



Hydrolysis of 4-(4-oxopentan-2-ylideneamino)benzoic acid and in-situ formation of nickel(II), zinc(II) and cadmium(II) complexes of 4-aminobenzoic acid

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ARTICLE INFO

Article history:

Received 30 July 2012

Received in revised form 6 September 2012

Accepted 8 September 2012

Available online 17 September 2012

Keywords:

Imine-hydrolysis

4-Aminobenzoic acid

Coordination effect

Five-coordinate complex

Coordination polymer

ABSTRACT

Metal(II) acetates of nickel, zinc and cadmium facilitate hydrolysis of 4-(4-oxopentan-2-ylideneamino)benzoic acid (*iminocarb*). In the case of nickel a dinuclear *iminocarb* complex is proposed as intermediate species to form nickel complex of 4-aminobenzoic acid (*Haben*). The nickel complex formed from hydrolysis has a composition $[\text{Ni}(\text{O}_2\text{CCH}_3)_2(\text{Haben})_2(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$. In this complex, the nitrogen atoms of the 4-aminobenzoic acid ligands coordinate to nickel ion and the benzoic acid end of the ligands remain free. A five coordinate $[\text{Zn}(\text{aben})_2(\text{py})(\text{H}_2\text{O})]$ complex is obtained from a similar reaction of *iminocarb* with zinc(II) acetate followed by reaction with pyridine (py). On the other hand the reaction of *iminocarb* with cadmium(II) acetate forms a coordination polymer $[\{\text{Cd}(\text{aben})_2(\text{H}_2\text{O})\} \cdot \text{H}_2\text{O}]_n$.

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1. Introduction

Imines are extensively used as ligands in coordination chemistry [1–3]. Many organic transformations of imines are catalysed by various metal complexes [2,4–11]. The conversion of an imine to a carbonyl compound is relatively easy and this transformation is used in protecting carbonyl groups [12–14]. Several pathways are possible for formation of a carbonyl compound from an imine. The hydrolysis [15–24], oxidative cleavage of C=N bond [25–28] and disproportionation reactions [29–31] are common paths for such transformations. Taking β -ketoimine as an example these paths are shown in Scheme 1. Ketoimines are well known for their ability to form interesting metal complexes [2,23]. We are interested to study the role of metal ions in hydrolytic cleavage of C=N bond of imines of 4-aminobenzoic acid, as they are formed from hydrolytic cleavage of some medicines that are used to evaluate pancreatic function and measured in urine [32]. We selected a model compound 4-(4-oxopentan-2-ylideneamino)benzoic acid (abbreviated as *iminocarb*) for such a study, as this ligand has a carboxylic acid group at one end and a β -ketoimine group at other end (Scheme 2). Both these groups efficiently bind to metal ions and are capable of forming chelates. We also felt that the presence of a binding site at a remote place from a hydrolysis site, would not only bind a metal ion but also assist in hydrolysis. In this article we report the hydrolysis of *iminocarb* leading to different metal complexes with 4-aminobenzoic acid (*Haben*). We discuss such

reactions to establish the coordination effect during *iminocarb* hydrolysis.

2. Experimental

2.1. Preparation of 4-(4-oxopentan-2-ylideneamino)benzoic acid (*iminocarb*)

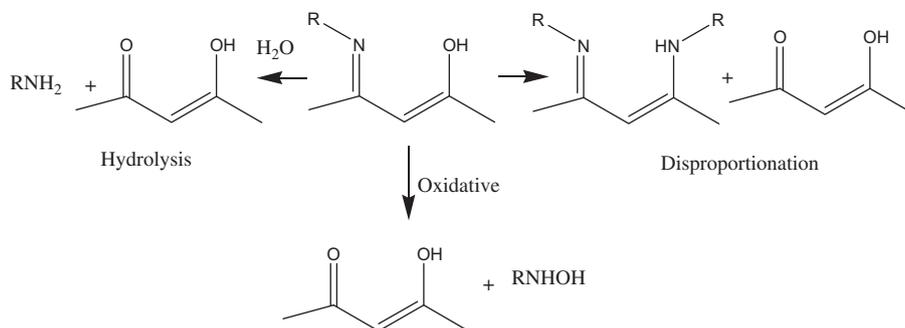
The ligand *iminocarb* was prepared by modifying reported procedure [33]. To a well stirred solution of 4-aminobenzoic acid (0.27 g, 2 mmol) in methanol (10 ml), acetylacetone (0.26 ml, 2 mmol) was added. The reaction mixture was refluxed for 6 h at 60 °C. A yellow precipitate was obtained upon cooling. The reaction mixture was filtered and the precipitate was collected. The product *iminocarb* was recrystallized from methanol. Isolated yield: 86%. IR (KBr, cm^{-1}): 3459 (b), 1697 (s), 1599 (s), 1557 (s), 1289 (s), 1253 (s), 1218 (s), 1179 (s). ^1H NMR (DMSO- d_6): 12.61 (s, 1H), 7.91 (d, 8.4 Hz, 2H), 7.28 (d, 8.4 Hz, 2H), 5.33 (s, 1H), 2.10 (s, 3H), 1.98 (s, 3H).

