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Journal of Organometallic Chemistry 689 (2004) 947–952

Journal of Organometallic Chemistry

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Coordination chemistry of a multidentate pyrrolylaldiminate ligand. X-ray crystal structure of double-helical bis-μ-[N,N'-ethylenedi(5-*tert*-butyl-pyrrol-2-ylaldiminate)]-dimagnesium

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Received 11 November 2003; accepted 9 December 2003

Abstract

The coordination chemistry of N,N'-ethylenedi(5-*tert*-butyl-pyrrol-2-ylaldiminate) dianion (L^{2-}) is described. The ligand precursor H_2L was prepared in high yield from the condensation reaction of ethylenediamine with 5-*tert*-butylpyrrole-2-carbaldehyde. Treatment of H_2L with $Zr(CH_2Ph)_4$ generated mononuclear $ZrL(CH_2Ph)_2$ (**1**), whereas the reaction of H_2L with Mg^nBu_2 or $ZnEt_2$ produced dinuclear M_2L_2 (M = Mg, **2**; Zn, **3**). In addition to spectroscopic characterizations of all new compounds, X-ray crystal structure of **2** was obtained. The X-ray study revealed a double-stranded helical structure in which the magnesium atoms are bound to two interwined L^{2-} ligands with a Mg···Mg separation of 4.116 Å.

Keywords: Zirconium; Magnesium; Zinc; Double-helix; Pyrrolylaldiminate

1. Introduction

Schiff bases are appealing ligands that have generated a wide spectrum of transition metal, main-group metal, and f-element complexes. Among the dianionic tetradentate Schiff bases reported to date, salen [1–9] and porphyrin [10–16] derivatives represent perhaps the most popular and significant examples for acyclic and macrocyclic ligands, respectively (Scheme 1). Intriguing chemistry evolved from metal complexes containing these ligands has emerged extensively. In comparison, chemistry involving dianionic tetradentate pyrrolylaldiminate ligands remains relatively undeveloped [17–24]. These compounds can be regarded as a tetraaza analogue of ubiquitous salen ligands or an acyclic variation of porphyrin derivatives. We chose to examine ligands that incorporate a substituent at the 5-position of the pyrrole rings [25], such as N,N'-ethylenedi(5-*tert*-butyl-pyrrol-2-ylaldiminate) dianion (L^{2-}). The 5-*tert*-butyl groups are anticipated to not only offer steric protection to the metal center but also preclude possible side reactions on the pyrrole rings. In this contribution, we aim to demonstrate the versatility of this ligand in binding to both transition and main-group metals. Of particular interest is the divergent coordination modes of L^{2-}, which acts as a tetradentate ligand for zirconium but contrarily a bis(bidentate) ligand for magnesium and zinc.

2. Experimental

2.1. General procedures

Unless otherwise specified, all experiments were performed under nitrogen using standard Schlenk or glovebox techniques. All solvents were reagent grade or better and purified by standard methods. All other chemicals were used as received from commercial

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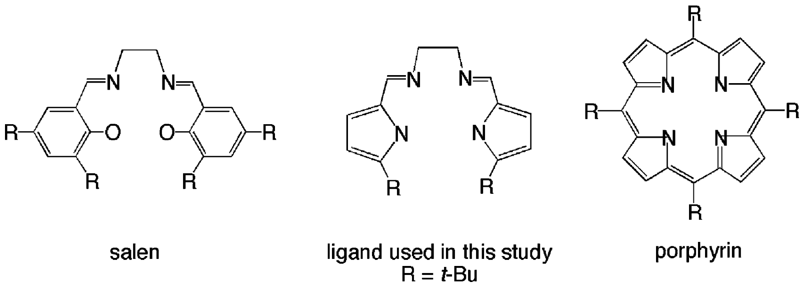
doi:10.1016/j.jorganchem.2003.12.035



Scheme 1. Representative examples of dianionic tetradentate Schiff bases.

vendors. The NMR spectra were recorded on Varian instruments. Chemical shifts (δ) are listed as parts per million downfield from tetramethylsilane and coupling constants (J) are in hertz. ^{1}H NMR spectra are referenced using the residual solvent peak at δ 7.16 for C_6D_6, and δ 7.27 for $CDCl_3$. ^{13}C NMR spectra are referenced using the residual solvent peak at δ 128.39 for C_6D_6. The assignment of the carbon atoms for all new compounds is based on the DEPT ^{13}C NMR spectroscopy. Routine coupling constants are not listed. All NMR spectra were recorded at room temperature in specified solvents. Low-resolution mass spectra were recorded on a Finnigan MAT 95XL Mass Spectrometer. High-resolution mass spectra were recorded on a Bruker Apex Mass Spectrometer. Elemental analysis was performed on a Heraeus CHN-O Rapid analyzer. Satisfactory analysis was hampered for compounds **1** and **2** due to their air- and moisture-sensitivity.

