Supplementary Material (ESI) for CrystEngComm

Supporting Information

Trapping Two Different $CdCl_2$ 1D-Layered Structures by a Cyclocarbophosphazene-based Ligand

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Experimental Details

General Method. Solvents and other general reagents used in this work were purified according to standard procedures.[1] $Na_2(NCN)_2$ and 4-Hydroxy pyridine (Fluka, Switzerland), PCl_5 (s. d. Fine Chemicals, India), $CdCl_2$ (Fluka, Switzerland were used as purchased. $[NCCl]_2[NPCl_2]$,[2] *N*,*N*,*N*',*N'*-tetramethylmethylenediamine,[3] and $[NC(NMe_2)]_2[NPCl_2]$[4] were prepared according to literature procedures. Melting points were measured using a JSGW melting point apparatus and are uncorrected. IR spectra were recorded as KBr pellets on a Bruker Vector 22 FT IR spectrophotometer operating from 400-4000 cm^{-1}. ^1H and $^{31}P\{^1H\}$ NMR spectra were recorded in $CDCl_3$ solutions on a JEOL JNM LAMBDA 400 model spectrometer operating at 400.0 and 161.7 MHz respectively. Chemical shifts are reported in ppm and are referenced with respect to internal tetramethylsilane (^1H) and external 85% H_3PO_4 (^{31}P). Elemental analyses were carried out using a Thermoquest CE instruments CHNS-O, EA/110 model elemental analyzer.

Synthesis

$[NC(NMe_2)]_2[NP(OC_5H_4N)_2]$ (L): In a 250 mL R.B flask $[NC(NMe_2)]_2[NPCl_2]$ (4.00 g, 17.6 mmol), 4-hydroxy pyridine (4.18 g, 44.0 mmol), and K_2CO_3 (12 g, 86.8 mmol) were taken along with 150 mL of acetone. The reaction mixture was stirred for 48 hours at 60 ˚C. After this acetone was removed by rotary evaporator at low pressure. An off-white residue was obtained which was redissolved in dichloromethane (100 mL) and filtered throw a G-4 frit. A light brown colored solution was obtained which was concentrated to 10 mL, and kept at 4˚C over night. Needle-shaped colorless crystalline product was isolated.

Yield: 5.93 g, 90.2 % (based on phosphorus). Mp 101 °C. IR (KBr) cm^{-1}: 3419 (b), 3028 (s), 2926 (s), 2866 (m), 2471 (w), 2339 (w), 1581 (s), 1367 (s), 1244 (s), 1195 (s), 1090 (s), 925 (s), 830 (s), 765 (s), 602 (s), 565 (s), 530 (s), 483 (s). ESI-MS m/z, ion : 374.14 $\{M+1\}^+$. Anal. Calcd for **L** (373.14): C, 51.47; H, 5.40; N, 26.26. Found: C, 51.76; H, 5.56; N, 26.23. ^{1}H NMR: δ 8.52 (s, 4 H, aromatic), 7.09 (s, 4 H aromatic), and 3.08 (s, 12 H, -CH_3). $^{31}P\{^1H\}$ NMR: δ 31.4 (s).

$[L_2(CdCl_2)_3.H_2O]$ (1) : Crystals of **1** were obtained by layering a methanolic solution of $CdCl_2$ (0.05 g, 0.248 mmol, in 5 mL) on to a benzene solution of the ligand (**L**) (0.0926 g, 0.248 mmol, in 5 mL). After 3 days the solution was filtered and colorless block-shaped crystals were isolated.

Yield: 82% (based on metal) for **1**. Mp > 250 ˚C. IR (KBr) cm^{-1}: 3433 (b), 3058 (m), 2936 (s), 2481 (m), 1596 (s), 1474 (s), 1236 (s) 1201 (s), 1089 (s), 1061 (s), 922 (s), 876 (s), 850 (s), 759 (s), 661 (s), 622 (s), 601 (s), 601 (s), 568 (s), 534 (s), 523 (s). Anal. Calcd for **1**: C, 48.00; H, 5.00; N, 19.60. Found: C, 48.36; H, 5.21; N, 19.32.

$[L_2(CdCl_2)_2]$ $4H_2O.CHCl_3$ (2) : Crystals of **2** were obtained by layering a methanolic solution of $CdCl_2$ (0.05 g, 0.248 mmol, in 5 mL) on to a chloroform solution of the ligand (**L**) (0.0926 g, 0.248 mmol, in 5 mL). After 2 days the solution was filtered and colorless block- shaped crystals were isolated.

