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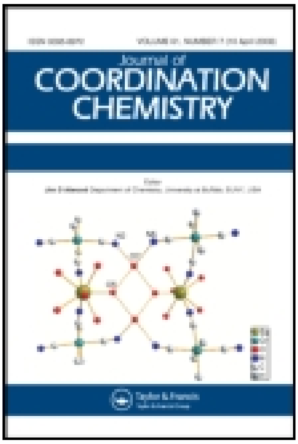

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gcoo20

Synthesis and characterization of Ni(II) complexes of O,N-donor Schiff bases derived from acyl pyrazolone analogues

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Published online: 10 Mar 2011.

To cite this article: Komal M. Vyas , Vrunda K. Shah & R.N. Jadeja (2011) Synthesis and characterization of Ni(II) complexes of O,N-donor Schiff bases derived from acyl pyrazolone analogues, Journal of Coordination Chemistry, 64:6, 1069-1081, DOI: 10.1080/00958972.2011.562290

To link to this article: http://dx.doi.org/10.1080/00958972.2011.562290

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Journal of Coordination Chemistry
Vol. 64, No. 6, 20 March 2011, 1069–1081

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Synthesis and characterization of Ni(II) complexes of O,N-donor Schiff bases derived from acyl pyrazolone analogues

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(*Received 19 July 2010; in final form 23 December 2010*)

Two bidentate Schiff bases, 5-methyl-2-*p*-tolyl-4-(1-*p*-tolylimino-propyl)-2H-pyrazol-3-ol (L1) and 2-(3-chloro-phenyl)-5-methyl-4-(1-*p*-tolylimino-propyl)-2H-pyrazol-3-ol (L2), were synthesized by condensation of 4-acyl pyrazolones with *p*-toluidine in ethanol. These ligands have been characterized by elemental analysis, infrared (IR), ^{1}H NMR, and mass spectra. A single crystal molecular structure of ligand L2 was also solved. Nickel(II) complexes of these ligands with general formula $[ML_2 \cdot 2H_2O]$ have been prepared by the interaction of aqueous solution of Ni-acetate with ethanolic solution of the appropriate ligand. The complexes were separated, analyzed, and their structures were elucidated on the basis of elemental analysis, Ni(II) determination, IR, UV-Vis, conductance, mass, and TGA-DTA data. Octahedral structure was proposed for the synthesized complexes.

Keywords: Acyl pyrazolone; Schiff bases; Ni(II) complexes; Pyrazolone derivatives

1. Introduction

Pyrazolones have been extensively studied due to their properties and applications. Pyrazolone sometimes refers to nonsteroidal anti-inflammatory agents. Pyrazolone derivatives, such as lactam-structure-related compounds, are also widely used in preparing dyes and pigments [1]. Acylated derivatives of pyrazolones (acyl pyrazolones), as heterocyclic β-diketones, have special characteristics such as extractability, colors suitable for photometric analyses, and intense biological activity [2]. These compounds display the majority of their properties as ligands in transition metal complexes. They are active as a pharmaceutical ingredient, especially in nonsteroidal anti-inflammatory agents used in the treatment of arthritis and other musculoskeletal and joint disorders. There are a number of reports in which the acyl pyrazolones bind with various metal ions resulting in structurally interesting coordination compounds [3, 4].

The chemistry of Schiff-base derivatives of 4-acylpyrazolones are less explored, eventhough these ligands also show appealing complexation properties [5–8]. Furthermore, Schiff-base formation extends the scope of the classical β-diketone

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2011 Taylor & Francis
DOI: 10.1080/00958972.2011.562290

system and provides an opportunity for constructing tridentate and tetradentate ligands from the bidentate 4-acylpyrazolone. Schiff bases of 4-acyl pyrazolones are therefore of interest [5, 6, 9–12]. There are few reports on Schiff-base Ni(II) complexes derived from acyl pyrazolone analogues [13–16].

The present study deals with the preparation of Ni chelates with 1-(5-hydroxy-3-methyl-1-*p*-tolyl-1H-pyrazol-4-yl)-propan-1-one and 1-[1-(3-chloro-phenyl)-5-hydroxy-3-methyl-1H-pyrazol-4-yl]-propan-1-one. The complexes obtained were characterized by elemental analysis, nickel determination, infrared (IR), TGA, FAB-MS, UV-Vis, magnetic moment, and molar conductance measurements.

