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### SYNTHESIS AND X-RAY CRYSTAL STRUCTURE OF A NEW $\mu_2$ -PHENOXY OXYGEN BRIDGED BINUCLEAR CADMIUM(II) CRYPTATE

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## SYNTHESIS AND X-RAY CRYSTAL STRUCTURE OF A NEW $\mu_2$ -PHENOXY OXYGEN BRIDGED BINUCLEAR CADMIUM(II) CRYPTATE

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A new *tris*(phenoxy oxygen) bridged bicadmium(II) polyaza cryptate has been synthesized by the [2 + 3] Schiff-base condensation of *tris*(3-aminopropyl)amine (trpn) with sodium 2,6-diformyl-4-chlorophenolate (sdcp) in the presence of Cd(ClO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O. The crystal structure of the cryptate [Cd<sub>2</sub>L](ClO<sub>4</sub>)·CH<sub>3</sub>OH has been determined by X-ray methods. Crystals are monoclinic, space group *P*2<sub>1</sub>/*n* with *a* = 16.076(4), *b* = 15.900(3), *c* = 18.590(4) Å,  $\beta$  = 95.100(14)°, *Z* = 4, *V* = 4719(4) Å<sup>3</sup>, *F*(000) = 2360, *D*<sub>c</sub> = 1.649 g cm<sup>-3</sup>,  $\mu$  = 11.9 cm<sup>-1</sup>, *R* = 0.0581, *R*<sub>w</sub> = 0.0682. Each cadmium(II) atom is bonded to three  $\mu_2$ -phenoxy oxygen atoms, three amine atoms and one bridgehead nitrogen atom. The coordination geometry around the Cd(II) is considered as being a capped trigonal prism.

**Keywords:** cadmium(II); binuclear cryptate; polyaza cryptand; X-ray structure

### INTRODUCTION

Polyaza cryptands have strong coordination ability and recognition functions for transition metal ions. 1-5 A new kind of macrocyclic ligand has been obtained by condensation of 2,6-diformyl-4-methylphenol with various diamines. 6 They have strong donors, namely, phenoxy oxygen atoms, as well as amine nitrogen atoms; therefore they play excellent roles in catalysis and biological simulation 7, 8 for their special coordination ability with transition metal ions. Recently Gagne,<sup>9</sup> Nelson<sup>10</sup> and others<sup>11</sup> synthesized several transition metal cryptates

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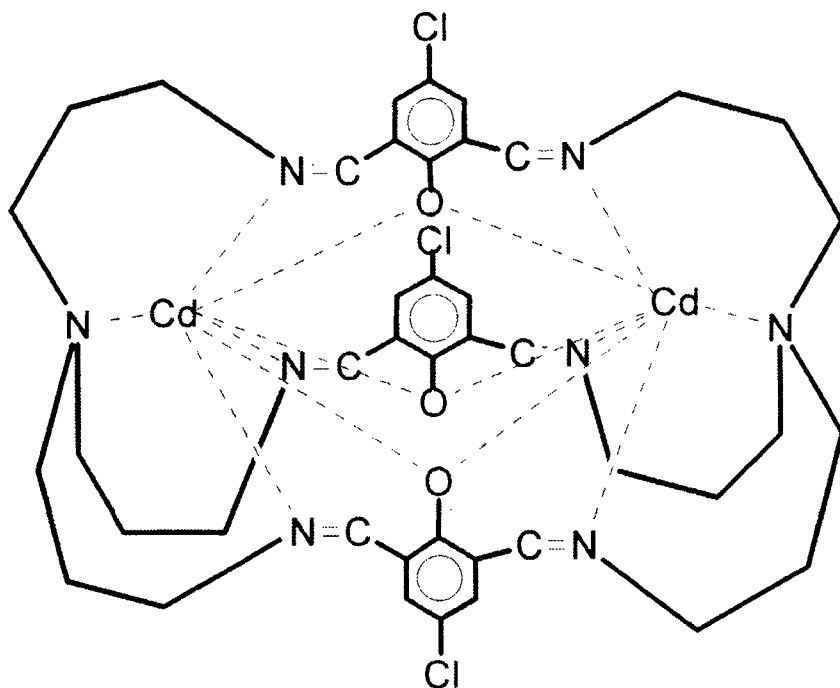


FIGURE 1 The binuclear cryptate cation  $[Cd_2L]^+$ .