Yield: 85% (based on metal) for **2**. Mp > 250 ˚C. IR (KBr) cm^{-1}: 3424 (b), 3095 (s), 2926 (m), 1606 (m), 1569 (m), 1476 (s), 1401 (m) 1256 (s), 1205 (s), 1085 (s), 1020 (s), 952 (s), 917 (s), 877 (m), 835 (s), 765 (s), 623(m), 529 (s). Anal. Calcd for **2**: C, 39.60; H, 4.10; N, 19.60. Found: C, 39.75; H, 4.17; N, 19.56.

X-ray crystallography. The crystal data and the cell parameters for compounds **L**, **1** and **2** are given in Table S1. Single crystals (**1** and **2**) suitable for X-ray crystallographic analyses were obtained by layering a methanol solution of the $CdCl_2$ on to a benzene or chloroform solution of the ligand (**L**) . The crystal data for compounds **L**, **1** and **2** have been collected on a Bruker SMART CCD diffractometer using a Mo Kα sealed tube. The program SMART [5a] was used for collecting frames of data, indexing reflections, and determining lattice parameters, SAINT [5b] for integration of the intensity of reflections and scaling, SADABS [5b] for absorption correction, and SHELXTL [5c,d] for space group and structure determination and least-squares refinements on F^2. All the structures were solved by direct methods using the program SHELXS [5e] and refined by full-matrix least squares methods against F^2 with SHELXL.[5e] Hydrogen atoms were fixed at calculated positions and their positions were refined by a riding model. Most of the non-hydrogen atoms were refined with anisotropic displacement parameters. Due to the variable disorder in carbon and nitrogen (C2, C5, C6, N2 and N5) atoms of cyclocarbophosphazene ring in complex **2** are isotropically refined. Several crystals of **2** were tested and measured on Bruker SMART CCD diffractometer using a Mo Kα sealed tube at different temperatures. The diffraction patterns of other crystals are exhibit lower quality in compare to what we report here. The disordered water molecules were refined isotropically. The crystallographic figures have been generated using *Diamond 3.1e* software. [5f] Bond angles and distances for compounds **1** and **2** are given in the table S2 and S3. The immediate coordination of the Cd atoms along with the bond distances and bond angles around these atoms in complexes **1** and **2** are listed in Table S4.

Table S1: Crystal and structure refinement parameters for compounds **L, 1** and **2**

Parameters	**L**	**1**	**2**
Empirical formula	$C_{16}H_{20}N_7O_2P$	$C_{32}H_{40}Cd_3Cl_6N_{14}O_5P_2$	$C_{33}H_{41}Cd_2Cl_7N_{14}O_8P_2$
Formula Weight	373.36	1312.62	1296.69
Temperature	293(2) K	293(2) K	293(2) K
Wavelength	0.71073 Å	0.71069 Å	0.71069 Å
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	P-1	P-1	P2(1)/c
Unit cell dimensions	a = 7.3777(16) Å b = 10.187(2) Å c = 12.570(3) Å α= 86.803(4)° β= 81.176(4)° γ = 74.745(4)°	a = 14.196(5) Å b = 15.367(5) Å c = 16.615(5) Å α= 76.167(5)° β= 87.919(5)° γ = 64.595(5)°	a = 13.957(5) Å b = 25.704(5) Å c = 15.827(5) Å α= 90.000(5)° β= 93.362(5)° γ = 90.000(5)°
Volume, Z	900.5(3) $Å^3$, 2	3170.0(18) $Å^3$, 2	5668(3) $Å^3$, 4
Density (Calculated)	1.377 Mg/m^3	1.375 Mg/m^3	1.520 Mg/m^3
Absorption coefficient	0.180 mm^{-1}	1.341 mm^{-1}	1.190 mm^{-1}
F (000)	392	1292	2584
Crystal size	0.11 x 0.09 x 0.06 mm^3	0.10 x 0.09 x 0.06 mm^3	0.19 x 0.10 x 0.08 mm^3
θ range for data collection	2.62 to 28.46°	2.11 to 26.00°	2.05 to 27.00°.
Limiting indices	$-8 \leq h \leq 9$, $-13 \leq k \leq 11$, $-16 \leq l \leq 16$	$-17 \leq h \leq 17$, $-18 \leq k \leq 18$, $-15 \leq l \leq 20$	$-17 \leq h \leq 17$, $-32 \leq k \leq 30$, $-20 \leq l \leq 15$
Reflections collected	5933	17670	33910
Independent reflections	4284 [R(int) = 0.0224]	12157 [R(int) = 0.0270]	12305 [R(int) = 0.0667]
Completeness to θ	94.2 %	97.5 %	99.5 %
Data/ restraints/ parameters	4284 / 0 / 240	12157 / 0 / 567	12305 / 22 / 570
Goodness - of - fit on F^2	1.135	1.093	1.033
Final R indices [I>2σ (I)]	R1 = 0.0598, wR2 = 0.1499	R1 = 0.0647, wR2 = 0.1853	R1 = 0.0829, wR2 = 0.2490
R indices (all data)	R1 = 0.0864, wR2 = 0.2082	R1 = 0.0810, wR2 = 0.2121	R1 = 0.1538, wR2 = 0.3149
Largest diff. peak and hole	0.345 and -0.511 e.$Å^{-3}$	3.275 and -0.939 e.$Å^{-3}$	3.128 and -0.700 e.$Å^{-3}$

