

Synthesis of complexes of Pb(II), Cd(II), Zn(II), Ni(II), La(III) and Cu(II) with a Schiff base macrocyclic ligand containing pyridine

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1,5-bis(2-formylphenyl)pentane (synthesised from 1,5 dibromopentane and salicylaldehyde). reacts with 2,6-diaminopyridine to give a new macrocyclic ligand that gives Cu(II), Ni(II), Pb(II), Zn(II), Cd(II) and La(III) complexes by reaction with Cu(NO₃)₂·3H₂O, Ni(NO₃)₂·6H₂O, Pb(NO₃)₂, Zn(NO₃)₂·6H₂O, Cd(NO₃)₂·4H₂O and La(NO₃)₃·6H₂O respectively. The ligand and its metal complexes have been characterised by elemental analysis, IR, ¹H and ¹³C NMR, UV-Vis spectra, magnetic susceptibility, conductivity measurements and mass spectra. All complexes are diamagnetic and the Cu(II) complex is binuclear.

Keywords: macrocyclic Schiff base complexes, 1,5-bis(2-formylphenyl)pentane, 2,6-diaminopyridine

Schiff base metal complexes have widely been studied because of their industrial, antifungal, and biological applications.^{1,2} Schiff base macrocycles have been of great importance in macrocyclic chemistry.³ The chemistry of macrocyclic complexes has received much attention in recent years on account of its applications in bioinorganic chemistry,^{4,5} in fundamental and applied sciences⁴⁻⁶ and its general importance in coordination chemistry.^{6,7} In the present work, we have synthesised a dialdehyde, 1,5-bis(2-formylphenyl)pentane, derived from 1,5-dibromopentane with salicylaldehyde and K₂CO₃, then a macrocyclic Schiff base (**L**) by reaction of 2,6-diaminopyridine and 1,5-bis(2-formylphenyl)pentane. Cu(II), Ni(II), Pb(II), Zn(II), Cd(II) and La(III) complexes of **L** have been synthesised by reaction of **L** and Cu(NO₃)₂·3H₂O, Ni(NO₃)₂·6H₂O, Pb(NO₃)₂, Zn(NO₃)₂·6H₂O, Cd(NO₃)₂·4H₂O and La(NO₃)₃·6H₂O respectively. Spectral and magnetic properties of the new compounds are reported in detail.

Experimental

Methods

1,5-bis(2-formylphenyl)pentane was prepared from 1,5-dibromopentane, salicylaldehyde and K₂CO₃ by a literature method.⁸⁻¹⁰ All other chemicals and solvents were of analytical grade and used as received. Elemental analysis was carried out on a LECO CHNS model 932 elemental analyser. ¹H NMR and ¹³C NMR spectra were recorded using a Bruker Avance DPX-400 NMR spectrometer. IR spectra were recorded on a Midac 1700 FTIR spectrometer as KBr discs in the wave number range 4000–400 cm⁻¹. Electronic spectral studies were conducted on a Shimadzu model 160 UV/visible spectrophotometer in the wavelength 200–800 nm. Molar conductivity was measured with a WTW LF model 330 conductivity meter, using a solution of the complex 10⁻³ M in DMSO. Electro spray ionisation mass spectrometric analysis (ESI-MS) was obtained on an Agilent 1100 MSD Spectrometer. Magnetic susceptibilities were determined on a Sherwood Scientific magnetic susceptibility balance (Model MK1) at room temperature (20 °C) using Hg[Co(SCN)₄] as calibrant; diamagnetic corrections were calculated from Pascal's constants.¹¹

Synthesis of 1,5-bis(2-formylphenyl)pentane

To a stirred solution of salicylaldehyde (24.4 g, 0.2 mol) and K₂CO₃ (13.8 g, 0.1 mol) in DMF (80 mL) was added dropwise 1,5-dibromopentane (23.0 g, 0.1 mol) in DMF (20 mL). The reaction was continued for 4 h at 150–155 °C and 4 h at room temperature (Fig. 1). After the addition was completed, distilled water (200 mL) was added and the mixture was put in a refrigerator; 1 h later the precipitate was filtered and washed with water (500 mL), dried in air and recrystallised from EtOH. Yield: 24.3 g (78%), m.p. 66–67 °C, Colour: white. Anal. Calcd for C₁₉H₂₀O₄: C, 73.1, H, 6.5. Found: C, 73.3, H, 6.6%. ¹³C NMR (DMSO-d₆, δ ppm): CH₂CH₂CH₂: 22.57, CH₂CH₂O: 28.66, CH₂CH₂O: 68.67, HC=O: 189.55, Aromatic: 113.98, 120.98, 124.71,