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2. Experimental

2.1. *Materials*

3-Methyl-1-(4′-methylphenyl)-2-pyrazolin-5-one and 3-methyl-1-(3′-chlorophenyl)-2-pyrazolin-5-one were obtained from Nutan Dye Chem Pvt Ltd, Sachin, Surat. Para-toluidine and dioxane were obtained from Sisco-Chem. Pvt Ltd, Mumbai and used after purification following a standard procedure [17]. Absolute alcohol was obtained from Baroda Chem. Industries Ltd, Baroda, and used after distillation. Calcium hydroxide and nickel acetate were obtained from LOBA Chem. Pvt Ltd, Mumbai.

2.2. *Instruments*

Elemental analyses (C, H, N) were performed on a model 2400 Perkin-Elmer elemental analyzer. IR spectra were recorded on a model RX 1 FTIR Perkin-Elmer as KBr pellets. NMR spectra were recorded on a model 400 MHz Bruker. FAB-MS were recorded on a Jeol SX 102/Da-600 mass spectrometer at room temperature using *m*-nitro benzyl alcohol as a matrix and Argon/Xenon as the FAB gas. Electronic spectra were recorded on a Perkin-Elmer Lambda 35 UV–Vis spectrometer. Simultaneous TG/DTA was recorded on a EXSTAR6000 TG/DTA6300 model. Conductivity of the complexes was measured on a Elico CM 180 conductivity meter. GC-MS spectra were recorded on Trace GC ultra DSQ II.

2.3. *Synthesis of ligands*

2.3.1. Preparation of 1-(5-hydroxy-3-methyl-1-*p*-tolyl-1H-pyrazol-4-yl)-propan-1-one and 1-[1-(3-chloro-phenyl)-5-hydroxy-3-methyl-1H-pyrazol-4-yl]-propan-1-one. For L1, 3-methyl-1-(4′-methylphenyl)-2-pyrazolin-5-one ($0.1\,\mathrm{mol\,L^{-1}}$) and for L2, 3-methyl-1-(3′-chlorophenyl)-2-pyrazolin-5-one ($0.1\,\mathrm{mol\,L^{-1}}$), were dissolved in hot dioxane ($80\,\mathrm{cm^3}$) in a flask equipped with a stirrer, separating funnel, and reflux condenser. Calcium hydroxide ($0.2\,\mathrm{mol\,L^{-1}}$) was added to this solution, followed by propanoyl chloride ($0.1\,\mathrm{mol\,L^{-1}}$) added dropwise with caution, as this reaction was exothermic. During this addition the whole mass was converted into a thick paste. After addition the reaction mixture was refluxed for 30 min and then poured into hydrochloric acid ($2\,\mathrm{mol\,L^{-1}}$). The colored crystals thus obtained were separated by

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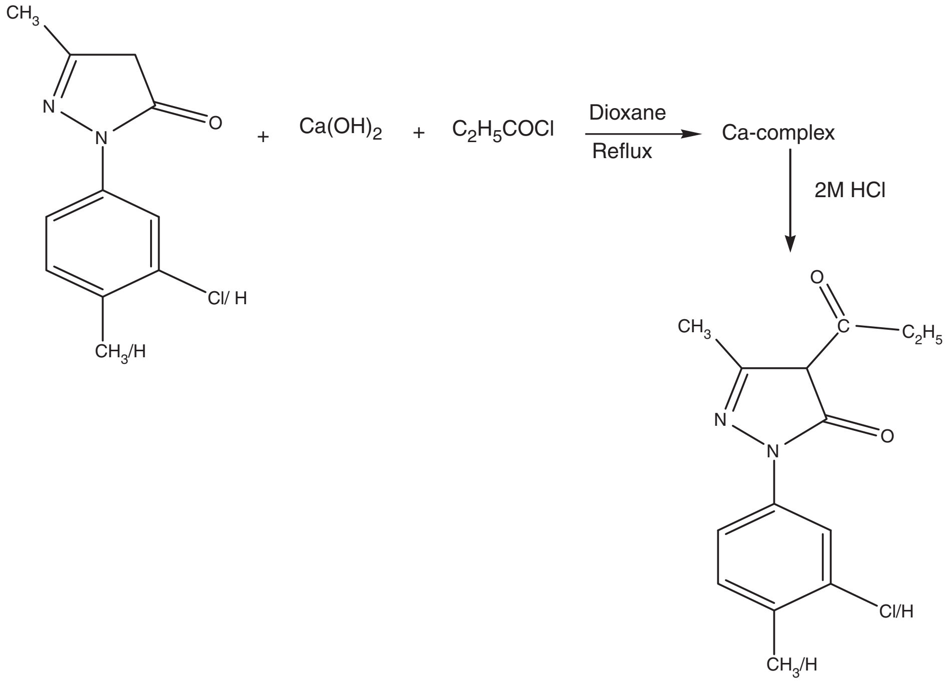

Scheme 1. Preparation of propyl pyrazolones.

filtration and recrystallized from an acidified methanol–water mixture [5]. Synthesis of these compounds can be summarized by reaction scheme 1.

