

Synthesis and spectral studies of macrocyclic Pb(II), Zn(II) and La(III) complexes by template reaction of 1,4-bis(3-aminopropoxy)butane with metal nitrate and salicylaldehyde derivatives

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Six new macrocyclic complexes are synthesised by template reaction of 1,4-bis(3-aminopropoxy)butane with metal nitrate and 1,7-bis(2-formylphenyl)-1,4,7-trioxahseptane or 1,10-bis(2-formylphenyl)-1,4,7,10-tetraoxadecane and their structures are proposed on the basis of elemental analysis, FT-IR, UV-Vis, molar conductivity measurements, ^1H NMR and mass spectra. The complexes are 1:2 electrolytes for Pb(II), Zn(II) complexes and 1:3 electrolytes for La(III) as shown by their molar conductivities (Λ_m) in DMSO at 10^{-3} mol L $^{-1}$. The configurations of La(III) and Zn(II) complexes are proposed to probably octahedral.

Keywords: macrocyclic Schiff base, macrocyclic complexes, 1,7-bis(2-formylphenyl)-1,4,7-trioxahseptane, 1,10-bis(2-formylphenyl)-1,4,7,10-tetraoxadecane and 1,4-bis(3-aminopropoxy)butane

Schiff-base condensations are used to form macrocyclic ligands from diamines and dicarbonyl compounds. It is normally necessary to use a metal ion to act as a template in bringing the amine and carbonyl compound together.^{1–3} Schiff base macrocycles play a large role in macrocyclic chemistry.⁴ The coordination chemistry of macrocyclic ligands is a fascinating area of intense study for inorganic chemists.^{5,6} Synthesis of these Schiff-base complexes are achieved through the template reaction or transmetallation reactions.^{7–9} There is continuing interest in synthesising macrocyclic complexes^{10–12} because of their potential applications in fundamental and applied sciences and importance in the area of coordination chemistry.^{13–15}

In this study, I have synthesised Pb(II), Zn(II) and La(III) complexes by template reaction of 1,4-bis(3-aminopropoxy)butane with salicylaldehyde derivatives and $\text{M}(\text{NO}_3)_n \cdot 6\text{H}_2\text{O}$ in methanol. Then, spectral properties of the new compounds are studied in detail.

Experimental

Methods

Elemental analysis is carried out on a LECO CHNS model 932 elemental analyzer. ^1H NMR spectra are recorded using a model Bruker Avance DPX-400 NMR spectrometer. IR spectra are recorded on a Perkin Elmer Spectrum RX1 FTIR spectrometer on KBr discs in the wave number range of 4000–400 cm^{-1} . Electronic spectral studies are conducted on a Shimadzu model 160 UV Visible spectrophotometer in the wavelength 200–800 nm. Molar conductivity is measured with a WTW LF model 330 conductivity meters, using prepared solution of the complex in DMSO. LC/MS-API-ES mass spectra are recorded using an AGILENT model 1100 MSD mass spectrophotometer.

Chemical and starting materials

The salicylaldehyde derivatives which are used in the synthesis are prepared as shown in Fig. 1.^{16,18} All the other chemicals and solvents are of analytical grade and used as received.

General synthesis of complexes

To a stirred solution of salicylaldehyde derivatives (2 mmol) and metal nitrate in methanol (50 mL) is added dropwise 1,4-bis(3-aminopropoxy)butane (2 mmol) in methanol (30 mL). The reaction is continued for 2h at 80 °C and 1 h at room temperature. After the reaction is completed, the coloured precipitate is filtered and washed with methanol and, dried in air. Yield: 33–14%.

$[\text{PbL}^1][\text{NO}_3]_2 \cdot 2\text{H}_2\text{O}$: Yield: 0.55 g (32.5%). Anal. Calcd for $\text{PbC}_{28}\text{H}_{38}\text{N}_4\text{O}_{11} \cdot 2\text{H}_2\text{O}$: C, 39.58; H, 4.95; N, 6.60. Found: C, 40.61; H, 5.09; N, 6.53%. ^1H NMR (DMSO- d_6 , δ ppm): δ = 3.85 (H1, t, 4H, J = 7.2), δ = 4.22 (H2, t, 4H, J = 6.9), δ = 3.64 (H3, t, 4H, J = 4.6), δ = 1.92 (H4, p, 4H, J = 7.4), δ = 3.62 (H5, t, 4H, J = 5.6), δ = 3.51 (H6,