by a [2 + 3] Schiff base condensation of *tris*(2-aminoethyl)amine (tren) with 2,6-diformyl-4-methylphenol (dmp) in the presence of transition metal ions. Few cryptates synthesized by condensation of *tris*(3-aminopropyl)amine (trpn) with dmp or its derivatives have been reported. It can be expected that these cryptates would be more versatile in host-guest properties for having a flexible, long chain in trpn. For example, we have obtained an interesting pentanuclear silver(I) cluster 12 by condensation of trpn with dmp in the presence of silver(I). In this paper, we report a new  $\mu$ -oxo bridged binuclear cadmium(II) cryptate (Figure 1) synthesized by a [2 + 3] Schiff base condensation of trpn with sodium 2,6-diformyl-4-chlorophenolate in the presence of  $Cd^{2+}$ . To our knowledge, only few of these kinds of structures have hitherto been reported.

## EXPERIMENTAL

All starting materials were of analytical purity. 2,6-Diformyl-4-chlorophenol (dcp) was prepared by a literature method. 13 Sodium 2,6-diformyl-4-chlor-

ophenolate was obtained by mixing equimolar amounts of 2,6-diformyl-4-chlorophenol and NaOH in absolute ethanol. *Tris*(3-aminopropyl)amine was prepared by a modified literature method. 14 Solution electrical conductivity was measured with a BDS-A conductometer (Jiangsu, China) with solution concentration of  $\sim 10^{-4}$  mol dm $^{-3}$  in acetonitrile. IR spectra were measured in KBr discs using a Nicolet 5DX FT-IR spectrophotometer. Electronic spectra were recorded on a Shimadzu UV-240 spectrophotometer.

#### *Preparation of [Cd<sub>2</sub>L](ClO<sub>4</sub>)·CH<sub>3</sub>OH*

To a stirred solution of sodium 2,6-diformyl-4-chlorophenolate (0.1549 g, 0.75 mmol) and Cd(ClO<sub>4</sub>)·6H<sub>2</sub>O (0.2621 g, 0.625 mmol) in 30 cm<sup>3</sup> of absolute methanol was added dropwise a solution of *tris*(3-aminopropyl)amine (0.094 g, 0.5 mmol) in 15 cm<sup>3</sup> of absolute methanol at 0°C. After stirring the solution for 2 h at 35°C, yellowish microcrystals of [Cd<sub>2</sub>L](ClO<sub>4</sub>)·CH<sub>3</sub>OH were filtered off, washed with methanol and dried in vacuum, with a yield of 70%. *Anal.* Calcd. for C<sub>43</sub>H<sub>52</sub>N<sub>8</sub>O<sub>8</sub>Cl<sub>4</sub>Cd<sub>2</sub> (%): C, 43.88; H, 4.46; N, 9.52; Cd, 19.20. Found: C, 44.03; H, 4.68; N, 9.83; Cd, 19.21. IR (cm<sup>-1</sup>): 3480 (br,  $\nu_{(\text{OH})}(\text{CH}_3\text{OH})$ ); 1637 (s,  $\nu_{(\text{C}=\text{N})}$ ); 1539 (s,  $\nu_{(\text{Ar-O})}$ ); 1103 (s,  $\nu_{(\text{ClO}_4)}$ ). UV-Vis ( $\lambda_{\text{max}}$  (nm), CH<sub>3</sub>CN): 240 (9407 M<sup>-1</sup>cm<sup>-1</sup>); 440 (3428).  $\Lambda_{\text{M}}$  (CH<sub>3</sub>CN, 291K): 90.7 S cm<sup>2</sup> mol<sup>-1</sup>. Yellowish crystals of [Cd<sub>2</sub>L](ClO<sub>4</sub>)·CH<sub>3</sub>OH suitable for the X-ray structure determination were obtained by slow evaporation of the above filtrate at room temperature.

**Caution.** Perchlorate salts of metal complexes with organic ligands are potentially explosive. Only small amounts of material should be prepared, and these should be handled with caution. The complexes described in this report have, so far, been found to be safe when used in small quantities.

#### *Crystallographic Data Collection and Solution of Structure*

Crystallographic data for the cryptate [Cd<sub>2</sub>L](ClO<sub>4</sub>)·CH<sub>3</sub>OH are summarized in Table I. Diffraction data were collected on SIEMENS P<sub>4</sub> diffractometer with graphite-monochromated MoK $\alpha$  radiation ( $\lambda = 0.71073$  Å) over the range  $2 < 2\theta < 48^\circ$ , with a  $\omega$ - $2\theta$  scan mode and with a  $\omega$  scan width of 0.8°, scan rate 10° min<sup>-1</sup>. The structure was solved by SHELXTL direct methods using the programs contained in the XSCANS package. The remaining non-hydrogen atoms were located from a difference Fourier map. Hydrogen atoms were introduced in calculated positions and refined. All calculations were performed using the programs contained in SHELXTL package. Lists of H atom positions,

TABLE I Crystal data collection parameters for the cryptate.

