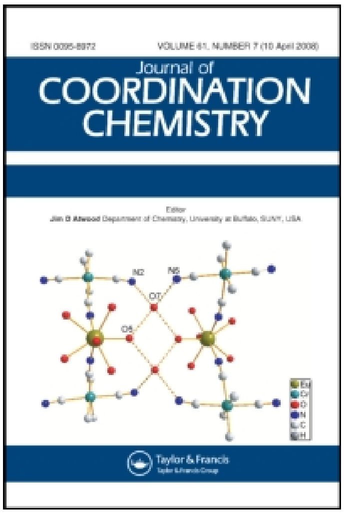

Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

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To cite this Article Chu, Zhaolian , You, Wei , Fan, Ying , Qian, Huifen and Huang, Wei(2009) 'Zinc(II) and cadmium(II) complexes having racemic and enantiomeric ligands: $[Zn(L_a)_2](NO_3)_2 \cdot CH_3CH_2OH$ and $[Cd(L_b)_2Cl](ClO_4)$ (L_a = D,L and L_b = D-(+)-1,2, 2-trimethylcyclopentane-1,3-diamine)', Journal of Coordination Chemistry, 62: 13, 2086 — 2094

To link to this Article: DOI: 10.1080/00958970902783584

URL: http://dx.doi.org/10.1080/00958970902783584

Journal of Coordination Chemistry
Vol. 62, No. 13, 10 July 2009, 2086–2094

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(*Received 31 March 2008; in final form 5 November 2008*)

Two d^{10} transition-metal complexes having racemic and enantiomeric 1,2,2-trimethylcyclopentane-1,3-diamine ligands, $[Zn(L_a)_2](NO_3)_2 \cdot CH_3CH_2OH$ (**1**) and $[Cd(L_b)_2Cl](ClO_4)$ (**2**) (L_a = D,L-1,2,2-trimethylcyclopentane-1,3-diamine, L_b = D-(+)-1,2,2-trimethylcyclopentane-1,3-diamine or (*1R,3S*)-1,2,2-trimethylcyclopentane-1,3-diamine), were synthesized and characterized by X-ray single-crystal diffraction. They crystallize in the *Pbca* and $P2_12_12_1$ space groups, respectively, and have different coordination numbers and coordination geometry (four-coordinate tetrahedron for Zn(II) in **1** and five-coordinate square-based pyramid for Cd(II) in **2**) mainly due to their different ionic radii.

Keywords: Zinc(II) and cadmium(II) complexes; Chiral and achiral ligands; Hydrogen bonding interactions; Supramolecular networks

1. Introduction

Enantiomeric and racemic camphoric acids have been widely used in organic syntheses and can serve as chiral bidentate ligands or building blocks in self-assembling supramolecular complexes with specific topologies and functions [1, 2]. Enantiomeric and racemic 1,2,2-trimethylcyclopentane-1,3-diamines, which can be prepared from their respective camphoric acids, are very useful in studies of asymmetric catalysis [3] and functional Schiff-base metal complexes when condensed with a variety of aldehydes [4].

In previous work, we synthesized a series of bifunctional compounds with camphoric backbones [5–8]. We also reported the crystal structures of two four-coordinate square planar nickel(II) complexes [9, 10] and two five-coordinate pyramidal copper(II)

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online
DOI: 10.1080/00958970902783584

complexes with the same chemical formula bearing D-(+)- and D,L-1,2,2-trimethylcyclopentane-1,3-diamine ligands, respectively [11]. In addition, we reported the sulfate salt of D-(+)-1,2,2-trimethylcyclopentane-1,3-diamine [12]. In this article, we report two d^{10} transition-metal complexes of enantiomeric and racemic 1,2,2-trimethylcyclopentane-1,3-diamines, *bis*(D,L-1,2,2-trimethylcyclopentane-1,3-diamine-κ^2N,N')-zinc(II) dinitrate ethanol solvate (**1**), and *bis*(D-(+)-1,2,2-trimethylcyclopentane-1,3-diamine-κ^2N,N')-cadmium(II) chloride perchlorate (**2**).