Table S2: Bond distances and bond angles for complex **1**

Cd(1)-N(7) 2.325(6)	Cd(2)-O(5) 2.330(7)	Cd(3)-Cl(6)* 2.5964(17)
Cd(1)-Cl(4) 2.5813(17)	Cd(2)-Cl(5) 2.543(2)	Cd(3)-Cl(4) 2.6141(18)
Cd(1)-Cl(1) 2.6037(19)	Cd(2)-Cl(1) 2.588(2)	Cd(3)-Cl(6) 2.6177(18)
Cd(1)-Cl(3) 2.6370(18)	Cd(2)-Cl(2) 2.6145(17)	Cd(3)-Cl(3) 2.6461(18)
Cd(1)-Cl(5)* 2.653(2)	Cd(2)-Cl(2)* 2.794(2)	Cl(2)-Cd(2)* 2.794(2)
Cd(1)-Cl(2) 2.6690(18)	Cd(3)-N(6)* 2.373(5)	Cl(5)-Cd(1)* 2.653(2)
Cd(2)-N(8) 2.319(6)	Cd(3)-N(14) 2.381(6)	Cl(6)-Cd(3)* 2.5964(17)
N(7)-Cd(1)-Cl(4) 90.82(15)	N(8)-Cd(2)-Cl(1) 88.03(16)	N(6)*-Cd(3)-Cl(6) 89.03(15)
N(7)-Cd(1)-Cl(1) 90.40(15)	O(5)-Cd(2)-Cl(1) 89.6(2)	N(14)-Cd(3)-Cl(6) 91.90(15)
Cl(4)-Cd(1)-Cl(1) 176.59(7)	Cl(5)-Cd(2)-Cl(1) 172.83(6)	Cl(6)*-Cd(3)-Cl(6) 84.17(6)
N(7)-Cd(1)-Cl(3) 88.59(15)	N(8)-Cd(2)-Cl(2) 164.12(17)	Cl(4)-Cd(3)-Cl(6) 175.97(5)
Cl(4)-Cd(1)-Cl(3) 84.71(6)	O(5)-Cd(2)-Cl(2) 100.26(19)	N(6)*-Cd(3)-Cl(3) 86.84(15)
Cl(1)-Cd(1)-Cl(3) 92.13(7)	Cl(5)-Cd(2)-Cl(2) 89.73(6)	N(14)-Cd(3)-Cl(3) 163.63(14)
N(7)-Cd(1)-Cl(5)* 89.39(16)	Cl(1)-Cd(2)-Cl(2) 85.15(6)	Cl(6)*-Cd(3)-Cl(3) 99.97(6)
Cl(4)-Cd(1)-Cl(5)* 85.47(6)	N(8)-Cd(2)-Cl(2)* 80.41(17)	Cl(4)-Cd(3)-Cl(3) 83.89(6)
Cl(1)-Cd(1)-Cl(5)* 97.73(7)	O(5)-Cd(2)-Cl(2)* 170.9(2)	Cl(6)-Cd(3)-Cl(3) 99.97(6)
Cl(3)-Cd(1)-Cl(5)* 169.95(6)	Cl(5)-Cd(2)-Cl(2)* 87.34(6)	Cd(2)-Cl(1)-Cd(1) 96.08(6)
N(7)-Cd(1)-Cl(2) 173.09(15)	Cl(1)-Cd(2)-Cl(2)* 97.30(7)	Cd(2)-Cl(2)-Cd(1) 93.87(6)
Cl(4)-Cd(1)-Cl(2) 95.23(6)	Cl(2)-Cd(2)-Cl(2)* 86.24(6)	Cd(2)-Cl(2)-Cd(2)* 93.76(6)
Cl(1)-Cd(1)-Cl(2) 83.74(6)	N(6)*-Cd(3)-N(14) 82.1(2)	Cd(1)-Cl(2)-Cd(2)* 89.31(6)
Cl(3)-Cd(1)-Cl(2) 95.26(6)	N(6)*-Cd(3)-Cl(6)* 171.09(14)	Cd(1)-Cl(3)-Cd(3) 93.48(6)
Cl(5)*-Cd(1)-Cl(2) 87.79(6)	N(14)-Cd(3)-Cl(6)* 92.35(15)	Cd(1)-Cl(4)-Cd(3) 95.56(6)
N(8)-Cd(2)-O(5) 94.0(3)	N(6)*-Cd(3)-Cl(4) 90.06(15)	Cd(2)-Cl(5)-Cd(1)* 95.29(6)
N(8)-Cd(2)-Cl(5) 98.16(16)	N(14)-Cd(3)-Cl(4) 84.09(15)	Cd(3)*-Cl(6)-Cd(3) 95.83(5)
O(5)-Cd(2)-Cl(5) 86.4(2)	Cl(6)*-Cd(3)-Cl(4) 96.33(6)	