2.2. Synthesis of N,N′-ethylenedi(5-tert-butyl-pyrrol-2-ylaldimine) (H_2L)

Ethylenediamine (0.447 mL, 6.613 mmol) was added to an ethanol solution (10 mL) of 5-*tert*-butyl-2-pyrrolecabaldehyde [26] (2 g, 13 mmol) at room temperature. One drop of glacial acetic acid was added and the reaction solution was stirred for 2 d. All volatiles were removed in vacuo. The resulting residue was extracted with boiling hexane (20 mL) and filtered. The filtrate was evaporated to dryness to afford the desired product as a yellowish orange solid; yield 1.706 g (79%). Recrystallization of the yellowish orange solid from diethyl ether gave yellow crystals; yield 1.468 g (68%). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.99 (s, 2, CH=N), 6.37 (d, 2, $J = 3.5$ Hz, pyrrole-CH), 5.96 (d, 2, $J = 3.5$ Hz, pyrrole-CH), 3.76 (s, 4, CH_2), 1.32 (s, 18, CMe_3), NH not found. ^{1}H NMR (C_6D_6, 500 MHz) δ 9.85 (br s, 1, NH), 7.83 (s, 2, CH=N), 6.37 (d, 2, $J = 3.5$ Hz, pyrrole-CH), 6.02 (d, 2, $J = 3.5$ Hz, pyrrole-CH), 3.76 (s, 4, CH_2), 1.02 (s, 18, CMe_3). ^{13}C NMR (C_6D_6, 125.5 MHz) δ 153.23 (CH=N), 146.73 (C, pyrrole), 130.32 (C, pyrrole), 115.01 (CH, pyrrole), 105.38 (CH, pyrrole), 62.36 (CH_2), 31.95 (CMe_3), 30.61 (CMe_3). LRMS (EI) Calc. for $C_{20}H_{30}N_4$ m/z 326, found m/z 326. Anal. Calc. for $C_{20}H_{30}N_4$: C, 73.58; H, 9.26; N, 17.16. Found: C, 73.58; H, 9.44; N, 16.60%.

2.3. Synthesis of $ZrL(CH_2Ph)_2$ (**1**)

Solid H_2L (140.4 mg, 0.43 mmol) was added to a diethyl ether solution (10 mL) of $Zr(CH_2Ph)_2$ [27] prepared in situ from the reaction of $PhCH_2MgCl$ (1.72 mL, 1 M in diethyl ether, Aldrich, 1.72 mmol) with $ZrCl_4$ (100 mg, 0.43 mmol) in the absence of light. The reaction mixture was stirred at room temperature overnight and filtered through a pad of Celite. The solution was evaporated to dryness to afford the product as a red solid; yield 239 mg (93%). Recrystallization from diethyl ether yielded orange-red crystals. ^{1}H NMR (C_6D_6, 500 MHz) δ 7.55 (s, 2, CH=N), 7.27 (t, 4, H_{meta} in $ZrCH_2Ph$), 7.18 (d, 2, $J = 3.5$ Hz, pyrrole-CH), 7.06 (t, 2, H_{para} in $ZrCH_2Ph$), 6.94 (d, 2, $J = 3.5$ Hz, pyrrole-CH), 6.90 (d, 4, H_{ortho} in $ZrCH_2Ph$), 3.37 (s, 4, NCH_2), 2.91 (s, 4, $ZrCH_2Ph$), 2.05 (s, 18, CMe_3). ^{13}C NMR (C_6D_6, 125.5 MHz) δ 167.27 (CH=N), 158.82 (CH), 144.03 (C), 138.12 (C), 130.62 (CH), 128.68 (CH), 124.29 (CH), 122.27 (CH), 113.40 (CH), 77.42 ($ZrCH_2$), 57.25 (NCH_2), 35.23 (CMe_3), 33.48 (CMe_3). LRMS (EI) Calc. for $C_{27}H_{37}N_4Zr$ ($[M–CH_2Ph]^+$) m/z 507, found m/z 507.