2.3.2. Preparation of 5-methyl-2-*p*-tolyl-4-(1-*p*-tolylimino-propyl)-2H-pyrazol-3-ol (L1). 1-(5-Hydroxy-3-methyl-1-*p*-tolyl-1H-pyrazol-4-yl)-propan-1-one was dissolved in minimum absolute alcohol. After complete dissolution, *p*-toluidine dissolved in alcohol was added to that solution. The reaction mixture was refluxed for 4 h. During the reflux a microcrystalline compound separated, this was isolated by filtration and dried in air.

2.3.3. Preparation of 2-(3-chloro-phenyl)-5-methyl-4-(1-*p*-tolylimino-propyl)-2H-pyrazol-3-ol (L2). It was prepared analogously from 1-[1-(3-chloro-phenyl)-5-hydroxy-3-methyl-1H-pyrazol-4-yl]-propan-1-one and crystals were collected and dried. The synthesis of these compounds can be summarized by reaction scheme 2.

2.4. *Analytical data and DART mass*

Analytical data of ligands are listed in table 1. The structure of the ligands can be confirmed from these data along with spectroscopic and crystallographic evidence discussed in section 3.1. The elemental analysis matches well with the empirical formula

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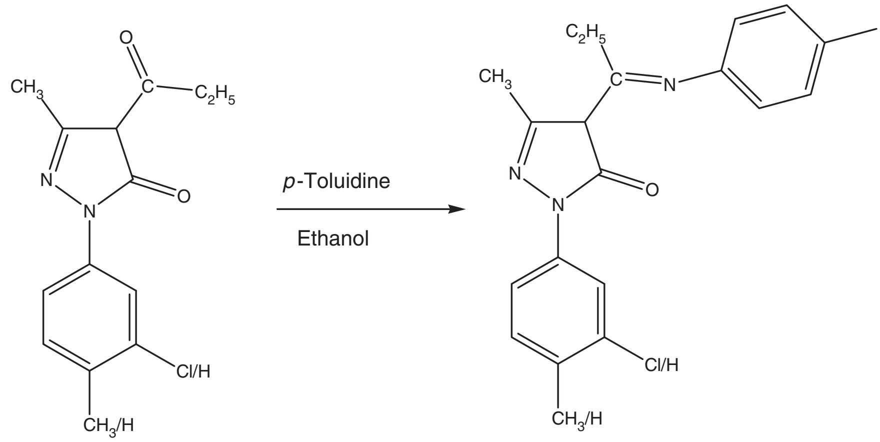

Scheme 2. Preparation of ligands.

Table 1. Analytical data of ligands.

Ligands	Color	Yield (%)	M.p. (°C)	Elemental analyses (%) C	H	N
L1	Yellow	75.18	162	74.13 (75.65)	7.09 (6.94)	12.55 (12.60)
L2	Brown	76.7	135	66.97 (67.88)	5.59 (5.69)	11.82 (11.87)

of the ligands. The ligand (L1) is identified by its DART mass spectrum, which shows molecular ion peak at $m/z = 333.19$ (Supplementary material).

2.5. *X-ray crystallography data collection and processing*

Crystallographic data for L2 are summarized in table 2. The data collection was carried out on a Bruker Mart Apex CCD diffractometer using graphite-monochromated Mo-Kα ($\lambda = 71{,}073$ Å) radiation at 293 K. An absorption correction based on SADABS [18] was also applied. The structure was solved by direct methods (SHELXTL) and refined by least-squares methods with atomic anisotropic thermal parameters for all non-hydrogen atoms [19, 20]. All hydrogens were either generated geometrically or obtained from the Fourier difference map and were assigned fixed isotropic thermal parameters in the structure factor calculations. Conformational and H-bonding analysis was performed using PLATON [21].