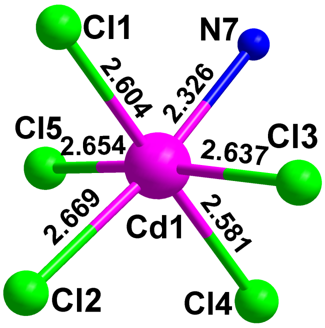
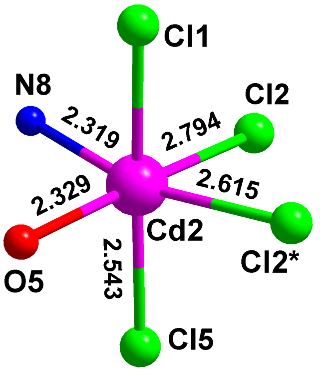
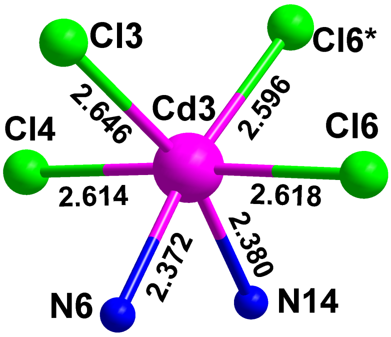
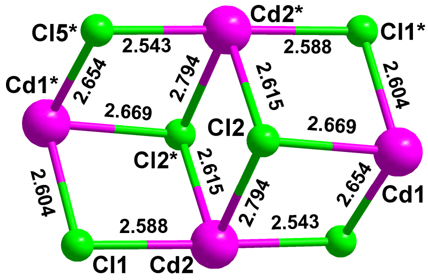
Symmetry transformations used to generate equivalent atoms: * -x+1,-y+1,-z+1 * x,y-1,z * -x+1,-y+1,-z+2 * x,y+1,z * x-1,y+1,z-1 * x+1,y-1,z+1

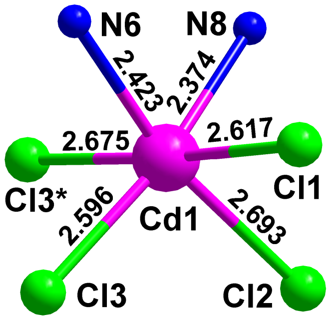
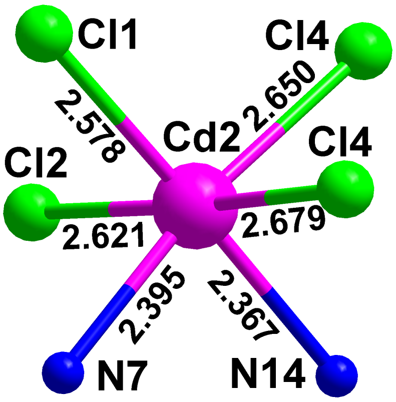
Table S3: Bond distances and bond angles for complex **2**