t, 4H, J = 7.1), δ = 1.38 (H7, t, 4H, J = 6.4), δ = 3.42 (H₂O), δ = 7.03–8.11 (m, 8H, ArH), δ = 10.36 (s, 2H, HC=N). Selected IR data (KBr, v cm^{-1}): 3368 $\text{v}(\text{H}_2\text{O})$, 1642 $\text{v}(\text{C}=\text{N})$, 1384 $\text{v}(\text{ionic NO}_3^-)$, 487 $\text{v}(\text{Pb}-\text{O})$, 439 $\text{v}(\text{Pb}-\text{N})$. $\Lambda_m = 183 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-vis (λ_{max} , nm) (DMSO): 278, 326, 382. Mass spectrum (m/z): [630, 2.6%, $[\text{PbL}^1-(\text{OCH}_2\text{CH}_2\text{O})^+]$].

$[\text{Zn}(\text{H}_2\text{O})\text{L}^1][\text{NO}_3]_2 \cdot 3\text{H}_2\text{O}$: Yield: 0.34 g (22.8%). Anal. Calcd for $\text{ZnC}_{28}\text{H}_{40}\text{N}_4\text{O}_{12} \cdot 3\text{H}_2\text{O}$: C, 45.22; H, 6.19; N, 7.54. Found: C, 45.37; H, 6.15; N, 7.58%. ^1H NMR (DMSO- d_6 , δ ppm): δ = 3.87 (H1, t, 4H, J = 6.1), δ = 4.26 (H2, t, 4H, J = 6.7), δ = 3.63 (H3, t, 4H, J = 5.6), δ = 1.94 (H4, p, 4H, J = 7.2), δ = 3.58 (H5, t, 4H, J = 5.4), δ = 3.50 (H6, t, 4H, J = 4.8), δ = 1.34 (H7, t, 4H, J = 6.4), δ = 3.42 (H₂O), δ = 7.02–8.07 (m, 8H, ArH), δ = 10.36 (s, 2H, HC=N). Selected IR data (KBr, v cm^{-1}): 3376 $\text{v}(\text{H}_2\text{O})$, 1646 $\text{v}(\text{C}=\text{N})$, 1384 $\text{v}(\text{ionic NO}_3^-)$, 515 $\text{v}(\text{Zn}-\text{O})$, 476 $\text{v}(\text{Zn}-\text{N})$. $\Lambda_m = 195 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-vis (λ_{max} , nm) (DMSO): 277, 325, 381. Mass spectrum (m/z): [691, 0.9%, $[\text{Zn}(\text{H}_2\text{O})\text{L}^1][\text{NO}_3]_2^+]$].

$[\text{La}(\text{H}_2\text{O})\text{L}^1][\text{NO}_3]_3 \cdot \text{H}_2\text{O}$: Yield: 0.25 g (14.8%). Anal. Calcd for $\text{LaC}_{28}\text{H}_{40}\text{N}_5\text{O}_{15} \cdot \text{H}_2\text{O}$: C, 39.86; H, 4.98; N, 8.30. Found: C, 40.07; H, 5.15; N, 8.51%. ^1H NMR (DMSO- d_6 , δ ppm): δ = 3.86 (H1, t, 4H, J = 6.4), δ = 4.27 (H2, t, 4H, J = 6.2), δ = 3.61 (H3, t, 4H, J = 5.2), δ = 1.95 (H4, p, 4H, J = 7.4), δ = 3.58 (H5, t, 4H, J = 6.2), δ = 3.52 (H6, t, 4H, J = 4.8), δ = 1.36 (H7, t, 4H, J = 6.3), δ = 3.41 (H₂O), δ = 7.01–8.09 (m, 8H, ArH), δ = 10.39 (s, 2H, HC=N). Selected IR data (KBr, v cm^{-1}): 3374 $\text{v}(\text{H}_2\text{O})$, 1641 $\text{v}(\text{C}=\text{N})$, 1384 $\text{v}(\text{ionic NO}_3^-)$, 493 $\text{v}(\text{La}-\text{O})$, 456 $\text{v}(\text{La}-\text{N})$. $\Lambda_m = 246 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-vis (λ_{max} , nm) (DMSO): 279, 327, 379. Mass spectrum (m/z): [827, 0.8%, $[\text{La}(\text{H}_2\text{O})\text{L}^1][\text{NO}_3]_3 + \text{H}^+]$].