2.6. *Preparation of the Ni(II)complexes*

The following general procedure was used in the synthesis of all the metal complexes (scheme 3). Known amount of appropriate ligand was dissolved in alcohol. Equimolar amount of nickel acetate was dissolved in water and then added to the

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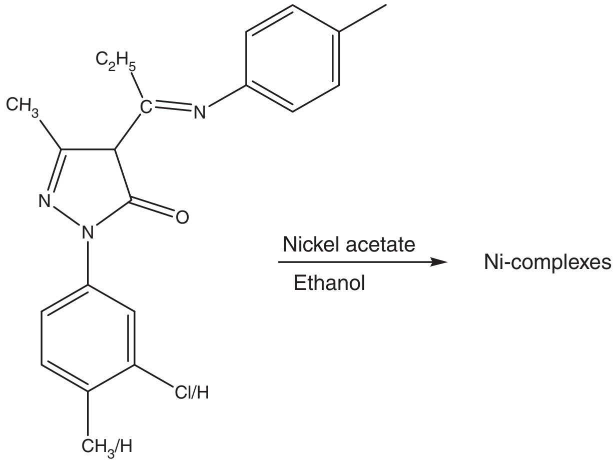
Table 2. Summary of crystallographic data for L2.

Compound	L2
Empirical formula	$C_{20}H_{20}ClN_3O$
Formula weight	353.84
Crystal system	Monoclinic
Space group	$P\ 2_1/n$
Unit cell dimensions (Å, °)	
a	8.1092(11)
b	18.034(2)
c	12.6225(16)
Volume (Å^3), Z	1835.7(4), 4
Calculated density (g cm^{-3})	1.280
Number of reflections	3607
Number of reflections $>2\sigma(I)$	2243
Absorption coefficient, μ (cm^{-1})	0.22
F(000)	744
No. of parameters refined	233
Final *R*	0.0733
wR(F2)	0.1243
Goodness-of-fit	1.081



Scheme 3. Preparation of Ni(II) complexes.

dissolved ligand. After addition, the reaction mixture was refluxed for 4 h. A light green microcrystalline solid separated, this was isolated by filtration, washed with hot water, and dried in air.

2.7. *Analytical data of Ni(II) complexes*

Analytical data of complexes are depicted in table 3. Conductances of complexes in DMF are from 9 to 11 Ω^{-1} cm^2 mol^{-1} indicating nonelectrolytes [22]. The data presented in table 3 along with the characterization establish the geometry of the metal complexes.

The nickel content of each of the complexes was determined gravimetrically and volumetrically. A known amount of sample was placed in silica crucible and

Table 3. Analytical data and physical properties of complexes.

Complexes	Color	Yield (%)	M.p. (°C)	Λ_M (Ω^{-1} cm^2 mol^{-1})	Ni estimation	Elemental analysis (%) C	H	N
$Ni(L1)_2 \cdot 2H_2O$	Light green	32.89	190	9.0	7.16 (7.72)	66.26 (66.41)	6.34 (6.38)	10.83 (11.06)
$Ni(L2)_2 \cdot 2H_2O$	Light green	40.04	145	11.0	6.89 (7.33)	59.94 (60.02)	5.27 (5.28)	10.04 (10.49)

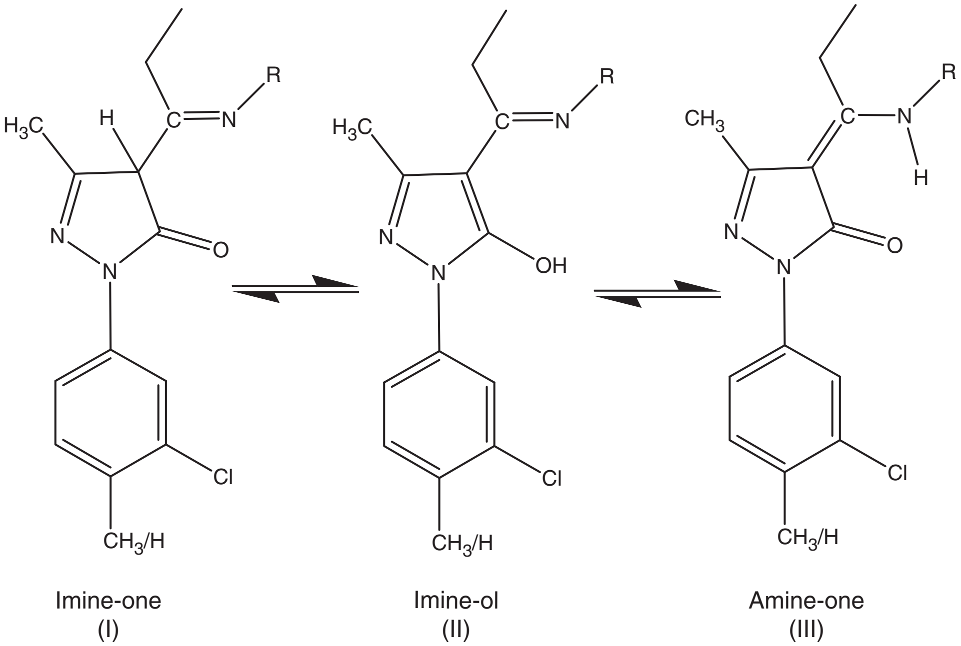


Figure 1. The existence of different tautomeric forms in the ligands.