128.03, 136.86, 161.53. ¹H NMR (DMSO-d₆, δ ppm): 1.66 (q, 2H, J = 8.4 Hz, CH₂CH₂CH₂), 1.87 (p, 4H, J = 7.6 Hz, CH₂CH₂O), 4.16 (t, 4H, J = 6.4 Hz, CH₂CH₂O), 7.03–7.70 (m, 8H, ArH), 10.39 (s, 2H, HC=N). Selected IR data (KBr, ν cm⁻¹): 3074, 3039 ν(Ar-CH), 2947, 2873 ν(Alf.-CH), 1682 ν(C=O), 1485, 1458 ν(Ar-C=C), 1284, 1242 ν(Ar-O), 1188, 1052 ν(R-O), 752 ν(substituted benzene). Mass spectra: 312 [M]⁺

Synthesis of macrocyclic Schiff Base L

The macrocyclic ligand (**L**) was prepared by the dropwise addition of a solution of 2,6-diaminopyridine (0.22 g, 2 mmol) in methanol (40 mL) to a stirred solution of 1,5-bis(2-formylphenyl)pentane (0.63 g, 2 mmol) in methanol (60 mL). After the addition was complete, stirring was continued for 2 h. The yellow-coloured precipitate was filtered, washed with methanol and dried (Fig. 2). Yield: 0.31 g (38.5%). Anal. Calcd for C₂₄H₂₃N₃O₂·H₂O: C, 74.8, H, 6.0, N, 10.9. Found: C, 75.0, H, 6.1, N, 10.8%. ¹³C NMR (DMSO-d₆, δ ppm): CH₂CH₂CH₂: 22.59, CH₂CH₂O: 28.66, CH₂CH₂O: 68.65, HC=N: 189.60, Aromatic: 112.68, 113.95, 120.99, 124.68, 128.06, 136.90, 155.60, 159.71, 161.53. ¹H NMR (DMSO-d₆, δ ppm): 1.66 (2H, CH₂CH₂CH₂), 1.86 (4H, CH₂CH₂O), 4.16 (4H, CH₂CH₂O), 7.05–7.68 (m, 11H, ArH), 10.40 (s, 2H, HC=N). Selected IR data (KBr, ν cm⁻¹): 3383 (H₂O), 3066, 3039 ν(Ar-CH), 2935, 2866 ν(Alf.-CH), 1683 ν(C=N), 1597 (C=N(pyridine)), 1489, 1454 ν(Ar-C=C), 1284, 1242 ν(Ar-O), 1160, 1053 ν(R-O), 752 ν(substituted benzene). UV-Vis (λ_{max}, nm) (DMSO): 265, 321, 373. Mass spectra: 385 [L]⁺.

Synthesis of complexes

To a stirred solution of **L** in chloroform (60 mL) was added dropwise M(NO₃)_n·nH₂O (2 mmol, if M = Cu 4 mmol) in methanol (40 mL). After the addition was complete, stirring was continued for 2 h, then the precipitate was filtered off, washed with CHCl₃ and methanol respectively and dried in air (Fig. 3).