Cd(1)-N(7) 2.374(6)	Cd(1)-Cl(6)* 2.677(2)	Cd(2)-Cl(4) 2.578(2)
Cd(1)-N(6) 2.417(6)	Cd(1)-Cl(5) 2.693(2)	Cd(2)-Cl(5) 2.622(2)
Cd(1)-Cl(6) 2.592(2)	Cd(2)-N(13) 2.369(7)	Cd(2)-Cl(7) 2.651(2)
Cd(1)-Cl(4) 2.614(2)	Cd(2)-N(14) 2.399(6)	Cd(2)-Cl(7)* 2.679(2)
N(7)-Cd(1)-N(6) 86.5(3)	Cl(6)-Cd(1)-Cl(5) 97.79(7)	Cl(4)-Cd(2)-Cl(7) 99.42(7)
N(7)-Cd(1)-Cl(6) 168.29(17)	Cl(4)-Cd(1)-Cl(5) 84.06(7)	Cl(5)-Cd(2)-Cl(7) 94.94(7)
N(6)-Cd(1)-Cl(6) 90.95(17)	Cl(6)*-Cd(1)-Cl(5) 98.82(7)	N(13)-Cd(2)-Cl(7)* 84.8(2)
N(7)-Cd(1)-Cl(4) 92.02(18)	N(13)-Cd(2)-N(14) 85.5(2)	N(14)-Cd(2)-Cl(7)* 92.83(18)
N(6)-Cd(1)-Cl(4) 86.46(18)	N(13)-Cd(2)-Cl(4) 171.7(2)	Cl(4)-Cd(2)-Cl(7)* 88.87(7)
Cl(6)-Cd(1)-Cl(4) 99.24(7)	N(14)-Cd(2)-Cl(4) 89.54(16)	Cl(5)-Cd(2)-Cl(7)* 174.81(6)
N(7)-Cd(1)-Cl(6)* 86.27(18)	N(13)-Cd(2)-Cl(5) 100.0(2)	Cl(7)-Cd(2)-Cl(7)* 87.47(7)
N(6)-Cd(1)-Cl(6)* 90.43(18)	N(14)-Cd(2)-Cl(5) 85.50(18)	Cd(2)-Cl(4)-Cd(1) 95.89(7)
Cl(6)-Cd(1)-Cl(6)* 82.32(7)	Cl(4)-Cd(2)-Cl(5) 86.21(7)	Cd(2)-Cl(5)-Cd(1) 92.98(7)
Cl(4)-Cd(1)-Cl(6)* 176.54(6)	N(13)-Cd(2)-Cl(7) 85.60(17)	Cd(1)-Cl(6)-Cd(1)* 97.68(7)
N(7)-Cd(1)-Cl(5) 86.51(16)	N(14)-Cd(2)-Cl(7) 171.04(16)	Cd(2)-Cl(7)-Cd(2)* 92.53(7)
N(6)-Cd(1)-Cl(5) 168.02(17)		

Symmetry transformations used to generate equivalent atoms: * -x,-y+2,-z+2 * -x+1,-y+2,-z+2 * -x, y-1/2,-z+3/2 * -x+1,y-1/2,-z+5/2 * -x+1,y+1/2,-z+5/2 * -x,y+1/2,-z+3/2

Table S4: Immediate coordination environment of different types of Cd atoms along with the bond distance and bond angle data around these atoms in complexes **1** and **2**.