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decomposed by gentle heating after adding 1–2 mL of concentrated HNO_3. This procedure was repeated 2–3 times giving NiO after decomposition and complete drying.

Known amount of NiO obtained as above was dissolved in a minimum of concentrated HCl, the solution obtained was diluted with distilled water to 100 mL in a volumetric flask and then titrated with standard EDTA solution using murexide indicator.

3. Results and discussion

3.1. *Characterization of ligands*

3.1.1. IR spectra. The ligands can exist in the tautomeric forms shown in figure 1. Important absorption frequencies for the ligands are listed in table 4. The IR spectra

Table 4. Relevant IR frequencies (cm^{-1}) for the ligands.

Ligands	$\nu_{C=N}$ (cyclic)	$\nu_{C=O}$ (pyrazolone ring)	$\nu_{C=N}$ (azomethine)	$\nu_{N–H}$
L1	1585 (s,sh)	1628 (s,sh)	1239 (s,sh)	3242 (m,sh)
L2	1584 (s,sh)	1638 (s,sh)	1240 (s,sh)	3244 (m,sh)

Note: br = broad, s = strong, sh = shoulder, m = medium.

Table 5. ^{1}H NMR spectral data for the aliphatic protons.

Ligands	Me1	Me2	Me3	Me4	$–CH_2–$	–OH/NH
L1	2.35 3H Singlet	2.45 3H Singlet	1.20 3H Triplet $J = 7.56$	2.40 3H Singlet	2.71 2H Quartet $J = 7.56$	12.97 1H Singlet
L2	–	2.44 3H Singlet	1.20 3H Singlet	2.40 3H Singlet	2.70 2H Quartet $J = 7.64$	12.85 1H Singlet

exhibit two characteristic bands at 3220–3245 and 1620–1640 cm^{-1}, which can be assigned to $\nu_{(O–H)}$ and $\nu_{(C=N)}$, respectively, for the imine-ol form (figure 1, II) or $\nu_{(N–H)}$ and $\nu_{(C=O)}$, respectively, for the amine-one form (figure 1, III) [6]. We assigned these to $\nu_{(N–H)}$ and $\nu_{(C=O)}$ for the latter form based on crystallographic studies (discussed later). IR spectra of both the ligands are presented in "Supplementary material" (figure S1).

3.1.2. ^{1}H NMR spectra.

The ^{1}H NMR spectra of all the ligands were recorded in $CDCl_3$. In all the spectra, integrations of each signal were in agreement with the number of different protons present. The NMR spectrum of L1 and L2 are shown as "Supplementary material" (figure S2). The much downfield signal observed at $\sim\delta$ 12.9 ppm is characteristic of intramolecular H-bonded OH/NH [5].

In the aliphatic region, a triplet corresponding to methyl protons of 4-propionyl at δ 1.19–1.25 ppm has coupling constant ~7.5. The methylene protons are a quartet at δ 2.70–2.79 ppm in spectra of the ligands. 5-Methyl of pyrazolone resonates at δ 2.45 ppm. The signal due to *p*-methyl of L1 and L2 appears at δ 2.39 ppm as a singlet (table 5; for atom numbering see figure 2).

In the aromatic region, aryl protons of benzene rings resonate from δ 7.12 to 8.13 ppm. Detailed assignments for each proton along with coupling constants are presented in table 6. From NMR data it is clear that the ligands exist in amine-one form (III) as solids and solutions.

3.1.3. Molecular structure of L2.

The molecular structure of L2 was determined from single crystal X-ray studies. The ORTEP diagram with the atom numbering scheme is shown in figure 3 and selected bond lengths and angles are listed in table 7.