Formula	C <sub>13</sub> H <sub>52</sub> N <sub>8</sub> O <sub>8</sub> Cl <sub>4</sub> Cd <sub>2</sub>
Molecular weight	1176.95
Colour	yellowish
Crystal system	monoclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i>
<i>a</i> (Å)	16.076(4)
<i>b</i> (Å)	15.900(3)
<i>c</i> (Å)	18.590(4)
$\beta$ (deg)	95.100(14)
<i>V</i> (Å <sup>3</sup> )	4719(4)
<i>Z</i>	4
<i>D</i> <sub>c</sub> (g cm <sup>-3</sup> )	1.65
<i>F</i> (000)	2360
$\mu$ (cm <sup>-1</sup> )	11.9
Crystal dimensions (mm)	0.28 0.30 0.36
Radiation (Å)	MoK $\alpha$ (0.71073)
Scan mode	$\omega$ -2 $\theta$
2 $\theta$ range(deg)	2 to 48
No. reflections measured	6198
No. reflections used ( <i>F</i> > 4 $\sigma$ ( <i>F</i> ))	3675
Final <i>R</i> , <i>R</i> <sub>w</sub>	0.058, 0.068
$w^{-1} = \sigma^2(F) + 0.0005F^2$	

anisotropic thermal parameter and observed and calculated structure factors are available from the authors on request.

## RESULTS AND DISCUSSION

### *Spectroscopic Characterization of the Binuclear Cryptate*

In the infrared spectrum, the strong peak at 1637 cm<sup>-1</sup> corresponds to the C=N stretch. Neither symmetric nor asymmetric stretching frequencies at 3280 and 3330 cm<sup>-1</sup> for NH<sub>2</sub> groups nor strong peaks at 1650 ~ 1700 cm<sup>-1</sup> for the carbonyl groups were found. This means that all the NH<sub>2</sub> and C=O groups have been condensed into C=N bonds. A broad band at 3480 cm<sup>-1</sup> characteristic of the OH group of CH<sub>3</sub>OH showed that CH<sub>3</sub>OH molecules exist in the cryptate, in agreement with the elemental analysis. The strong peak at 1103 cm<sup>-1</sup> indicates that ClO<sub>4</sub><sup>-1</sup> does not take part in coordination with Cd(II). This was also confirmed by the molar conductivity of the cryptate which is characteristic of a 1:1 electrolyte; 15 this indicates that the three phenoxy oxygens are also coordinated to Cd(II). Two intense peaks in the UV region of the electronic spectrum are assigned to  $\pi$ - $\pi^*$  transitions of the K band of the benzene rings and of C=N groups, respectively. 16

*Description of the Structure of  $[Cd_2L](ClO_4) \cdot CH_3OH$* 

A perspective view of the cation  $[Cd_2L]^+$  is shown in Figure 2. Positional parameters, selected bond distances and angles are given in Tables II and III, respectively. In Figure 2 it is seen that each Cd(II) atom is enclosed within the polyaza cryptand. Each is coordinated by three imino nitrogens, one tertiary amine and three oxygen atoms of phenoxy groups to form a capped trigonal prismatic geometry. Around Cd(1), the phenoxy oxygens O(1), O(2) and the imino nitrogen N(8) form one trigonal face of the capped trigonal prism.