2.2. Complex $[\text{Ni}(\text{O}_2\text{CCH}_3)_2(\text{Haben})_2(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$ (1)

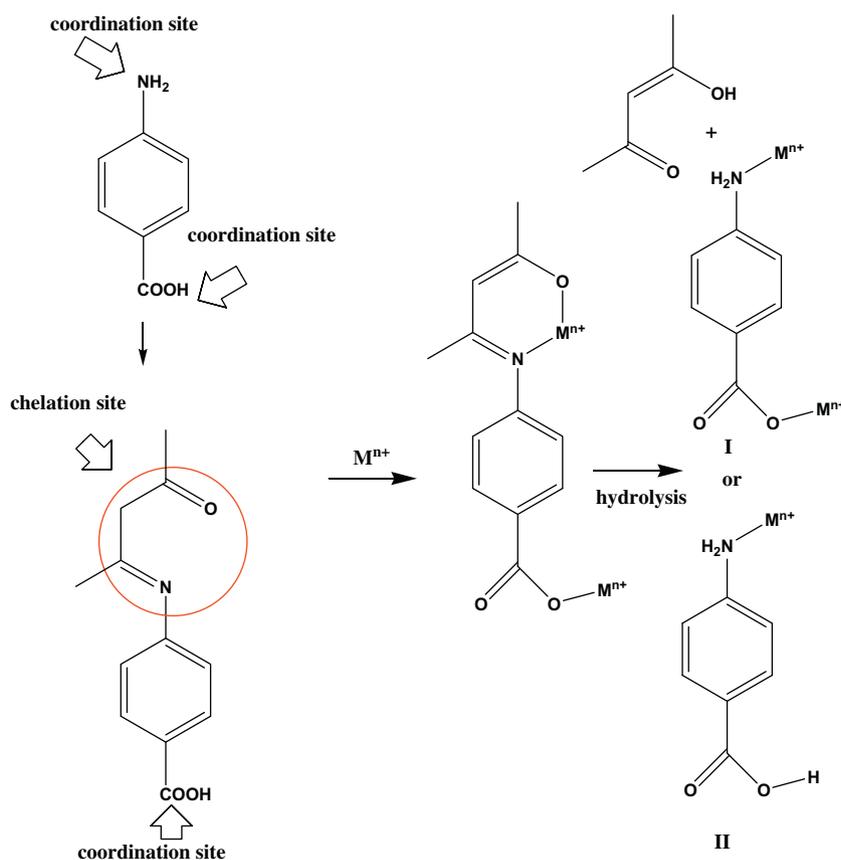
To a methanol solution of *iminocarb* (0.04 g, 0.2 mmol), nickel(II) acetate tetrahydrate (0.05 g, 0.2 mmol) was added, a dark green precipitate was obtained. The precipitate was dissolved in a minimum amount of dimethylformamide and filtered. The filtrate was kept for crystallization for a week; dark green crystals were obtained. Isolated yield: 62%, IR (KBr, cm^{-1}): 3407 (b), 1600 (s), 1394 (s), 1310 (s), 1281 (s), 1188 (s), 1113 (s), 1024 (m), 924 (s).

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Scheme 1. Some possible paths of carbonyl group formation from an imine.



4-(4oxopentan-2-ylideneamino)benzoic acid

Scheme 2. One possible intermediate metal complex during hydrolysis of *iminocarb* and its products.

Molar conductance (methanol, RT) $27.2 \text{ S cm}^2 \text{ mol}^{-1}$. Visible (λ_{max} , methanol) 673 nm ($\epsilon = 99 \text{ L mol}^{-1} \text{ cm}^{-1}$); 725 ($\epsilon = 86 \text{ L mol}^{-1} \text{ cm}^{-1}$).