[Cu₂(L)(NO₃)₂][NO₃]₂·H₂O: Brown. Yield: 0.65 g (42.1%). Anal. Calcd for Cu₂C₂₄H₂₃N₃O₁₄·H₂O : C, 37.0, H, 3.2, N, 12.6. Found: C, 37.4, H, 3.4, N, 12.3%. ¹H NMR (DMSO-d₆, δ ppm): 1.68 (CH₂CH₂CH₂), 1.84 (CH₂CH₂O), 4.15 (CH₂CH₂O), 7.02–7.74 (ArH), 10.41 (HC=N). Selected IR data (KBr, ν cm⁻¹): 3346 (H₂O), 3067 ν(Ar-CH), 2933, 2864 ν(Alf.-CH), 1616 ν(C=N), 1598 (C=N(pyridine)), 1488, 1453 ν(Ar-C=C), 1243 ν(Ar-O), 1105 ν(R-O), 753 ν(substituted benzene), 1384 ionic ν(NO₃⁻). Λ_M = 193 Ω⁻¹·mol⁻¹·cm². UV-Vis (λ_{max}, nm) (DMSO): 274, 324, 374. Mass spectra (m/z): 715 [[Cu₂(L)(NO₃)₂][NO₃]-H]⁺. (M.W. = 778 g mol⁻¹).

[Ni(L)(NO₃)₂]₂·2H₂O: Yellow. Yield: 0.44 g (35.2%). Anal. Calcd for NiC₂₄H₂₃N₃O₈·2H₂O: C, 47.7, H, 4.5, N, 11.6. Found: C, 48.1, H, 4.7, N, 11.7%. ¹H NMR (DMSO-d₆, δ ppm): 1.66 (CH₂CH₂CH₂), 1.93 (CH₂CH₂O), 4.16 (CH₂CH₂O), 6.96–7.68 (ArH), 10.41 (HC=N). Selected IR data (KBr, ν cm⁻¹): 3367 (H₂O), 3066 ν(Ar-CH), 2928, 2866 ν(Alf.-CH), 1653 ν(C=N), 1598 (C=N(pyridine)), 1489, 1453 ν(Ar-C=C), 1286, 1246 ν(Ar-O), 1106, 1048 ν(R-O), 752 ν(substituted benzene), Λ_M = 39 Ω⁻¹·mol⁻¹·cm². UV-Vis (λ_{max}, nm) (in DMSO): 276, 328, 376. Mass spectra(m/z): 603 [Ni(L)(NO₃)₂-H]⁺. (M.W. = 604 g mol⁻¹).

[Pb(L)(NO₃)][NO₃]₂·H₂O: Yellow. Yield: 0.41 g (28.0%). Anal. Calcd for PbC₂₄H₂₃N₃O₈·H₂O: C, 39.2, H, 3.4, N, 9.5. Found: C, 39.5,

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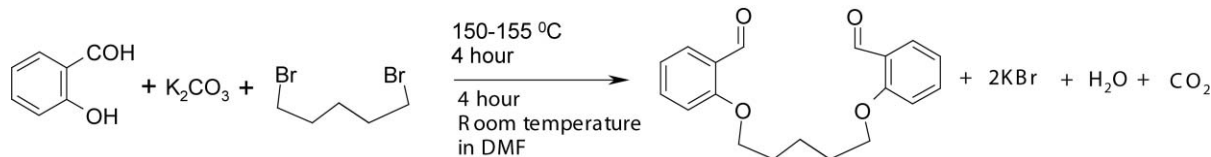


Fig. 1 Synthesis of 1,5-bis(2-formylphenyl)pentane.

H, 3.4, N, 9.7%. ^1H NMR could not be taken because of the low solubility. Selected IR data (KBr, ν cm^{-1}): 3429 (H_2O), 3068 $\nu(\text{Ar-CH})$, 2932, 2877 $\nu(\text{Alf.-CH})$, 1635 $\nu(\text{C=N})$, 1598 ($\text{C=N}(\text{pyridine})$), 1486, 1467 $\nu(\text{Ar-C=C})$, 1243 $\nu(\text{Ar-O})$, 1105 $\nu(\text{R-O})$, 751 $\nu(\text{substituted benzene})$, 1384 ionic $\nu(\text{NO}_3^-)$. $\Lambda_{\text{M}} = 86 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-Vis (λ_{max} , nm) (in DMSO): 274, 327, 377. Mass spectra (m/z): 654 $[\text{Pb}(\text{L})(\text{NO}_3)]^+$. (M.W. = 734 g mol^{-1}).

