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To cite this article: Yasunori Yamada, Takeshi Imari & Daishin Koori (2015) Syntheses, crystal structures, and some spectroscopic properties of zinc(II) complexes with N₂O₂ ligands derived from m-phenylenediamine and m-aminobenzylamine, Journal of Coordination Chemistry, 68:8, 1433-1440, DOI: [10.1080/00958972.2015.1018196](https://doi.org/10.1080/00958972.2015.1018196)

To link to this article: <http://dx.doi.org/10.1080/00958972.2015.1018196>



Accepted author version posted online: 12 Feb 2015.
Published online: 09 Mar 2015.



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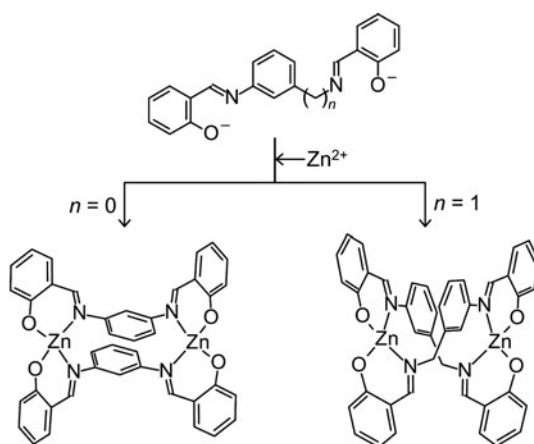
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Syntheses, crystal structures, and some spectroscopic properties of zinc(II) complexes with N_2O_2 ligands derived from *m*-phenylenediamine and *m*-aminobenzylamine

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(Received 21 November 2014; accepted 29 January 2015)



The reaction of bis(salicylidene)-*m*-phenylenediamine with zinc(II) ion affords a 2 : 2 dinuclear zinc(II) complex formulated as $[Zn_2(L^1)_2]$. A similar 2 : 2 dinuclear zinc(II) complex, $[Zn_2(L^2)_2]$, can be obtained by reaction of bis(salicylidene)-*m*-aminobenzylamine with zinc(II) ion. These two dinuclear complexes slightly differ in their crystal structures, especially coordination environments around the zinc(II) centers, depending on the dissimilar flexibilities of the two ligands. The differences between the two complexes are reflected in their diffuse reflectance and photoluminescence behaviors.

Keywords: Dinuclear zinc(II) complexes; N_2O_2 Ligands; Crystal structures; Emission

1. Introduction

Aliphatic and aromatic diamine compounds, such as ethylenediamine and phenylenediamine, are useful precursors for syntheses of multidentate ligands chelating various metal

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centers [1–19]. The N_2O_2 -type ligand, which is obtained by condensation of phenylenediamine with salicylaldehyde, can coordinate either one metal center or several metal centers depending on the positional relation between two amino groups in precursor [1–12, 20–24]. For instance, the N_2O_2 ligand derived from *o*-phenylenediamine is a tetradentate chelating agent that forms a mononuclear complex [1–13]. The ligand obtained by the reaction of *p*-phenylenediamine functions as a bis-bidentate ligand providing a dinuclear unit [20, 21]. Similar to the ligand derived from *p*-phenylenediamine, the one from *m*-phenylenediamine cannot ligate a single metal center but two metal centers [22–24]. In the absence of co-ligands, however, the *m*-phenylenediamine-derived ligands tend to provide finite polynuclear structures while the *p*-phenylenediamine-derived ligands are likely to form infinite or rather dissimilar polynuclear structures. For the *m*-phenylenediamine-derived ligands, a dinuclear Co(II) complex has been reported [22]. In such a phenylenediamine-derived ligand with lower flexibility around two nitrogens, as the above facts indicate, directionality of the two atoms is an important factor governing the structure of the resulting complex. In the case of the analogous ligand derived from *m*- or *p*-aminobenzylamine, additional methylene group in the benzylamine causes an increase of flexibility as a ligand. As a result, *p*-aminobenzylamine-derived ligands lead to formations of finite polynuclear structures, besides *m*-aminobenzylamine-derived ligands [25–27]. Although several types of metal complexes with *p*-aminobenzylamine-derived ligands have been reported for their crystal structures and some properties, investigations on *m*-aminobenzylamine-derived ligands are more limited than for *m*-phenylenediamine-derived ligands. Among complexes with such ligands, Zn(II) complexes attract attention to their spectroscopic properties as well as characteristic structures [25, 28, 29]. In this article, we describe syntheses, crystal structures, and some spectroscopic properties of Zn(II) complexes with the N_2O_2 ligands derived from *m*-phenylenediamine and *m*-aminobenzylamine.