2.3. Complex $[\text{Zn}(\text{aben})_2(\text{py})(\text{H}_2\text{O})]$ (2**)**

To a well stirred solution of *iminocarb* (0.04 g, 0.2 mmol) in methanol (10 ml), zinc(II) acetate dihydrate (0.04 g, 0.2 mmol) was added, after 1 hr the resulting reaction mixture was filtered and to the filtrate pyridine (1 ml) was added. The transparent liquid obtained was kept for crystallization. After 10 days colorless crystals were obtained. Isolated yield: 60%. IR (KBr, cm^{-1}): 3375 (w), 3304 (w), 2926 (w), 1605 (w), 1553 (m), 1511 (w), 1434 (w), 1398 (s), 1256 (w), 1182 (w), 1020 (w), 787 (m), 699 (m). ^1H NMR (DMSO- d_6): 7.86 (t, 7.6 Hz, 3H), 7.62 (d, 8.4 Hz, 4H), 7.46 (t,

7.2 Hz, 2H), 6.51 (d, 8.8 Hz, 4H), 5.55 (s, 4H). Molar conductance (methanol, RT) $5.9 \text{ S cm}^2 \text{ mol}^{-1}$.

2.4. Complex $[\{\text{Cd}(\text{aben})_2(\text{H}_2\text{O})\}_n \cdot \text{H}_2\text{O}]_n$ (3**)**

To a well stirred solution of *iminocarb* (0.04 g, 0.2 mmol) in methanol (10 ml), cadmium(II) acetate dihydrate (0.05 g, 0.2 mmol) was added, a colorless precipitate was obtained. The precipitate was dissolved in minimum amount of methanol. The reaction mixture was filtered and the transparent liquid was kept for crystallization. After 1 week colorless crystals were obtained. Isolated yield: 67%. IR (KBr, cm^{-1}): 3365 (s), 3291 (w), 3159 (w), 1607 (s), 1519 (s), 1409 (s), 1256 (w), 1176 (w), 1093 (w), 1012 (w), 943 (m). $^1\text{HNMR}$ (DMSO- d_6): 8.62 (bs, 2H), 7.86 (t, 7.6 Hz,

Table 1
Crystallographic parameters of the complexes **1** and **2**.

Compound No.	1	2
Formulae	C ₁₈ H ₃₀ N ₂ NiO ₁₄	C ₁₉ H ₁₉ N ₃ O ₅ Zn
Mol. wt.	557.15	434.74
Crystal system	monoclinic	monoclinic
Space group	P2 ₁ /n	P2 ₁ /n
T (K)	296(2)	296(2)
λ (Å)	0.71073	0.71073
a (Å)	6.7822(3)	6.8027(4)
b (Å)	18.1737(9)	27.7694(17)
c (Å)	10.6188(5)	10.3788(6)
α (°)	90.00	90.00
β (°)	97.332(2)	96.079(3)
γ (°)	90.00	90.00
V (Å ³)	1298.15(11)	1949.6(2)
Z	2	4
D (Mg m ⁻³)	1.425	1.481
Absorption	0.815	1.295
Coefficient (mm ⁻¹)		
Absorption correction	none	none
F(000)	584	896
Total reflections	3221	3378
Reflections, I > 2σ(I)	2953	2728
Maximum θ (°)	28.50	25.25
Ranges (h, k, l)	-9 ≤ h ≤ 9, -24 ≤ k ≤ 24, -14 ≤ l ≤ 14	-8 ≤ h ≤ 8, -30 ≤ k ≤ 30, -12 ≤ l ≤ 9
Complete to 2θ (%)	98.0	95.7
Data/restrain/parameter	3221/7/183	3378/3/274
Goodness-of-fit (F ²)	1.053	1.042
R ₁ [I > 2σ(I)]	0.0351	0.0372
wR ₂ [I ≥ 2σ(I)]	0.1008	0.0947
R ₁ (all data)	0.0378	0.0468
wR ₂ (all data)	0.1031	0.0988

1H), 7.62 (d, 8.0 Hz, 4H), 7.48 (t, 7.2 Hz, 2H), 6.48 (d, 8.0 Hz, 4H), 5.53 (s, 4H). Molar conductance (DMF, RT) 2.4 S cm² mol⁻¹.

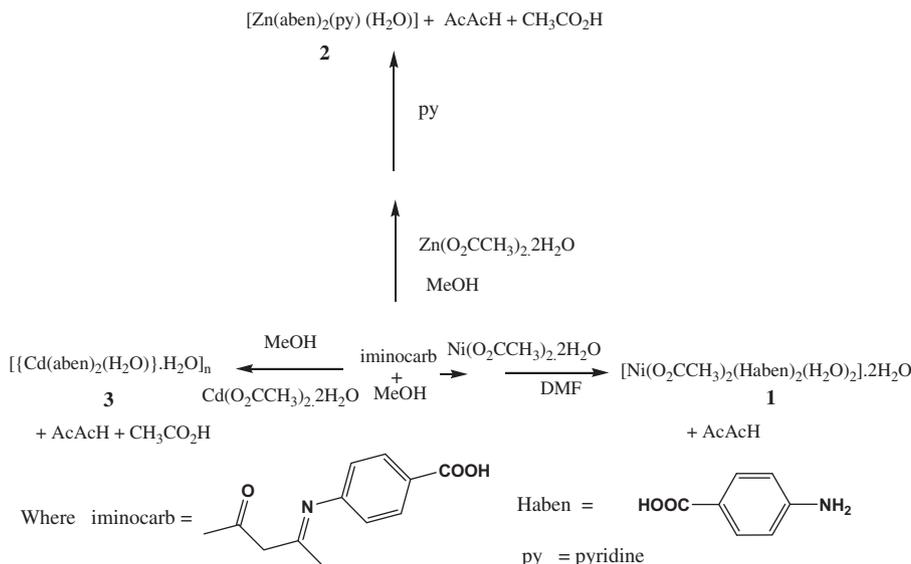
The crystallographic parameters of the complexes **1** and **2** are listed in Table 1.