2.4. Synthesis of Mg_2L_2 (**2**)

Solid H_2L (200 mg, 0.613 mmol) was dissolved in diethyl ether (6 mL) and cooled to −35 °C. To this was added Mg^nBu_2 (0.61 mL, 1 M in heptane, Aldrich, 0.61 mmol) dropwise via a syringe. The reaction mixture was naturally warmed to room temperature and stirred for 2 d. After being filtered through a pad of Celite, the reaction solution was concentrated under reduced pressure to ca. 2 mL. The concentrated solution was then cooled to −35 °C to afford the product as red crystals; yield 288 mg (62%). X-ray quality crystals were grown from a concentrated diethyl ether solution at room temperature. ^{1}H NMR (C_6D_6, 500 MHz) δ 7.16 (s, 4, CH=N), 6.80 (d, 4, $J = 3.5$ Hz, pyrrole-CH), 6.43 (d, 4, $J = 3.5$ Hz, pyrrole-CH), 3.00 (d, 4, $^2J_{HH} = 10.5$ Hz, CH_AH_B), 2.92 (d, 4, $^2J_{HH} = 10.5$ Hz, CH_AH_B), 1.30 (s, 36, CMe$_3$). ^{13}C NMR (C$_6$D$_6$, 500 MHz) δ 162.99 (CH=N), 161.02 (C, pyrrole), 135.78 (C, pyrrole), 122.67 (CH, pyrrole), 109.85 (CH, pyrrole), 54.29 (CH$_2$), 33.73 (C Me$_3$), 31.69 (CMe_3). HRMS (EI) Calc. for $C_{40}H_{56}Mg_2N_8$ m/z 696.4329, found m/z 696.4332. Anal. Calc. for $C_{40}H_{56}Mg_2N_8$: C, 68.88; H, 8.09; N, 16.06. Found: C, 67.05; H, 8.14; N, 15.67%.

2.5. Synthesis of Zn_2L_2 (**3**)

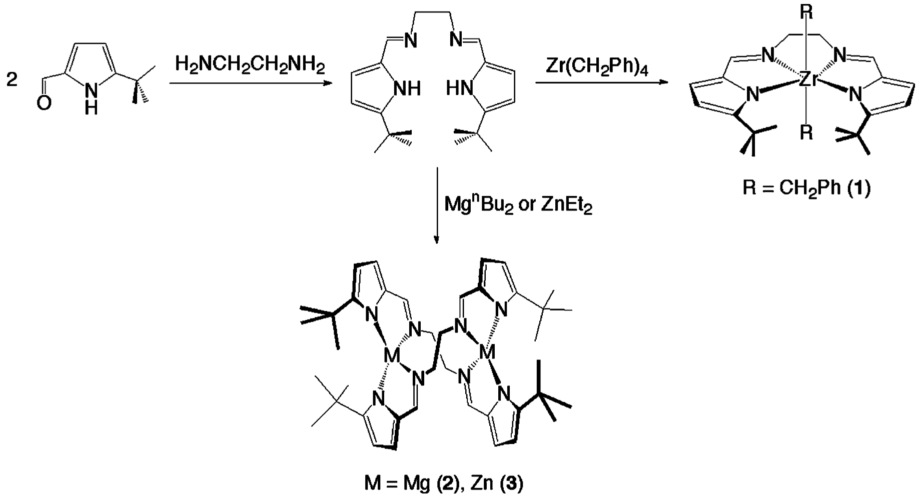
Solid H$_2$L (200 mg, 0.613 mmol) was dissolved in pentane (10 mL) and cooled to −35 °C. To this was added ZnEt$_2$ (0.61 mL, 1 M in hexane, Aldrich, 0.61 mmol) dropwise via a syringe. The reaction mixture was naturally warmed to room temperature and stirred for 2 d. After being filtered through a pad of Celite, the reaction solution was concentrated under reduced pressure to ca. 2 mL. The concentrated solution was then cooled to −35 °C to afford the product as an off-white solid, which was isolated and dried in vacuo; yield 134 mg (56%). ^{1}H NMR (CDCl$_3$, 200 MHz) δ 7.37 (s, 4, CH=N), 6.69 (d, 4, J = 3.6 Hz, pyrrole-CH), 6.26 (d, 4, J = 3.6 Hz, pyrrole-CH), 3.50 (d, 4, $^2J_{HH}$ = 11.6 Hz, CH_AH$_B$), 3.30 (d, 4, $^2J_{HH}$ = 11.6 Hz, CH$_A$$H_B$), 1.20 (s, 36, CMe$_3$). ^{1}H NMR (C$_6D_6$, 500 MHz) δ 7.07 (s, 4, CH=N), 6.85 (d, 4, J = 3.5 Hz, pyrrole-CH), 6.53 (d, 4, J = 3.5 Hz, pyrrole-CH), 3.32 (d, 4, $^2J_{HH}$ = 12.5 Hz, CH_AH$_B$), 3.12 (d, 4, $^2J_{HH}$ = 12.5 Hz, CH$_A$$H_B$), 1.37 (s, 36, CMe$_3$). ^{13}C NMR (C$_6D_6$, 500 MHz) δ 160.13 (CH=N), 159.28 (C, pyrrole), 135.95 (C, pyrrole), 120.50 (CH, pyrrole), 110.21 (CH, pyrrole), 54.38 (CH$_2$), 33.61 (C Me$_3$), 31.44 (CMe_3). HRMS (EI) Calc. for $C_{40}H_{56}Zn_2N_8$ m/z 776.3211, found m/z 776.3221. Anal. Calc. for $C_{40}H_{56}Zn_2N_8$: C, 61.62; H, 7.24; N, 14.37. Found: C, 61.47; H, 7.22; N, 14.30%.

