968
Crystal size	0.20 x 0.08 x 0.02 mm^3
Theta range for data collection	2.68 to 27.10.
Index ranges	$-20 \leqq h \leqq 20$, $-11 \leqq k \leqq 11$, $-14 \leqq I \leqq 14$
Reflections collected	20018
Independent reflections	3321 [R(int) = 0.0645]
Completeness to theta = 30.060	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.3 and 0.05
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3321/0/199
Goodness-of-fit on F^2	1.041
Final R indices for 3037 ref 1. [I>2sigma(I)]	Rl = 0.0368, wR2 = 0.0870
R indices (all data)	R1 = 0.0394, wR2 = 0.0889
Largest diff. Peak, hole	2.6610, −2.152 e. Å^{-3}

2.4 Preparation of $[Cd(L)_2Cl_2]$ and $[Hg(L)(SCN)_2]$ complexes

We prepared the complexes, $[Cd(L)_2Cl_2]$, and $[Hg(L)(SCN)_2]$, in the same way as the $[Zn(L)_2](NO_3)_2$ complex.

$[Cd(L)_2Cl_2]$: Reacting materials: N-(2-pyridyl)carbonylaniline (L), cadmium(II) chloride (1:2). − White crystals; Mp = 190 °C. Yield: 0.32g, 60 %.

Analysis for $C_{24}H_{20}N_4O_2Cl_2Cd$(579.32);C 49.4 (calc. 49.7); H 3.3 (3.5); N 9.9 (9.7) %.

IR (cm^{-1}) selected bands: ν(N-H) 3210; ν(C-H)$_{ar}$ 3010; ν(C=C), ν(C=N) 1610, 1580; ν(C=O) 1640. **^{1}H NMR** ([D6]Aceton) δ: 7.1-7.1 (m, 1H), 7.3-9.4 (m, 2H), 7.5 (m, 1H), 7.9(m, 2H), 7.9-8.0 (m, 1H), 8.2-8.6 (m, 1H), 10.2 (s, 1H). **^{13}C-{^{1}H} NMR** ([D6]Aceton) δ: 119.4, 121.6, 123.5, 126.2, 128.3, 137.4, 138.1, 147.8, 149.6, 161.5.

$[Hg(L)(SCN)_2]$: Reacting materials: N-(2-pyridyl)carbonylaniline (L), mercury(II) thiocyanate (1:1). − Yellow-white crystals. Mp = 210 °C. Yield: 0.26g, 50 %

Analysis for $C_{14}H_{10}N_4OS_2Hg$ (514.72):C 32.8 (calc. 32.8); H 1.6 (1.9); N 10.1 (10.9) %.

IR (cm^{-1}) selected bands: ν(N-H) 3216; ν(C-H)$_{ar}$ 3030; ν(C=C), ν(C=N) 1615, 1570; ν(C=O) 1650, ν(SCN) 2030. **^{1}H NMR** ([D6]Aceton) δ: 7.1-7.2 (m, 1H), 7.3-9.4 (m, 2H), 7.5 (m, 1H), 7.9 (m, 2H), 7.9-8.1 (m, 1H), 8.2-8.6 (m, 1H), 10.2 (s, 1H). **^{13}C-{^{1}H} NMR** ([D6]Aceton) δ: 119.3, 121.6, 123.7, 126.3, 128.3,131.2, 137.4, 138.0, 147.80, 149.5, 161.5.

2.5 Determination of the crystal structure

Crystallographic measurements were carrried out at 120(2) K for $[Hg(L)(SCN)_2]$ using a Siemens R3m/V diffractometer. The intensity data was collected in the range, $2.68 \leq \theta \leq 27.10°$ using graphite monochromated $Mo-K_\alpha$ radiation (λ=0.71073 Å). Accurate unit cell parameters and orientation matrix for data collection are ob-

Table 2 Selected bond lengths /Å and angles /° for $[Hg(L)(SCN)_2]$.