3. Results and discussion

The reaction of *iminocarb* with metal(II) acetates of nickel, zinc and cadmium led to formation of 4-aminobenzoic acid com-

plexes of these metal ions (Scheme 3). These complexes are formed through hydrolysis of the *iminocarb*. Since all the complexes obtained are from hydrolysis of *iminocarb*, we can alternatively say that the hydrolysis of the *iminocarb* is facilitated by these metal(II) acetates. Due to the strong binding ability of β-ketoimine, the *iminocarb* is expected to form metal complexes with these metal ions; we could not isolate any such stable complex. However, solution study has led us to propose intermediacy of such complex. At this juncture it is worth mentioning that these metal acetates independently react with *Haben* to form insoluble metal complexes. Similar situations were reported during the preparation of metal-aromatic carboxylate complexes [34–36] and in such cases ancillary ligands were used to overcome the insolubility problem. The *iminocarb* forms a stable complex with tin(IV) [33]. The tin(IV) complex has relatively more covalent Sn–O and Sn–N bonds which resists hydrolysis of the complex.

The nickel(II) acetate tetrahydrate hydrolyses *iminocarb* leading to the formation of di-(κ-N-4-aminobenzoic acid)di-aqua nickel(II) acetate dihydrate (**1**). It is an acetate complex (Fig 1a) having two 4-aminobenzoic acid ligands. The non-ionic nature of the compound is reflected by its molar conductance (27.2 S cm² mol⁻¹). The complex has an octahedral geometry with two aqua-ligands, two amino groups from the *Haben* and two acetate ligands. The complex **1** has a highly symmetric structure. The structure has a mirror plane of symmetry which is also apparent from its space group (P2₁/n). The structure of the compound is shown in Fig. 1a and selected bond parameters are shown in the caption of Fig. 1a. The complex possesses intra-molecular hydrogen bond between a carbonyl oxygen of acetate with one coordinated aqua ligand through O5–H···O4 interaction (d_{D···A}, 2.64 Å, ∠D–H···A 143.1°). The lattice water molecules form hydrogen bonds with acid group of the *Haben* through O7–H···O2 and O7–H···O2 interactions (d_{D···A}, 2.81 Å and d_{O7···O1}, 3.00 Å; ∠O7–H···O2, 178.0°; and ∠O7–H···O1, 160.5°). The water molecules in the crystal lattice are also linked to carboxylates of acetate groups through O6–H···O4 interactions (d_{O6···O4}, 2.78 Å ∠O7–H···O2 154.3°). The N1–H···O1 interactions (d_{N1···O1}, 2.30 Å ∠N1–H···O1 158.0°), as well as C2–H···O4 (d_{C2···O4}, 3.30 Å ∠C2–H···O4 135.7°) are pertinent hydrogen bonds in the crystal lattice (Fig. 1b). Each molecule of the complex in the lattice can be visualized to be surrounded by six other molecules as shown in Fig. 1c. Thus a central molecule is held to six-other through supramolecular interactions.



Scheme 3. Formation of metal complexes of *Haben* from hydrolysis of *iminocarb*.