2. Experimental

2.1. *Materials and measurements*

All solvents and chemicals were of analytical grade and used without purification. D-(+)- and D,L-1,2,2-trimethylcyclopentane-1,3-diamine dihydrogenchlorates were prepared according to the literature method using D-(+)- and D,L-camphoric acids as starting materials [3]. Elemental analyses (EA) were measured with a Perkin–Elmer 1400C analyzer. Infrared (FT-IR) spectra (4000–400 cm^{-1}) were recorded on a Nicolet FT-IR 170X spectrophotometer.

Caution: Although no problem was encountered in all our experiments, transition metal perchlorates are potentially explosive and should be handled in small quantities.

2.2. *Preparation of $[Zn(L_a)_2](NO_3)_2\cdot CH_3CH_2OH$ (1)*

NaOH (0.160 g, 4.0 mmol) was dissolved in 20 cm^3 ethanol, and it was added to a solution of D,L-1,2,2-trimethylcyclopentane-1,3-diamine dihydrochloride (0.431 g, 2.0 mmol) dissolved in 20 cm^3 ethanol. After 15 min of stirring, a solution of $Zn(NO_3)_2 \cdot 6H_2O$ (0.297 g, 1.0 mmol) in 10 cm^3 ethanol was added. The mixture was refluxed for 1 h and cooled to room temperature. The mixture was evaporated to nearly 5 cm^3 under reduced pressure and the colorless precipitates were collected, washed with a small amount of ethanol, and dried *in vacuo* (yield: 0.364 g, 70%). EA calculated for $C_{18}H_{42}N_6O_7Zn$: C, 41.58; H, 8.14; N, 16.16%. Found: C, 41.55; H, 8.11; N, 16.12%. Main FT-IR absorptions (KBr, cm^{-1}): 3425(s), 3267(s), 3230(s), 1589(s), 1473(m), 1384(s), and 669(s). Single crystals of **1** suitable for X-ray diffraction were grown from a mixture of ethanol and acetonitrile (2 : 1 v/v) by slow evaporation in air at room temperature.

2.3. *Preparation of $[Cd(L_b)_2Cl](ClO_4)$ (2)*

NaOH (0.160 g, 4.0 mmol) was dissolved in 20 cm^3 ethanol and added to a solution of (*1R,3S*)-1,2,2-trimethylcyclopentane-1,3-diamine dihydrochloride (0.431 g, 2.0 mmol) dissolved in 20 cm^3 ethanol. After 15 min of stirring, a solution of $Cd(ClO_4)_2 \cdot 6H_2O$ (0.419 g, 1.0 mmol) in 10 cm^3 ethanol was added. The mixture was refluxed for 1 h, cooled to room temperature, evaporated to 5 cm^3 under reduced pressure, the colorless precipitate collected, washed with a small amount of ethanol, and dried *in vacuo* (yield: 0.473 g, 89%). EA calculated for $C_{16}H_{36}N_4O_4Cl_2Cd$: C, 36.14; H, 6.82; N, 10.54%.

Found: C, 36.23; H, 6.91; N, 10.47%. Main FT-IR absorptions (KBr, cm^{-1}): 3432(s), 3303(s), 3266(s), 2965(m), 2882(m), 1589(s), 1470(m), 1110(vs), 1063(vs), and 623(m). Single crystals of **2** suitable for X-ray diffraction were grown from a mixture of ethanol and water (3 : 1 v/v) by slow evaporation in air at room temperature.

2.4. *X-ray data collection and refinement*

Single crystals of **1** and **2** were mounted on glass fibers and measured on a Bruker SMART 1K CCD area detector at 291(2) K using graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). The collected data were reduced by using SAINT [13] and empirical absorption correction was done by using SADABS [14]. The crystal systems were determined by Laue symmetry and the space groups were assigned on the basis of systematic absences using XPREP. The structures were solved by direct methods and refined by least squares on F^2_{obs} using the SHELXTL-PC [15] software package.