Hg(1)-N(1)	2.4(6)	S(1B)-Hg(1)-O(1)	99.6(1)
Hg(1)-O(1)	2.5(3)	N(1)-Hg(1)-N(1B)#1	83.3(2)
Hg(1)-S(1A)	2.4(2)	S(1A)-Hg(1)-N(1B)#1	89.8(1)
Hg(1)-S(1B)	2.5(2)	S(1B)-Hg(1)-N(1B)#1	85.2(1)
Hg(1)-N(1B)#1	2.8(6)	O(1)-Hg(1)-N(1B)#1	151.1(2)
Hg(1)-N(1A)#2	3.2(6)	N(1)-Hg(1)-N(1A)#2	142.9(2)
N(1)-Hg(1)-S(1A)	119.3(1)	S(1A)-Hg(1)-N(1A)#2	63.8(1)
N(1)-Hg(1)-S(1B)	96.6(1)	S(1B)-Hg(1)-N(1A)#2	93.8(1)
N(1)-Hg(1)-O(1)	67.9(2)	O(1)-Hg(1)-N(1A)#2	75.4(1)
S(1A)-Hg(1)-O(1)	102.3(1)	N(1B)#1-Hg(1)-N(1A)#2	133.1(2)
S(1B)-Hg(1)-S(1A)	142.9(7)		

Symmetry codes #1: x+1, −y+5/2, z+1/2 #2: x, −y+5/2, z−1/2

Table 3 Hydrogen bond and shortest intermolecular contact parameters (distances /Å and angles /°) for the $[Hg(L)(SCN)_2]$.

	D-H		d(D-H)	d(H···A)	d(D...A)	<(DHA) A
N(8)-H(8A)	0.86	2.3	3.1(8)		160.8	N(1A) [x, −y+3/2, z−1/2]
C(10)-H(10)	0.97	2.9	3.6(7)		131.8	N(4) [x, −y, z+1/2]
S(1A)			3.1(6)			N(1A) [x, 2.5−y, 0.5+z]

Table 4 Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters ($Å^2$ × 10^3) for $[Hg(L)(SCN)_2]$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Hg(1)	1919(1)	11119(1)	9348(1)	29(1)
O(1)	2670(3)	9433(5)	8160(4)	33(1)
N(1)	1729(3)	8487(6)	9744(5)	29(1)
C(2)	1225(4)	8028(7)	10466(6)	29(1)
C(3)	988(4)	6523(7)	10562(6)	32(1)
C(4)	1260(4)	5451(7)	9838(6)	34(1)
C(5)	1782(4)	5975(8)	9077(7)	38(1)
C(6)	2014(4)	7413(7)	9072(6)	30(1)
C(7)	2616(4)	8059(7)	8320(5)	25(1)
N(8)	3079(3)	7000(6)	7880(5)	29(1)
C(9)	3682(4)	7272(7)	7156(6)	27(1)
C(10)	3642(4)	8566(7)	6431(6)	29(1)
C(11)	4224(4)	8702(7)	5688(6)	32(1)
C(12)	4834(4)	7570(8)	5660(6)	35(1)
C(13)	4880(4)	6303(7)	6400(7)	35(1)
C(14)	4311(4)	6152(5)	7147(6)	31(1)
S(1A)	3121(1)	12496(2)	10630(1)	31(1)
N(1A)	2964(4)	11552(6)	12958(5)	33(1)
C(1A)	3113(4)	11931(7)	12005(6)	29(1)
S(1B)	513(1)	11464(2)	7836(2)	33(1)
N(1B)	945(4)	13449(7)	6100(5)	37(1)
C(1B)	794(4)	12641(7)	6828(6)	30(1)

tained from a least−squares refinement. The intensities of 20018 unique reflections were measured, from which 3037 with I>2σ (I) were used for the refinement. The structure was solved by direct methods and refined by full-matrix least-squares techniques on F^2.