$[\text{PbL}^2][\text{NO}_3]_2 \cdot \text{H}_2\text{O}$: Yield: 0.27 g (15.4%). Anal. Calcd for $\text{PbC}_{30}\text{H}_{42}\text{N}_4\text{O}_{12} \cdot \text{H}_2\text{O}$: C, 41.10; H, 5.02; N, 6.39. Found: C, 41.46; H, 5.19; N, 6.33%. ^1H NMR (DMSO- d_6 , δ ppm): δ = 3.59 (H1, s, 4H), δ = 3.85 (H2, t, 4H, J = 8.1), δ = 4.21 (H3, t, 4H, J = 7.6), δ = 3.56 (H4, t, 4H, J = 6.4), δ = 1.87 (H5, p, 4H, J = 4.3), δ = 3.23 (H6, t, 4H, J = 6.8), δ = 3.35 (H7, t, 4H, J = 7.3), δ = 1.42 (H7, t, 4H, J = 5.3), δ = 3.43 (H₂O), δ = 7.01–8.10 (m, 8H, ArH), δ = 10.38 (s, 2H, HC=N). Selected IR data (KBr, v cm^{-1}): 3376 $\text{v}(\text{H}_2\text{O})$, 1647 $\text{v}(\text{C}=\text{N})$, 1384 $\text{v}(\text{ionic NO}_3^-)$, 482 $\text{v}(\text{Pb}-\text{O})$, 436 $\text{v}(\text{Pb}-\text{N})$. $\Lambda_m = 207 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-vis (λ_{max} , nm) (DMSO): 277, 324, 378. Mass spectrum (m/z): [628, 1.3%, $[\text{PbL}^2-(\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{O})^+]$].

$[\text{ZnL}^2][\text{NO}_3]_2 \cdot \text{H}_2\text{O}$: Yield: 0.24 g (16.3%). Anal. Calcd for $\text{ZnC}_{30}\text{H}_{42}\text{N}_4\text{O}_{12} \cdot \text{H}_2\text{O}$: C, 48.98; H, 5.99; N, 7.62. Found: C, 49.19; H, 6.11; N, 7.50%. ^1H NMR (DMSO- d_6 , δ ppm): δ = 3.88 (H1, t, 4H, J = 6.1), δ = 4.26 (H2, t, 4H, J = 6.7), δ = 3.61 (H3, t, 4H, J = 5.6), δ = 1.96 (H4, p, 4H, J = 7.2), δ = 3.56 (H5, t, 4H, J = 5.4), δ = 3.51 (H6, t, 4H, J = 4.8), δ = 1.33 (H7, t, 4H, J = 6.4), δ = 3.41 (H₂O), δ = 6.93–8.08 (m, 8H, ArH), δ = 10.38 (s, 2H, HC=N). Selected IR data (KBr, v cm^{-1}): 3373 $\text{v}(\text{H}_2\text{O})$, 1649 $\text{v}(\text{C}=\text{N})$, 1384 $\text{v}(\text{ionic NO}_3^-)$, 523 $\text{v}(\text{Zn}-\text{O})$, 481 $\text{v}(\text{Zn}-\text{N})$. $\Lambda_m = 177 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$. UV-vis (λ_{max} , nm) (DMSO): 279, 327, 382. Mass spectrum (m/z): [735, 1.0%, $[\text{ZnL}^2][\text{NO}_3]_2 + \text{H}^+]$].

$[\text{LaL}^2][\text{NO}_3]_3 \cdot 2\text{H}_2\text{O}$: Yield: 0.32 g (18.0%). Anal. Calcd for $\text{LaC}_{30}\text{H}_{42}\text{N}_5\text{O}_{16} \cdot 2\text{H}_2\text{O}$: C, 40.54; H, 5.18; N, 7.88. Found: C, 41.12; H, 5.31; N, 7.73%. ^1H NMR (DMSO- d_6 , δ ppm): δ = 3.81 (H1, t, 4H, J = 6.7), δ = 4.28 (H2, t, 4H, J = 7.3), δ = 3.62 (H3, t, 4H, J = 6.2),