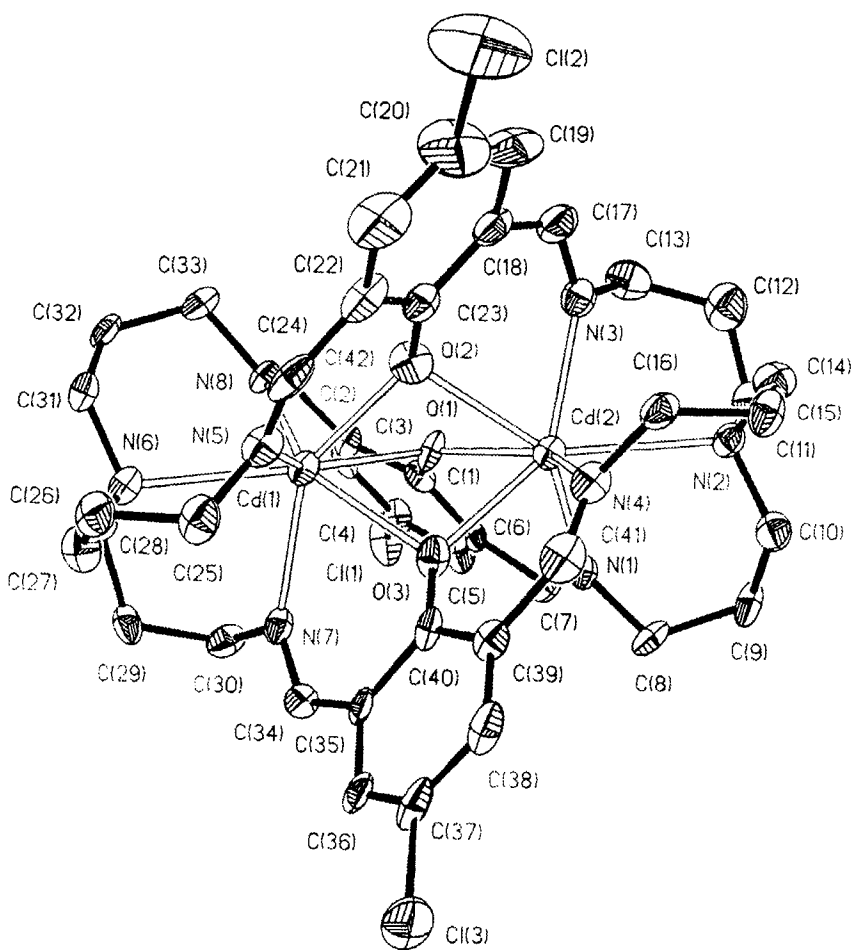


FIGURE 2 A perspective view of the cryptate cation  $[cd_2L]^+$ .

TABLE II Positional parameters for the cryptate.

Atom	$x/a$	$y/b$	$z/c$	$U(eq)$
Cd(1)	-755(1)	-2335(1)	-1622(1)	51(1)
Cd(2)	592(1)	-3875(1)	-2221(1)	52(1)
Cl(1)	-3173(2)	-6074(2)	-699(2)	105(1)
Cl(2)	1187(2)	-882(2)	-5048(2)	254(1)
Cl(3)	2986(2)	-1480(2)	891(2)	120(1)
O(1)	-762(2)	-3827(2)	-1907(2)	62(1)
O(2)	-175(2)	-2619(2)	-2666(2)	76(1)
O(3)	551(2)	-2935(2)	-1279(2)	64(1)
N(1)	537(2)	-4918(2)	-1292(2)	50(1)
N(2)	1585(2)	-4982(2)	-2670(2)	58(1)
N(3)	73(2)	-4110(2)	-3445(2)	71(1)
N(4)	1852(2)	-3102(2)	-2213(2)	67(1)
N(5)	-13(2)	-1091(2)	-1861(2)	70(1)
N(6)	-1728(2)	-1192(2)	-1135(2)	65(1)
N(7)	-654(2)	-2553(2)	-360(2)	57(1)
N(8)	-2075(2)	-2704(2)	-2143(2)	51(1)
C(1)	-1286(2)	-4329(2)	-1605(2)	51(1)
C(2)	-2155(2)	-4114(2)	-1686(2)	45(1)
C(3)	-2717(2)	-4692(2)	-1426(2)	58(1)
C(4)	-2436(2)	-5389(2)	-1035(2)	60(1)
C(5)	-1601(2)	-5560(2)	-916(2)	56(1)
C(6)	-1000(2)	-5030(2)	-1199(2)	39(1)
C(7)	-116(2)	-5236(2)	-1028(2)	62(1)
C(8)	1352(2)	-5170(2)	-972(2)	69(1)
C(9)	1829(2)	-5755(2)	-1430(2)	76(1)
C(10)	2158(2)	-5312(2)	-2089(2)	73(1)
C(11)	1063(3)	-5697(2)	-2961(2)	91(1)
C(12)	505(3)	-5597(3)	-3642(2)	109(1)
C(13)	-197(2)	-4970(2)	-3650(2)	89(1)
C(14)	2078(2)	-4679(2)	-3243(2)	79(1)
C(15)	2700(2)	-3952(2)	-3050(2)	77(1)
C(16)	2286(2)	-3121(2)	-2885(2)	71(1)
C(17)	54(2)	-3554(2)	-3967(2)	95(1)
C(18)	231(2)	-2620(3)	-3840(2)	78(1)
C(19)	528(3)	-2236(3)	-4437(2)	126(1)
C(20)	741(3)	-1386(3)	-4339(3)	158(1)
C(21)	627(3)	-959(3)	-3723(2)	126(1)
C(22)	321(2)	-1357(2)	-3138(2)	81(1)
C(23)	107(2)	-2217(2)	-3165(2)	63(1)
C(24)	271(2)	-892(2)	-2446(2)	83(1)
C(25)	161(2)	-507(2)	-1281(2)	86(1)
C(26)	-630(2)	-45(2)	-1085(2)	83(1)
C(27)	-1243(2)	-521(2)	-693(2)	85(1)
C(28)	-2355(2)	-1571(2)	-677(2)	73(1)
C(29)	-2000(2)	-1956(2)	42(2)	73(1)
C(30)	-1441(2)	-2710(2)	-15(2)	72(1)
C(31)	-2203(2)	-779(2)	-1771(2)	66(1)
C(32)	-2845(2)	-1330(2)	-2208(2)	69(1)
C(33)	-2488(2)	-2032(2)	-2609(2)	67(1)
C(34)	5(2)	-2489(2)	88(2)	68(1)
C(35)	862(2)	-2365(2)	-121(2)	55(1)
C(36)	1479(2)	-2029(2)	393(2)	71(1)
C(37)	2261(2)	-1902(2)	231(2)	81(1)
C(38)	2529(2)	-2114(2)	-442(2)	86(1)
C(39)	1936(2)	-2477(2)	-964(2)	58(1)
C(40)	1080(2)	-2619(2)	-823(2)	58(1)
C(41)	2209(2)	-2678(2)	-1689(2)	77(1)
C(42)	-2464(2)	-3393(2)	-2090(2)	52(1)
C(43)	4294(3)	-2782(3)	-954(3)	112(1)
Cl(4)	4949(2)	-2876(2)	-2651(2)	158(1)
O(4)	4918(3)	-2546(3)	-1941(2)	492(1)
O(5)	5338(3)	-3630(2)	-2605(3)	341(1)
O(6)	4195(2)	-2904(3)	-3097(3)	410(1)
O(7)	5486(2)	-2295(3)	-3027(2)	339(1)
O(8)	3976(3)	-3672(3)	-985(3)	251(1)