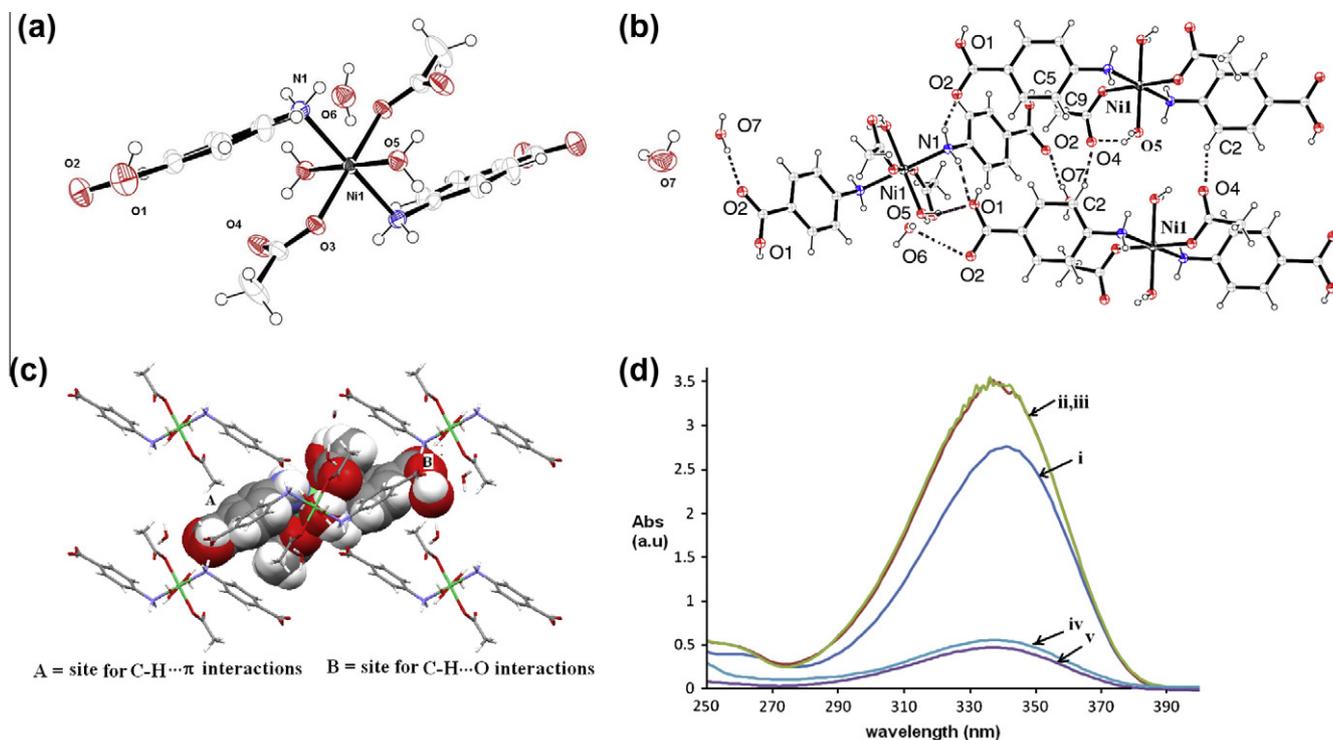


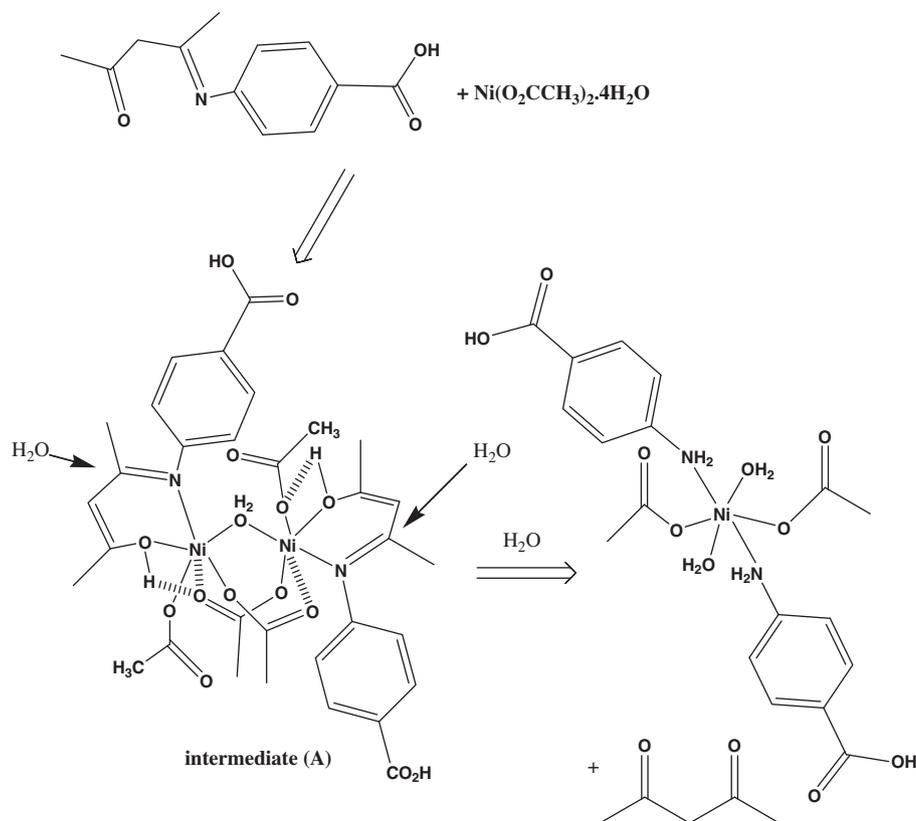
Fig. 1. (a) Structure of $[\text{Ni}(\text{O}_2\text{CCH}_3)_2(\text{Haben})_2(\text{H}_2\text{O})_2]\cdot 2\text{H}_2\text{O}$ (**1**) (ORTEP drawn with 50% thermal ellipsoids, water of crystallisations are omitted) Selected bond-distances and bond-angles are Ni1–O5, 2.0670(12) Å; Ni1–O3, 2.0583(11) Å; Ni1–N1, 2.1593(14) Å; $\angle\text{N1–Ni1–O5}$, 90.87(6) $^\circ$; $\angle\text{N1–Ni1–O3}$, 87.75(5) $^\circ$; $\angle\text{O3–Ni1–O5}$ 85.52(5) $^\circ$. (b) Weak interactions in the crystal lattice of **1** (30% thermal ellipsoids). (c) Packing diagram showing a molecule of **1** (CPK) surrounded by six other molecules. (d) UV–Vis spectra of (i) the *iminocarb* in methanol (10^{-4} M, 2 ml); (ii) immediately after addition of nickel(II) acetate tetrahydrate (10^{-4} M) to (i). Same solution of *iminocarb* with nickel(II) acetate after (iii) 1 day, (iv) 3 days and (v) 4 days.