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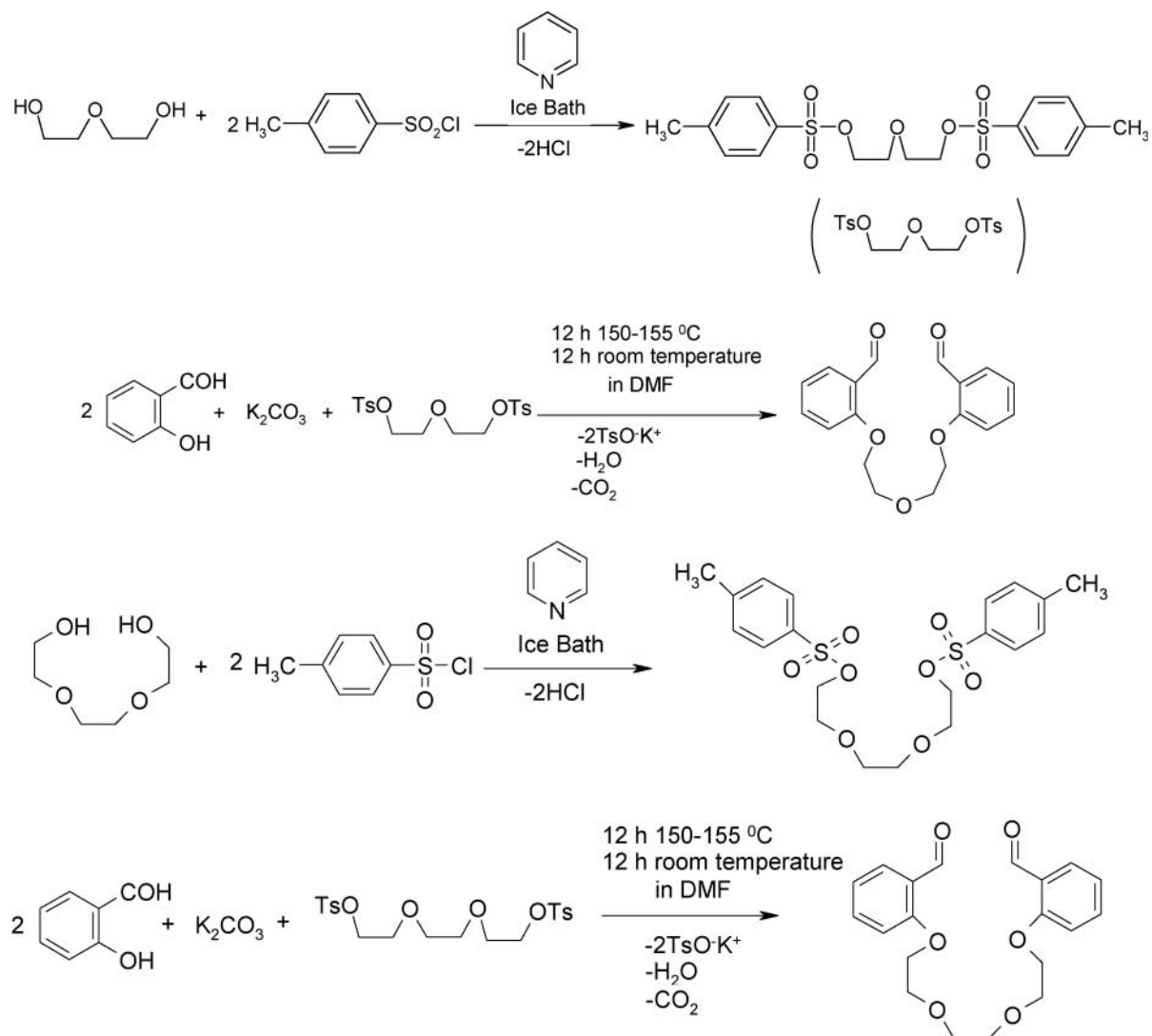


Fig. 1 Synthesis of 1,7-bis(2-formylphenyl)-1,4,7-trioxaheptane and 1,10-bis(2-formylphenyl)-1,4,7,10-tetraoxadecane.

$\delta = 1.96$ (H4, p, 4H, $J = 7.4$), $\delta = 3.57$ (H5, t, 4H, $J = 6.6$), $\delta = 3.54$ (H6, t, 4H, $J = 8.1$), $\delta = 1.35$ (H7, t, 4H, $J = 5.8$), $\delta = 3.41$ (H₂O), $\delta = 7.00$ – 8.04 (m, 8H, ArH), $\delta = 10.40$ (s, 2H, HC=N). Selected IR data (KBr, ν cm⁻¹): 3369 ν (H₂O), 1644 ν (C=N), 1384 ν (ionic NO₃⁻), 495 ν (La-O), 462 ν (La-N). $\Lambda_M = 262 \Omega^{-1}\text{mol}^{-1}\text{cm}^2$. UV-vis (λ_{max} , nm) (DMSO): 277, 326, 376. Mass spectrum (m/z): [852, 1.1%, [LaL₂][NO₃]₃]⁺].