All nonhydrogen atoms were refined on F^2 by full-matrix least-squares procedure using anisotropic displacement parameters. Hydrogens were inserted in calculated positions, assigned fixed isotropic thermal parameters at 1.2 times the equivalent isotropic U of the atoms to which they are attached (1.5 times for the methyl and amino groups), and allowed to ride on their respective parent atoms. The methyl group and the hydrogen bonded to the chiral carbons (C1 and C3) of one 1,2,2-trimethylcyclopentane-1,3-diamine ligand in **1** are refined disorderly with equal site occupancy factors. Each chiral carbon is bonded to half hydrogen atom and half methyl group, which is the same as for D,L-1,2,2-trimethylcyclopentane-1,3-diamine copper(II) complex [11]. For the chiral carbons (C9 and C11) of the other diamine, the methyl and hydrogen cannot be refined as disorder because the program gives site occupancy factors of 0.9926 : 0.0174 for C14 and C14′ atoms by using the free variable refinement method. All calculations and molecular graphics were carried out on a PC computer with the SHELXTL PC program package. The summary of the crystal data, experimental details, and refinement results for **1** and **2** are listed in table 1, while selected bond distances and angles are given in table 2.

3. Results and discussion

3.1. *Synthesis and spectral characterization*

Comparative studies were carried out by treatment of D-(+)- and D,L-1,2,2-trimethylcyclopentane-1,3-diamines and Zn(II) or Cd(II) salts with different counter-ions ($Zn(NO_3)_2 \cdot 6H_2O$, $Zn(ClO_4)_2 \cdot 6H_2O$, $Cd(NO_3)_2 \cdot 4H_2O$, and $Cd(ClO_4)_2 \cdot 6H_2O$), respectively. In the preparation of these eight d^{10} metal complexes, NaOH was used to neutralize the dihydrogenchlorides of D-(+)- and D,L-1,2,2-trimethylcyclopentane-1, 3-diamines; however, the chloride remained in the system and can coordinate with the central ion in some cases. They show very little difference in synthetic yields, FT-IR, and EA results, although single-crystal samples of the other six species suitable for X-ray diffraction are very difficult to obtain. For example, D-(+)- and D,L-1,2, 2-trimethylcyclopentane-1,3-diamine $Cd(NO_3)_2 \cdot 4H_2O$ complexes have identical FT-IR and analyses, while D-(+)- and D,L-1,2,2-trimethylcyclopentane-1,3-diamine

Table 1. Crystal data and structural refinements for **1** and **2**.

Compound	**1**	**2**
Formula	$ZnC_{18}H_{42}N_6O_7$	$CdC_{16}H_{36}N_4O_4Cl_2$
Formula weight	519.97	531.79
T (K)	291	291
Crystal size (mm^3)	$0.10 \times 0.12 \times 0.20$	$0.10 \times 0.12 \times 0.14$
Crystal system	Orthorhombic	Orthorhombic
Space group	*Pbca*	$P2_12_12_1$
Units of dimensions (Å, °)		
a	13.4490(9)	7.8449(4)
b	11.8264(8)	14.1098(7)
c	31.953(2)	20.0957(11)
V (Å^3)	5082.2(6)	2224.4(2)
Z	8	4
ρ_{Calcd} ($g\,cm^{-3}$)	1.359	1.588
$F(000)$	2224	1096
μ (mm^{-1})	1.013	1.250
Max/min h, k, l	−16/16, −14/10, −39/37	−10/10, −18/15, −26/24
Min/max transmission	0.8230/0.9054	0.8444/0.8852
Collected reflections	25,724	14,000
Unique reflections	4994	5337
Reflections with $I > 2\sigma(I)$	2715	4606
Parameters	304	250
R_{int}	0.050	0.041
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Flack parameter	–	−0.041(19)
R_1/wR_2 [$I > 2\sigma(I)$]	0.0590/0.1597	0.0276/0.0464
R_1/wR_2 (all data)	0.0950/0.1691	0.0357/0.0483
GOF on F^2	0.924	0.906
Max/min. ρ (e Å^{-3})	0.862/−0.570	0.306/−0.323

Note: $R_1 = \Sigma\|F_o| - |F_c\|/\Sigma|F_o|$, $wR_2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma w(F_o^2)^2]^{1/2}$.

Table 2. Selected bond distances (Å) and angles (°) for **1** and **2**.