$[\text{Cd}(\text{L})][\text{NO}_3]_2 \cdot 2\text{H}_2\text{O}$: Yellow. Yield: 0.43 g (32.4%). Anal. Calcd for $\text{CdC}_{24}\text{H}_{23}\text{N}_5\text{O}_8 \cdot 2\text{H}_2\text{O}$: C, 43.8, H, 4.1, N, 10.6. Found: C, 44.5, H, 4.2, N, 10.8%. ^1H NMR (DMSO- d_6 , δ ppm): 1.67 ($\text{CH}_2\text{CH}_2\text{CH}_2$), 1.92 ($\text{CH}_2\text{CH}_2\text{O}$), 4.17 ($\text{CH}_2\text{CH}_2\text{O}$), 7.03–7.72 (ArH), 10.39 (HC=N). Selected IR data (KBr, ν cm^{-1}): 3378 (H_2O), 3069 $\nu(\text{Ar-CH})$, 2932, 2874 $\nu(\text{Alf.-CH})$, 1639 $\nu(\text{C=N})$, 1598 ($\text{C=N}(\text{pyridine})$), 1486, 1455 $\nu(\text{Ar-C=C})$, 1243 $\nu(\text{Ar-O})$, 1092 $\nu(\text{R-O})$, 754 $\nu(\text{substituted benzene})$, 1384 ionic $\nu(\text{NO}_3^-)$. $\Lambda_{\text{M}} = 168 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-Vis (λ_{max} , nm) (in DMSO): 274, 325, 377. Mass spectra (m/z): 657 $[[\text{Cd}(\text{L})](\text{NO}_3)_2]^+$. (M.W. = 657 g mol^{-1}).

$[\text{La}(\text{L})(\text{NO}_3)_3(\text{H}_2\text{O})] \cdot \text{H}_2\text{O}$: Yellow. Yield: 0.57 g (39.0%). Anal. Calcd for $\text{LaC}_{24}\text{H}_{25}\text{N}_5\text{O}_{12} \cdot \text{H}_2\text{O}$: C, 38.6, H, 3.6, N, 11.3. Found: C, 39.1, H, 3.9, N, 11.4%. ^1H NMR (DMSO- d_6 , δ ppm): 1.68 ($\text{CH}_2\text{CH}_2\text{CH}_2$), 1.91 ($\text{CH}_2\text{CH}_2\text{O}$), 4.17 ($\text{CH}_2\text{CH}_2\text{O}$), 6.97–7.66 (ArH), 10.41 (HC=N). Selected IR data (KBr, ν cm^{-1}): 3356 (H_2O), 3066 $\nu(\text{Ar-CH})$, 2933, 2863 $\nu(\text{Alf.-CH})$, 1646 $\nu(\text{C=N})$, 1597 ($\text{C=N}(\text{pyridine})$), 1488, 1453 $\nu(\text{Ar-C=C})$, 1286, 1243 $\nu(\text{Ar-O})$, 1096, 1048 $\nu(\text{R-O})$, 753 $\nu(\text{substituted benzene})$. $\Lambda_{\text{M}} = 27 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-Vis (λ_{max} , nm) (in DMSO): 274, 326, 376. Mass spectra (m/z): 648 $[\text{La}(\text{L})(\text{NO}_3)_2]^+$. (M.W. = 746 g mol^{-1}).

$[\text{Zn}(\text{L})(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$: Yellow. Yield: 0.52 g (42.6%). Anal. Calcd for $\text{ZnC}_{24}\text{H}_{23}\text{N}_5\text{O}_8 \cdot 2\text{H}_2\text{O}$: C, 47.2, H, 4.4, N, 11.5. Found: C, 47.5, H, 4.8, N, 11.4%. ^1H NMR (DMSO- d_6 , δ ppm): 1.67 ($\text{CH}_2\text{CH}_2\text{CH}_2$), 2.01 ($\text{CH}_2\text{CH}_2\text{O}$), 4.16 ($\text{CH}_2\text{CH}_2\text{O}$), 6.94–7.69 (ArH), 10.40 (HC=N). Selected IR data (KBr, ν cm^{-1}): 3371 (H_2O), 3067 $\nu(\text{Ar-CH})$, 2938, 2874 $\nu(\text{Alf.-CH})$, 1644 $\nu(\text{C=N})$, 1598 ($\text{C=N}(\text{pyridine})$), 1488, 1454 $\nu(\text{Ar-C=C})$, 1292, 1242 $\nu(\text{Ar-O})$, 1105, 1048 $\nu(\text{R-O})$, 756 $\nu(\text{substituted benzene})$. $\Lambda_{\text{M}} = 34 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-Vis (λ_{max} , nm) (in DMSO): 276, 326, 374. Mass spectra (m/z): 575 $[\text{Zn}(\text{L})(\text{NO}_3)_2 + \text{H}]^+$. (M.W. = 610 g/mol).