Result and discussion

Macrocyclic Schiff base complexes

In the reaction between synthesis of 1,7-bis(2-formylphenyl)-1,4,7-trioxaheptane or 1,10-bis(2-formylphenyl)-1,4,7,10-tetraoxadecane, metal nitrate and 1,4-bis(3-aminopropoxy)butane in methanol, the [1+1] macrocyclic Schiff-base complexes are formed as the product. The macrocyclic complexes are characterised by elemental analysis, UV-vis spectra, conductivity measurements, mass, ¹H NMR and IR spectra. The mass spectrum of complexes plays an important role in confirming the monomeric [1+1] (dicarbonyl and diamine) nature of complexes. As the crystals are unsuitable for single-crystal, X-ray analyses of the complexes cannot be done. The complexes are insoluble in most common solvents, including water, ethanol, ethyl acetate, and acetonitrile (Table 1).

FT-IR spectra

The characteristic IR spectrum data are given in the experimental section. IR spectra of the complexes are recorded in the KBr pellet from 4000 to 400 cm⁻¹. The broad bands within the range *ca* 3370 cm⁻¹ for all complexes can be attributed to stretching vibrations of water molecule ν (H₂O).¹⁹ The absence of a ν (C=O) peak at around 1700 cm⁻¹

and ν (NH₂) peak at around 3300 cm⁻¹ are indicative of Schiff's base condensation. A strong band observed in the IR spectra of the complexes at *ca* 1645 cm⁻¹ region which is attributed to the ν (C=N) stretch, indicating coordination of the azomethine nitrogen to metal.²⁰ The presence of several bands in the region associated with nitrate vibrations clearly identifies these species as containing nitrate groups. The absorptions of the nitrate counterions, at *ca* 1460–1452 (ν_3), 1300 (ν_1) and 1040 (ν_2) cm⁻¹, suggest the presence of nitrate groups: a intense band at *ca* 1384 cm⁻¹ attributable to ionic nitrate, is also present.²¹ In the spectra of all the complexes are dominated by bands between 2965 and 2855 cm⁻¹ due to ν (Alph.-CH) groups.²² Conclusive evidence of the bonding is also shown by the observation that new bands in the IR spectra of the complexes appear at 525–485 cm⁻¹ and 481–435 cm⁻¹ assigned to ν (M–O) and ν (M–N) stretching vibrations²³ (Table 2).

Electronic spectra

Electronic absorption spectral data of complexes in DMSO at room temperature are presented in the experimental section. The absorption bands below *ca* 300 nm are practically identical and can be attributed to $\pi \rightarrow \pi^*$ transitions in the benzene ring and azomethine (–C=N) groups. The absorption bands observed *ca* the 300–330 nm range are most probably due to the transitions of $n \rightarrow \pi^*$ of imine groups.^{22–24}

Molar conductivity and mass spectra

The complexes are 1:2 electrolytes for Pb(II) and Zn(II) complexes as shown by their molar conductivities (Λ_M) in DMSO at 10⁻³ M, which are in the range 140–200 $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$ and 1:3 electrolytes for La(III) complexes as shown by their molar conductivities (Λ_M) in DMSO at 10⁻³ M, which are in the range 200–250 $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$.^{25–27}

Table 1 Physical characterisation, analytical, molar conductance and mass data of the complexes