The positions of hydrogen atoms were idealized and included in the calculations of the structure factors as fixed contributions. Each hydrogen atom was assigned an isotopic thermal parameter. The values of R, R_w, with a goodness of fit on F^2 of 0.980 are 0.0368 and 0.0889, respectively. The final difference density map showed a maximum peak and hole of 2.661 and −2.152 e·Å^{-3}. Corrections for the Lorentz and polarization effects as well as the empirical correction for absorption were applied using the semi-empirical method. All structural calculations were carried out with a PDP − 11/23+ computer using the SDP − PLUS program package [23−24].

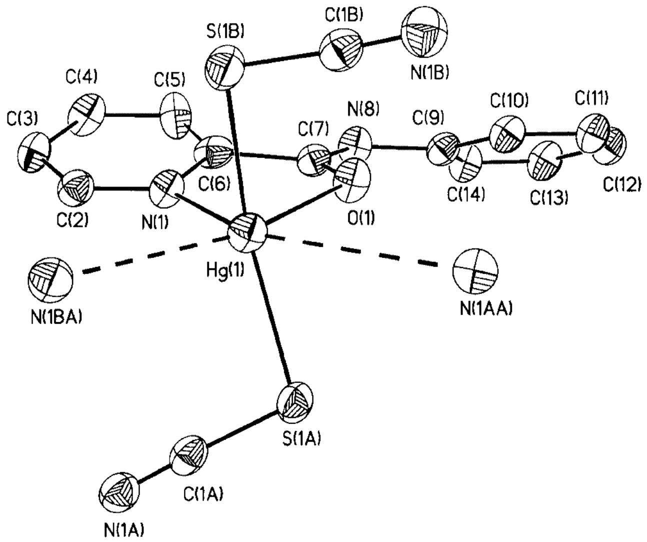

Fig. 1 ORTEP drawing of $[Hg(L)(SCN)_2]$

Crystal data and structure refinement and selected bond lengths and angles are given in Tables 1 and 2. Atomic coordination are given in Table 4. Anisotropy thermal parameters, observed and calculated structure factors, full lists of bond distances, bond angles and torsion angles are given in the supplementary material. An ORTEP drawingdiagram and a perspective view of the packing in the unit cell are shown in Figures 1 and 2.

3 Results and Discussion

3.1 Synthesis and spectroscopic properties of the complexes

The reaction of $Hg(SCN)_2$, $Zn(ClO_4)_2$, and $CdCl_2$ with N-(2-pyridyl)carbonylaniline (L) provided powder materials analyzed as $[Hg(L)(SCN)_2]$, $[Zn(L)_2](NO_3)_2$, and $[Cd(L)_2Cl_2]$, respectively. The IR spectrum of the $[Hg(L)(SCN)_2]$ complex shows ν(SCN) band at 2030 cm^{-1}; the spectrum of $[Zn(L)_2](NO_3)_2$ complex shows a band for $\nu(NO_3)$ at 1370 cm^{-1}. Attempts to isolate $[Hg(L)_2(SCN)_2]$ were failed; each time, a 1:1 adduct was isolated. The comparison of IR spectra of the free ligand with that of the complexes shows a shift in the ν(C=O) band of the amide substituent for the complexes from 1675 cm^{-1} for the ligand L to 1640 cm^{-1} for $[Cd(L)_2Cl_2]$, 1658 cm^{-1} for $[Zn(L)_2](NO_3)_2$, and 1650 cm^{-1} for [Hg(L)(SCN)2], this indicates that the oxygen atom of the amide substituent is coordinated to the metal atoms [25].

3.2 Crystal structure of $[Hg(L)(SCN)_2]$

An ORTEP drawing of $[Hg(L)(SCN)_2]$ is shown in Figure 1 and selected bond lengths and angles are given in Table 2. The complex is built up of a monomeric $Hg(SCN)_2$ unit, [Hg(1)-S(1A)=2.4 Å and Hg(1)-S(1B)=2.5 Å], with one N-(2-pyridyl)carbonylaniline ligand (L) coordinated to the Hg atom via one pyridine nitrogen and one carbonyl oxygen atom to form a distorted tetrahedron, Hg(1)-N(1) 2.4 Å