\*Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

TABLE III Selected bond distances (Å) and angles (°) for the cryptate [Cd<sub>2</sub>L](ClO<sub>4</sub>)-CH<sub>3</sub>OH.

Cd(1)-O(1)	2.430(4)	Cd(2)-O(1)	2.305(4)
Cd(1)-O(2)	2.271(4)	Cd(2)-O(2)	2.452(4)
Cd(1)-O(3)	2.342(4)	Cd(2)-O(3)	2.307(4)
Cd(1)-N(5)	2.372(4)	Cd(2)-N(1)	2.402(4)
Cd(1)-N(6)	2.612(4)	Cd(2)-N(2)	2.566(4)
Cd(1)-N(7)	2.364(4)	Cd(2)-N(3)	2.383(4)
Cd(1)-N(8)	2.327(4)	Cd(2)-N(4)	2.368(4)
O(1)-Cd(1)-O(2)	67.3(1)	O(1)-Cd(1)-O(3)	69.4(1)
O(2)-Cd(1)-O(3)	74.1(1)	O(1)-Cd(1)-N(5)	140.0(1)
O(2)-Cd(1)-N(5)	75.8(1)	O(3)-Cd(1)-N(5)	86.4(1)
O(1)-Cd(1)-N(6)	139.8(1)	O(2)-Cd(1)-N(6)	137.6(1)
O(3)-Cd(1)-N(6)	137.6(1)	N(5)-Cd(1)-N(6)	79.1(1)
O(1)-Cd(1)-N(7)	94.1(1)	O(2)-Cd(1)-N(7)	145.3(1)
O(3)-Cd(1)-N(7)	71.9(1)	N(5)-Cd(1)-N(7)	108.5(1)
N(6)-Cd(1)-N(7)	75.4(1)	O(1)-Cd(1)-N(8)	71.1(1)
O(2)-Cd(1)-N(8)	91.3(1)	O(3)-Cd(1)-N(8)	140.6(1)
N(5)-Cd(1)-N(8)	126.0(1)	N(6)-Cd(1)-N(8)	76.6(1)
N(7)-Cd(1)-N(8)	110.8(1)	O(1)-Cd(2)-O(2)	66.5(1)
O(1)-Cd(2)-O(3)	72.2(1)	O(2)-Cd(2)-O(3)	71.4(1)
O(1)-Cd(2)-N(1)	75.2(1)	O(2)-Cd(2)-N(1)	139.1(1)
O(3)-Cd(2)-N(1)	84.1(1)	O(1)-Cd(2)-N(2)	137.2(1)
O(2)-Cd(2)-N(2)	139.3(1)	O(3)-Cd(2)-N(2)	138.8(1)
N(1)-Cd(2)-N(2)	79.8(1)	O(1)-Cd(2)-N(3)	89.3(1)
O(2)-Cd(2)-N(3)	71.5(1)	O(3)-Cd(2)-N(3)	142.7(1)
N(1)-Cd(2)-N(3)	123.1(1)	N(2)-Cd(2)-N(3)	75.9(1)
O(1)-Cd(2)-N(4)	143.9(1)	O(2)-Cd(2)-N(4)	89.1(1)
O(3)-Cd(2)-N(4)	75.1(1)	N(1)-Cd(2)-N(4)	116.1(1)
N(2)-Cd(2)-N(4)	78.5(1)	N(3)-Cd(2)-N(4)	108.3(1)
Cd(1)-O(1)-Cd(2)	95.8(1)	Cd(1)-O(2)-Cd(2)	96.1(2)
Cd(1)-O(3)-Cd(2)	98.2(1)	Cd(1)-O(1)-C(1)	119.2(3)
Cd(1)-O(1)-C(1)	137.7(3)	Cd(1)-O(2)-C(23)	137.7(3)
Cd(2)-O(2)-C(23)	117.6(3)	Cd(1)-O(3)-C(40)	124.0(3)
Cd(2)-O(3)-C(40)	135.3(3)		