2. Experimental

2.1. Materials and measurements

All purchased chemicals were of reagent grade and used without purification. The ligands, bis(salicylidene)-*m*-phenylenediamine (H_2L^1) and bis(salicylidene)-*m*-aminobenzylamine (H_2L^2), were prepared using modified literature methods [22–24, 26]. Elemental analyses (C, H, N) were performed with a Perkin-Elmer 2400 CHN Elemental Analyzer. IR spectra were recorded on a Bruker VERTEX 70 FT/IR spectrometer. Diffuse reflectance spectra were measured with a Perkin-Elmer Lambda 900 spectrophotometer equipped with an integrating sphere apparatus. Solid-state photoluminescence spectra were recorded with the OceanOptics Maya2000ProHC spectrometer equipped with the LLS-365 light source.

2.2. Synthesis of $[Zn_2(L^1)_2]$

Triethylamine (0.020 g, 0.2 mM) was added to a solution containing H_2L^1 (0.032 g, 0.1 mM) and $Zn(ClO_4)_2 \cdot 6H_2O$ (0.037 g, 0.1 mM) in 1 : 1 mixed solvent of ethanol and dichloromethane (100 cm³). After the mixture was allowed to stand at 25 °C for several days, the resulting yellow crystals were collected by filtration. Yield = 0.063 g (83% based on Zn). Anal. Calcd for $C_{40}H_{28}N_4O_4Zn_2$ ($[Zn_2(L^1)_2]$) (%): C, 63.26; H, 3.72; N, 7.38.

Found (%): C, 63.28; H, 3.76; N, 7.29. Characteristic IR data [cm^{-1}]: 1604^s (strong), 2600^{br} (broad). Diffuse reflectance spectrum [λ_{max} , nm]: 235.0, 270.0, 300.0, 372.5. Photoluminescence spectrum [λ_{max} , nm]: 520.0. Caution! Perchlorate salts are potentially dangerous explosives. Care must be exercised in handling these perchlorate salts and only small quantities should be used to avoid any untoward incident.

2.3. Synthesis of $[\text{Zn}_2(\text{L}^2)_2]$

This complex was prepared by a similar method to that for $[\text{Zn}_2(\text{L}^1)_2]$, using H_2L^2 instead of H_2L^1 . Yield = 0.059 g (75% based on Zn). Anal. Calcd for $\text{C}_{42}\text{H}_{32}\text{N}_4\text{O}_4\text{Zn}_2$ ($[\text{Zn}_2(\text{L}^2)_2]$) (%): C, 64.09; H, 4.10; N, 7.12. Found (%): C, 64.10; H, 4.14; N, 7.01. Characteristic IR data [cm^{-1}]: 1612^s, 1624^s, 2600^{br}, 2908^w. Diffuse reflectance spectrum [λ_{max} , nm]: 238.0, 269.5, 326.0, 385.5. Photoluminescence spectrum [λ_{max} , nm]: 509.0.

2.4. Crystallographic data collection and structure determination

Single crystals of $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$ and $[\text{Zn}_2(\text{L}^2)_2]$ were hand-picked directly out of the reaction mixture in a 1 : 1 mixed solvent of ethanol and dichloromethane, and used for data collection on a Rigaku VariMax Saturn CCD 724 system with graphite-monochromated $\text{MoK}\alpha$ radiation ($\lambda = 0.71075 \text{ \AA}$). The crystal data and experimental parameters are summarized in table 1. The structures were solved by direct methods and expanded using Fourier techniques [30]. The non-hydrogen atoms were refined anisotropically. All hydrogens, except those of one of the dichloromethane molecules in $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$ which were not included in any of the structural models because their positions could not be determined precisely, were placed in calculated positions and refined with a riding model. The final cycle of full-matrix least-squares refinement on F^2 was based on observed reflections and variable parameters, and converged with unweighted and weighted agreement factors of

Table 1. Crystallographic data for $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$ and $[\text{Zn}_2(\text{L}^2)_2]$.