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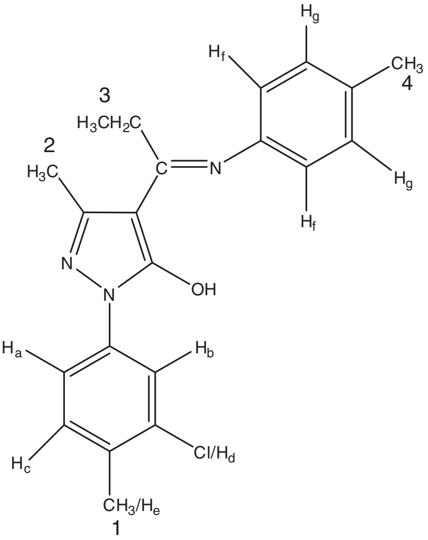

Figure 2. Proton numbering scheme for NMR.

Table 6. ^{1}H NMR spectral data for the aromatic protons.

Ligands	Hb	Ha	Hf	Hc	Hd	Hg	He
L1	7.88 Doublet 2H *J* = 8.36			7.20 Multiplet 4H		7.12 Doublet 2H *J* = 8.12	–
L2	8.13 Singlet 1H		7.12 Doublet 3H *J* = 8.2	7.30 Triplet 1H *J* = 8.16	–	7.25 Doublet 2H *J* = 8.12	7.99 Doublet 1H *J* = 7.4

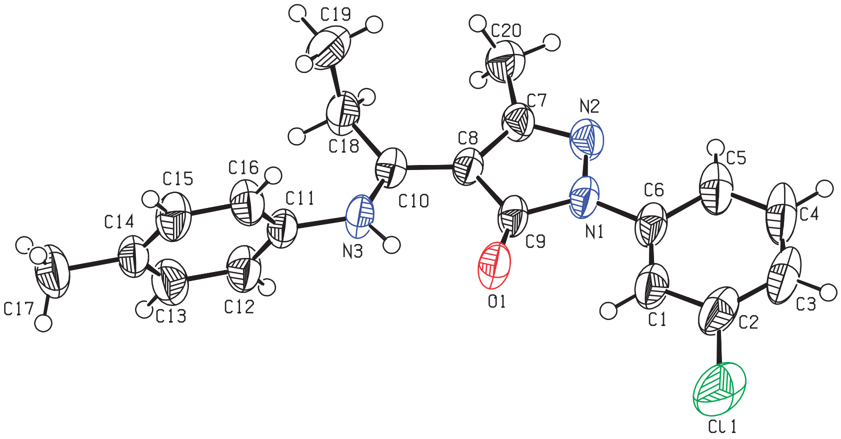

Figure 3. ORTEP diagram of L2.

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Table 7. Selected bond lengths and angles in L2.

Bond distances (Å) with esd in parentheses		Bond angles (°) with esd in parentheses	
N1 C9	1.380(4)	C9 N1 N2	112.4(2)
N1 N2	1.393(3)	C9 N1 C6	129.0(3)
N1 C6	1.417(4)	N2 N1 C6	118.5(2)
N2 C7	1.312(4)	C7 N2 N1	106.1(2)
O1 C9	1.247(3)	C10 N3 C11	127.0(3)
N3 C10	1.321(4)	O1 C9 N1	126.2(3)
N3 C11	1.437(4)	O1 C9 C8	129.7(3)
C10 C8	1.400(4)	N1 C9 C8	104.2(3)
C1 C6	1.375(4)	C16 C11 C12	119.6(3)
C10 C18	1.498(4)	C16 C11 N3	120.5(3)
C18 C19	1.525(5)	C12 C11 N3	119.8(3)
C14 C17	1.513(4)	N3 C10 C8	118.4(3)

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Existence of acyl pyrazolone derivatives in different tautomeric forms (figure 1) as solids is well known and many studies establish the geometry of these derivatives [6, 11, 12, 23]. The Schiff-base derivatives prepared from 4-acetyl-5-methyl-2-phenyl-2,4-dihydro-pyrazol-3-one and alkyl amines remain exclusively in the amine-one tautomeric form in chloroform at room temperature, proved recently using a combination of 1H, 13C, APT, COSY, HMQC, and HMBC NMR spectroscopic methods [23]. The single crystal X-ray diffraction study clearly indicates that the present compound exists in amine-one form without any ambiguity. In fact, the solid-state structure supports strong hydrogen bonding between amine hydrogen and the pyrazolone C3 carbonyl to stabilize the amine-one tautomer.