Compound	(Calcd) Found %C	%H	%N	Λ_M (ohm ⁻¹ cm ² mol ⁻¹)	Formula weight	MS/EI	Assignment
[PbL ¹][NO ₃] ₂ ·2H ₂ O	(39.58) 40.61	(4.95) 5.09	(6.60) 6.53	183	850	630	[PbL ¹ ·(OCH ₂ CH ₂ O)] ⁺ .
[Zn(H ₂ O)L ¹][NO ₃] ₂ ·3H ₂ O	(45.22) 45.37	(6.19) 6.15	(7.54) 7.58	195	744	691	[Zn(H ₂ O)L ¹][NO ₃] ₂ ⁺
[La(H ₂ O)L ¹][NO ₃] ₃ ·H ₂ O	(39.86) 40.07	(4.98) 5.15	(8.30) 8.51	246	844	827	[La(H ₂ O)L ¹][NO ₃] ₃ +H ⁺]
[PbL ²][NO ₃] ₂ ·H ₂ O	(41.10) 41.46	(5.02) 5.19	(6.39) 6.33	207	876	628	[PbL ² ·(OCH ₂ CH ₂ OCH ₂ CH ₂ O)] ⁺
[ZnL ²][NO ₃] ₂ ·H ₂ O	(48.98) 49.19	(5.99) 6.11	(7.62) 7.50	177	734	735	[ZnL ²][NO ₃] ₂ ·H ₂ O+H ⁺
[LaL ²][NO ₃] ₃ ·2H ₂ O	(40.54) 41.12	(5.18) 5.31	(7.88) 7.73	262	888	852	[LaL ²][NO ₃] ₃ ⁺

Table 2 IR (cm⁻¹) spectral data for the complexes

Compound	$\nu(\text{H}_2\text{O})$	$\nu(\text{C}=\text{N})$	ionic $\nu(\text{NO}_3^-)$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$
[PbL ¹][NO ₃] ₂ ·2H ₂ O	3368 s	1642 m	1384 m	487 w	439 w
[Zn(H ₂ O)L ¹][NO ₃] ₂ ·3H ₂ O	3369 s	1636 m	1384 m	515 w	476 w
[La(H ₂ O)L ¹][NO ₃] ₃ ·H ₂ O	3364 s	1635 m	1384 m	493 w	456 w
[PbL ²][NO ₃] ₂ ·H ₂ O	3333 s	1635 m	1384 m	482 w	436 w
[ZnL ²][NO ₃] ₂ ·H ₂ O	3348 s	1634 m	1383 m	523 w	481 w
[LaL ²][NO ₃] ₃ ·2H ₂ O	3349 s	1635 m	1384 m	495 w	462 w

m, medium; s, strong; w, weak.

The mass spectrum of the complexes are given in experimental section and shown in Table 1.²⁵⁻²⁷

NMR spectra

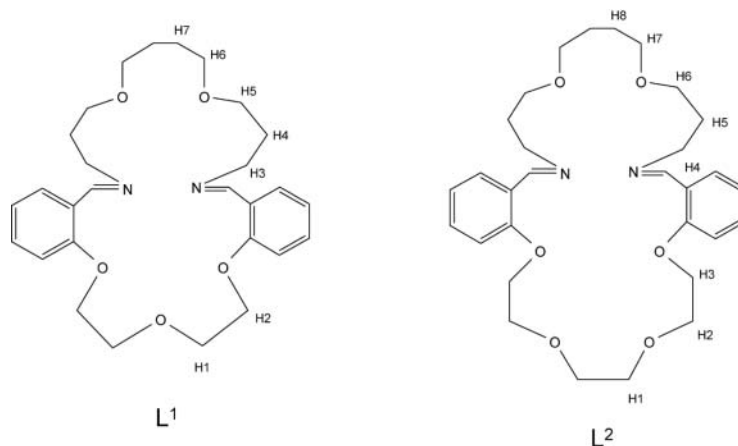
The ¹H NMR spectra are run in DMSO-d₆ and gives the expected simple spectrum, indicating the integrity of the complex in that solvent. The ¹H NMR spectra of the complexes show a multiplet in the range 7.1–8.0 ppm due to aromatic protons, 3,4 ppm due to H₂O protons, around 1.99–4.32 ppm due to CH₂, OCH₂ and OCH₂Ph protons and 10.4 ppm corresponding to the imine protons, but no signals corresponding to the formyl or amine protons are present.²⁶ Also ¹H NMR spectral data of the complexes are given in experimental section (Fig. 2).