	$[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$	$[\text{Zn}_2(\text{L}^2)_2]$
Formula	$\text{C}_{43}\text{H}_{34}\text{Cl}_6\text{N}_4\text{O}_4\text{Zn}_2$	$\text{C}_{42}\text{H}_{32}\text{N}_4\text{O}_4\text{Zn}_2$
Formula weight	1014.24	787.50
Crystal size (mm)	$0.31 \times 0.21 \times 0.13$	$0.31 \times 0.23 \times 0.19$
Space group	$P\bar{1}$	$C2/c$
a (Å)	10.040(3)	15.387(4)
b (Å)	10.123(3)	16.895(4)
c (Å)	10.870(4)	14.723(4)
α (°)	87.213(13)	90
β (°)	85.923(15)	117.918(4)
γ (°)	71.963(9)	90
V (Å ³)	1047.4(5)	3382.1(16)
Z	1	4
D_c (g cm ⁻³)	1.608	1.546
μ (cm ⁻¹)	15.762	14.707
Trans. factors	0.646–0.815	0.669–0.756
Tot. reflections	4710	3812
No. of variables	292	235
R_1 [$I > 2\sigma(I)$]	0.0477	0.0307
R (all data)	0.0526	0.0348
wR_2 (all data)	0.1330	0.0776
Goodness-of fit on F^2	1.136	1.063

Table 2. Selected bond distances (Å) and angles (°) for $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$ and $[\text{Zn}_2(\text{L}^2)_2]$.

	$[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$	$[\text{Zn}_2(\text{L}^2)_2]$
Zn(1)–O(1)	1.910(2)	1.9141(13)
Zn(1)–O(2)* ^a	1.914(2)	1.9312(18)
Zn(1)–N(1)	2.025(2)	2.0163(17)
Zn(1)–N(2)*	2.015(3)	2.012(2)
O(1)–Zn(1)–O(2)*	110.09(9)	117.53(7)
O(1)–Zn(1)–N(1)	94.87(10)	98.41(6)
O(1)–Zn(1)–N(2)*	132.35(8)	112.23(7)
O(2)*–Zn(1)–N(1)	122.18(8)	112.93(8)
O(2)*–Zn(1)–N(2)*	96.05(10)	95.55(8)
N(1)–Zn(1)–N(2)*	104.08(10)	121.62(7)

^aSymmetry transformations used to generate equivalent atoms: asterisk for $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2 = -x, -y, -z$; asterisk for $[\text{Zn}_2(\text{L}^2)_2] = -x + 2, y, -z + 1/2$.

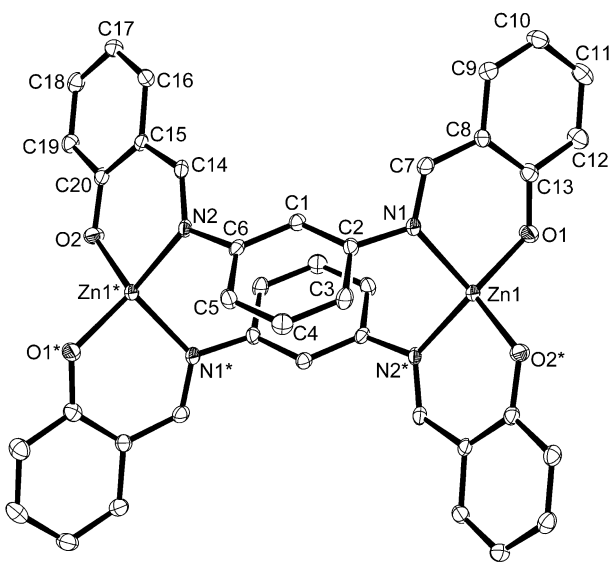


Figure 1. Perspective view of $[\text{Zn}_2(\text{L}^1)_2]$ with the atom labeling scheme (50% probability ellipsoids). Symmetry codes for the atoms with asterisks: $-x, -y, -z$.