Result and discussion

Macrocyclic Schiff base complexes

The ligand and its complexes have been synthesised and characterised by elemental analysis, IR, ^1H and ^{13}C -NMR data, electronic spectra, magnetic susceptibility measurements, molar conductivity and mass spectra. The complexes have no clearly defined melting point and begin to decompose in the temperature range 250–350 $^{\circ}\text{C}$. Ligand **L** is soluble in DMSO, DMF, CHCl_3 , CH_2Cl_2 and CH_3CN but insoluble in H_2O , EtOH and MeOH. The complexes are air stable, partly soluble in DMF, DMSO but insoluble in CHCl_3 , CH_2Cl_2 and CH_3CN and the crystals were unsuitable for single-crystal X-ray structure determination.

Molar conductivity

The conductivities of the Pb(II) and Cd(II) complexes in DMSO are 86 $\Omega^{-1} \text{mol}^{-1} \text{cm}^2$ and 168 $\Omega^{-1} \text{mol}^{-1} \text{cm}^2$ respectively, which indicate that

they are 1:1 and 1:2 electrolytes respectively in DMSO. These results verify different binding modes of **L** in the case of the Cd(II) and Pb(II) metal ions. As expected, in the case of the relatively small Ni(II) and Zn(II) metal ions, the ligand behaves as a bidentate ligand using the lone electron pairs of the azomethine nitrogen atoms with NO_3^- ligands also. The conductivity measurements showed that these complexes are nonelectrolytes.^{12–14}

FTIR spectra

The IR spectrum of the ligand (**L**) shows $\nu(\text{C=N})$ at 1683 cm^{-1} , absence of a (C=O) peak at around 1700 cm^{-1} and $\nu(\text{NH}_2)$ at around 3300 cm^{-1} , indicative of Schiff's base condensation. The IR spectra of all complexes show $\nu(\text{C=N})$ at 1666–1653 cm^{-1} and it is found that the $\nu(\text{C=N})$ in the complexes is shifted by about 67–30 cm^{-1} to lower energy compared to that in the free ligand.^{12–14} This phenomenon appears to be due to the coordinated of azomethine nitrogen to the metal ion.¹⁵ A strong (H_2O) band at about 3380 cm^{-1} is observed in **L** and its complexes.¹⁶ Also, the ionic metal complexes exhibit an intense band at 1384 cm^{-1} assigned to NO_3^- .¹⁷ The IR spectra of the complexes clearly demonstrated that the COC and CCO stretching vibrations are altered compared to **L** due to conformational changes. The fact that the C–O–C absorptions of the complexes are shifted to lower wave numbers compared to those of **L** also confirms the complex formation.¹⁸ The spectra of all the metal complexes are dominated by bands between 2955 and 2828 cm^{-1} due to $\nu(\text{Alph.-CH})$. A band appearing in the 1598 cm^{-1} region is assigned to $\nu(\text{C=N})_{(\text{pyridine})}$ ¹⁹ and its value did not change in the complexes, thus the azomethine group in the pyridine did not bind the metal ions.²⁰

NMR spectra

^1H NMR and ^{13}C NMR of the 1,5-bis(2-formylphenyl)pentane and ligand and ^1H NMR of the complexes in DMSO- d_6 solution show that they are NMR active. The ^1H NMR spectra of the complexes exhibited almost the same values as **L**. Although we expected a shift on the position of the CH=N signal for the NMR spectra of the complexes, no significant shift could not be observed, but this resonance is observed in very low intensity owing to solubility problems.¹⁹