 **Complex 1**	**Bond Angles(/ °):**			
	N(7)-Cd(1)-Cl(4)	90.82(15)	Cl(4)-Cd(1)-Cl(5)*	85.47(6)
	N(7)-Cd(1)-Cl(1)	90.40(15)	Cl(1)-Cd(1)-Cl(5)*	97.73(7)
	Cl(4)-Cd(1)-Cl(1)	176.59(7)	Cl(3)-Cd(1)-Cl(5)*	169.95(6)
	N(7)-Cd(1)-Cl(3)	88.59(15)	N(7)-Cd(1)-Cl(2)	173.09(15)
	Cl(4)-Cd(1)-Cl(3)	84.71(6)	Cl(4)-Cd(1)-Cl(2)	95.23(6)
	Cl(1)-Cd(1)-Cl(3)	92.13(7)	Cl(1)-Cd(1)-Cl(2)	83.74(6)
	N(7)-Cd(1)-Cl(5)*	89.39(16)	Cl(3)-Cd(1)-Cl(2)	95.26(6)
			Cl(5)*-Cd(1)-Cl(2)	87.79(6)
 **Complex 1**	**Bond Angles(/ °):**			
	N(8)-Cd(2)-O(5)	94.0(3)	Cl(5)-Cd(2)-Cl(2)	89.73(6)
	N(8)-Cd(2)-Cl(5)	98.16(16)	Cl(1)-Cd(2)-Cl(2)	85.15(6)
	O(5)-Cd(2)-Cl(5)	86.4(2)	N(8)*-Cd(2)-Cl(2)*	80.41(17)
	N(8)-Cd(2)-Cl(1)	88.03(16)	O(5)-Cd(2)-Cl(2)*	170.9(2)
	O(5)-Cd(2)-Cl(1)	89.6(2)	Cl(5)-Cd(2)-Cl(2)*	87.34(6)
	Cl(5)-Cd(2)-Cl(1)	172.83(6)	Cl(1)-Cd(2)-Cl(2)*	97.30(7)
	N(8)-Cd(2)-Cl(2)	164.12(17)	Cl(2)-Cd(2)-Cl(2)*	86.24(6)
	O(5)-Cd(2)-Cl(2)	100.26(19)		
 **Complex 1**	**Bond Angles(/ °):**			
	N(6)*-Cd(3)-N(14)	82.1(2)	N(14)-Cd(3)-Cl(6)	91.90(15)
	N(6)*-Cd(3)-Cl(6)*	171.09(14)	Cl(6)*-Cd(3)-Cl(6)	84.17(6)
	N(14)-Cd(3)-Cl(6)*	92.35(15)	Cl(4)-Cd(3)-Cl(6)	175.97(5)
	N(6)*-Cd(3)-Cl(4)	90.06(15)	N(6)*-Cd(3)-Cl(3)	86.84(15)
	N(14)-Cd(3)-Cl(4)	84.09(15)	N(14)-Cd(3)-Cl(3)	163.63(14)
	Cl(6)*-Cd(3)-Cl(4)	96.33(6)	Cl(6)*-Cd(3)-Cl(3)	99.97(6)
	N(6)*-Cd(3)-Cl(6)	89.03(15)	Cl(4)-Cd(3)-Cl(3)	83.89(6)
			Cl(6)-Cd(3)-Cl(3)	99.97(6)
 **Complex 1**	**Bond Angles(/ °):**			
	Cd(2)-Cl(1)-Cd(1)	96.08(6)	Cd(1)-Cl(3)-Cd(3)	93.48(6)
	Cd(2)-Cl(2)-Cd(1)	93.87(6)	Cd(1)-Cl(4)-Cd(3)	95.56(6)
	Cd(2)-Cl(2)-Cd(2)*	93.76(6)	Cd(2)-Cl(5)-Cd(1)*	95.29(6)
	Cd(1)-Cl(2)-Cd(2)*	89.31(6)	Cd(3)*-Cl(6)-Cd(3)	95.83(5)

 **Complex 2**	**Bond Angles(/ °):** N(7)-Cd(1)-N(6) 86.5(3) N(7)-Cd(1)-Cl(6) 168.29(17) N(6)-Cd(1)-Cl(6) 90.95(17) N(7)-Cd(1)-Cl(4) 92.02(18) N(6)-Cd(1)-Cl(4) 86.46(18) Cl(6)-Cd(1)-Cl(4) 99.24(7) N(7)-Cd(1)-Cl(6)* 86.27(18)	N(6)-Cd(1)-Cl(6)* 90.43(18) Cl(6)-Cd(1)-Cl(6)* 82.32(7) Cl(4)-Cd(1)-Cl(6)* 176.54(6) N(7)-Cd(1)-Cl(5) 86.51(16) N(6)-Cd(1)-Cl(5) 168.02(17) Cl(6)-Cd(1)-Cl(5) 97.79(7) Cl(4)-Cd(1)-Cl(5) 84.06(7) Cl(6)*-Cd(1)-Cl(5) 98.82(7)
 **Complex 2**	**Bond Angles(/ °):** N(13)-Cd(2)-N(14) 85.5(2) N(13)-Cd(2)-Cl(4) 171.7(2) N(14)-Cd(2)-Cl(4) 89.54(16) N(13)-Cd(2)-Cl(5) 100.0(2) N(14)-Cd(2)-Cl(5) 85.50(18) Cl(4)-Cd(2)-Cl(5) 86.21(7) N(13)-Cd(2)-Cl(7) 85.60(17) N(14)-Cd(2)-Cl(7) 171.04(16) Cl(4)-Cd(2)-Cl(7) 99.42(7)	Cl(5)-Cd(2)-Cl(7) 94.94(7) N(13)-Cd(2)-Cl(7)* 84.8(2) N(14)-Cd(2)-Cl(7)* 92.83(18) Cl(4)-Cd(2)-Cl(7)* 88.87(7) Cl(5)-Cd(2)-Cl(7)* 174.81(6) Cl(7)-Cd(2)-Cl(7)* 87.47(7) Cd(2)-Cl(4)-Cd(1) 95.89(7) Cd(2)-Cl(5)-Cd(1) 92.98(7) Cd(1)-Cl(6)-Cd(1)* 97.68(7) Cd(2)-Cl(7)-Cd(2)* 92.53(7)