R and R_w . All calculations were performed using the CrystalStructure crystallographic software package except for refinement, which was performed using SHELXL-97 [31, 32]. Bond distances and angles are presented in table 2.

3. Results and discussion

3.1. Crystal structures

An X-ray structural analysis for $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$ revealed the presence of a dinuclear Zn(II) complex and two CH_2Cl_2 molecules of crystallization. As shown in figure 1, each

Zn(II) center in $[\text{Zn}_2(\text{L}^1)_2]$ is coordinated with two NO-chelating sites from two different L^1 ligands. The dihedral angle between the two ZnNO planes in each Zn(II) center is 73.89° , indicating a distorted tetrahedral geometry. Such structural characteristic for $[\text{Zn}_2(\text{L}^1)_2]$ is similar to that for $[\text{Co}_2(\text{L}^1)_2]$ [22]. Therefore, it can be regarded that the distortion of the complex can be attributable to less flexibility around the azomethine groups in the ligand. In fact, the two salicylideneaminophenolate planes are not coplanar with the central phenyl plane, forming a dinuclear structure with considerable distorted dihedral angles of 45.57° and 60.30° . In $[\text{Zn}_2(\text{L}^1)_2]$, the interplane distance and angle between the two central phenyl groups are 3.3153 \AA and 0.000° , respectively. This indicates an intramolecular π - π stacking interaction in $[\text{Zn}_2(\text{L}^1)_2]$. Although, two absolute configurations, Δ and Λ , are possible for each Zn(II) center, the configurations around Zn1 and Zn1* are stereoselectively unified to Λ and Δ , respectively. This implies that $[\text{Zn}_2(\text{L}^1)_2]$ adopts a dinuclear mesocate structure.

$[\text{Zn}_2(\text{L}^2)_2]$ contains only a dinuclear Zn(II) complex without any molecule of crystallization in contrast to $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$. In $[\text{Zn}_2(\text{L}^2)_2]$, each Zn(II) center is surrounded by two distinct NO-chelating sites from two L^2 ligands (figure 2). The Zn–Zn distance in $[\text{Zn}_2(\text{L}^2)_2]$ is 6.111 \AA , while that in $[\text{Zn}_2(\text{L}^1)_2]$ is 7.357 \AA . This implies that the structure of $[\text{Zn}_2(\text{L}^2)_2]$ is more compact than that of $[\text{Zn}_2(\text{L}^1)_2]$. The dihedral angle between two ZnNO planes in each Zn(II) center is 84.47° , which is clearly different from the corresponding angle in $[\text{Zn}_2(\text{L}^1)_2]$ and rather closer to 90° . This implies that the tetrahedral geometry of $[\text{Zn}_2(\text{L}^2)_2]$ is less distorted than that of $[\text{Zn}_2(\text{L}^1)_2]$. The less distortion in $[\text{Zn}_2(\text{L}^2)_2]$ is due to the introduction of flexible methylene group into the ligand. Actually, the salicylideneaminophenolate connected to the central phenyl group retains a coplanarity with each other (dihedral angle: 3.93°), while that with an intervening methylene group is almost perpendicular to the central phenyl plane (dihedral angle: 89.73°). Similarly to the case of $[\text{Zn}_2(\text{L}^1)_2]$, the interplane distance and angle between the two central planes composed of the N1, C1, C2, C3, C4, C5, C6, and C7 are 3.3342 \AA and 11.524° , respectively. This suggests that stabilization of the dinuclear structure of $[\text{Zn}_2(\text{L}^2)_2]$ is due to intramolecular π - π stacking