The IR spectra of the *Haben* has sharp absorptions at 3461 ($\nu_{\text{N–H}}$) cm^{-1} , 3364 ($\nu_{\text{O–H}}$) cm^{-1} , 1666 ($\nu_{\text{C=O}}$) cm^{-1} , 1624 ($\nu_{\text{N–Hscissoring}}$) cm^{-1} and 1601 ($\nu_{\text{C=C}}$) cm^{-1} , whereas the IR of the nickel complex **1** has broad absorption at 3407 cm^{-1} . The sharp absorption at 3407 cm^{-1} is attributed to N–H and O–H stretching frequencies of the coordinated amino group and free carboxylic acid. The IR absorptions observed at 1697 ($\nu_{\text{C=O}}$) cm^{-1} and 1599 ($\nu_{\text{C=N}}$) cm^{-1} for *iminocarb* are not present in **1** but it has a strong absorption at 1600 ($\nu_{\text{C=O}}$) cm^{-1} . This shows that carboxylate group has highly delocalized electrons. In fact the complex has free carboxylic acid with equal C7–O1 and C7–O2 distances (1.259 Å each). This is due to formation of hydrogen bond by the carboxylic acid with lattice water molecules. The interesting feature of complex **1** is the monodentate coordination mode of acetate group. Generally, nickel(II) acetate undergoes ligand replacement reactions with benzoates [37]. The nickel-*aben* complex having dioxime ligands are reported earlier [38]. N-functionalized nickel-*aben* complex shows carboxylate binding [39]. There is an example of an ionic complex having N-coordinated *Haben* with nickel(II) chloride [40]. The complex **1** on the other hand is a neutral complex, having carboxylates within the coordination sphere. The IR spectra of the insoluble product from reaction of nickel(II) acetate with *Haben* has similarity to the IR spectra of cadmium complex **3** and it suggests that they have similar structure. Thus, direct mixing of nickel(II) acetate with *Haben* leads to a coordination polymer with carboxylate and amino groups as bridges.

The complex **1** absorbs at 673 and 725 nm due to *d–d* transitions (assigned wide extinction coefficient). Formation of the complex (**1**) from *iminocarb* was monitored by UV–Vis spectroscopy. The *iminocarb* has absorption maximum at 342 nm. The absorption at 342 nm of a solution of *iminocarb* increases on addition of nickel(II) acetate solution (Fig. 1d). The increase is accompanied by a shift in the absorbance to 339 nm. It is reported that an alcoholic