3.2. *Characterization of complexes*

3.2.1. IR spectra. The IR spectra of the metal complexes (table 8) exhibit no band for $\nu_{(N\text{–}H)}$, indicating removal of N–H in metal complexes. Complexes show absorptions at 2800–2900 cm^{-1} due to $\nu_{C\text{–}H}$ and a band at 3400–3500 cm^{-1} due to coordinated water. The cyclic C=N does not change in complexes, indicating no C=N (cyclic) coordination [9]. The weak band at 400–500 cm^{-1} is the Ni–O stretch [15, 24]. By comparing IR spectra of ligand and complexes the ligand binds to nickel *via* N and O, bidentate. Coordinated water satisfies the coordination number. The IR spectra of both complexes are shown in figure S3.

3.2.2. Thermal analysis. The TG curves were recorded at 10°C min^{-1} from 100°C to 550°C. The TG curves of Ni complexes (Supplementary material) have an initial mass loss at >150°C attributed to loss of water of coordination [25]. For $Ni(L1)_2 \cdot 2H_2O$ mass loss at 161–172°C corresponds to loss of 6.1% (Calcd 6.2%) for two waters (table 9). For $Ni(L2)_2 \cdot 2H_2O$ mass loss at 170–180°C corresponds to loss of 5.82% (Calcd 5.77%) for two waters. Thus, there are two coordinated waters in both complexes.

Table 8. Relevant IR frequencies (cm^{-1}) for the complexes.

Complexes	$\nu_{(O\text{–}H)}$ coordinated H_2O	$\nu_{(C=N)}$ (Cyclic)	$\nu_{(CO)}$
$Ni(L1)_2 \cdot 2H_2O$	3431 (m,br)	1585 (s,sh)	1246 (s,sh)
$Ni(L2)_2 \cdot 2H_2O$	3429 (m,br)	1585 (s,sh)	1245 (s,sh)

Note: br = broad, s = strong, sh = shoulder, m = medium.

Table 9. Thermal analysis data of Ni(II) complexes.

	Temperature (°C)			Mass loss (%)		
Metal chelates	TGA range	DTA	DTG	Found	Calculated	Assignment
$Ni(L1)_2 \cdot 2H_2O$	100–200	160, 349,	161, 256,	6.1	6.2	$2H_2O$
	300–350	371, 537	363, 455	27.74	21.66	$2CH_2$, $6CH_3$
	400–500			20.98	20.62	2CO, 2O
$Ni(L2)_2 \cdot 2H_2O$	100–170	107, 177,	145, 366,	5.82	5.77	$2H_2O$
	270–350	374, 518	393, 443, 558	23.52	27.052	CH_2, $4CH_3$,2Cl
	400–500			19.14	20.53	2CO, 2O

3.2.3. Electronic spectral studies. Diffuse reflectance spectra of the Ni(II) complexes are typical octahedral Ni(II) spectra consisting of bands from 400 to 900 nm assignable to $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ and $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$. The electronic spectra in DMF solutions were also recorded from 300 to 900 nm, with bands at ~440–460 nm assignable to $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ [26].

3.2.4. FAB-MS. The FAB-MS of $[Ni(L1)_2 \cdot 2H_2O]$ was recorded in *m*-nitro benzyl alcohol as a matrix. The parent ion can be observed at $m/z = 723$, formed by the complex when two waters are removed. A peak corresponding to L1 appears at $m/z = 333$. The fragmentation pattern of the nickel complex is presented in scheme 4.

4. Conclusion

IR data suggest O, N coordination of the ligands. The X-ray single crystal analysis of the ligand confirms the molecular structure with amine-one tautomer, which on deprotonation coordinates with Ni(II) as a bidentate ligand. The TGA curves of both complexes show mass loss at >150°C corresponding to loss of two coordinated waters. The analytical data correspond to $[ML_2 \cdot 2H_2O]$ and molar conductances in DMF are consistent with nonelectrolytes. The proposed structure is depicted in figure 4.