3. X-ray crystallography

Data for compound **2** were collected on a Bruker SMART 1000 CCD diffractometer with graphite monochromated Mo-Kα radiation (λ = 0.7107 Å). Structures were solved by direct methods and refined by full matrix least squares procedures against F_2 using SHELXTL. All full-weight non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in calculated positions. One of the *tert*-butyl groups is disordered with the methyl substituents being in the ratio ca. 1:1 over two conformations.

4. Results and discussion

The ligand precursor H$_2$L was readily prepared by conventional condensation method from the reaction of ethylenediamine with two equivalents of 5-*tert*-butyl-2-pyrrolecabaldehyde [26] in ethanol (Scheme 2). Addition of H$_2$L to an ethereal solution of Zr(CH$_2$Ph)$_4$ [27] generated high yield of ZrL(CH$_2$Ph)$_2$ (**1**) as orange crystals. Solution NMR spectroscopic data of **1** are consistent with a molecule having a C_{2v} symmetry. For instance, the ^{1}H NMR spectroscopy reveals a singlet resonance at 2.98 ppm for the ethylene backbone, a singlet at 2.52 ppm for the benzylic protons, and a singlet at 1.67 ppm for the t-butyl groups. The imine protons of the L^{2-} ligand are shifted upfield as compared to those of H$_2$L, whereas the aromatic hydrogen atoms on the pyrrole rings show downfield shift. Similar trend in the change of chemical shifts is also reported for other group 4 metal complexes containing a pyrrolylaldiminate ligand [25]. The zirconium center and the tetraaza ligand likely adopt an approximately coplanar geometry and the two benzyl-groups are *trans* to each other. The



Scheme 2.