solution of sodium salt of acetylacetonate shows $\pi\text{--}\pi^*$ transition at 288 nm; whereas the nickel(II) acetylacetonate has similar absorption peak at 294 nm [41]. Based on the analogy of the structural backbone of acetylacetonate and *iminocarb*, the absorbances observed at 342 and 339 nm are attributed to the $\pi\text{--}\pi^*$ transition of *iminocarb* in respective cases. The change in absorbance is attributed to the formation of an intermediate complex **A** as depicted in Scheme 4. From Fig. 1d it is clear that as time progresses peak intensity of 339 nm reaches a maximum and then decreases. The isolated complex **1** does not show absorption at this wavelength. On the basis of these observations, we propose a mechanism for hydrolysis of *iminocarb* as illustrated in Scheme 4. We propose a binuclear aqua-bridged nickel(II) complex **A** as an intermediate species for the hydrolysis. The intermediate has a highly symmetric structure which can lead to the final product **1**. It is an established fact that the nickel(II) acetate forms aqua-bridged binuclear complexes with nitrogen donor ancillary ligands [37]. Such complexes have structural similarity to biologically important metal hydrolases [42,43]. Thus, in another sense we can suggest this mechanism to be a bio-mimicking process [44]. Our hydrolytic reaction is sensitive to pH and works at near neutral conditions (pH of about 7.5–8.5). The reaction is also anion specific. The use of halide or nitrate salt for similar reactions did not give satisfactory result. Further, addition of sodium acetate in excess from an external source also did not facilitate the hydrolysis of *iminocarb*. Thus, it is presumed that the acetate ions remain bound to the metal during the course of the reaction as shown in the reaction mechanism. The hydrolysis of *iminocarb* by nickel(II) acetate was also monitored by GC–mass. The reaction takes place under catalytic condition (10 mol% of nickel(II) acetate) at room temperature in methanol.

The reaction of zinc(II) acetate with *iminocarb* followed by treatment with pyridine (py) in methanol led to the formation of



Scheme 4. A plausible mechanism of nickel(II) mediated hydrolysis reaction of iminocarb.

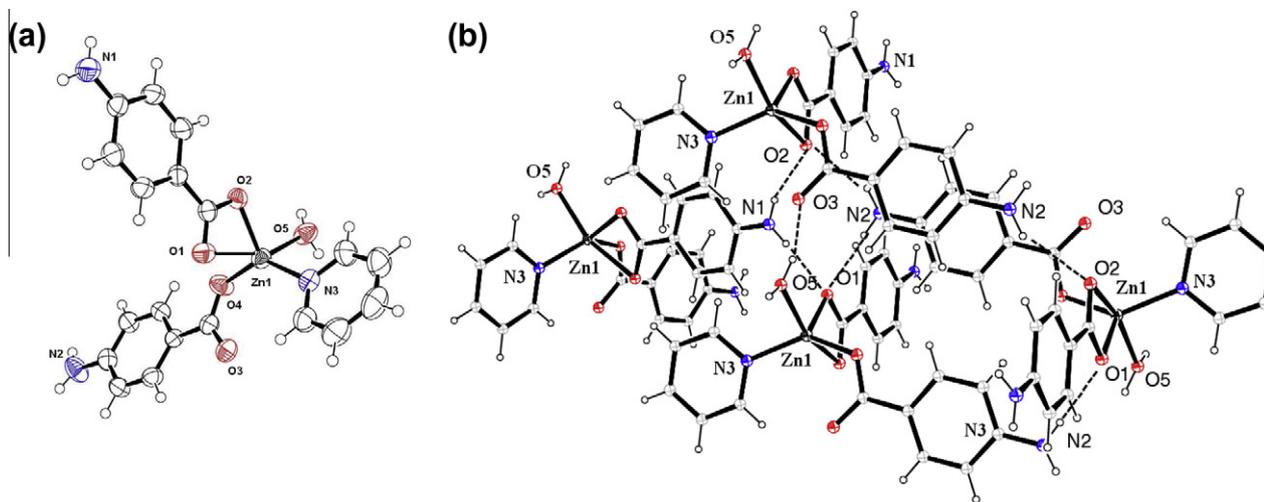


Fig. 2. (a) The structure of $[Zn(aben)_2(py)(H_2O)]$ (**2**) (drawn with 50% thermal ellipsoids). Selected bond distances and bond angles are Zn1–O1, 2.012(16) Å; Zn1–O2, 2.412(17) Å; Zn1–O4, 1.911(17) Å; Zn1–O5, 2.056(18) Å; Zn1–N3, 2.021(2) Å; $\angle O1-Zn1-O2$, 58.41(6)°; $\angle O5-Zn1-O2$, 154.47(7)°; $\angle O1-Zn1-N3$, 109.04(7)°; $\angle O4-Zn1-N3$, 135.59(8)°. (b) The weak interactions among different molecules of **2** to form self-assembly.