		1	
Zn1–N1	2.002(3)	N1–Zn1–N2	99.88(15)
Zn1–N2	1.998(4)	N1–Zn1–N3	110.72(14)
Zn1–N3	2.018(3)	N1–Zn1–N4	116.06(14)
Zn1–N4	2.014(3)	N2–Zn1–N3	113.75(13)
N1–C1	1.497(6)	N2–Zn1–N4	117.31(13)
N2–C3	1.498(6)	N3–Zn1–N4	99.70(13)
N3–C9	1.492(5)		
N4–C11	1.502(5)		
		2	
Cd1–Cl1	2.495(1)	Cl1–Cd1–N1	99.29(6)
Cd1–N1	2.326(2)	Cl1–Cd1–N2	102.52(6)
Cd1–N2	2.315(2)	Cl1–Cd1–N3	106.28(6)
Cd1–N3	2.331(2)	Cl1–Cd1–N4	110.62(6)
Cd1–N4	2.303(2)	N1–Cd1–N2	84.57(8)
N1–C1	1.502(3)	N1–Cd1–N3	85.68(8)
N2–C3	1.482(4)	N1–Cd1–N4	149.99(8)
N3–C9	1.498(3)	N2–Cd1–N3	150.72(8)
N4–C11	1.483(4)	N2–Cd1–N4	91.21(8)
		N3–Cd1–N4	83.66(8)

$Cd(NO_3)_2 \cdot 4H_2O$ and $Cd(ClO_4)_2 \cdot 6H_2O$ complexes only exhibit different strong absorptions at 1110(vs) and 1063(vs) cm^{-1} for typical perchlorate anions and 1384(s) cm^{-1} for typical nitrate anions.

For **1**, the chloride is not present and Zn(II) is four-coordinate, while Cl^- coordinates with Cd(II) in **2**. The possible reason is that Cd(II) is larger than Zn(II), allowing for two bidentate diamine ligands and one chloride to form a five-coordinate configuration.

Compared with broad peaks from 2500 to 3500 cm^{-1} in the starting materials, two sharp peaks were observed in IR spectra of the complexes at 3267 and 3230 cm^{-1} (**1**) and 3303 and 3266 cm^{-1} (**2**), exhibiting complexation between the amino groups and zinc(II) and cadmium(II). In addition, very strong IR peaks at 1110 and 1063 cm^{-1} in **2** are typical for perchlorate. The asymmetric stretching mode of $\delta_{N\text{–}H}$ (both at 1589 cm^{-1}) as well as the typical alkyl absorptions are observed in **1** and **2**.

3.2. *Structural description of 1*

The atom-numbering scheme of **1** is shown in figure 1. The asymmetric unit of **1** consists of a four-coordinate zinc(II) with two independent nitrates and an uncoordinated ethanol. It crystallizes in the orthorhombic space group *Pbca* with zinc(II) in a distorted tetrahedron from two diamine ligands. The Zn–N bond lengths are in the range of 1.997(3)–2.017(3) Å. The dihedral angle between the least-squares plane defined by N1, N2, and Zn1 and the plane defined by N3, N4, and Zn1 is 89.2(1)°. The zinc(II) is coordinated to two 1,2,2-trimethylcyclopentane-1,3-diamine ligands; one refined as disordered where the methyl and hydrogen bonded to the chiral carbons (C1 and C3) are positioned at two places with equal site occupancy, while the other (*1R,3S*)-1,2,2-trimethylcyclopentane-1,3-diamine cannot be refined as disordered because the SHELXTL program gives only 0.0174 site occupancy factor for the

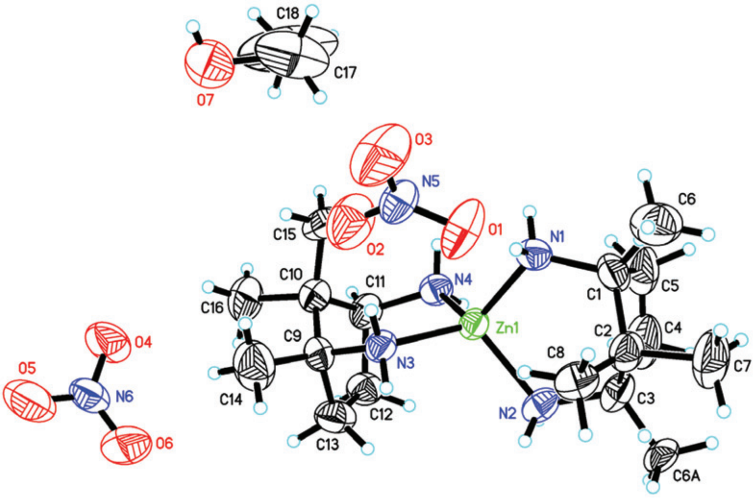

Figure 1. An ORTEP of **1** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

methyl group bonded to C14 by using free variable refinement method. Using racemic ligand, this kind of disorder for the diamine ligand is not uncommon and the same disorder has been observed in D,L-1,2,2-trimethylcyclopentane-1,3-diamine copper(II) [11].