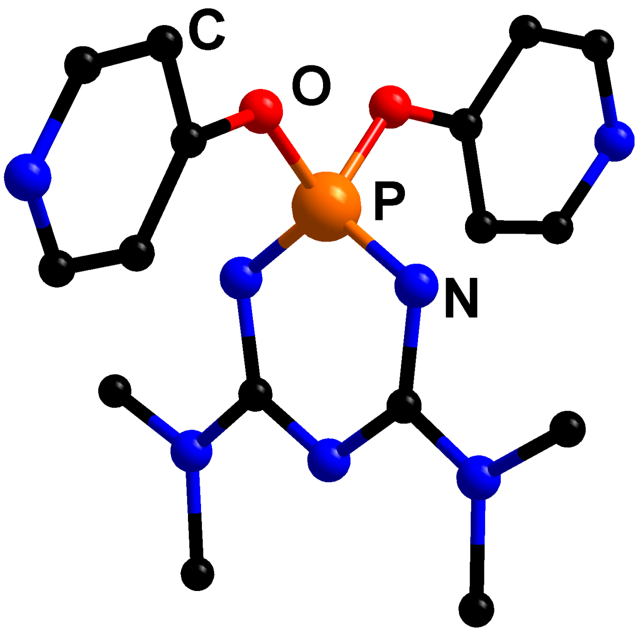


Figure S1: Molecular structure of **L.**

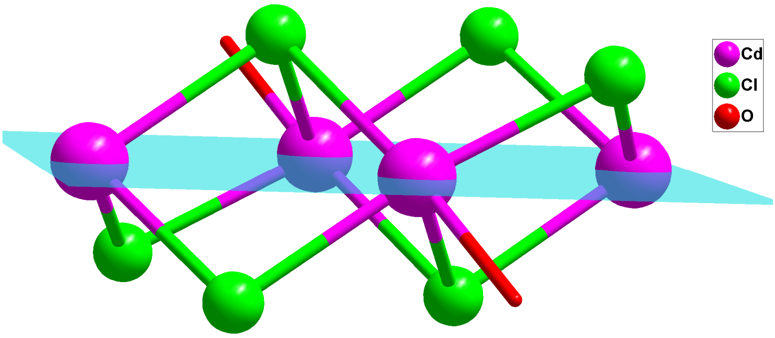


Figure S2: Motif A present in complex **1**.