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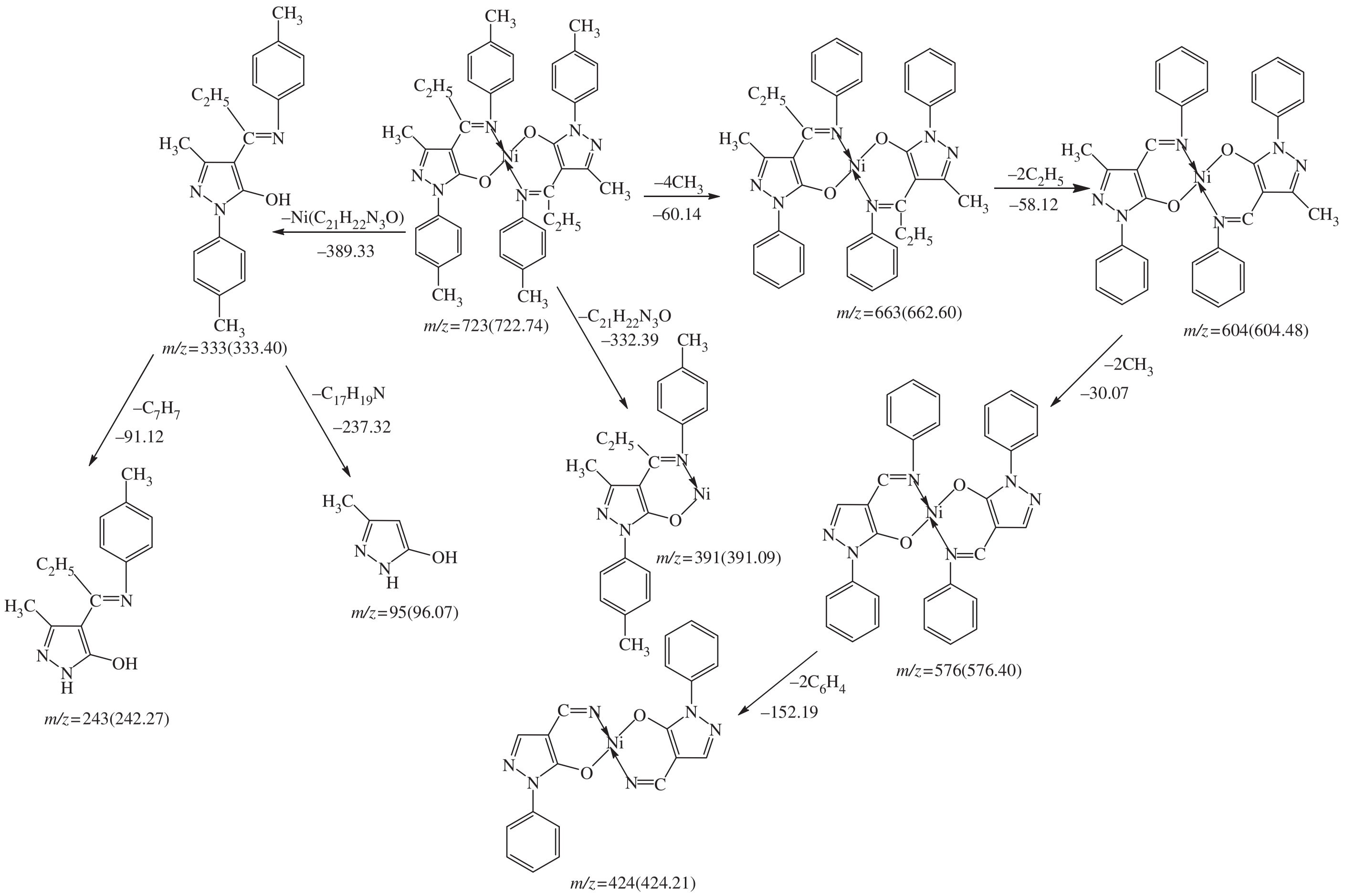

Scheme 4. Fragmentation pattern for L1 and its Ni complex.

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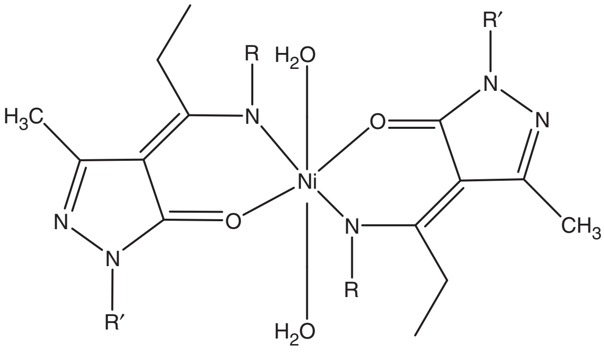

Figure 4. Suggested structure for the nickel(II) complexes.

Supplementary material

CCDC 782562 contains the supplementary crystallographic data for ligand L2. This data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; (Fax +44-1223/336-033; E-mail: deposit@ccdc.ac.uk). IR, NMR, TGA and Mass spectra of ligand/complexes have been submitted as a supplementary figures and are available in online version of the article.

Acknowledgments

The authors are thankful to University Grants Commission, New Delhi for the financial assistance to this work in terms of major research project to RNJ. They are also thankful to the Head, Department of Chemistry for providing necessary facilities required to carry out this work.

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