In the crystal structure of **1**, all the N atoms of diamines contribute to the formation of intermolecular N–H $\cdots$ O hydrogen bonds (table 2). A 1-D chain along the crystallographic *a*-axis is formed from multiple N–H $\cdots$ O hydrogen bonds between nitrogens of diamines and oxygens of nitrates (figure 2). Ethanol oxygen O7 as both donor and acceptor forms intermolecular O–H $\cdots$ O and N–H $\cdots$ O hydrogen bonds with the nitrate oxygen O5 and the amine nitrogen N4, respectively. Two weak C–H $\cdots$ N hydrogen-bond contacts are present between amine nitrogens N1, N3, and methyl carbons C8, C15. Thus, a 3-D network structure is constituted with hydrogen bonds.

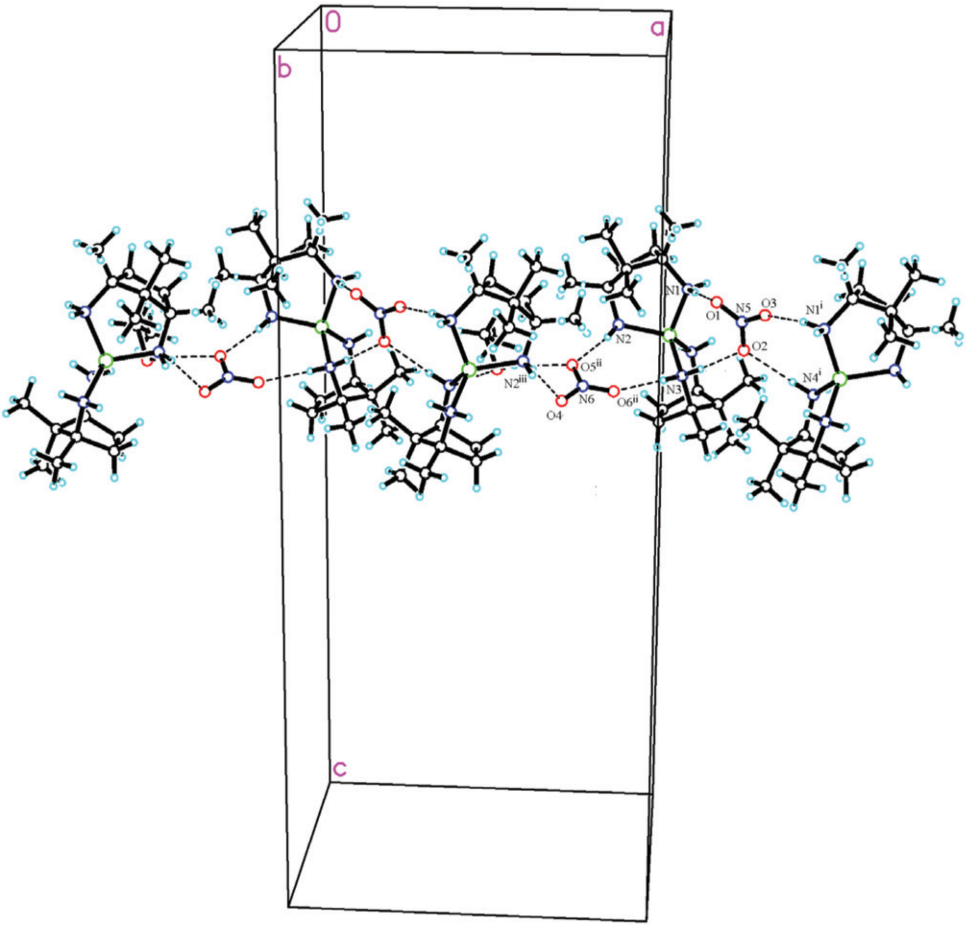

Figure 2. View of the intermolecular N–H $\cdots$ O hydrogen bonding interactions together with the unit cell. The hydrogen bonds are shown as dashed lines (Symmetry codes: (i) $-x+1/2$, $y+1/2$, z; (ii) $-x$, $-y+1$, $-z+1$; (iii) $x-1/2$, $-y+3/2$, $-z+1$).