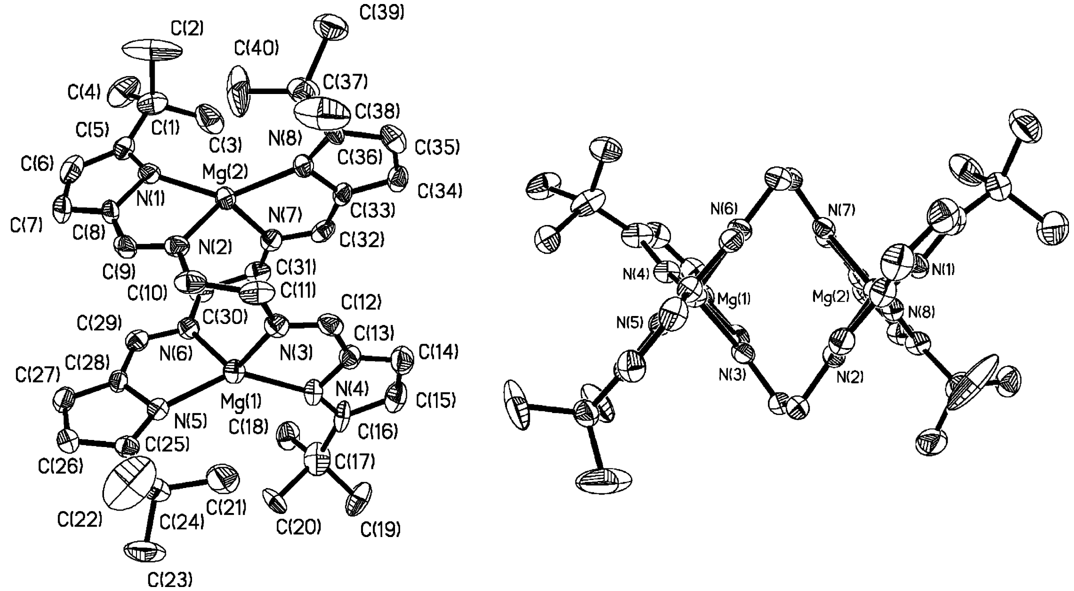


Fig. 1. Two views of the molecular structure of Mg_2L_2 (**2**), featuring the double helicate (left) and the cavity (right).

ortho protons of the benzyl groups appear as a doublet resonance at 6.90 ppm, suggesting η^1-coordination mode. The η^1-benzyl complex is also described previously for six-coordinate group 4 metal complexes that contain a tetradentate ligand [28].

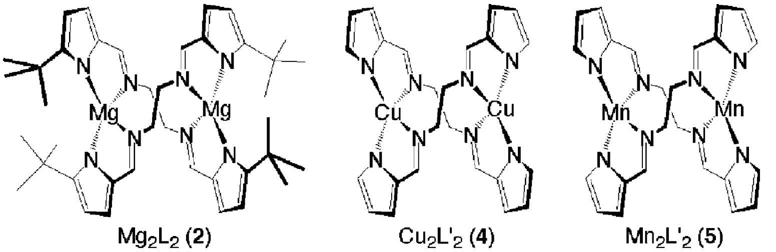
The reaction of H_2L with Mg^nBu_2 in diethyl ether at −35 °C generated red crystals of dimeric Mg_2L_2 (**2**). In contrast to that found in **1**, the tetraaza ligand in **2** is a binucleating bis(bidentate) ligand. An X-ray study of **2** revealed a double-stranded helicate, as illustrated in Fig. 1. Crystallographic details are summarized in Table 1, and selected bond distances and angles are listed in Table 2. The $Mg–N_{imine}$, $Mg–N_{pryyolr}$ distances and the $N_{imine}–Mg–N_{pyrrole}$ binding angle are all unexceptional for pyrrolylaldiminate complexes of magnesium [29]. Crystallographically characterized double helical complexes of pyrrolylaldiminate are extremely rare. To date, compound **2** represents the third example as indicated by the Cambridge structural database. Scheme 3 depicts the magnesium compound **2** and the previously reported dinuclear copper (**4**) [19] and manganese (**5**) [18] complexes supported by the pyrrolylaldiminate ligands (L'^{2-}) that contain a proton rather than a *t*-butyl group at the 5-position. Similar to those observed in **4** and **5**, complex **2** possesses a crystallographically D_2 symmetry. The two magnesium atoms in **2** are displaced out of the mean pyrrolylaldiminate planes by a distance ranging from 0.2215 to 0.5389 Å. The coordination about the magnesium atoms is roughly tetrahedral, with dihedral angles between the two pyrrolylaldiminate-planes of 85.3° and 85.6° for Mg(1) and Mg(2), respectively. Notably these values are much larger than the corresponding angles found in **4** (35.0° average) and **5** (76.1° average). The distance

Table 1
Crystallographic data for Mg_2L_2 (**2**)

Formula	$C_{40}H_{56}Mg_2N_8$
M	697.55
Crystal size (mm^3)	0.39 × 0.26 × 0.18
Crystal system	Monoclinic
Space group	$P2_1/c$
a (Å)	13.855(5)
b (Å)	22.727(9)
c (Å)	13.176(6)
β (°)	99.143(9)
V (Å^3)	4096(3)
Z	4
D_{calc} (g cm^{-3})	1.131
$2\theta_{max}$ (°)	55.58
T (K)	150(2)
Total reflections	14,642
Independent reflections	6986
T_{max}	0.9486
T_{min}	0.4001
Absorption coefficient (mm^{-1})	0.096
R_{int}	0. 0604
Goodness-of-fit	0.959
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0668$, $wR_2 = 0.1639$
R indices (all data)	$R_1 = 0.1536$, $wR_2 = 0.1853$