Conclusion

The novel six Schiff base macrocyclic complexes are synthesised by template reaction and characterised by elemental analyses, FTIR and UV-Vis spectra, conductivity measurements, ¹H NMR and mass spectra. The complexes have no clearly defined melting point and begin to decompose in the temperature range 250–350 °C.²⁷ The complexes are air stable,

soluble in DMF, DMSO and insoluble CHCl₃, CH₂Cl₂ and less soluble MeOH, EtOH, CH₃CN. As the crystals are unsuitable for single-crystal, X-ray analyses of the complexes cannot be done.²⁸ Absolute structure of the complexes can be determined using X-ray diffraction method. In this study only suggested structure of the complexes are shown in Fig. 3. The La(III) and Zn(II) complexes probability show octahedral geometry.²⁹ As the Pb(II) is large, the ligand behaves as a heptadentate or octadentate ligand with the lone electron pairs of two azomethine nitrogen atoms and the lone electron pairs of five or six oxygen atoms in ether groups. As La(III) and Zn(II) are small, La(III) and Zn(II) coordinate to three oxygen atoms in L¹, lone electron pairs of two azomethine nitrogen atoms and one H₂O, four oxygen atoms in L² lone electron pairs of two azomethine nitrogen atoms.

In brief, the Pb(II) metal ion can bind seven or eight atoms and Zn(II) and La(III) ions can bind six atoms in the complexes. Structure of the Pb(II) complexes are different from structure of the Zn(II) and La(III) complexes. Similar cases can be seen in the literature.³⁰⁻³⁴

**Fig. 2** Structure of the ligands.

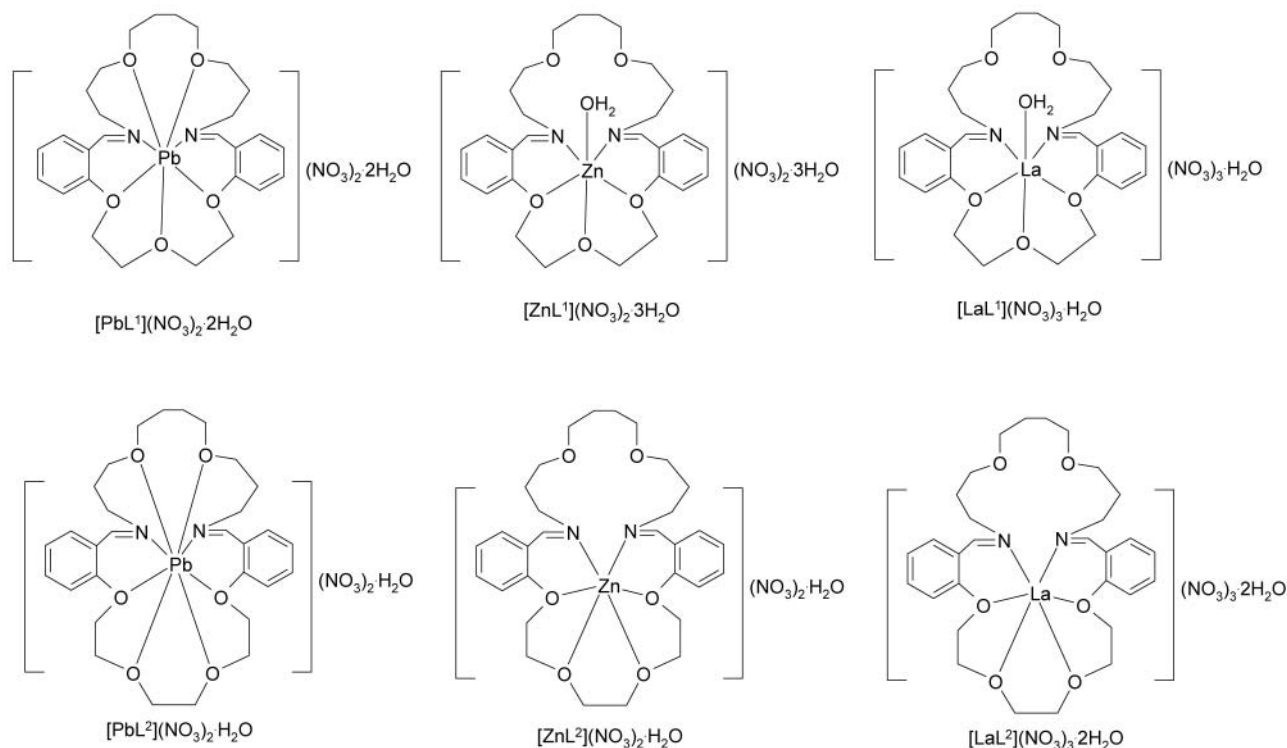


Fig. 3 Suggested structure of the complexes.

Received 13 September 2009; accepted 27 November 2009
 Paper 090784 doi: 10.3184/030823409X12609007112282
 Published online: 20 January 2010

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