imino nitrogens N(5), N(7) and the phenoxy oxygen O(3) form another trigonal face. The bridgehead nitrogen N(6) is located in the cap position. Around Cd(2), the phenoxy oxygens O(1), O(2) and the imino nitrogen N(3) form one trigonal face. The imino nitrogens N(1), N(4) and the phenoxy oxygen O(3) form another trigonal face. The bridgehead nitrogen N(2) is located in the cap position. The dihedral angles between plane 1 (Cd(1), O(1), Cd(2)), plane 2 (Cd(1), O(2), Cd(2)) and plane 3 (Cd(1), O(3), Cd(2)) are  $\angle 1,2 = 69.3^\circ$ ,  $\angle 1,3 = 122^\circ$  and  $\angle 2,3 = 52.7^\circ$ , respectively. The three bond distances Cd(1)-N(imino) and Cd(2)-N(imino) are in the range 2.327 ~ 2.402 Å, which is slightly longer than the standard Cd-N bond distance of  $2.33 \pm 0.005$  Å.<sup>17</sup> This means that these imino groups are weakly coordinated to Cd(II). The two bond distances Cd(1)-N(6) and Cd(2)-N(2) are 2.612 and 2.566 Å, respectively, implying a weaker interaction



between Cd(II) and bridgehead nitrogen atoms. In the binuclear cadmium(II) cryptate  $[\text{Cd}_2\text{L}'](\text{ClO}_4)$  synthesized by condensation of *tris*(2-aminoethyl)amine (tren) with 2,6-diformyl-4-methylphenol, <sup>10</sup> however, the corresponding Cd-N (bridgehead) distance is 2.787 Å, longer than that reported in this paper. The Cd-N(bridgehead) distance is much shorter than that of its analogue  $[\text{Cd}_2\text{L}']^+$  in which the substituent in the benzene ring is bromo instead of chloro, its corresponding distance is 3.051 Å. The long Cd(1)...Cd(2) distance of 3.514 Å indicates that there is no interaction between the two metal atoms. N(2)-N(6) is 7.446 Å, being much shorter than the corresponding distance 9.01 Å in  $[\text{Cd}_2\text{L}']^+$ . Cd(1)-O(phenoxy oxygen) and Cd(2)-O(phenoxy oxygen) are in the range 2.271 ~ 2.452 Å, close to those reported by Drew *et al.*,<sup>10</sup> also implying that Cd(1) is similar to Cd(2) in coordination environment but slightly different to Cd(2) in distortion geometry. Therefore, in our cryptate the chloro substituent changed the coordination environment of Cd(II) from that in the bromo substitute in which Cd(II) is located at the centre of a distorted octahedron.

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