Electronic spectra

The electronic spectrum of **L** in DMSO shows absorption bands at ca 280, 320 and 370 nm arising from benzene and other chromophore moieties present in the ligand. The absorption bands of the complexes are shifted to longer wave numbers compared to those of **L** as expected.^{18–20} No d–d transitions for the complexes were observed probably due to the low solubility of complexes. A moderately intensive band observed in the range of 320–380 nm is due to $n-\pi^*$ transitions, and the strong band observed in the range of 270–280 nm is due to $\pi-\pi^*$ transitions²¹ for these complexes.

Magnetic measurements and mass spectra

The observed room-temperature magnetic moment value for the binuclear Cu(II) compound and the mononuclear complexes show them to be diamagnetic. The diamagnetic behaviour of the binuclear complex may be explained by a very strong anti-ferromagnetic interaction in the Cu–Cu pair.²² Their mass spectra confirm the monomeric [1+1] (dicarbonyl and diamine) nature of the complexes.²³

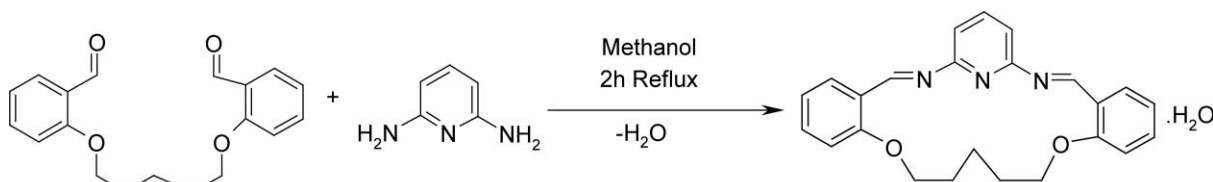


Fig. 2 Synthesis of **L**.

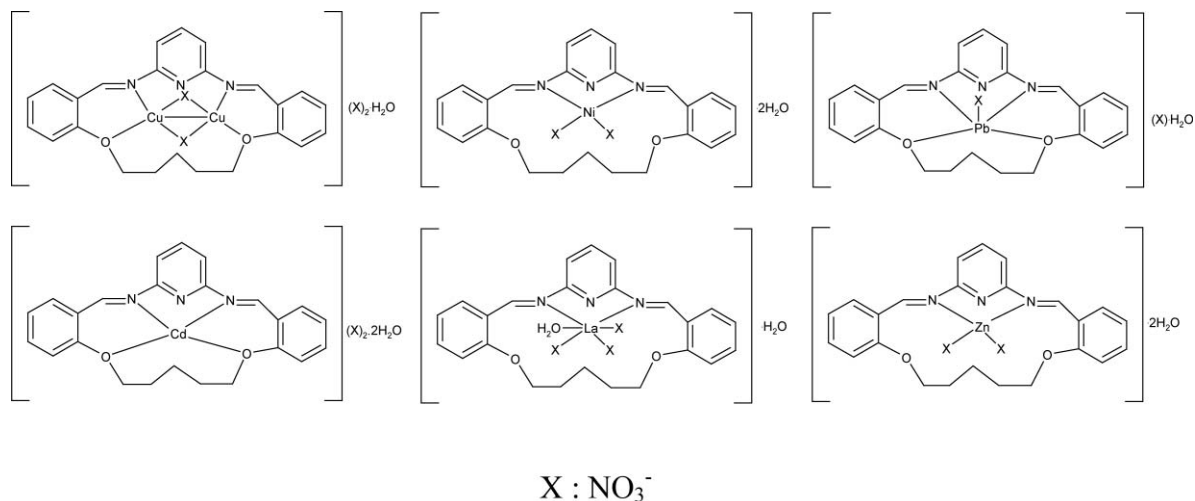


Fig. 3 Suggested structures of the complexes.