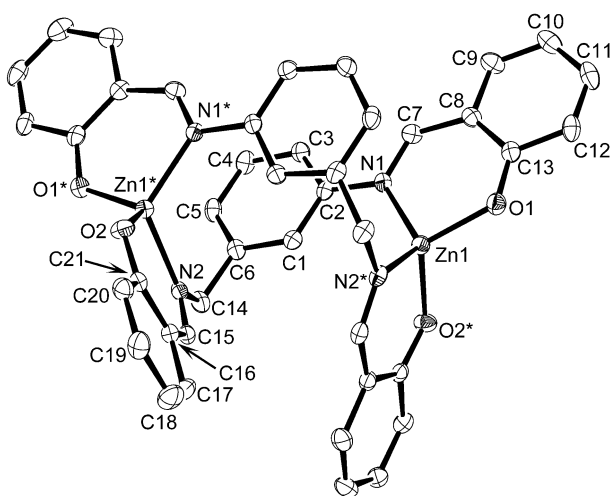


Figure 2. Perspective view of $[\text{Zn}_2(\text{L}^2)_2]$ with the atom labeling scheme (50% probability ellipsoids). Symmetry codes for the atoms with asterisks: $-x + 2, -y, -z + 1/2$.

interaction. It is noteworthy that the two ligands in $[\text{Zn}_2(\text{L}^2)_2]$ intersect each other along the intramolecular Zn–Zn axis to afford a double-stranded helical structure. This also reflects the introduction of flexible methylene group into L^2 .

3.2. Characterization

The complexes, $[\text{Zn}_2(\text{L}^1)_2]$ and $[\text{Zn}_2(\text{L}^2)_2]$, were readily prepared by the reactions of $\text{Zn}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ with H_2L^1 and H_2L^2 , respectively. These complexes crystallize as a yellow crystals with or without CH_2Cl_2 molecules as $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$ or $[\text{Zn}_2(\text{L}^2)_2]$. The CH_2Cl_2 molecules of crystallization in $[\text{Zn}_2(\text{L}^1)_2] \cdot 2\text{CH}_2\text{Cl}_2$ are easily lost when the crystal is exposed to air for a long time. Although these complexes are sparingly soluble in common organic solvents, partial decompositions occur immediately.

The *m*-phenylenediamine-derived ligand H_2L^1 has two equivalent azomethine groups, while the *m*-aminobenzylamine-derived H_2L^2 has two distinguishable azomethine groups. These differences are retained in their complexes, $[\text{Zn}_2(\text{L}^1)_2]$ and $[\text{Zn}_2(\text{L}^2)_2]$, reflecting their infrared spectral behaviors. In the infrared spectrum, $[\text{Zn}_2(\text{L}^1)_2]$ (H_2L^1) shows only a single band due to the $-\text{Ph}-\text{N}=\text{CH}-$ vibration at 1604 cm^{-1} (1618 cm^{-1}). On the other hand, $[\text{Zn}_2(\text{L}^2)_2]$ (H_2L^2) exhibits a similar band at 1612 cm^{-1} (1618 cm^{-1}) accompanied by the additional band at 1624 cm^{-1} (1632 cm^{-1}) assigned as the $-\text{CH}_2-\text{N}=\text{CH}-$ vibration besides the $-\text{Ph}-\text{N}=\text{CH}-$ vibration. It should be noted that these bands for the complexes appear in the lower wavenumber sides compared with the corresponding bands for the ligands. These shifts indicate that the ligands coordinate with zinc(II) centers through the imine nitrogens. Although H_2L^1 and H_2L^2 exhibit broad bands around 2600 cm^{-1} due to the intramolecular hydrogen bonding, the corresponding bands cannot be seen in the spectra of $[\text{Zn}_2(\text{L}^1)_2]$ and $[\text{Zn}_2(\text{L}^2)_2]$ [26, 33]. In the region lower than 600 cm^{-1} , the spectra of $[\text{Zn}_2(\text{L}^1)_2]$ and $[\text{Zn}_2(\text{L}^2)_2]$ exhibit additional bands, not observed for H_2L^1 and H_2L^2 . These facts support the formations of the complexes. The *m*-aminobenzylamine-derived H_2L^2 and its complex show weak bands around 2900 cm^{-1} , while the *m*-phenylenediamine-derived ligand H_2L^1