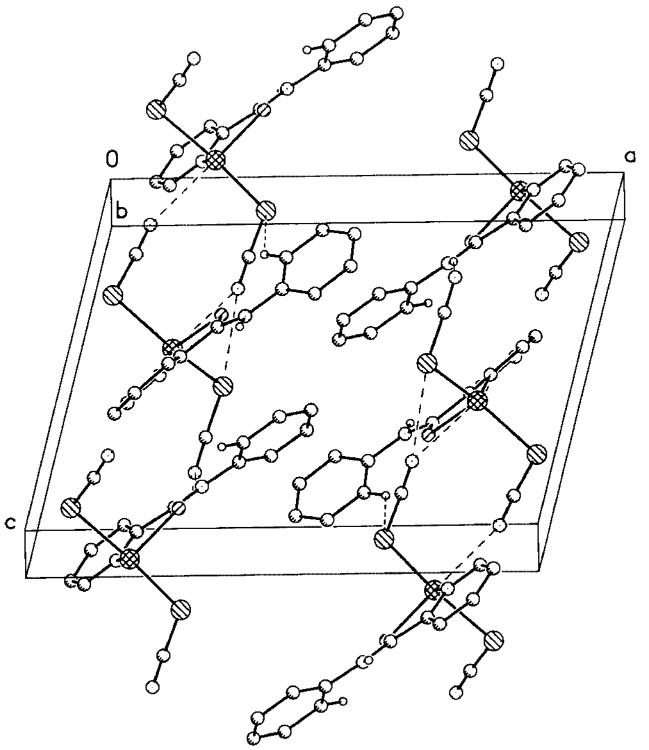

Fig. 2 The unit cell showing the π-π stacking in $[Hg(L)(SCN)_2]$

and Hg(1)-O(1) 2.5 Å. The N-(2-pyridyl)carbonylaniline ligand (L) is thus bidentate forming a five-membered heterocycle. The smallest and largest bond angles around the Hg atoms are N(1)-Hg(1)-O(1) 67.9° and S(1B)-Hg(1)-S(1A) 142.9(7)°. The intermolecular interaction between Hg(1) and two N atoms of two neighboring molecules (one nitrogen atom of thiocyanate anion and one amide nitrogen atom of the N-(2-pyridyl)carbonylaniline ligand (L) (Hg(1)-N(1B)>#1 2.8 Å and Hg(1)-N(1A)>#2 3.2 Å) turn the distorted Hg(1) tetrahedron to a distorted pseudo-octahedron. The tetrahedra are linked together by hydrogen bonding (Figure 2). The coordinated N-(2-pyridyl)carbonylaniline ligand (L) provides hydrogen atom donors for hydrogen bonding to the S and N atoms from SCN^- anion as potential hydrogen-bond acceptors. There is a short intermolecular contact between the S and N atoms (Table 3). As shown in Figure 2, the hydrogen bonding and shortest intermolecular distances between the S and N atoms yield infinite chains parallel to the crystallographic axis *c*. Each $[Hg(L)(SCN)_2]$ unit is bonded to two neighbors to form the molecules into a one-dimensional chain.

The thiocyanate ion in the lead(II) complexes are coordinated via the N atom [26−28]. In contrast, here the thiocyanate ions are coordinated via S atoms. This shows that Hg^{2+} ion is softer than Pb^{2+} ion. There is also π-π stacking interaction (charge-transfer arrays) [29−30] between the parallel aromatic rings of adjacent chains (Figure 2). The planar species, in which the mean molecular planes are almost parallel and separated by a distance of ~ 3.5 Å, are close to that of the planes in graphite.

Supplementary material: Complete bond lengths and angles, co-ordinates and displacement parameters have been deposited at Cambridge Crystallography Data Center. Supplementary data are available from CCDC, 12 Union Road,Cambridge CB21EZ, UK on request,by quoting the deposition number CCDC 208608.

Acknowledgements. Support of this investigation by Zanjan University Research Council [Synthesis of N^2-phenyl-2-pyridinecarboxamide and investigation of its complexation with metal ions (ZURC 8146)] is gratefully acknowledged.

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