$[Zn(aben)_2(py)(H_2O)]$ (**2**). The complex has very interesting structural features (Fig. 2a). It has five-coordinate geometry around zinc ion, which is relatively less common in zinc chemistry. It has an intermolecular hydrogen bonded structure. The coordinated water molecule and amino groups of the *aben* form very interesting cyclic hydrogen bond $R_4^4(8)$ motifs. The motifs are comprise of N1–H···O2–H–N2–H···O1···H–N1 interactions ($d_{N1···O2}$, 2.95 Å; $d_{N2···O1}$, 3.05 Å; $d_{N1···O1}$, 2.93 Å and $d_{N2···O2}$, 3.06 Å; $\angle N1-H···O2$, 163.0°; $\angle N2-H···O1$, 176.8° $\angle N1-H···O1$, 156.9°; $\angle N2-$

H···O2, 148.9°). These cyclic motifs arise from interactions of four molecules of the complex **2**. The cyclic structure can be compared to a rectangle having two oxygen atoms of carboxylate placed at two corners and two nitrogen atoms of two amino groups at other two corners. Further to this, there is O5–H···O3 interactions among aqua ligand and monodentate carboxylate of a neighboring molecule ($d_{D···A}$, 2.72 Å, $\angle D-H···A$ 157.9°). The important weak interactions responsible for making self-assemble structure are shown in Fig. 2b. The complex **2** is presumably formed during

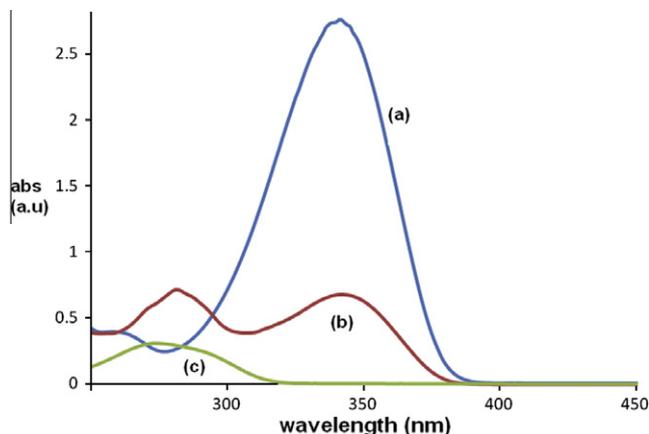


Fig. 3. UV-Vis spectra of a solution of (a) *iminocarb* in methanol (10^{-4} M, 2 ml); (b) just after addition of cadmium(II) acetate (10^{-4} M); (c) coordination polymer **3** in methanol (10^{-4} M).

crystallization. The pyridine possibly replaces the coordinated nitrogen of *aben* during crystallization. In fact the IR spectrum of the product before dissolving it in pyridine has similarities to the IR spectra of the product **3** (whose structure is polymeric). Unfortunately, we are unable to obtain crystalline product in this case without pyridine. The zinc(II) acetate quenches the absorption of a methanol solution of *iminocarb*. During the course of measurement there is oscillation in absorbance with time. The trend in increase or decrease is not consistent, it may be due to the equilibration between C=N and C=O bond formation along with the crystallization of the product. Thus, a complete conclusion is not reached. A polymeric *aben*-zinc complex is reported in literature [45]. The *aben* act as bridge zinc(II) complexes [46,47]. These examples suggest that the binding of zinc is very sensitive to environment. The complex **2** is non-ionic as it shows negligible molar conductance.

The reaction of cadmium(II) acetate with *iminocarb* in methanol gave the 4-aminobenzoate complex $\{[Cd(aben)_2(H_2O)] \cdot H_2O\}_n$ (**3**). The compound **3** has a similar structure to the already reported one [48]. The cadmium complex **3** is also non-ionic with very low molar conductance. The hydrolysis of *iminocarb* by cadmium is apparent from its UV-Vis spectra. It is observed that immediately after addition of cadmium(II) acetate to *iminocarb* forms **3** as shown in Fig. 3. The absorption of *iminocarb* at 342 nm gets drastically quenched on addition of cadmium acetate and new absorption peak at 282 nm appears. This new peak matches with the peak of the coordination polymer **3**. The complete conversion of *iminocarb* to **3** in solution does not take place even after four days. Thus, it is suggested that there is an equilibrium between C=N cleavage and formation of C=O bond. The precipitation during the equilibration process is the driving force for the formation of **3**. The bridging binding mode of the **3** suggests that it is formed through a cadmium-*iminocarb* intermediate complex. Such intermediate is possible as the size of cadmium is relatively bigger and can expand its coordination number [34].

In conclusion, the coordination to metal namely Ni, Zn and Cd ions facilitate the hydrolysis of *iminocarb*. The coordination modes of ligands in the isolated metal complexes provide evidences towards coordination effect. A dinuclear aqua-bridged model having resemblance to biological system is proposed to explain the nickel mediated hydrolysis. The structure of cadmium-*aben* complex shows the formation of bridged structure involving *iminocarb* as intermediate species. Isolation of a five-coordinate zinc-*aben* complex is due to coordination effect during hydrolysis of *iminocarb*.

Acknowledgements

One of the author (N.P.) thanks Council of Scientific and Industrial Research (New Delhi) for a Junior Research Fellowship.

Appendix A. Supplementary material

CCDC 868737 and 868738 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.ica.2012.09.005>.

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