Conclusion

Compound **L** and its six complexes have been prepared and characterised. The binding mode of **L** in the Pb(II), Cd(II) and Cu(II) complexes in the solid state differs from the other complexes (Fig. 3). In the first case, **L** behaves as a tetradentate ligand using the lone electron pairs of the azomethine nitrogen atoms the two oxygens in the ether groups. In the second case, the ligand behaves as a bidentate ligand using only the azomethine nitrogen atoms. The long distance binding process can be favoured for the large Cd(II) and Pb(II) metal ions but not for smaller metal ions where coordination is then satisfied by, for example, two or three NO_3^- and one H_2O for the La(III) complex. Similar binding modes were observed in the literature for Pb(II) and Cd(II) metal ions.¹² The general structures proposed for the solid complexes are shown in Fig. 3. Most probably the La(III) complexes show octahedral geometry, the Zn(II) and Cd(II) complexes tetrahedral geometry, the Ni(II) complex square planar geometry and the Pb(II) complex pyramidal geometry around the central metal ions.^{23–25}

Received 29 December 2009; accepted 7 May 2010

Paper 100970 doi: 10.3184/030823410X12744563061343

Published online: 1 July 2010

References

- C. Celik, M. Tumer and S. Serin, *Synth. React. Inorg. Met. Org. Chem.*, 2002, **32** (10), 1839.

- R. Herzfeld and P. Nagy, *Spectrosc. Lett.*, 1999, **31** (1), 57.
- S. Tamburini, P. A. Vigato, M. Gatos, L. Bertolo and U. Casellato, *Inorg. Chim. Acta*, 2006, **359**, 183.
- N. Ranan and C. Thangaraja, *Trans. Met. Chem.*, 2005, **30**, 317.
- K.R. Krishnapriya and M. Kandaswamy, *Polyhedron*, 2005, **24**, 113.
- V. Alexander, *Chem. Rev.*, 1995, **95**, 273.
- D.E. Fenton and P. A. Vigato, *Chem. Soc. Rev.*, 1988, **17**, 69.
- S. İlhan and H. Temel, *J. Mol. Struct.*, 2008, **891**, 157.
- S. İlhan and H. Temel, *J. Coord. Chem.*, 2009, **62**, 456.
- İ. Yılmaz, S. İlhan, H. Temel and A. Kılıç, *J. Inc. Phenom Macrocyclic Chem.*, 2009, **63**, 163.
- A. Earnshaw, *Introduction to magnetochemistry*, Academic Press, London, 1968, p.4.
- S. İlhan, *J. Coord. Chem.*, 2009, **62**, 456.
- S. İlhan, H. Temel, İ. Yılmaz and M. Sekerci, *J. Organomet. Chem.*, 2007, **692**, 3855.
- S. İlhan, H. Temel and S. Paşa, *Chin. Chem. Lett.*, 2009, **20**, 339.
- S. İlhan, H. Temel and A. Kılıç, *Chin. J. Chem.*, 2007, **25**, 1547.
- S. İlhan, H. Temel, İ. Yılmaz and A. Kılıç, *Trans. Metal Chem.*, 2007, **32**, 3.
- S. İlhan and H. Temel, *Trans. Metal Chem.*, 2007, **32**, 1039.
- H. Temel, H. Hoşgören and M. Boybay, *Spectrosc. Lett.*, 2001, **34**, 1.
- S. İlhan, H. Temel, A. Kılıç and E. Tas, *Trans. Metal Chem.*, 2007, **32**, 1012.
- S. İlhan, *Indian J. Chem., A (IJC-A)*, 2008, **47A**, 374.
- S. İlhan, *Russ. J. Coord. Chem.*, 2009, **35**, 347.
- S. İlhan, *J. Coord. Chem.*, 2008, **61**, 2884.
- S. İlhan, H. Temel, R. Ziyadanogullari and M. Sekerci, *Trans. Metal Chem.*, 2007, **32**, 584.
- S. İlhan, H. Temel, İ. Yılmaz and M. Şekerci, *Polyhedron*, 2007, **12**, 2795.
- H. Temel, H. Alp, S. İlhan, B. Ziyadanogullari and İ. Yılmaz, *Monatsh. Chem.*, 2007, **138**, 1199.