Table 2
Selected bond distances (Å) and angles (°) for Mg_2L_2 (**2**)

Mg(1)–N(4)	2.042(4)	Mg(2)–N(1)	2.026(4)
Mg(1)–N(5)	2.042(4)	Mg(2)–N(8)	2.041(4)
Mg(1)–N(3)	2.085(3)	Mg(2)–N(2)	2.094(4)
Mg(1)–N(6)	2.102(3)	Mg(2)–N(7)	2.100(4)
N(4)–Mg(1)–N(5)	139.99(15)	N(1)–Mg(2)–N(8)	141.16(15)
N(4)–Mg(1)–N(3)	83.40(16)	N(1)–Mg(2)–N(2)	83.39(15)
N(5)–Mg(1)–N(3)	118.08(15)	N(8)–Mg(2)–N(2)	119.46(15)
N(4)–Mg(1)–N(6)	116.16(15)	N(1)–Mg(2)–N(7)	114.13(14)
N(5)–Mg(1)–N(6)	83.94(15)	N(8)–Mg(2)–N(7)	83.50(15)
N(3)–Mg(1)–N(6)	119.30(14)	N(2)–Mg(2)–N(7)	118.93(14)



Scheme 3.

between the two metal atoms of 4.116 Å in **2** is longer than those found for **4** (3.29 Å) and **5** (3.997 Å), a result that is ascribable to the distinct coordination geometries found in these complexes.

In contrast to **4** and **5**, the solution structure of **2** can be readily characterized by ^{1}H and ^{13}C NMR spectroscopy due to its diamagnetic property. In solution, compound **2** remains the double-helical structure as indicated by NMR spectroscopy. For instance, the imino protons are observed as a singlet resonance in the ^{1}H NMR spectroscopy, so are the *t*-butyl groups. The four methylene-moieties are all chemically equivalent as only one CH$_2$ signal is observed at 54.3 ppm in the ^{13}C{^{1}H} and DEPT ^{13}C NMR spectroscopy. The ^{1}H NMR spectroscopy reveals an AB resonance pattern for the diastereotopic ethylene proton atoms (CH_AH_B), a result that is consistent with a D_2-symmetric molecule and notably different from that of the C_{2v}-symmetric zirconium complex **1**. High-resolution mass spectroscopy unambiguously shows the expected signal for the molecular ion of dimeric complex **2**.

Analogously, the reaction of H_2L with $ZnEt_2$ produced dinuclear zinc complex Zn_2L_2 (**3**) as an off-white solid. Analytically pure compound **3** can be conveniently isolated by precipitation of a concentrated pentane solution at −35 °C followed by filtration. Unlike **1** and **2**, the zinc compound **3** is not sensitive to air and moisture. It is stable in protic solvents such as methanol. Attempts to grow X-ray quality crystals of **3**, however, were not successful. Nevertheless, the spectroscopic data of **3** are all suggestive of a double-helical structure similar to that observed for **2**. For instance, high-resolution mass spectroscopy confirms the dimeric feature of **3**. The ^{1}H NMR spectrum of **3** exhibits singlet signals for both imino protons and *tert*-butyl groups and an AB resonance pattern for the diastereotopic methylene protons of the ligand backbone. Similar result was also reported for dimeric $Zn_2L'_2$ on the basis of mass spectroscopy [17].

5. Conclusion

We have demonstrated the versatility of an acyclic, pyrrole-derived tetraaza ligand for binding to both transition and main-group metals. With selected metallic elements, L^{2-} can be a tetradentate ligand for a mononuclear complex or a bis(bidentate) ligand that bridges between two metal atoms. The formation of a double-helical complex of magnesium is of particular interest. The spectroscopic data suggest that the helical structure maintain in solution on the NMR time scale. Further studies directed to the reactivity chemistry of these compounds and the synthesis of new double-helical molecules of pyrrolylaldiminate derivatives are currently underway.

6. Supplementary information

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 222727 for compound **2**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1 EZ UK (Fax: +44-1223-336-033 or e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

Acknowledgements

We thank the National Science Council (NSC 89-2113-M-110-038) of Taiwan for financial support of this research.

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