3.3. *Structural description of 2 and comparative study of related complexes*

The atom-numbering scheme of **2** is shown in figure 3. The asymmetric unit consists of a mononuclear five-coordinate Cd(II) with coordinated chloride and uncoordinated perchlorate. The Cd(II) is square pyramidal, where four nitrogen atoms from two D-(+)-1,2,2-trimethylcyclopentane-1,3-diamine ligands constitute the basal plane with the Cd–N bond lengths in the range of 2.303(2)–2.331(2) Å. The apical position is occupied by chloride with Cd–Cl bond length at 2.495(1) Å. These bond lengths are longer than those in similar Cu(II) complexes $[Cu(L_a)_2Cl]BF_4$ and $[Cu(L_b)_2Cl]BF_4$ (2.010–2.025 Å for the basal plane Cu–N bonds and 2.655(1)–2.665(1) Å for the apical Cu–Cl bonds) due to the larger radius of Cd(II). The Cd(II) is 0.590 Å above its tetra-N least-squares basal plane.

Metal complexes having enantiomeric or racemic chiral ligands crystallize in different space groups with different crystal symmetry. A low Flack parameter of 0.041(19) in the case of **2** exhibits the correct assignment of the $P2_12_12_1$ space group [16] with the chiral ligand. Both five-membered carbon rings of bidentate D-(+)-1,2,2-trimethylcyclopentane-1,3-diamine ligands adopt the *trans* configuration relative to the coordination plane, similar to those in $[Ni(L_b)_2](NO_3)_2$, $[Cu(L_a)_2Cl]BF_4$, and $[Cu(L_b)_2Cl]BF_4$ but different from $[Ni(L_a)_2]Cl_2 \cdot 2H_2O$ where the *cis* configuration is observed, although **2** and $[Ni(L_a)_2]Cl_2 \cdot 2H_2O$ crystallize in the same $P2_12_12_1$ space group.

Intermolecular N–H···O hydrogen bonds are observed between the two groups of oxygens of perchlorate (O1, O2 and O1, O4) and the two adjacent hydrogens of amino groups from different chiral diamine ligands, forming two neighboring eight-membered $N_2H_4ClO_2Cd$ hydrogen-bond rings (figure 4). An interesting consequence of the large number of N–H···O hydrogen bonds is that the lattice perchlorate is not disordered. In addition, N–H···Cl and C–H···O hydrogen bonding interactions are found between contiguous molecules (table 3) to form a 3-D network.

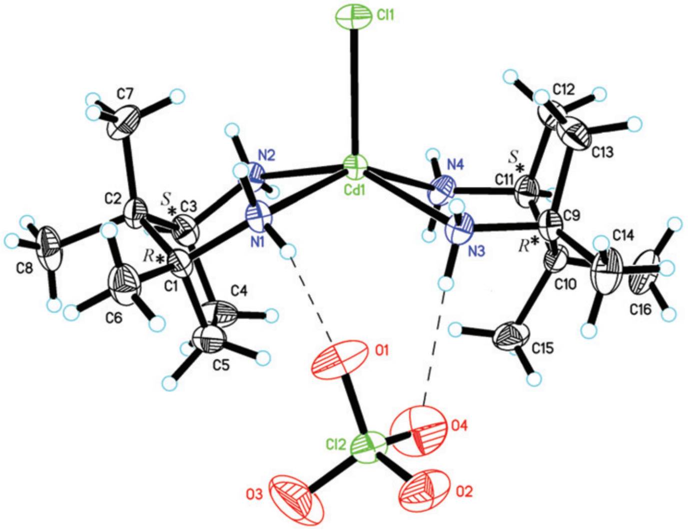

Figure 3. An ORTEP of **2** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and the eight-membered hydrogen bonding ring is also shown.