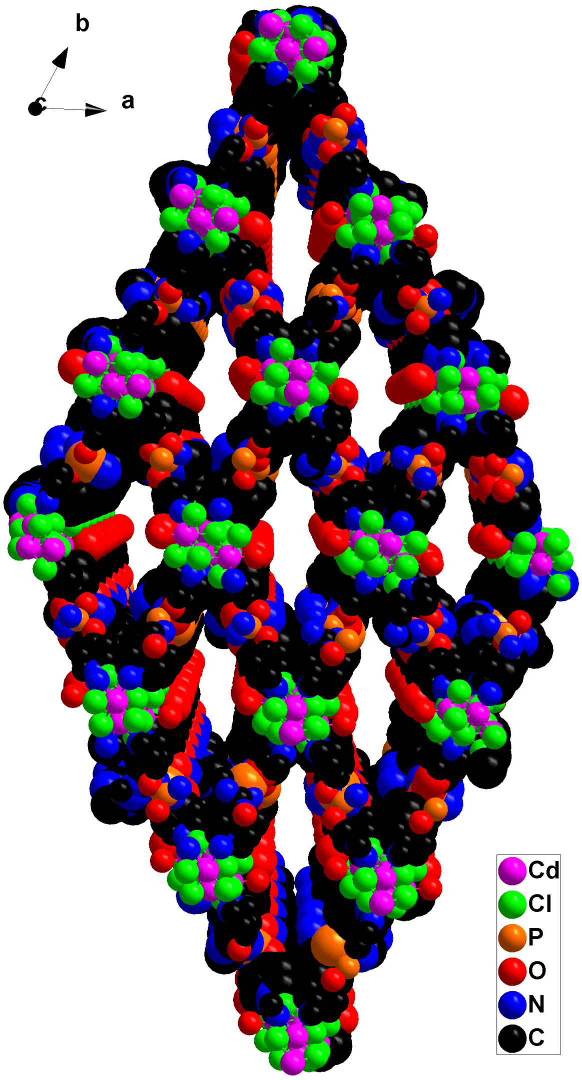


Figure S3: Space filling 3D-structure of **1.**

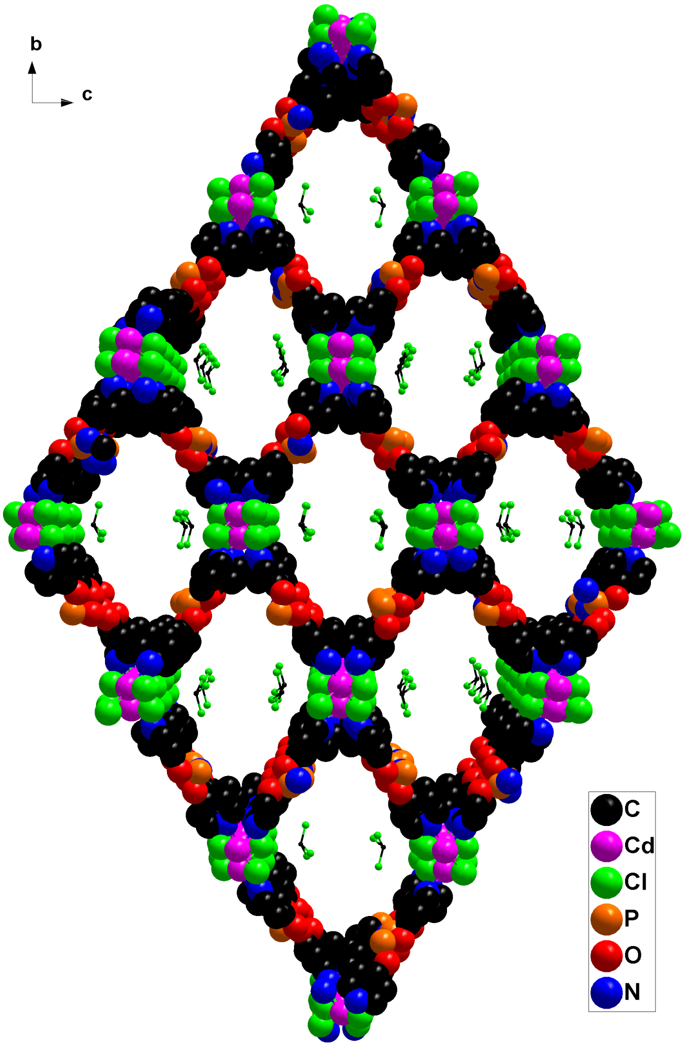


Figure S4: Space filling 3D-structure of **2.**

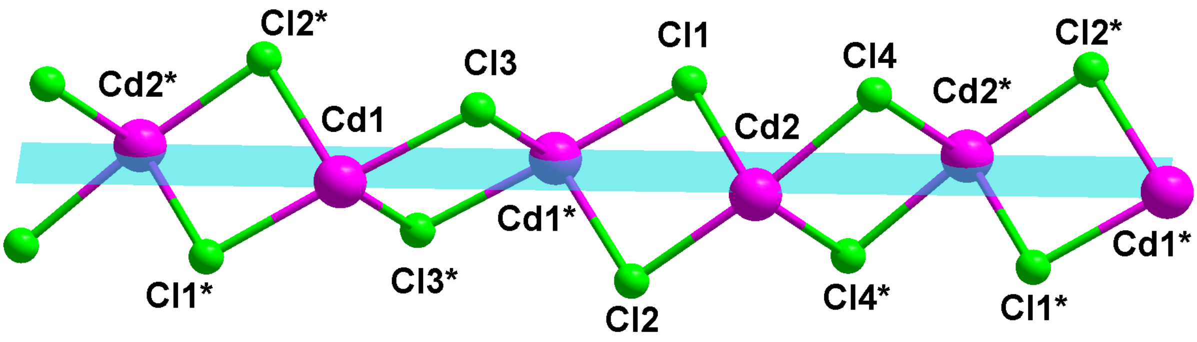


Figure S5: CdCl2 layer present in **2**.

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