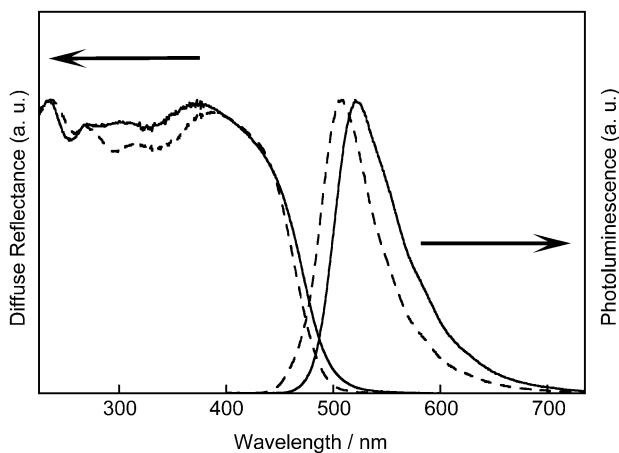


Figure 3. Diffuse reflectance and photoluminescence spectra of $[\text{Zn}_2(\text{L}^1)_2]$ (solid line) and $[\text{Zn}_2(\text{L}^2)_2]$ (broken line) in the crystalline state at $25 \text{ }^\circ\text{C}$.

and its complex have no corresponding bands. This implies that the bands around 2900 cm^{-1} of H_2L^2 and $[\text{Zn}_2(\text{L}^2)_2]$ are assigned as $-\text{CH}_2-$ vibrations.

As shown in figure 3, the diffuse reflectance spectrum of $[\text{Zn}_2(\text{L}^1)_2]$ is composed of four bands at 235.0, 270.0, 300.0, and 372.5 nm. In the spectrum of $[\text{Zn}_2(\text{L}^2)_2]$, the corresponding bands to $[\text{Zn}_2(\text{L}^1)_2]$ appear at 238.0, 269.5, 326.0, and 385.5 nm, respectively, among which the 326.0 and 385.5 nm bands are considerably shifted to longer wavelength in comparison with $[\text{Zn}_2(\text{L}^1)_2]$. This reflects the differences in electronic structures between the two complexes. The edge of the lowest energy band for $[\text{Zn}_2(\text{L}^1)_2]$ extends to a longer wavelength side than that for $[\text{Zn}_2(\text{L}^2)_2]$. This implies that the lowest excited state of $[\text{Zn}_2(\text{L}^1)_2]$ is lying on a lower energy level than that of $[\text{Zn}_2(\text{L}^2)_2]$. Solid-state photoluminescence spectrum of $[\text{Zn}_2(\text{L}^1)_2]$ exhibits a single band at 520.0 nm upon excitation at 365 nm (figure 3). The corresponding emission band for $[\text{Zn}_2(\text{L}^2)_2]$ shifts toward the shorter wavelength side and appears at 509.0 nm. Such a difference in the emission maxima between the two complexes is essentially consistent with the result of diffuse reflectance spectra.

4. Conclusion

The N_2O_2 ligands, H_2L^1 and H_2L^2 , can act as bis-bidentate ligands and react with zinc(II) ions, which prefer four-coordinate tetrahedral environment, to form dinuclear complexes formulated as $[\text{Zn}_2(\text{L}^1)_2]$ and $[\text{Zn}_2(\text{L}^2)_2]$, respectively. Both of the zinc(II) centers in $[\text{Zn}_2(\text{L}^1)_2]$ are considerably distorted from the ideal tetrahedron, while the distortions in $[\text{Zn}_2(\text{L}^2)_2]$ are less due the flexibilities around the azomethine groups in H_2L^2 . Although $[\text{Zn}_2(\text{L}^1)_2]$ adopts a dinuclear mesocate structure, $[\text{Zn}_2(\text{L}^2)_2]$ affords a double-stranded helical structure. These two complexes, $[\text{Zn}_2(\text{L}^1)_2]$ and $[\text{Zn}_2(\text{L}^2)_2]$, are yellowish-green light emitters on exposure to ultraviolet light. The present complexes, $[\text{Zn}_2(\text{L}^1)_2]$ and $[\text{Zn}_2(\text{L}^2)_2]$, are potentially applicable to the models for the active sites of the hydrolytic enzymes, where the zinc(II) centers are ligated by hard donors such as nitrogen and/or oxygen [34].

Supplementary materials

CCDC 1035184 for $[\text{Zn}_2(\text{L}^1)_2]\cdot 2\text{CH}_2\text{Cl}_2$ and 1035185 for $[\text{Zn}_2(\text{L}^2)_2]$ contain the supplementary crystallographic data for this article. These 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.cam.ac.uk).

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