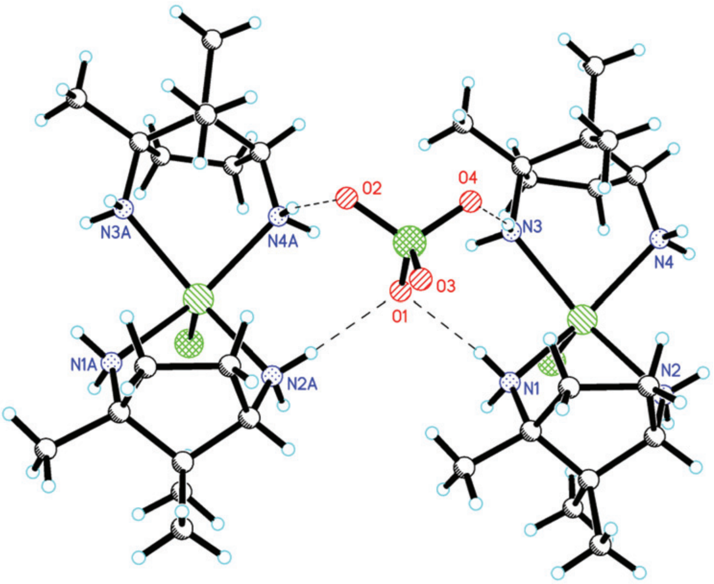

Figure 4. View of the intermolecular N–H···O hydrogen bonding interactions. The hydrogen bonds are indicated as dashed lines (Symmetry code: $1 + x$, y, z).

Table 3. Hydrogen bonding interactions (Å, °) in **1** and **2**.

			1		
N1–H1A···O3	0.90	2.05	2.922(6)	162	$1/2 - x$, $1/2 + y$, z
N2–H2A···O5	0.90	2.08	2.960(6)	167	$-x$, $1 - y$, $1 - z$
N2–H2B···O4	0.90	2.14	3.009(5)	164	$-1/2 + x$, $3/2 - y$, $1 - z$
N3–H3A···O6	0.90	2.09	2.972(6)	167	$-x$, $1 - y$, $1 - z$
N4–H4D···O7	0.90	1.99	2.885(6)	175	$1/2 - x$, $1/2 + y$, z
N4–H4C···O2	0.90	2.29	3.171(5)	165	$1/2 - x$, $1/2 + y$, z
O7–H7···O5	0.82	2.19	2.867(7)	140	$1 - x$, $1 - y$, $1 - z$
N1–H1B···O1	0.90	2.07	2.963(6)	174	
N3–H3B···O2	0.90	2.18	3.080(5)	177	
			2		
N1–H1A···O1	0.90	2.25	3.141(3)	169	
N2–H2B···O1	0.90	2.18	3.076(3)	174	$1 + x$, y, z
N3–H3A···Cl1	0.90	2.60	3.461(3)	159	$-1/2 + x$, $3/2 - y$, $2 - z$
N3–H3B···O4	0.90	2.41	3.291(3)	167	
N4–H4C···O2	0.90	2.33	3.219(3)	171	$1 + x$, y, z
N4–H4D···Cl1	0.90	2.60	3.432(3)	154	$1/2 + x$, $3/2 - y$, $2 - z$
C8–H8A···O4	0.96	2.50	3.424(5)	162	$2 - x$, $1/2 + y$, $3/2 - z$

4. Conclusion

Chirality effects for forming two d^{10} transition metals **1** and **2** by using enantiopure and racemic ligands were carried out in this article. Due to the different radii of Zn(II) and Cd(II), different coordination numbers and geometries were obtained. Compound **1** crystallizes in the *Pbca* space group in a four-coordinate tetrahedral geometry. Compound **2** crystallizes in the $P2_12_12_1$ space group in a five-coordinate square pyramid having D-(+)-1,2,2-trimethylcyclopentane-1,3-diamine ligands.

Versatile hydrogen bonding interactions are observed in the crystal packing structures of **1** and **2** forming 3-D supramolecular networks.

Supplementary material

CCDC reference numbers 680254 & 680255 for **1** and **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 Centre, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44-1223/336-033; Email: deposit@ccdc.cam.ac.uk].

Acknowledgments

W. Huang would like to acknowledge the Major State Basic Research Development Programs (No. 2007CB925101 and No. 2006CB806104), the National Natural Science Foundation of China (No. 20871065 and No. 20301009), and the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry for financial aid.

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